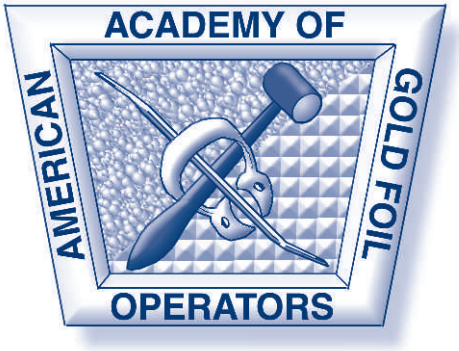
OPERATIVE DENTISTRY



july/august 2003 • volume 28 • number 4 • 325-476

(ISSN 0361-7734)

OPERATIVE DENTISTRY

JULY/AUGUST 2003 • VOLUME 28 • NUMBER 4 • 325-476

Aim and Scope

Operative Dentistry publishes articles that advance the practice of operative dentistry. The scope of the journal includes conservation and restoration of teeth; the scientific foundation of operative dental therapy; dental materials; dental education; and the social, political, and economic aspects of dental practice. Review papers, book reviews, letters, and classified ads for faculty positions are also published.

Subscriptions: Fax (317) 852-3162

Operative Dentistry (ISSN 0361-7734) is published bimonthly by *Operative Dentistry*, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186. Periodicals postage paid at Indianapolis, IN, and additional mailing offices. **Postmaster:** Send address changes to: *Operative Dentistry*, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186.

Current pricing for individual, institutional and dental student subscriptions (both USA and all other countries) can be found at our website: www.jopdent.org, or by contacting our subscription manager:

Fax 317/852-3162
E-mail: jmatis@indy.rr.com

Information on single copies, back issues and reprints is also available. Make remittances payable (in US dollars only) to *Operative Dentistry* and send to the above address. Credit card payment (Visa, MasterCard) is also accepted by providing card type, card number, expiration date, and name as it appears on the card.

Contributions

Contributors should study the instructions for their guidance printed in this journal and should follow them carefully.

Permission

For permission to reproduce material from *Operative Dentistry* please apply to *Operative Dentistry* at the above address.

The views expressed in *Operative Dentistry* do not necessarily represent those of the Academies or of the Editors.

Editorial Office

Operative Dentistry
Indiana University School of Dentistry, Room S411
1121 West Michigan Street, Indianapolis, IN 46202-5186
Telephone: (317) 278-4800, Fax: (317) 278-4900
URL: http://www.jopdent.org/

Editorial Staff

Editorial Board

©Operative Dentistry, 2003, **28-4**, 325-326

Editorial

Focus

Excellence is what (I would like to believe) all health care professionals strive for in their treatment of patients. During our dental education, the goal of doing our very best is constantly reinforced as we are taught to examine, diagnose, treat and even refer to specialists, when necessary. After graduation, we spend countless hours and dollars attending continuing education courses to be able to provide cutting-edge therapy in all phases of dental treatment. However, if you look carefully at the curriculum in dental schools and the majority of the more popular post-graduate course offerings, the emphasis is primarily on the repair of the sequellae of dental disease, not the treatment of the disease itself.

This approach is not without merit, since the greater part of the treatment rendered in dental practice involves restoring our patients' dentition to healthy form and function by correcting, repositioning, restoring or replacing damaged hard and soft oral structures. We also spend considerable time repairing or replacing existing restorations that do not meet our criteria of excellence. Certainly, this aspect of our profession has been and will continue to be of great importance and an essential focus of education and research. The question is, would much of this treatment be necessary if our focus was more on treating and possibly eliminating the disease rather than just repairing the resulting damage?

Of course, we have known for a long time that dental caries is an infectious disease and can be treated as such. We also know that dental restorations do not cure the disease, and generally do not even repair the damage permanently (particularly, if we examine the available data on restoration replacement). We are aware of the ongoing research on early diagnosis and monitoring of caries, the available adjuncts such as salivary flow and bacterial testing and the 1995 ADA policy statement that the use of a sharp explorer to detect dental caries should be discontinued (due to the high incidence of iatrogenic cavitation and bacterial transfer).

Unfortunately, knowing something and making it an integral part of everyday practice are two entirely different things, particularly when we receive so many mixed messages from our educational institutions, educators, researchers and both the manufacturing and insurance industries. A great deal of discussion and "lip service" are given to prevention and caries control, but it is often practically limited to periodic prophylaxis and "oral hygiene" lectures that seem to have limited effect on the patients who could benefit the most. Dental schools are just beginning to incorporate caries risk assessment into the curriculum and only a few have actual requirements or outcome assessment. The importance is lost on busy students who are trying to complete a myriad of clinical requirements that are worth far more credit hours. Continuing education courses are available, but they are a distant choice when compared to those which deal with cosmetic dentistry procedures. Research is promising in developing technology such as Quantitative Laser Fluorescence to detect caries at an early stage and monitor the success of non-restorative treatment, but widespread, practical utilization is still somewhere in the future. We also have a patient population that has been educated on the importance of restorative and cosmetic therapies but has not responded as readily to the possibilities and necessities of prevention and disease control. Finally, the demand for such technology is not strong enough to drive industry to develop and promote products at a high level, and dental insurance companies are only beginning to consider supporting proper remuneration for such procedures.

The dental profession has an enviable track record for working to eliminate dental caries, but we can't rest on the laurels of fluoride. For years we have lived with the debate as to whether dentists are really doc-

tors or just technicians who place restorations. Obviously, we need to be an amalgam of both. From my perspective, good dentists are a combination of diagnostician, healer, technician and artist… but if we want to truly be health care providers, our goal of excellence must be focused on the elimination of dental disease and not just treatment of its ravages.

After all, if you had a bacterial infection in your hand, would you want treatment to cure the disease or would you opt for a nice cosmetic prosthetic replacement?

Michael A Cochran
Editor

©Operative Dentistry, 2003, **28-4,** 327-333

Clinical Research

The Effectiveness of Bonded Composite Restorations in the Treatment of Painful, Cracked Teeth: Six-Month Clinical Evaluation

NJM Opdam • FJM Roeters

Clinical Relevance

Bonded resin composite restorations can be effective in treating painful, cracked teeth with or without cuspal coverage.

SUMMARY

This study investigated the clinical efficacy of a bonded resin composite restoration with and without cuspal coverage for the treatment of painful, cracked teeth. Patients in a private dental practice who presented with complaints were selected. Inclusion criteria were sensitivity to cold, biting and a clinically-visible crack after removal of the existing restoration. All 40 teeth were restored with a three-step total etch system (Phosphoric acid/Clearfil SA primer/PhotoBond), 20 with cuspal coverage and 20 without. Patients were interviewed at one week, five weeks and six months regarding the presence of pain. In addition, the teeth were clinically examined after six months to reveal any sensitivity. At one week, patients reported that 12 teeth (30%) were free of pain and 28 teeth (70%) still had symptoms. At five weeks, patients reported that 25 teeth (62.5%) were free of pain and 13 teeth (32.5%) still had symptoms. Two teeth (5%) needed endodontic treatment after two and five weeks. At six months, patients reported that 30 teeth (75%) were functioning without any complaints. Upon clinical examination, only 20 teeth (50%) were free of symptoms.

No statistically significant difference between the results of the teeth treated with and without cuspal coverage could be shown (Fischer's exact test at $p<0.05$).

*Niek JM Opdam, DDS, PhD, Department of Cariology and Endodontology, University of Nijmegen, The Netherlands

Joost M Roeters, DDS, PhD, associate professor, Department of Cariology and Endodontology University of Nijmegen, The Netherlands

*Reprint request: PO Box 9101, NL 6500 HB Nijmegen, The Netherlands; e-mail: n.opdam@}dent.dent.umch.nl

INTRODUCTION

The phenomenon of a "cracked tooth" was first described by Cameron (1964), who defined a cracked tooth as "an incomplete fracture of a vital posterior tooth involving dentin and possibly pulpal tissue." Patients complain about pain when cold and hot drinks or food come into contact with the tooth, combined with a painful sensation when chewing, especially on hard food (Homewood, 1998; Christensen, 1996). In those teeth, microcracks have developed at the base of weakened cusps, mostly due to large preparations and repeated functional occlusal loading. In most cases, the crack is visible in the dentin at the base of the involved

cusp when the existing restorations are removed. The presence of pain upon loading is explained by dentinal tubular fluid flow, which is a result of the movement of the two fracture sites away from and towards each other (Brännström, 1986). The pulp of those teeth may become inflamed because of irritation resulting from microleakage. Subsequently, patients complain about pain when cold and hot food and beverages come into contact with the tooth. Eventually, further propagation of the crack may result in loss of the cusp or irreversible pulpitis, which requires endodontic treatment.

Several therapies have been described as treatment for painful cracked teeth. In one study (Geurtsen & García Godoy, 1999), the authors stated that bonded amalgam or resin-based composite restorations do not sufficiently increase fracture resistance of teeth with wide occlusal-proximal cavities. They recommend that weakened, cracked teeth with wide cavities should be strengthened by full cuspal coverage with cast or ceramic restorations, by bonded ceramic inlays or by indirectly-fabricated bonded composite inlays.

As a temporary, diagnostic treatment, the cementation of a stainless steel orthodontic band was recommended to splint the involved tooth in order to evaluate whether the complaints disappear (Ehrmann & Tyas, 1990). In a study by Homewood (1998), the author recommended that the treatment of cracked teeth involve cuspal coverage. The author stated that glass-ionomer cements would not be strong enough and doubted whether resin composite would be effective. Cuspal coverage with metal, according to Homewood, allows for the absorbtion of fracture energies three times higher than that of intact teeth. In a clinical study by the same author, overlaying the cusps with amalgam or composite was successful in most cases (Homewood, 1998). In a study by Davis and Overton (2000), the cusps were covered with adhesive bonded or pin-retained amalgam restorations. These restorations were reported to be successful at one-year recall. However, the adhesive bonded amalgam restorations were more successful than the pin-retained amalgam restorations in reducing sensitivity to application of a cold stimulus. Another study also reported on the successful treatment of cracked teeth with bonded amalgam restorations (Bearn, Saunders & Saunders, 1994).

A bonded resin composite restoration has the potential to connect the weakened cusp(s) with the restoration. From several *in-vitro* studies, large preparations are known to reduce the fracture strength of teeth (Reeh, Messer & Douglas, 1989; Salis & others, 1987) and adhesive bonded resin composite restorations are able to improve or recover the strength of weakened teeth (Ausiello & others, 1997; Wendt, Harris & Hunt, 1987). Cuspal stiffness of a tooth weakened by a preparation will increase, especially when a hybrid composite is used in combination with a total etch technique (Ausiello & others, 1997). However, studies have also been published where authors do not agree with this and question the strengthening capacity of bonded composite restorations (Reel & Mitchell, 1989; Steele & Johnson, 1999).

Therefore, a bonded hybrid composite restoration may be effective as a therapy for painful, cracked teeth. However, no clinical reports regarding the suitability of this technique for this specific indication are available. When placing a direct composite restoration, a further option is to reduce the height of the cusp and cover it with resin composite. It is unknown whether such a restoration needs coverage of the "cracked" cusp, as has been found to be beneficial when placing an amalgam restoration. Furthermore, it is questionable whether a bonded resin composite restoration will prevent movement of the two parts of the tooth at the fracture site and reduce pulpal irritation. In one clinical study, three composite restorations and 48 amalgam restorations with cuspal coverage were placed, the number being too low to draw conclusions regarding the effectiveness of resin composite restorations (Homewood, 1998). Therefore, this study evaluated the efficacy of treating painful, cracked teeth with a bonded resin composite restoration with and without cuspal coverage.

METHODS AND MATERIALS

Patients were selected for this study from those in the authors' dental practice who presented with a toothache. Forty teeth in 39 patients were treated, 14 male (ranging in age between 24 and 68 years) and 26 female (ranging in age between 20 and 56 years), one of which had two cracked teeth. All patients reported tooth sensitivity when cold food and beverages came in contact with certain teeth. They also reported pain when chewing on hard food. Upon clinical investigation, the typical features of a cracked tooth had to be present. A thermal test involving application of a cold stimulus was performed using a cotton pellet soaked with Ethyl Chloride. All posterior teeth in the quadrant were tested in order to localize the sensitive tooth. Only teeth that were more sensitive to cold compared to the adjacent teeth, according to the patient, were included. When the sensitive tooth was localized, this tooth and the adjacent teeth were tested for the presence of pain upon loading using a plastic biting device called Tooth Slooth (Cypres, CA, USA). Of those teeth, all cusps were randomly tested, and when pain upon loading was recorded twice for the involved tooth and cusp, the diagnosis "cracked tooth" was made. Then, patients were asked to participate in the study. After explaining the goal and design of the study, informed consent was obtained from all patients. Under local anesthesia, the existing restoration was removed using a diamond bur in a high-speed handpiece with three-point water-spray. As

a third inclusion criterion, a fracture line had to be visible at the bottom of the preparation in the dentin of the involved cusp after removing the existing amalgam restoration. When a tooth was included in the study, it was randomly assigned to one of two experimental groups. In one group, the preparation only included removing the restoration and placing a bevel on the gingival, buccal and lingual margins of the boxes. When the preparation ended cervically in dentin, no bevel was placed along that margin. In the other group (control), the preparation was ground as in Group 1, but in addition, the involved cusp(s) were reduced to a level about 0.5-1 mm coronally from the onset of the microcracks. Then, the outline was beveled as in Group 1.

After the preparation was finished, isolation was performed with cotton rolls and a saliva ejector. A metal matrix was placed and fixed with interdental wooden wedges. The preparations were then etched for 15 seconds with 38% phosphoric acid (DMG, Hamburg, Germany), rinsed for 10 seconds and gently air dried for three seconds. The dentin was thoroughly wetted with a dentin primer (SA primer, Kuraray, Osaka, Japan) and dried with a gentle air stream. The adhesive (PhotoBond-Kuraray, Osaka Japan) was mixed and applied to the cavity and the ethanol-solvent was evaporated using a gentle stream of air. The adhesive layer was cured for 10 seconds. The preparations were restored with a compact-filled hybrid composite, Clearfil Photo Posterior (Kuraray) that was injected into the cavity by means of a preloaded tip (Hawe Neos, Boggio, Switzerland). The resin composite was placed in increments not exceeding 2 mm in thickness and each increment was cured for 20 seconds. A halogen light-curing device (Optilux 500, Demetron, light-intensity 800mW/cm^2) was used to cure the adhesive and the resin composite. After curing the final increment of resin composite, the matrix and wedges were removed and the restorations were post-cured for 20 seconds from the buccal and the lingual side. Subsequently, the restorations were contoured and finished using fine grit diamond burs and Sof-Lex discs (3M, St Paul, MN, USA). All restorations were placed by the same operator in a single dental practice.

After one week, all patients were contacted by the dental nurse via telephone and asked if they still experienced complaints from their restored tooth. When pain was still present, the interviewer asked the patients if they experienced pain upon loading the tooth or when the tooth came into contact with cold food and beverages. Furthermore, patients were asked if spontaneous pain was experienced and, if the level of pain had diminished, was the level of pain the same or had it become more severe since placing the restoration. At five weeks this interview was repeated.

At six months, the patients came to the dental office for recall examinations. Interviews were repeated and, in addition, the treated teeth were examined clinically. The affected teeth and adjacent teeth, which functioned as a control, were tested for pain upon cold application, using a cotton pellet soaked with Ethyl Chloride and they were tested for pain upon loading using the Tooth Slooth. The teeth and cusps were tested according to the same protocol used during the inclusion procedure. Care was taken so that the teeth were tested in a random range and patients were unaware whether the involved tooth or a control tooth was tested. When the experimental teeth were experienced as being more sensitive by the patient, the teeth were evaluated as still being sensitive. When there was no difference between the treated tooth and the adjacent teeth, the tooth was evaluated as being symptom-free.

Differences in performance between the restorations with and without cuspal coverage were analyzed statistically using Fischer's exact test at a significance level of p=0.05.

RESULTS

All patients could be followed during the six-month recall period. Table 1 shows distribution of the cracked teeth. Of all the teeth, the molars were most involved. All the teeth in this study had amalgam restorations: three had occlusal restorations, 10 had two-surface and 27 had three or more surface restorations. Of the 18 maxillary teeth involved, 15 had fractures indicated in the buccal cusps. Of the 22 mandibular teeth in the study, 18 had fractures located in the lingual cusps. Most of the teeth in the study showed signs of bruxism and clenching activity, including wear facets on the involved cusps and impressions of opposing cusps in the central fossae.

Figures 1-3 present a clinical case of a maxillary molar with amalgam restoration that shows incomplete fracture of the mesio-buccal cusp upon removal of the existing restoration. Figure 3 shows the new bonded composite restoration without cusp-capping.

Table 2 shows the results of the presence of sensitivity by patients who were interviewed at one week, five weeks and six months. At one week, 12 teeth (30%) were free of symptoms and 28 teeth (70%) still had symptoms. Of those teeth, four gave intermittent, spontaneous pain. Fifteen teeth showed sensitivity upon biting and after contact with cold food and beverages. Eleven teeth were still sensitive after contact with cold food and beverages and two teeth were sensitive upon biting. Two patients experienced more pain compared to the preoperative situation and two patients experienced no difference in pain level compared to the preoperative situation. All other teeth (36) were less painful after one week.

Table 1: *Distribution of Cracked Teeth (n=40)*

involved jaw	maxilla								mandibula							
involved tooth	2nd molar		1st molar		2nd premolar				2nd molar		1st molar		2nd premolar			
involved cusp(s)	lingual	buccal	lingual	buccal	lingual	buccal			lingual	buccal	lingual	buccal	lingual	buccal		
Class I amalgam		1		1					1							
Class II/ 2-surface amalgam				2	1				4		3					
Class II/ 3-surface amalgam		5	2	4		2			4		5	3	1	1		
total cusps	0	6	2	7	1	2			9	0	8	3	1	1		
total teeth	**6**		**9**		**3**				**9**		**11**		**2**			

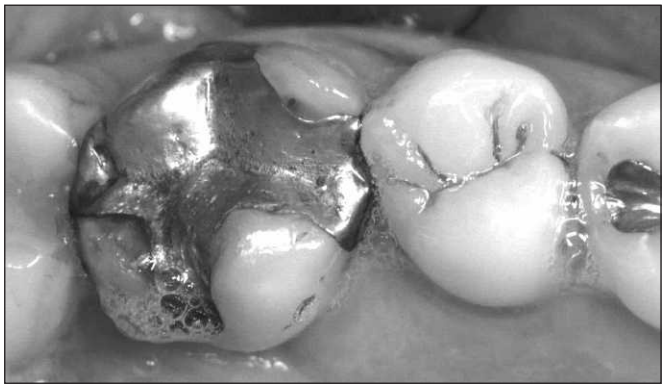


Figure 1. *First upper molar of a patient complaining about pain at cold drinks and when chewing on hard food.*

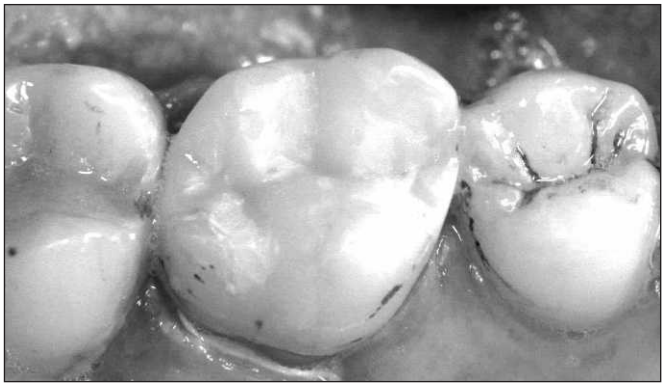


Figure 3. *This tooth was restored with a composite restoration without cusp-capping.*

At five weeks, 25 teeth (62.5%) were reported to be completely free of complaints by patients. Two patients (5%) needed endodontic treatment, one after two and the other after five weeks. Both patients suffered from increasing pain levels and the presence of spontaneous pain. When the endodontic opening was completed, a fracture line in the pulpal wall was observed in both teeth. These fracture lines, which were visible after removing the existing amalgam restorations, were situated more centrally in these teeth compared to the most common site of the fracture line at the base of the

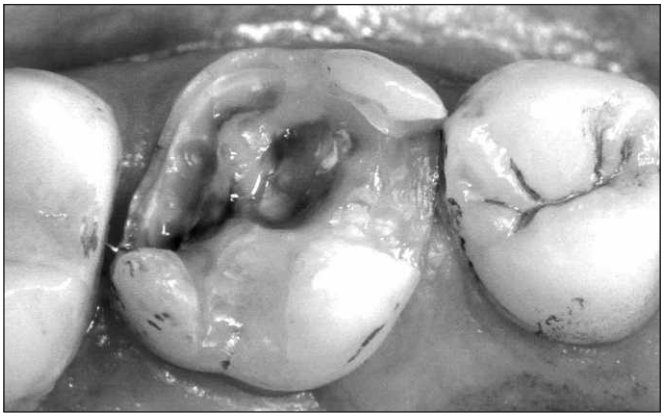


Figure 2. *At removal of the amalgam restoration, a crack can be seen at the mesiobuccal cusp.*

cusp. The endodontically-treated teeth became free of pain shortly after treatment.

At five weeks, the 38 teeth that remained vital were evaluated. Thirteen (32.5%) still had symptoms according to the patients, nine were sensitive upon contact with cold food and beverages and four patients still had complaints related to chewing and upon contact with cold food and beverages. Thirty-seven evaluated teeth were reported to be less sensitive compared to the preoperative situation. For one tooth, no differences existed between the preoperative and postoperative situation. This patient was seen in the office and it was discovered that the adjacent tooth also had symptoms of a crack. After replacing the amalgam restoration with a bonded composite restoration, this patient reported the pain had diminished considerably after four weeks.

At six months, 38 teeth that remained vital were evaluated. Thirty teeth (75%) were functioning without any complaints. One tooth was sensitive upon contact with cold food and three patients still had complaints related to chewing and upon contact with cold food and beverages. Four teeth were sensitive only upon chewing. Thirty-seven vital teeth were reported to be less sensitive compared to the preoperative situation. With one tooth, no improvement between the situation at five

weeks and six months, was reported. Upon clinical examination of the involved first mandibular molar, no sensitivity related to biting and cold application could be recorded. However, the adjacent second molar showed sensitivity upon cold application and both the lower first and second molars appeared to be sensitive upon percussion. Thus, the diagnosis was made that the teeth were sensitive due to heavy loading, probably related to clenching activity and both teeth were slightly reduced to be out of occlusion.

Table 3 shows the comparison of the six-month results of patient interviews and clinical examination. Upon clinical examination, 20 teeth (50%) were free of symptoms. Six teeth were sensitive upon application of ethylchloride, seven teeth were sensitive both upon application of Ethyl Chloride and when loading the cusps using the Tooth Slooth. Five teeth were just sensitive upon loading.

Table 4 shows the results of cuspal coverage. At six months, no statistically significant differences could be noted between the results of the teeth treated with cusp coverage and those teeth treated without cuspal coverage (Fisher's exact test $p<0.05$).

Table 2: *Results of Patient Interviews*

Time Interval	Baseline	1-Week	5-Weeks	6-Months
Number of teeth (n=40)				
Failure (endo)			2	2
Sensitive for cold and biting	40	15	4	3
Only sensitive for cold		11	9	1
Only sensitive for biting		2		4
No symptoms		12	25	30

Table 3: *Results at Six Months*

	Interview	Clinical Examination
Number of teeth (n=40)		
Failure (endo)	2	2
Sensitive for cold and biting	3	7
Only sensitive for cold	1	6
Only sensitive for biting	4	5
No symptoms	30	20

Table 4: *Results of Cuspal Coverage of Cracked Teeth*

	6 Month-Interview Symptoms		6-Month Clinical Evaluation Symptoms	
	Absent	Present	Absent	Present
Number of teeth (n=40)				
Including cuspal coverage	16	4	8	12
Without cuspal coverage	14	6	12	8
	p=0.34*		p=0.79*	

**Fischer's exact test*

DISCUSSION

This study established the effectiveness of a bonded composite to restore painful, cracked teeth. For that purpose, 40 teeth with evident symptoms of a "cracked tooth" were treated, 20 teeth received a restoration with cuspal coverage and 20 received a restoration without cuspal coverage. The results of this study show that bonded composite restorations can be successful in treating painful, cracked teeth. Furthermore, it is shown that no statistically significant effect of cuspal coverage could be demonstrated. This may lead to the conclusion that cuspal coverage is not necessary when treating cracked teeth with a bonded composite restoration. However, six months is not enough time to make definite conclusions regarding whether bonding between the fractured cusp and the restoration is strong enough to withstand the forces applied on heavily loaded teeth in the long-term. As a result, it is possible that the covered cusps will exhibit greater longevity compared to bonded-only cusps. In the coming years, restorations from this study will be examined to establish this possible effect. For clinical treatment decision-making, the choice to cusp-cap will lead to sacrificing a small amount of sound tooth structure to avoid the possible risk of cusp fracture in the long-term.

In this study, patients with painful, cracked teeth were treated with an adhesive resin composite restoration. To be sure of the diagnosis, only patients who experienced pain both on contact with cold food and beverages, as well as upon biting hard food, were included. As a third inclusion criterium, a crack had to be visible upon removal of the existing amalgam restoration. In the other available studies on cracked teeth (Homewood, 1998; Davis & Overton, 2000), the diagnosis was based solely on pain due to biting. The criteria in this study would have prevented a faulty diagnosis due to a defective restoration or supra-contacts. However, it also resulted in the exclusion of "cracked teeth" cases that exhibited pain only upon biting and no pain due to cold application as a symptom. This may have influenced the outcome of the study. After six months, some sensitivity upon biting or cold application was recorded in some cases. Also, the two failures that required endodontic treatment were recorded. Endodontic treatments were also reported as a final treatment in other studies (Homewood, 1998). Davis and Overton (2000) included patients solely on the basis of chewing pain

response and reported no failures resulting in endodontic treatment. However, they excluded patients with severe reactions upon the application of cold. Furthermore, Davis and Overton (2000) reported that pain upon biting was recorded by interviewing the patient and it was not established clinically by using a fracture-finding device. This might explain the somewhat better results obtained by Davis and Overton (2000), who showed that cracked teeth could be successfully treated by cusp-covering amalgam restorations. As described in the two cases above, sometimes patients experience pain not from the involved tooth, but as a result of problems with adjacent teeth. This phenomenon may influence a study's results and emphasizes the need for additional clinical examination.

The two teeth that needed endodontic treatment are considered to be failures in this study. A retrospective examination of the slides made of the preparation showed that these cracks were more centrally located and, in both teeth, the fracture lines were also detectable from inside the pulp chamber during endodontic treatment. Therefore, it can be assumed that teeth showing such a crack leading toward the pulp have a poor prognosis for preservation of the vitality. In this study, all the teeth that were included had amalgam restorations that indicated this may be an etiological factor for the development of a cracked tooth. Reel and Mitchell (1989) showed in an *in-vitro* study that especially MOD-restored teeth were weakened significantly compared to sound teeth.

In this study, most cracks were found in molars, with more cracks found in mandibular molars (48%) than in maxillary molars (38%). Cameron (1976) even found a crack percentage of 67% in mandibular molars and 22% in maxillary molars. This high incidence of cracks in molars, which was also described by Cameron (1976), may also be attributed to parafunctional activity of the patient (clenching). These teeth have a shorter distance to the temporomandibular joint compared to premolars, which may result in high forces applied, particularly on the second molar during clenching ("nutcracker phenomenon"). Among the clinical cases in this study, many teeth showed symptoms of bruxism and clenching, such as impressions of antagonistic cusps in the central fossa or wear facets on the involved cusps. Therefore, it can be assumed that these parafunctional activities play a major role in the etiology of cracked teeth. In this study, 14 males and 26 females were involved. However, this figure cannot be considered as an epidemiological finding since the patient population in this study was not representative of the entire population. However, the finding that cracked teeth are more prevalent among females is consistent with findings in other studies (Homewood, 1998; Cameron, 1976). It may reflect a higher number of female patients or a reduced tendency of male patients to report pain to the dentist. Also, as symptoms of craniomandibular dysfunction are more common among females than males (De Kanter & others, 1993), an explanation might be that women more often have clenching habits that can also cause cracking of the posterior teeth, especially when larger amalgam restorations are present.

The resin composite and adhesive systems used in this study have been successful in clinical studies (Wendt & Leinfelder, 1992; Prati, 2000). The composite is a heavily filled, high E-modulus composite that is suitable for large posterior restorations. The adhesive system is a three-step, total-etch system. As posterior resin composite restorations are sometimes associated with postoperative sensitivity (Christensen, 1998), it is interesting to note that this study shows that sensitive teeth can be treated with a resin composite restoration placed with a total-etch technique. As self-etching primer systems have proven to result in less postoperative sensitivity (Opdam & others, 1998; Unemori & others, 2000), whether these adhesive systems would lead to better results compared to the system used in this study makes for an interesting question. These self-etching primers are sometimes associated with a lower adhesion to enamel compared to the total-etch technique with phosphoric acid (Opdam, Roeters & Verdonschot, 1997). In this study, the adhesion to etched enamel may have played an important role in the success of the treatment. The results at six months show that more teeth were evaluated as still being painful when examined clinically compared to the results of the patient interview. One explanation for these findings may be the fact that the teeth were examined very accurately. As a result, when patients only reported a slight difference in sensitivity of teeth upon loading or cold application compared to the control teeth, the teeth were recorded as still being sensitive. It is possible that patients were not aware of these slight differences when the involved teeth function normally in the mouth. However, it is also possible that patients unconsciously avoid the painful teeth.

The literature offers several treatment options for cracked teeth. A full cusp-covering cast restoration as the restoration of choice is recommended (Geurtsen & García Godoy, 1999), but, direct restorations including cuspal coverage are also mentioned as possible options (Homewood, 1998). Cuspal coverage with metal will allow for absorbtion of fracture energies that are three times higher than intact teeth (Homewood, 1998). Cuspal coverage with direct restorations, mostly amalgams, was reported to be successful in several studies and reports (Homewood, 1998; Davis & Overton, 2000; Bearn & others, 1994). The results of this study show that, based on a six-month evaluation, bonded composite restorations can be successful in the treatment of cracked teeth, both when cusps are covered and without cuspal coverage.

As cracked teeth can be considered to have a high risk for complications, it is questionable whether an indirect restoration is the optimal treatment option. An indirect restoration will result in more loss of sound tooth tissue and will result in the need for a temporary restoration. Both factors will increase the risk for pulpal complications. When looking at cost-effectiveness, a direct restoration should be preferred to starting an endodontic treatment after placing the casting and is a considerable risk when painful, cracked teeth have to be treated. Whenever an indirect restoration is indicated, the authors recommend that it should be done when a tooth is initially treated with a direct restoration and when it has been shown to be free of symptoms for a longer period of time. Alternatively, the cracked tooth can be treated temporarily with an orthodontic band placed around the tooth as reported by Ehrmann and Tyas (1990). However, an interim direct resin composite restoration can also serve as a core for the cast restoration, when needed.

Furthermore, since many of the patients with cracked teeth exhibit symptoms of clenching, the authors are concerned about the longevity of an indirect restoration with occlusal porcelain. In such a case, it is probably better to place a metal cast restoration, which will be more fracture resistant and less abrasive to antagonistic teeth. In a total treatment concept, restoring cuspid-guidance may help to protect posterior teeth during articulation.

CONCLUSIONS

Within the limitations of this study, it can be concluded that cracked teeth may be successfully treated with a bonded composite restoration. From the results of this six-month study, it appears that cusp-capping is not necessary.

Acknowledgements

The authors thank Mrs Ten Brink for interviewing all patients. They also thank the manufacturers for supplying the materials.

(Received 2 August 2003)

References

Ausiello P, de Gee AJ, Rengo S & Davidson CL (1997) Fracture resistance of endodontically-treated premolars adhesively restored *American Journal of Dentistry* **10(5)** 237-241.

Bearn DR, Saunders EM & Saunders WP (1994) The bonded amalgam restoration-A review of the literature and report of its use in the treatment of four cases of cracked-tooth syndrome *Quintessence International* **25(5)** 321-326.

Brännström M (1986) The hydrodynamic theory of pain: Sensation in preparations, caries and the dentinal crack syndrome *Journal of Endodontics* **12(10)** 453-457.

Cameron CE (1964) Cracked tooth syndrome: Additional findings *Journal of the American Dental Association* **68** 405-411.

Cameron CE (1976) The cracked tooth syndrome, additional findings *Journal of the American Dental Association* **93** 971-975.

Christensen GJ (1996) Tooth sensitivity related to Class I and II resin restorations *Journal of the American Dental Association* **127(4)** 497-498.

Christensen GJ (1998) Preventing sensitivity in Class II composite resin restorations *Journal of the American Dental Association* **129(10)** 1469-1470.

Davis R & Overton JD (2000) Efficacy of bonded and non-bonded amalgam in the treatment of teeth with incomplete fractures *Journal of the American Dental Association* **131(4)** 469-478.

De Kanter RJ, Truin GJ, Burgersdijk RC, Van'tHof MA, Battistuzzi PG, Kalsbeek H & Kayser AF (1993) Prevalence in the Dutch adult population and a meta-analysis of signs and symptoms of temporomandibular disorder *Journal of Dental Research* **72(11)** 1509-1518.

Ehrmann EH & Tyas MJ (1990) The cracked tooth syndrome: Diagnosis, treatment and correlation between symptoms and post-extraction findings *Australian Dental Journal* **35(2)** 105-112.

Geurtsen W & García-Godoy F (1999) Bonded restorations for the prevention and treatment of the cracked-tooth syndrome *American Journal of Dentistry* **12(6)** 266-270.

Homewood Cl (1998) Cracked tooth syndrome—incidence, clinical findings and treatment *Australian Dental Journal* **43(4)** 217-222.

Opdam NJ, Roeters FJ & Verdonschot EH (1997) Adaptation and radiographic evaluation of four adhesive systems *Journal of Dentistry* **25(15)** 391-397.

Opdam NJ, Feilzer AJ, Roeters JJ & Smale I (1998) Class I occlusal composite resin restorations: *In-vivo* post-operative sensitivity, wall adaptation, and microleakage *American Journal of Dentistry* **11(5)** 229-234.

Prati C (2000) *Advanced Adhesive Dentistry*, 3rd International Kuraray symposium Cirimido-ltaly: Grafiche Erredue 267-278.

Reeh ES, Messer HH & Douglas WH (1989) Reduction in tooth stiffness as a result of endodontic and restorative procedures *Journal of Endodontics* **15(11)** 512-516.

Reel DC & Mitchell RJ (1989) Fracture resistance of teeth restored with Class II composite restorations *Journal of Prosthetic Dentistry* **61(12)** 177-180.

Salis SG, Hood JA, Kirk EE & Stokes AN (1987) Impact-fracture energy of human premolar teeth *Journal of Prosthetic Dentistry* **58(1)** 43-48.

Steele A & Johnson BR (1999) *In vitro* fracture strength of endodontically treated premolars *Journal of Endodontics* **25(1)** 6-8.

Unemori M, Matsuya Y, Akashi A, Goto Y & Akamine (2001) A composite resin restoration and postoperative sensitivity: Clinical follow-up in an undergraduate program *Journal of Dentistry* **29(1)** 7-13.

Wendt SL Jr, Harris BM & Hunt TE (1987) Resistance to cusp fracture in endodontically treated teeth *Dental Materials* **3(5)** 232-235.

Wendt SL Jr & Leinfelder KF (1992) Clinical evaluation of Clearfil photoposterior: 3-year results *American Journal of Dentistry* **5(3)** 121-125.

©Operative Dentistry, 2003, **28-4**, 334-340

Intracoronal Bleaching of Discolored Non-Vital Teeth

M Bizhang • A Heiden • U Blunck
S Zimmer • R Seemann • JF Roulet

Clinical Relevance

The modified new technique "intracoronal bleaching technique with 10% carbamide peroxide in an open chamber" was as effective as the modified walking bleach technique after six months.

SUMMARY

This clinical study compared the effectiveness of bleaching non-vital teeth with an open pulp chamber during bleaching using 10% carbamide peroxide compared to the modified walking bleach technique and extracoronal bleaching. Sixty discolored, non-vital teeth were treated. They were divided into three groups. Each group was treated with one of the bleaching materials and methods: extracoronally using 10% carbamide peroxide for two weeks as negative control (Group A), intracoronally using sodium perborate mixed with 3% hydrogen peroxide (modified walking bleach technique) (Rotstein, Mor & Friedman, 1993) for four weeks (Group B) and intracoronally and extracoronally using 10% carbamide peroxide for two weeks (Group C) (Liebenberg, 1997). Tooth color was measured at baseline, (BL), immediately post-bleaching (IP) and six months post-bleaching (SP) with a colorimeter (Castor, Sigma, Germany) using a tooth-positioning jig. The color was determined according to the CIELAB system, which records lightness as L* and chromaticity coordinates as a* and b*. The difference in L* and b* among the three groups was significant between BL and IP examination. The post-bleaching, whitening effect in Group C was significantly better, but after six months, in Group C, it was as effective as in Group B.

*Mozhgan Bizhang, dr med dent, Charité, Humboldt—University Berlin, Germany

Anke Heiden, Charité, Humboldt—University Berlin, Germany

Uwe Blunck, dr med dent, Charité, Humboldt—University Berlin, Germany

Stefan Zimmer, PD dr med dent, Charité, Humboldt—University Berlin, Germany

Rainer Seemann, dr med dent, Charité, Humboldt—University Berlin, Germany

Jean-François Roulet, prof dr med dent, Charité, Humboldt—University Berlin, Germany

*Reprint request: Center for Dental Medicine, Dept of Operative and Preventive Dentistry, Augustenburger Platz 1, 13353 Berlin, Germany; e-mail: mozhgan.bizhang@charite.de

INTRODUCTION

Discoloration of non-vital teeth is an aesthetic deficiency that requires an effective treatment. The most common cause of tooth discoloration is intracoronal blood decomposition (Walton, 1989). Incomplete root canal therapy means that necrotic debris in the pulp horns, filling materials located in the pulp chamber and endodontic sealer that lines the chamber walls could cause discoloration or a change in translucency (Brown, 1965; van der Burgt & Plasschaert, 1986). Intracoronal bleaching of non-vital teeth has offered a conservative therapy for this problem. However, external cervical

root resportion following bleaching of non-vital teeth is an important aspect to be considered. It is not possible to determine the exact cause of the root resorption, but several hypotheses have been discussed. Friedman and others (1988) found that after six months there was a resorption incidence of 6.9% in 58 teeth bleached with 30% hydrogen peroxide and heat. Heat was considered one possible reason. A second hypothesis was that hydrogen peroxide may have penetrated through the dentinal tubules to the cervical periodontal ligaments and initiated a local inflammatory reaction and caused a dental resorptive process. This phenomenon is known to be related to the type of technique and the materials used (Harrington & Natkin, 1979; Cvek & Lindvall, 1985; Madison & Walton, 1990). The most popular way to bleach is the walking bleach technique that is also safer than the thermocatalytic technique (Rotstein & others, 1993). The risks of the walking bleach technique are penetration of hydrogen peroxide through the dentinal tubules, possibility of fracture during the multiple treatment steps and over-bleaching. To overcome these problems, a new, modified technique was used for this study. The modified technique was used on a non-vital tooth with an open pulp chamber while simultaneously bleaching the tooth extra-coronally with 10% carbamide peroxide. This study compared the effectiveness of this modified technique with the modified walking bleach and the extra-coronal bleaching technique, alone.

METHODS AND MATERIALS

Subjects for the study were adult volunteers from the dental clinic in Berlin who agreed to the terms and conditions of the study and who signed an approved human-subjects consent form. The study was approved by the Ethics Committee of Charité Berlin. The subjects had to be at least 18 years old and have at least one dark tooth that they wished to have lightened. Inclusion criteria included the absence of caries. A periapical radiograph of the tooth indicated successful endodontic treatment without periapical or periodontal lesions. The subjects for the study were 43 adults (30 women and 13 men). Sixty-one discolored, non-vital teeth were treated. They were divided into three groups. Tooth color was measured at baseline (BL), immediately post-bleaching (IP) and six months post-bleaching (SP) with a colorimeter (Castor, Sigma, Germany) using a tooth-positioning jig. The color parameters were recorded in the L*, a*, b* color space as established by the Commission Internationale de L'Eclairage (CIE) in 1978 (Commission Internationale de L´Eclairage, 1978). This system is related to human color perception in all three dimensions or directions of color space. L* is a lightness variable similar to Munsell's Value, a* and b* are chromaticity coordinates. The a* coordinate corresponds to the red/green axis in the Munsell color space. A positive a* value relates to a red color, whereas a negative a* value denotes a color that is more green. Similarly, the b* coordinate corresponds to the yellow-blue axis. A positive b* value relates to a yellow color, whereas a negative b* value denotes a blue color. ΔE^* is accepted as the difference in color value, which is determined by including each of the three parameters $\Delta E^*=(\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$.

The color measurements were repeated six times and the color coordinate values were averaged for each tooth.

The shades of the teeth were also recorded with a value-oriented shade guide (Vita Zahnfabrik). The sequence of light-to-dark shade tabs, as ordered by the manufacturer, was numbered from 1 to 16 (Table 1).

Group A: Twenty teeth were selected for this group. An alginate impression (Palga Plus, 3M ESPE AG, Seefeld, Germany) was taken of the arch in which the discolored tooth was located. The impression was poured with a dental stone (Röconit, Röhrich & Co GmbH, Berlin, Germany) and a custom-fitted tray was fabricated. Light-cured resin material (LC Blockout, Ultradent Products, Inc, South Jordan, UT, USA) was used to create a reservoir on the facial surface of the non-vital tooth to accommodate a supplementary subject-applied nightguard bleaching technique. A heat and vacuum tray-forming machine (Sta-Vac, Buffalo Mfg, Syosset, NY, USA) was used to fabricate the trays. The tray was 0.9-mm thick (Sof-Tray, Ultradent Products, Inc) and covered all teeth. The teeth were bleached extracoronally for two weeks with 10% carbamide peroxide (Opalescence, 10% CP Ultradent Products, Inc) using the described custom-fitted tray.

Group B: Twenty teeth were bleached intracoronally. After rubber dam placement, the access restoration was removed. Approximately 3 mm of root canal filling material was removed in an apical direction beyond the clinical height of the crown (incisogingival height). This procedure has a twofold purpose: to create space for application of the cervical seal and to expose dentinal tubules directed toward the cervical region of the teeth. A prophylactic calcium hydroxide base approximately 1.0-mm thick was applied in direct contact with the root canal filling material. A base of a glass-ionomer cement approximately 2-mm thick was applied on top of the

Table 1: *Ordering of Vital Shade Guide by Value (Light-to-Dark Ranking by Manufacturer)*

Tab	B1	A1	B2	D2	A2	C1	C2	D4	A3	D3	B3	A3.5	B4	C3	A4	C4
Rank	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16

calcium hydroxide. Sodium perborate (Mereck Company, Darmstadt, Germany) mixed with 3% hydrogen peroxide (Geyer Company, Hamburg, Germany) was applied to the pulp chamber. The lingual access was sealed with a glass ionomer cement. Every seven days, the bleaching agent was replaced by fresh material. After 28 days, the access restoration was removed, the pulp chamber was flushed generously with water to remove the bleaching agent (Walton, 1989) and the pulp chamber was obturated with a calcium hydroxide and water paste to be left in the chamber for seven days. This procedure is intended to render the pH alkaline in the cervical region of the teeth (Kehoe, 1987). After that time, the coronal access cavities were sealed with resin composite.

Group C: Twenty-one teeth were bleached intracoronally and extracoronally. They were prepared as in the modified "walking bleach" technique, so that the root canal was sealed from the pulp chamber. A calcium hydroxide base, approximately 1 mm and a glass-ionomer cement base, approximately 2 mm in thickness, was applied to the root canal orifice. Ten percent carbamide peroxide (Opalescence, 10% CP Ultradent Products, Inc) was injected into the pulp chambers of the non-vital teeth and simultaneously loaded into the custom-fitted tray for all teeth. Subjects were instructed in the technique of inserting a cotton pellet into the access cavity of the tooth during the day and changing the pellet after meals, using tweezers. This was done to prevent food impaction in the orifice. The first three days the subjects were encouraged to limit application of bleaching agents to daylight hours. The subjects wore the tray for three consecutive hours daily and replaced the bleaching agents at the beginning of each hour, the rest of each day they wore the tray unloaded. If any allergic reaction occurred, they were to discontinue the bleaching. At bedtime, the cotton was removed and the tooth was irrigated as before. Then, 10% carbamide peroxide was loaded into the bleaching tray and injected into the orifice. They applied fresh bleach agent nightly for 11 days.

Data Analysis

Data analysis was accomplished using Kruskal-Wallis and Mann-Whitney U Tests (SPSS 10.0). Changes in the L*, a* and b* color parameters between the BL, IP and SP examination for each group were compared. The Tests were performed with p=0.05 to test for significant differences among the three groups.

RESULTS

The drop-out quota for this study at recall was 3.2% (two teeth in one subject after six months). The mean age was 38.06 ± 14.73 in Group A, 35.80 ± 11.35 in Group B and 36.00 ± 7.54 in Group C. Thirty-two subjects bleached one tooth, seven subjects two teeth, two subjects three teeth, one subject bleached four teeth and one subject five teeth. The teeth included 54 maxillary incisors, three maxillary canines, two maxillary premolars and two mandibulary incisors. No fractures were observed during the treatment periods, nor were any external cervical resorptions observed after six months post-treatment in any of the subjects.

For each technique, a Friedman test and Wilcoxon two-sample test was used. The mean and standard deviation (SD) of L*, a* and b* at baseline, post-bleaching and six months post-bleaching are shown for each group in Table 2 (p<0.05).

Table 3 reflects the mean SD of ΔL*, Δa* and Δb* from baseline to post-bleaching (BL-IP), from baseline to six months post-bleaching (BL-SP) and from post-bleaching to six month post-bleaching (IP and SP) for each group.

The Kruskal-Wallis test and Mann-Whitney test of color change determined that there were changes in L* between BL and IP (ΔL^*_0) that were statistically significant between Groups A and C and between Group B and C (Figure 1).

However, no statistically significant differences were observed in changes of the color parameter between BL and IP (Δa^*_0), between BL and SP (Δa^*_1) and between IP and SP (Δa^*_2) for each group (Figure 2).

There were changes in the b* color between BL and IP (Δb^*_0) that were statistically significant between Groups B and C, between IP and SP (Δb^*_2), between Groups A and C and between Groups B and C (Figure 3).

The Table 4 shows the mean and S of ΔE* (overall changes in 3-D color space) from baseline to post-bleaching (BL-IP), from baseline to six month post-bleaching (BL-SP) and from post-bleaching to six month post-bleaching (IP-SP) for each group (p<0.05).

There were ΔE* between post-bleaching and six months post-bleaching (IP-SP) that were statistically significant between Groups A and C and between Groups B and C (p<0.05). There was a tendency for significant differences (p=0.054) between baseline and post-bleaching (BL-IP) between Groups B and C (Figure 4).

For each technique, the shades of the teeth were recorded using Vita-Lumin baseline (BL), post-bleaching (IP), and six months post-bleaching of shades of the teeth with Vita Zahnfabrik were recorded. Subjects from Groups B and C had a statistically significant difference for the change in shade guide between BL and IP, between BL and SP and between IP and SP, but there was no statistically significant difference between each examination for Group A (Table 5, p<0.05).

Table 2: *Mean and (SD) of Tooth Color from Pretreatment Baseline Measurement (BL), Immediately Post-Bleaching (IP) and Six Months Post-Bleaching for Each Group (Friedman & Wilcoxon Tests)*

Mean	L*			a*			b*		
(SD)	BL	IP	SP	BL	IP	SP	BL	IP	SP
Group A	54.76[a,b] (3.13)	59.21[a,c] (5.92)	57.84[b,c] (5.68)	0.76[a,b] (1.87)	-0.22[a] (1.52)	-0.02[b] (1.60)	0.20[a,b] (7.47)	-5.53[a] (7.79)	-4.50[b] (7.82)
Group B	56.93[a,b] (6.03)	61.30[a] (5.95)	59.27[b] (5.78)	0.00[a,b] (1.66)	-1.23[a] (1.28)	-1.04[b] (1.32)	-5.55 (5.64)	-7.10 (5.42)	-7.58 (5.68)
Group C	56.69[a,b] (5.54)	67.34[a] (8.37)	60.23[b] (6.05)	-0.46[a] (1.08)	-1.71[a,b] (0.91)	-1.00[b] (0.84)	-3.86[a,c] (6.44)	-12.78[a,b] (5.72)	-9.36[b,c] (5.91)

Values by the same letter [(a,b,c)] within rows having the same superscript were significantly different (p<0.05).

Table 3: *Mean and (SD) of Tooth Color from Baseline Measurement to Post-Bleaching (BL-IP), from Baseline to Six Months Post-Bleaching (BL-SP), and From Post-Bleaching to Six Months Post-Bleaching (IP-SP) for Each Group (Friedman & Wilcoxon Tests)*

Mean	ΔL*			Δa*			Δb*		
(SD)	BL-IP	BL-SP	IP-SP	BL-IP	BL-SP	IP-SP	BL-IP	BL-SP	IP-SP
Group A	4.45[a,b] (5.00)	3.20[a,c] (4.32)	-1.42[b,c] (1.62)	-0.97[a] (1.16)	-1.02[b] (0.94)	0.09[a,b] (0.52)	-5.73[a] (7.86)	-5.45[b] (7.75)	0.66[a,b] (3.45)
Group B	4.37[a,b] (6.84)	2.34[a] (4.74)	-2.03[b] (6.75)	-1.23[a] (1.20)	-1.04[b] (1.33)	0.19[a,b] (0.95)	-1.55 (4.98)	-2.03 (5.33)	-0.48 (5.15)
Group C	10.65[a,b] (8.85)	3.54[a,c] (4.46)	-7.11[b,c] (9.71)	-1.26[a,b] (1.48)	-0.54[a,c] (1.28)	0.71[b,c] (0.96)	-8.92[a,b] (5.85)	-5.50[a,c] (3.69)	3.43[b,c] (3.54)

Values by the same letter [(a,b,c)] within rows having the same superscript were significantly different (p<0.05).

Table 4: *Mean (SD) of DE Between Baseline and Post-Bleaching (BL-IP), Between Baseline and Six Months Post-Bleaching (BL-SP), and Between Post-Bleaching and Six Months Post-Bleaching (IP-SP) for Each Group*

Mean (SD)	ΔE		
	BL-IP	BL-SP	IP-SP
Group A	8.53[a] (8.24)	7.89[b] (7.56)	3.16[a,b] (2.61)
Group B	8.28 (5.01)	6.63 (4.20)	6.58 (5.65)
Group C	14.66[a,b] (9.67)	8.17[a] (3.29)	9.86[b] (8.46)

Values by the same letter [(a,b,c)] within rows having the same superscript were significantly different (p<0.05).

Table 5: *Mean and (SD) of Change in Shade Guide Color Measurement From Baseline Measurement (BL), Immediately Post-Bleaching (IP), and Six Months Post-Bleaching (SP) for Each Group*

Mean (SD)	BL	IP	SP
Group A	12.35 (4.60)	8.80 (4.80)	8.39 (4.89)
Group B	11.50[a,b] (3.43)	3.45[a,c] (2.50)	6.85[b,c] (4.66)
Group C	12.14[a,b] (5.12)	2.62[a,c] (1.63)	5.95[b,c] (3.63)

Values by the same letter [(a,b,c)] within rows having the same superscript were significantly different (p<0.05).

DISCUSSION

Non-vital tooth bleaching has some risks. A few authors have reported cervical root resportion associated with intracoronal bleaching (Goon, Cohen & Borer, 1986; Latcham, 1986; Friedman & others, 1988). The exact cause of the phenomenon has not been fully clarified. A possibility for preventing cervical resorption may be reducing the concentration of bleaching material. McCormick, Weine and Maggio (1983) found that polymorphonuclear leukocytes and osteoclasts function best at a slightly acidic pH, promoting acid hydrolysis, which leads to demineralization of hard tissue components and prevents new hard tissue formation. If this pH change occurs in the micro-environment of the cervical periodontal ligament *in vivo*, external inflammatory root resorption of the tooth could result. The pH-value of 30% hydrogen peroxide is 2-3; of sodium perborate, it is 10-12. The walking bleach agents in this study were sodium perborate and 3% hydrogen peroxide. The agents were combined to hopefully prevent external root resportion. In the modified method, a neutral pH, 10% carbamide

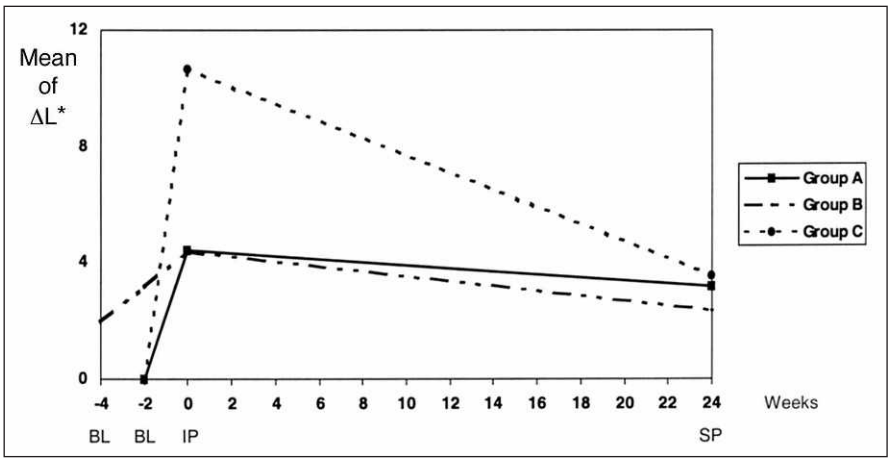


Figure 1. *Mean ΔL* from baseline measurement (BL, t=-4 weeks, -2 weeks), post-bleaching (IP, t=0 week) and six months post-bleaching (SP, t=24 weeks) for each group.*

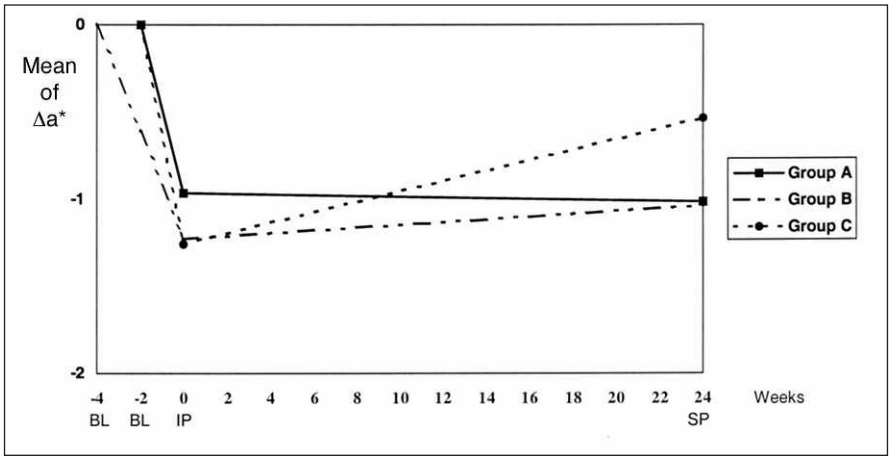


Figure 2. *Mean Δa* from baseline measurement (BL, t=-4 weeks, -2 weeks), post-bleaching (IP, t=0 week) and six months post-bleaching (SP, t=24 weeks) for each group.*

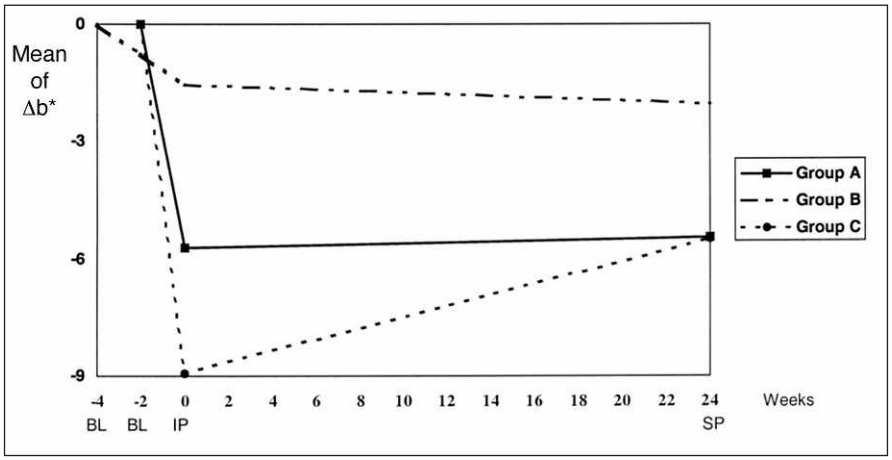


Figure 3. *Mean Δb* from baseline measurement (BL, t=-4 weeks, -2 weeks), post-bleaching (IP, t=0 week) and six months post-bleaching (SP, t=24 weeks) for each group.*

peroxide was used. Ten percent carbamide peroxide breaks down to 3.4% hydrogen peroxide and is classified as an "oral antiseptic" by the US Food and Drug Administration (FDA) monograph of 1988 and, therefore, minimizes the risks (Haywood, 1993). The base of a calcium hydroxide and a glass-ionomer cement were placed on top of a gutta-percha filling before non-vital bleaching procedures B and C were started. This has two effects: first, to prevent diffusion of the bleaching agent through the tooth to the periodontal ligaments and second, to infiltrate through the root canal to the periapical region of the teeth. Calcium hydroxide paste was used as a temporary dressing in the pulp chamber after bleaching. The rationale for its use is based on the buffering potential of calcium hydroxide, which can avoid the decrease in pH caused by bleaching with hydrogen peroxide (Kehoe, 1987). Since changes in pH have been implicated in the external root resorption process, temporary dressing with calcium hydroxide could help to prevent this. Interaction between residual peroxide or peroxide-related substances and the restorative material could interfere with its adhesion to enamel (Titley & others, 1993). However, such temporary dressing with calcium hydroxide has not been investigated in relation to microleakage of adhesive restorations. As adhesion has been related to microleakage, lower values for bond strength after tooth bleaching may facilitate microleakage around restorations (Retief, Mandras & Russel, 1994). Barkhordar, Kempler and Plesh (1997) detected increased microleakage when composite restorations were placed after the walking bleach treatment. They also demonstrated that more microleakage was observed when longer bleaching treatments were performed.

Another benefit of the modified technique is that the subject controls the bleaching effect on his teeth. Overbleaching can be avoided. Repeated replenishment at home allows a comfortable treatment for both office staff and subjects. The subject can receive treatments for a longer period of time without having to return to the office.

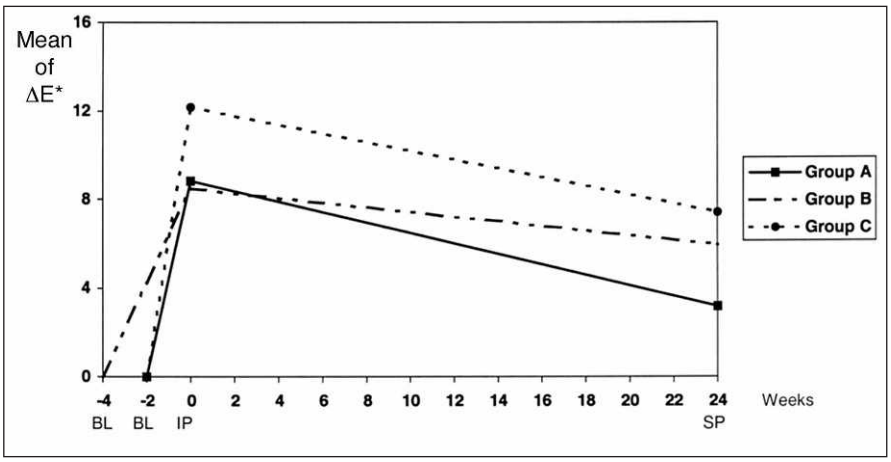


Figure 4. *Mean ΔE* from baseline measurement (BL, t=-4 weeks, -2 weeks), post-bleaching (IP, t=0 week), and six months post-bleaching (SP, t=24 weeks) for each group.*

During the bleaching process with the modified technique, the tooth had no restoration and, consequently, there was a risk of fracture. To prevent fracture, the subject should be advised to chew carefully in that region. The disadvantage of this relates to subject compliance since it is essential in the subject-applied technique for the subject to return for a final restoration. An open chamber cavity has a higher risk of fracture and a higher risk of periradicular inflammation than a cavity with restoration (Ray & Trope, 1995). The open teeth can easily succumb to caries and be lost if the tooth remains unrestored. There is no concern about caries during the process because 10% carbamide peroxide materials have anticariogenic properties and elevate the pH higher than the area of carious activity (Leonard & others, 1994).

In 1965, Brown found that 25% of the teeth bleached intracoronally with hydrogen peroxide failed over a period of one-to-five years. The color regression suggests that long-term bleaching prognosis is time-dependent (Brown, 1965; Rotstein & others, 1993).

The immediate aesthetic results of bleaching treatments with the modified technique were better than the walking bleach technique, but after six months, there was a color regression. In a clinical *in vivo* study, Carrillo, Arredondo Trevino and Haywood (1998) used the same technique. They also reported that this technique, immediately after bleaching, can provide an effective method to lighten non-vital teeth. They observed an average shade change of 14.9 in the non-vital teeth. The multiple treatment steps in the walking bleach technique are a major disadvantage. The bleaching results with sodium perborate and 3% hydrogen peroxide may even increase the number of treatments. The period of walking bleach treatment in this study was four weeks (Holmstrup, Palm & Lambjerg Hansen, 1988; Brown, 1965; Walton, 1989). The modified technique consists of application of the bleaching agent by the subject within and outside the tooth simultaneously for two weeks (Liebenberg, 1997; Carrillo & others, 1998). Regarding bleaching results, there was no difference between the modified technique and the walking bleach. However, the treatment time could be reduced by 50% using the modified technique.

Remarkable color changes usually observed in the tooth bleaching procedure are an increase of L* (lightness) and a decrease of b* (less yellow) (Rosenstiel, Gegauff & Johnston, 1991). Because the most important color change is an increase in whiteness (Frysh & others, 1995), in this study, the immediate changes in L* and b* were better for the modified technique, but after six months they were less evident. For the modified technique, the bleaching effect after six months was as effective as the walking bleach technique.

CONCLUSIONS

The results in the study showed that the modified technique using 10% carbamide peroxide was effective in bleaching discolored, non-vital teeth within two weeks. After six months, the modified technique was still as effective as the walking bleach technique. Yet, the decision issue was the time factor. The modified technique reduced the treatment time in comparison to the walking bleach technique by 50%.

(Received 16 August 2002)

References

Barkhordar RA, Kempler D & Plesh O (1997) Effect of non-vital tooth bleaching on microleakage of resin composite restorations *Quintessence International* **28(5)** 341-344.

Brown G (1965) Factors influencing successful bleaching of the discolored root-filled tooth *Oral Surgery* **20** 238-244.

Carrillo A, Arredondo Trevino MV & Haywood VB (1998) Simultaneous bleaching of vital tooth and an open-chamber non-vital teeth with 10% carbamide peroxide *Quintessence International* **29(10)** 643-648.

Commission Internationale de L´Eclairage (1978) Recommendations on uniform color spaces. Color difference equations. Psychometric color terms. Paris: Bureau Central de la CIE suppl 2 to CIE publication 15 (E-13.1) 1971/(TC-1.3).

Cvek M & Lindvall AM (1985) External root resorption following bleaching of pulpless teeth with oxygen peroxide *Endodontics and Dental Traumatology* **1(2)** 56-60.

Friedman S, Rotstein I, Libfeld H, Stabholz A & Heling I (1988) Incidence of external root resorption and esthetic results in 58 bleached pulpless teeth *Endodontics and Dental Traumatology* **4(1)** 23-26.

Frysh H, Bowles WH, Baker F, Rivera-Hidalgo F & Guillen G (1995) Effect of pH on hydrogen peroxide bleaching agents *Journal of Esthetic Dentistry* **7(3)** 130-133.

Goon WW, Cohen S & Borer RF (1986) External cervical root resorption following bleaching *Journal of Endodontics* **12** 414-418.

Harrington GW & Natkin E (1979) External resorption associated with bleaching of pulpless teeth *Journal of Endodontics* **5(11)** 344-348.

Haywood VB (1993) The Food and Drug Administration and its influence on home bleaching *Current Opinion in Cosmetic Dentistry* 12-18.

Holmstrup G, Palm AM & Lambjerg Hansen H (1988) Bleaching of discoloured root-filled teeth *Endodontics and Dental Traumatology* **4(5)** 197-201.

Kehoe JC (1987) pH reversal following *in vitro* bleaching of pulpless teeth *Journal of Endodontics* **13(1)** 6-9.

Latcham NL (1986) Postbleaching cervical resorption *Journal of Endodontics* **12** 262-264.

Leonard RH Jr, Austin SM, Haywood VB & Bentley CD (1994) Change in pH of plaque and 10% carbamide peroxide solution during nightguard vital bleaching treatment *Quintessence International* **25(12)** 819-823.

Liebenberg WH (1997) Intracoronal lightening of discolored pulpless teeth: A modified walking bleach technique *Quintessence International* **28(12)** 771-777.

Madison S & Walton R (1990) Cervical root resorption following bleaching of endodontically treated teeth *Journal of Endodontics* **16(12)** 570-574.

McCormick JE, Weine FS & Maggio JD (1983) Tissue pH of developing periapical lesions in dogs *Journal of Endodontics* **9(2)** 47-51.

Ray HA & Trope M (1995) Periapical status of endodontically-treated teeth in relation to the technical quality of the root filling and the coronal restoration *International Endodontic Journal* **28(1)** 12-18.

Retief DH, Mandras RS & Russel CM (1994) Shear bond strength required to prevent microleakage of the dentin/restoration interface *American Journal of Dentistry* **7(1)** 44-46.

Rosenstiel SF, Gegauff AG & Johnston WM (1991) Duration of tooth color change after bleaching *Journal of the American Dental Association* **122(4)** 54-59.

Rotstein I, Mor C & Friedman S (1993) Prognosis of intracoronal bleaching with sodium perborate preparation *in vitro*: 1-year study *Journal of Endodontics* **19(1)** 10-12.

Titley KC, Torneck CD, Ruse ND & Krmec D (1993) Adhesion of a resin composite to bleached and unbleached human enamel *Journal of Endodontics* **19(3)** 112-115.

van der Burgt TP & Plasschaert AJ (1986) Bleaching of tooth discoloration caused by endodontic sealers *Journal of Endodontics* **12(6)** 231-234.

Walton R (1989) Bleaching discolored teeth: Internal and external in Walton RE TMe (ed) *Principles and Practice of Endodontics* Saunders Philadelphia p 389-397.

©Operative Dentistry, 2003, **28-4,** 341-345

Laboratory Research

Fluorescence-Aided Caries Excavation (FACE) Compared to Conventional Method

ÁM Lennon

Clinical Relevance

A new caries excavation method that uses a fluorescence diagnostic procedure during excavation allows the operator to identify and remove bacterially-infected dentin more successfully than with the conventional method that uses visual tactile criteria for identification of caries.

SUMMARY

A recent study showed that orange-red fluorescence in carious dentin could be used to detect residual caries (Lennon & others, 2002). This study compared the ability of a new fluorescence-aided caries excavation technique (FACE) with the conventional method. Forty extracted teeth with occlusal dentin caries were selected. The teeth were bisected longitudinally through the center of the lesion. Lesion depth and width were measured and the teeth were divided into two groups of 20, each with the same average lesion size. The tooth halves were reassembled and fixed by embedding the roots in acrylic resin. Access cavities were prepared using a high-speed handpiece and diamond fissure bur. In the FACE group, violet light (370–420 nm) was fed into the fiber optics of a slow-speed handpiece, so that it illuminated the operating field. The cavity was observed through a 530-nm high-pass filter and orange-red fluorescing areas were removed. In the conventional group, a sharp probe was used to detect soft dentin, which was removed. One-half of each tooth was stained for bacteria using Ethidium Bromide and examined using Confocal Laser Scanning Microscopy (CLSM). Bacteria were present in significantly (p=0.037) fewer FACE samples (3) compared to conventional samples (9). It can be concluded that the new method is more effective than conventional caries excavation.

*Áine M Lennon, b dent sc, dr med dent, Department of Operative Dentistry, Preventive Dentistry and Periodontology, Georg-August-University Göttingen, Göttingen, Germany; Oral Health Research Institute, Department of Preventive and Community Dentistry, Indiana University School of Dentistry, Indianapolis, IN

*Reprint request: Robert-Koch-Str 40, 37075 Göttingen, Germany; e-mail: lennon@med.uni-goettingen.de

INTRODUCTION

Most of the dental restorations placed in Scandinavia, the UK and the USA during the last 20 years were replacements rather than initial restorations (Deligeorgi, Mjör & Wilson, 2001). If infected dentin is not completely removed before placing a restoration, caries can recur. Most commonly, dentists decide whether dentin should be excavated or not based on the color and hardness of the tissue. This decision is often difficult clinically and recurrent caries is still one of the major reasons for restoration replacement (Dahl & Eriksen, 1978; Pink, Minden & Simmonds, 1994).

Caries detector dyes were introduced in the 1970s to help identify infected dentin (Sato & Fusayama, 1976). Kidd, Joyston-Bechal and Beighton (1993) have shown that there is no difference in the level of infection of dye-stained and dye-unstained sites at the Dentin Enamel Junction (DEJ) and concluded that the use of a caries detector dye on hard and stain-free dentin will result in unnecessary tissue removal. Since these dyes also stain normal circumpulpal dentin, their use may result in unnecessary removal of healthy tissue (McComb, 2000). Chemomechanical systems recently introduced for caries removal have also been investigated. However, their usefulness at the Dentin Enamel Junction (DEJ) appears to be limited (Cederlund, Lindskog & Blomlöf, 1999a) and there are concerns that they may damage the collagenous component of dentin which is needed for adhesive restorations (Cederlund, Lindskog & Blomlöf, 1999b). Therefore, an accurate and reliable method for residual caries detection is still needed.

Because bacteria in dentin are not visible to the observer, methods for detection of residual caries have focused until now on identifying tissue that has already been damaged by the caries process, for example, demineralized dentin (caries detector dyes) or dentin where the collagen has been denatured (chemomechanical methods [Carisolv]). However, several oral microorganisms are known to produce fluorescing molecules or "fluorophores" that emit in the yellow to red area of the visible spectrum under certain excitation wavelengths (König & Schneckenburger, 1994).

A recent *in vitro* study showed that exciting carious dentin with violet blue light caused visible orange-red fluorescence that could be used successfully to identify residual caries (Lennon & others, 2002).

This study evaluated a new caries excavation technique that is based on the previously described fluorescence diagnostic procedure.

METHODS AND MATERIALS

Specimen Preparation

Extracted human premolars and permanent molars with occlusal caries were collected and stored in 0.01% thymol solution. All teeth were sectioned longitudinally under continuous water cooling (Ultraslice 2000, Ultratec, Santa Ana, CA, USA) through the center of the lesion in a mesiodistal direction. After examining the tooth halves using stereomicroscopy, 40 samples with caries at least 1 mm into dentin and 1 mm clear of the pulp were chosen. The selected teeth had a single occlusal lesion and were free of any restorations.

Lesion depth and width were measured and the teeth were distributed into two groups of 20 teeth, each group having the same average and total lesion size. Tooth halves were reassembled and fixed by embedding the roots in acrylic resin (Samplquik, Buhler, USA) to the level of the DEJ.

Excavation

Caries excavation was carried out by one operator for both groups. No magnification was used. Samples were removed from the storage solution for excavation. They were then replaced in storage solution immediately after excavation.

FACE Group

Access cavities were prepared using a 557-diamond bur in a high-speed handpiece (Star Dental, Lancaster, PA, USA) under continuous water-cooling. Violet light (370–420 nm) was generated using a 35-watt Xenon discharge lamp and a blue band pass filter with peak transmission at 370 nm (taken from QLF system, Inspektor Research Systems BV, Amsterdam, The Netherlands). This light was fed into the fibreoptics of a slow-speed handpiece (Star Dental) so that it illuminated the operating field during excavation (Figure 1). The operator observed the cavity through a 530-nm yellow glass filter (OG530, Schott, Mainz, Germany) in a darkened room. Areas exhibiting orange-red fluorescence were selectively removed using stainless steel round burs sizes 4 and 6.

Conventional Excavation Group

Access cavities were prepared using a 557-diamond bur in a high-speed handpiece (Star Dental) under continuous water-cooling. During excavation samples were illuminated using a standard dental unit light. Brown and yellow stained dentin and/or softened dentin detected using a sharp explorer (EX85, Hu-Friedy Inc, Chicago, IL, USA) were removed from the DEJ. Soft dentin was removed from the rest of the cavity. Caries

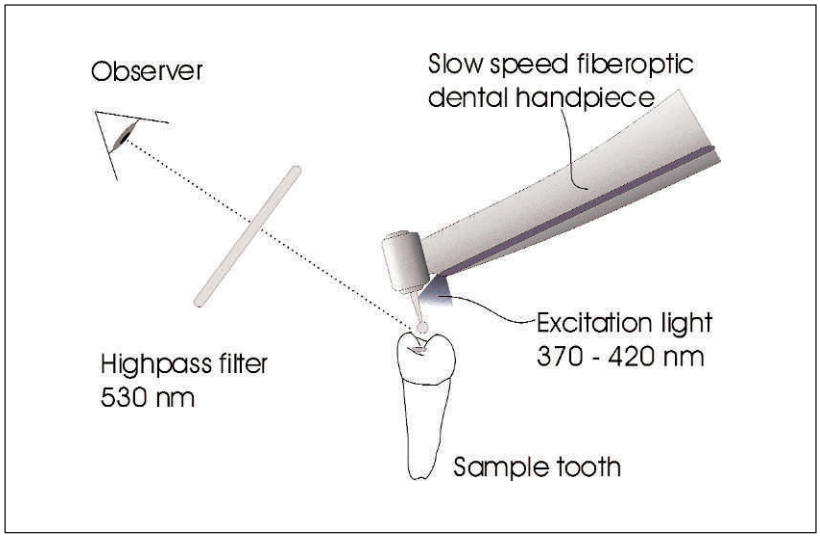


Figure 1. *FACE excavation method.*

was removed using stainless steel round burs sizes 4 and 6 in a slow-speed handpiece (Star Dental).

Histology

The acrylic base and roots of each sample were removed (Ultraslice 2000, Ultratec, Santa Ana, CA, USA) and the teeth were disassembled into two halves. One half of each sample was used for histology. Samples were fixed in 70% ethyl alcohol, then rinsed extensively in phosphate buffered saline, pH 7.2 (PBS). Samples were placed in a 1:500 concentration of 10 mg/ml ethidium bromide (Molecular Probes, Eugene, OR, USA), vortexed and incubated at 37°C for 20 minutes. Samples were then rinsed extensively in PBS.

The tooth halves were analyzed for the presence of fluorescent stain using a confocal laser-scanning microscope (Odessey, Noran Instruments, Middleton, WI, USA). A 488 nm argon ion laser was used for excitation. A 515-nm barrier filter, a 15 µm confocal detection slit and a 60X oil immersion objective were used for detection. Scans were made at a depth of approximately 10 µm below the cut surface and along the cavity outline. The sample edge was excluded to avoid false positives due to surface contaminants.

Samples were scored positive for residual caries when bacteria were identified. Samples were scored negative for residual caries when no bacteria were identified.

Statistical Analyses

The difference between the two groups was statistically analyzed using the Pearson Chi squared test. The level of significance was set at $p<0.05$.

Data were analyzed using SPSS software version 10.1 for windows.

RESULTS

A cross-tabulation of the results is presented in Table 1. No pulp exposures occurred. All samples had sufficient dentin remaining after excavation to allow staining and analysis using CLSM. In the conventional group, residual caries was detected in nine samples (Figure 3). The remaining 11 samples were caries free. In the FACE group, residual caries was detected in three samples (Figure 2), while 17 samples were caries free. Residual caries was detected in significantly fewer FACE samples than conventionally excavated samples ($p=0.037$).

DISCUSSION

This paper describes, for the first time, the use of a new dental handpiece that combines a fluorescence diagnostic procedure with caries removal. In a previous study, it was shown that visible orange-red fluorescence in carious dentin could be used to detect residual caries (Lennon & others, 2002). In the earlier study, however, flat samples were used and caries was removed by grinding. In contrast, both the samples and the excavation procedure used in the current study

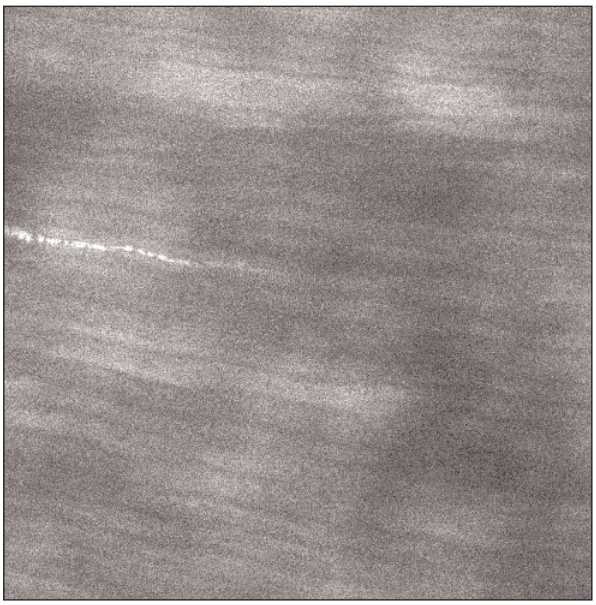


Figure 2. *Confocal micrograph of sample with residual caries following FACE excavation (1720x).*

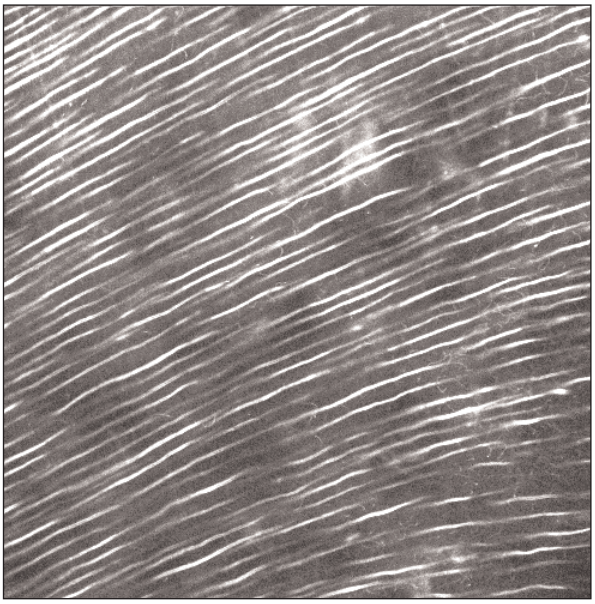


Figure 3. *Confocal micrograph of sample with residual caries following conventional excavation (1720x).*

Table 1: *Cross-tabulation of Results*

Group	Caries Free	Residual Caries	Total
FACE	17	3	20
Conventional excavation	11	9	20
Total	28	12	40

were three-dimensional and therefore similar to the clinical situation.

Porphyrins and metalloporphyrins produced by some oral microorganisms as metabolic byproducts are thought to be responsible for orange-red fluorescence in carious dental tissues (König, Flemming & Hibst, 1998). These fluorophores typically have absorption maxima between 398 and 421 nm and emission maxima between 530 and 633 nm (König & Schneckenburger, 1994). Because emission occurs in the visible portion of the electromagnetic spectrum, it can be detected by visual inspection using the appropriate high-pass filters (Lennon & others, 2002).

In this current study, teeth with caries extending very close to the pulp were excluded to avoid the possibility of pulp exposure and because samples should still have sufficient dentin after caries removal to allow staining and CLSM examination of the dentin tubules.

It is recommended that infected dentin be completely removed before a restoration is placed (Weerheijm & others, 1999). Demineralized but not infected dentin is thought to be remineralizable and should be conserved rather than removed (Fusayama & Kurosaki, 1972). Therefore, laboratory techniques to evaluate the success of excavation techniques should specifically detect infected dentin remaining after excavation (residual caries) rather than demineralization.

Confocal microscopy has been used in conjunction with immunofluorescent labels to identify bacteria in caries lesions in the past (Gonzalez-Cabezas & others, 1999). The disadvantage of using a specific antibody label for detection of residual caries is that only one bacterial species can be labeled. The microflora of dentin caries is complex (Pekovic & others, 1987) and, therefore, it is preferable to use a technique that reveals the presence of bacteria regardless of species. Ethidium bromide is a fluorescent nucleic acid stain that has been used extensively for identification of bacteria in plaque regardless of species (Netuschil, 1983). Because the odontoblast nucleus is situated at the border of the pulp chamber and not within dentin itself, a nucleic acid stain can be used to label bacterial nucleic material within the dentin tubules.

Although the new method was more successful in removing bacterially-infected dentin than conventional excavation, bacteria were still detected in three samples after FACE excavation. The main difference between CLSM and FACE is that CLSM specifically identifies bacteria, whereas, FACE detects fluorescence produced by bacterial by-products. Another difference is the specificity of the methods. The gold standard (CLSM) is capable of identifying a single bacterium, whereas, the FACE method relies on the abilities of the human eye and may not detect very small amounts of fluorescence. The numbers of bacteria present in the positive FACE samples appeared to be much less than that in the positive conventional samples. However, bacteria present in the samples were not quantified and this would be an interesting question for future studies.

The gold standard used in this study tested the ability of the respective excavation methods to remove bacteria-infected dentin. An accurate excavation technique should, however, not only successfully remove infected tissue but also conserve sound tissue, and this aspect should also be addressed in the future.

CONCLUSIONS

Within the limitations of this *in vitro* investigation, it can be concluded that excavation using FACE results in significantly fewer cases of residual caries than conventional excavation.

(Received 7 June 2002)

References

Cederlund A, Lindskog S & Blomlöf J (1999a) Efficacy of Carisolv-assisted caries excavation *International Journal of Periodontics Restorative Dentistry* **19(5)** 464-469.

Cederlund A, Lindskog S & Blomlöf J (1999b) Effect of a chemomechanical caries removal system (Carisolv) on dentin topography of non-carious dentin *Acta Odontolgica Scandinavica* **57(4)** 185-189.

Dahl J & Eriksen H (1978) Reasons for replacement of amalgam dental restorations *Scandinavian Journal of Dental Research* **86(5)** 404-407.

Deligeorgi V, Mjör I & Wilson N (2001) An overview of reasons for the placement and replacement of restorations *Primary Dental Care* **8(1)** 5-11.

Fusayama T & Kurosaki N (1972) Structure and removal of carious dentin *International Dental Journal* **22(3)** 401-411.

Gonzalez-Cabezas C, Li Y, Gregory R & Stookey G (1999) Distribution of three cariogenic bacteria in secondary carious lesions around amalgam restorations *Caries Research* **33(5)** 357-365.

Kidd E, Joyston-Bechal S & Beighton D (1993) The use of a caries detector dye during cavity preparation: A microbiological assessment *British Dental Journal* **174(7)** 245-248.

König K, Flemming G & Hibst R (1998) Laser-induced auto-fluorescence spectroscopy of dental caries *Cellular Molecular Biology* (Noisy-le-grand) **44(8)** 1293-1300.

König K & Schneckenburger H (1994) Laser-induced auto-fluorescence for medical diagnosis *Journal of Fluorescence* **4(3)** 17-40.

Lennon Á, Buchalla W, Switalski L & Stookey G (2002) Residual caries detection using visible fluorescence *Caries Research* **36(5)** 315-319.

McComb D (2000) Caries-detector dyes-how accurate and useful are they? *Journal of the Canadian Dental Association* **66(4)** 195-198.

Netuschil L (1983) Vital staining of plaque microorganisms using fluorescein diacetate and ethidium bromide *Deutsche Zahnärztliche Zeitschrift* **38(10)** 914-917.

Pekovic D, Adamkiewicz V, Shapiro A & Gornitsky M (1987) Identification of bacteria in association with immune components in human carious dentin *Journal of Oral Pathology* **16(5)** 223-233.

Pink F, Minden N & Simmonds S (1994) Decisions of practitioners regarding placement of amalgam and composite restorations in general practice settings *Operative Dentistry* **19(4)** 127-132.

Sato Y & Fusayama T (1976) Removal of dentin by fuchsin staining *Journal of Dental Research* **55(4)** 678-683.

Weerheijm K, Kreulen C, de Soet J, Groen H & van Amerongen W (1999) Bacterial counts in carious dentine under restorations: 2-year *in vivo* effects *Caries Research* **33(2)** 130-134.

©Operative Dentistry, 2003, **28-4**, 346-351

Dentinal Composition and Knoop Hardness Measurements of Cavity Floor Following Carious Dentin Removal with Carisolv

M Hossain • Y Nakamura • Y Tamaki
Y Yamada • JA Jayawardena • K Matsumoto

Clinical Relevance

In the clinic, it is possible to remove carious dental hard tissues using Carisolv without affecting the dentinal composition of the treated cavities, and when a proper clinical guide is used, complete removal of carious dentin is also no longer difficult with the Carisolv system, alone.

SUMMARY

This study evaluated the dentinal composition and Knoop hardness measurements of the cavity floor following the removal of carious dentin by the Carisolv chemo-mechanical caries removal system, *in vitro*. The carious dentin of 25 extracted human teeth was removed by using Carisolv for one minute with instruments and excavation that was performed until the gel was clear. Caries removal with a sharp explorer was verified according to the color and hardness of the lesion, then, by means of DIAGNOdent. Atomic analysis of treated cavities was performed by energy dispersive x-ray spectroscopy (SEM-EDX) and the Knoop hardness number (KHN) of the cavity floor was determined. Surface characteristics were observed by the scanning electron microscope (SEM). Adjacent sound dentin was used as a control reference. No significant differences were found between the quantities of calcium content (Ca weight %), phosphorus content (P weight %) and the Ca/P weight ratio of Carisolv cavities with that of the adjacent, sound dentin ($p<0.01$). KHN of the Carisolv cavity floor was almost similar to that of the adjacent sound dentin. SEM analysis revealed an extremely rough or irregular surface, and there remained a minimal debris-like smear layer; most of the dentinal tubules were opened. The results indicated that Carisolv does not produce any adverse side effects on dentinal

Mozammal Hossain, BDS, PhD, JSPS postdoctoral, Department of Endodontics, School of Dentistry, Showa University, Tokyo, Japan

*Yukio Nakamura, DDS, PhD, associate professor, Department of Endodontics, School of Dentistry, Showa University, Tokyo, Japan

Yukimichi Tamaki, DDS, PhD, associate professor, Department of Oral Biomaterials & Technology, School of Dentistry, Showa University, Tokyo, Japan.

Yoshishige Yamada, DDS, PhD, assistant professor, Department of Endodontics, School of Dentistry, Showa University, Tokyo, Japan

Jayanetti Asiri Jayawardena, BDS, PhD, visiting research fellow, Department of Endodontics, School of Dentistry, Showa University, Tokyo, Japan

Koukichi Matsumoto, DDS, PhD, professor and chairman, Department of Endodontics, School of Dentistry, Showa University, Tokyo, Japan

*Reprint request: Showa University, 2-1-1 Kitasenzoku, Ohta-ku, Tokyo 145-8515, Japan; e-mail: yukio@senzoku.showa-u.ac.jp

compositions of the treated cavities. The possibility of remaining residual softened dentin was also minimal in this study.

INTRODUCTION

Caries removal in decayed teeth has conventionally been performed using the mechanical cutting and drilling system. However, mechanical preparation often induces pain, and local anesthesia is therefore needed (Berggren & Meynert, 1984). It is often difficult to establish exactly how much tooth material should be removed, which often leads to overextended cavities (Fusayama, 1988). As possible alternatives to the conventional technique, previous studies have reported several chemo-mechanical caries removal systems for caries removal. Habib, Kronman and Goldman (1975) reported a chemo-mechanical caries removal system (GK-101) using the pharmacodynamic action of sodium hypochlorite. Caridex system that consists of N-monochloro-DL-2-aminobutyrate (NMAB) is formed by mixing equal parts sodium hypochlorite and aminobutyric acid, which is used to dissolve the carious dentin (Schutzbank & others, 1978). Its high patient acceptance was reported by Zinck and others (1988). Recently developed, Carisolv, which contains sodium hypochlorite and three kinds of amino acids (glutamic acid, leucine and lysine), has shown the capability to minimize the disadvantages of traditional cavity preparation (Ericson & others, 1998; Ericson, 1999a). Carisolv does not need a mechanical device and uses specially designed blunt edge excavators used for "gentle excavation" (Ericson & others, 1999b).

In recent years, the potential applications of Carisolv on carious dental hard tissue removal or cavity preparation for restorations have already been explored by a number of investigators. Animal histological studies showed that no sign of pulp cell damage could be identified after placing Carisolv into the cavities (Dammaschk & others, 2001); in addition, Carisolv does not produce adverse side-effects or give rise to any deleterious effects on the pulp nerve supply (Young & Bongenhielm, 2001). Studies on the surface characteristics of human dentin following carious dentin removal by Carisolv demonstrated a rough surface, a similar effect of acid etching (Wennerberg, Sawase & Kultje, 1999) and, when modern bonding systems are used, Carisolv has no adverse effect on bonding to caries-affected dentin (Haak, Wicht & Noack, 2000). The adhesive restorative techniques of today, together with a chemo-mechanical method for caries removal, may further reduce patient discomfort and decrease unnecessary removal of sound dental tissue (Ericson & others, 1999b). Clinical studies have confirmed that compared to conventional spoon excavators, the Carisolv system appeared to be more comfortable for patients; no pain/discomfort was reported by 68% of participants and only 3% requested local anesthesia (Nadanovsky, Cohen Carneiro & Souza de Mello, 2001). However, there are still no reports on the compositional changes of dentin and microhardness of the cavity floor following carious dentin removal by Carisolv.

This study investigated the compositional changes of human dentin by SEM-EDX and Knoop hardness measurements of the cavity floor following the removal of carious dentin using the Carisolv system, *in vitro*.

METHODS AND MATERIALS

Experimental Procedure

Twenty-five extracted human permanent teeth with dentin caries on the proximal surface were used. Each carious lesion was analyzed according to the color and hardness of the lesion. Carious lesions with a brown-to-black color and medium consistency (it was resistance to probing but readily penetrated when tested with a sharp probe) were selected for this study (Figure 1). All lesions had no enamel coverage and dentin was easily accessible through the cavity openings. The extent of the carious lesions was further assessed by means of KaVo DIAGNOdent 2095 (KaVo Dental GmbH, Jena, Germany) that provided a pulsed 655-nm laser beam directed into the tooth. When the incident light encountered a change in tooth substances, it stimulated fluorescent light of a different wavelength. This was translated, through the handpiece, into a number from 0 to 99. For the selection of carious dentin in this study, the following criteria suggested by Ross (1999) were used: 0-20=no caries; 20+, the caries was deeper into the enamel or into the dentin. Carious lesions that scored higher than 26 with this laser ray were used. The authors employed a tapered fiber-optic tip (Tip A) for measurements. Before obtaining the measurements, the DIOGNOdent was calibrated against a ceramic standard following the manufacturer's instructions.

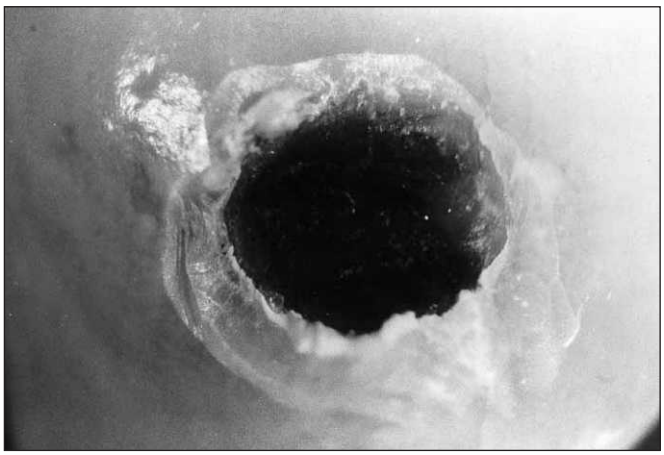


Figure 1. *Preoperative photograph of a carious lesion. Carious dentin with a brown to black color and a medium consistency was selected (DIAGNOdent reading 87).*

The Carisolv (Medi Team, Göteborg AB, Sweden) system was applied according to the manufacturer's instructions using the Carisolv hand instruments. Carisolv was applied on the surface of the carious lesions for one minute with instruments and excavation was performed until the gel was clear. Gross caries removal was verified according to the color and hardness of the lesion by checking the hardness of the dentin with a dental explorer until a leather-hard texture was reached or a sharp scratching sound was heard as suggested by previous studies (Splieth, Rosin & Gellissen, 2001; Cederlund, Lindskog & Blomlöf, 1999a). As carious dentin removal progressed and the treated cavity floor became deeper and closer to the underlying intact dentin layer, the treated cavity was carefully assessed by means of DIAGNOdent. The procedure was repeated until DIAGNOdent showed a value less than 20.

Twenty cavities were cross-sectioned perpendicularly to the tooth axis through the middle of the treated cavity; one-half was used for atomic analysis by SEM-EDX and the other half were subjected to the Knoop Hardness test. Adjacent sound dentin (areas at least 1000 µm beneath the cavity floor) of the same samples was used as a control reference. The remaining five cavities were observed morphologically.

Atomic Analysis by SEM-EDX

Atomic analysis by SEM-EDX was performed according to a previous study (Kimura & others, 2001). Cut sections were dehydrated through a graded ethanol series (70, 80, 90, 95 and 100%) for 24 hours at each concentration, then embedded in a polyester resin block (Rogolac, Nisshin, Tokyo, Japan). After the treated areas were flattened as much as possible by polishing, they were sputter-coated using a carbon-coating device (HUS-5GB, HITACHI, Tokyo, Japan) and examined by SEM-EDX (S-2500CX, HITACHI) at 10 kV accelerating voltage, tilt angle at 35°, and 3,000x magnification linked to a personal computer. Fluorapatite [$Ca_{10}(PO_4)F_6$] was used as a standard during the measurements because of the greater structural stability of synthetic fluorapatite compared to hydroxyapatite (HAP). The calcium content (Ca weight %), phosphorus content (P weight %), Ca/P ratio from the five measurement points in the treated cavity floor and adjacent sound dentin (reference control) were recorded and statistically analyzed using Mann-Whitneys U test. Results were expressed as a mean ± standard deviation (SD) and a value of $p<0.01$ was considered to be significant.

Knoop Hardness Measurements of the Cavity Floor

For measuring Knoop Hardness of the treated cavity floor, cavity sections were embedded in epoxy resin and the cross-sectional surfaces were polished. Since obtaining a Knoop Hardness measurement of the cavity surface was impossible, recordings were obtained below the cavity floor; the hardness of the subsurface at a point 25 µm below the cavity floor was used as that of the cavity floor (Harnirattisai & others, 1992). The Knoop Hardness number (KHN) was measured at five points in each treated cavity by application of a 50-g load for 15 seconds by means of a hardness tester. To determine the degree of residual softened dentin, the hardness change of the adjacent sound dentin (reference control) on the same specimens was evaluated. The mean of the measurements was used as the KHN of the dentin and a statistically significant difference between the KHN of the Carisolv cavity floor and adjacent sound dentin was determined by Mann Whitneys U test; a value of $p<0.01$ was considered significant.

Surface Characteristics of the Prepared Cavity

Five Carisolv-treated cavities were observed macroscopically using a stereoscope (SMZ-10, Nikon, Tokyo, Japan). For further investigation, specimens were cross-sectioned, dehydrated with a graded series of ethanol, dried to a critical point with CO_2 and mounted on aluminum stubs. Specimens were then sputter-coated with platinum at a thickness of 15 µm for scanning electron microscopic examination at 20 kV (SEM) (JSM-T220A, JOEL, Tokyo, Japan).

RESULTS

Atomic Analysis by SEM-EDX

Table 1 shows the results of atomic analysis following the removal of carious dentin with the Carisolv chemomechanical system. Measurements were performed at the cavity floor and adjacent sound dentin (reference control). No significant differences were found between the quantities of Ca (Ca weight %) and P (P weight %) or Ca/P ratio of Carisolv cavities with that of the adjacent sound dentin. The mean of Ca (weight %) of Carisolv and the adjacent sound dentin was 26.29 ± 2.55 and 27.10 ± 2.15, respectively. The mean of P (P weight %), on the other hand, was 13.00 ± 2.26 and 13.20 ± 2.17, respectively. The Ca/P ratio of Carisolv (2.02) and the adjacent sound dentin (2.05) did not show any statistically significant differences ($p<0.01$).

Knoop Hardness Measurements of the Cavity Floor

Table 1 also shows the results of KHN of Carisolv and the adjacent sound dentin. The results revealed that

Table 1: *Summary of the Results Found in This Study (Mean ± SD)*

	Carisolv Cavity	Adjacent Sound Dentin
Ca (weight%)	26.29 ± 2.55	27.10 ± 2.15
P (weight%)	13.00 ± 2.26	13.20 ± 2.17
Ca/P	2.02	2.05
Knoop Hardness (km/cm^2)	60.30 ± 4.16	62.50 ± 5.05

No statistically significant difference was found between Carisolv and the adjacent sound dentin (p<0.01).

the KHN of the cavity floor prepared by Carisolv ranged from 58 to 62 kg/cm^2 (mean ± S D: 60.30 ± 4.16), which was slightly lower than the KHN of the adjacent sound dentin (from 59 to 64 kg/cm^2, mean ± S D: 62.50 ± 5.50) but not statistically significant.

Surface Characteristics of the Prepared Cavity

Morphologically, the cavity surfaces treated by Carisolv were extremely rough and irregular. There remained a minimal debris-like smear layer and most of the dentinal tubules were opened (Figures 2 & 3).

DISCUSSION

In this study, the compositional change of human dentin following carious dentin removal by Carisolv was assessed by SEM-EDX and Knoop Hardness measurement of the cavity floor was determined *in vitro*. The methodology followed in this study allowed different forms of data (atomic analysis and Knoop hardness measurements of the cavity floor) to be collected from the same individual sample. This, in turn, permitted accurate analysis of the results, which indicated a correlation between dentinal compositions and the hardness of the treated area or the adjacent tissues following removal of carious dentin by Carisolv.

Atomic Analysis by SEM-EDX

The results of SEM-EDX showed that no significant differences were found between Ca (weight %) and P (weight %) or Ca/P ratio of Carisolv cavities with that of the adjacent sound dentin. This confirms that there were minimal chemically-induced changes of dentin components after carious dentin removal or cavity preparation with Carisolv. Since this study is the first to examine the effect of Carisolv solution on inorganic dentinal compositions, the results found in this study corresponded to some previous studies of other dental structures. Dammaschk and others (2001) did not identify any sign of pulp cell damage following Carisolv treatment; other studies have shown that Carisolv did not produce any adverse side effects or give rise to any deleterious effects on the pulp nerve supply (Young & Bongenhielm, 2001). Moreover, Dammaschk and others (2002) reported that Carisolv causes destruction of cellular components of the odontoblastic process but does not attack healthy collagen fibrils. Therefore, based on this and previous studies, it can be suggested that cavities could be treated with Carisolv without damaging the dentin compositions and the surrounding tissues or the dental pulp.

Knoop Hardness Measurements of the Cavity Floor

Previous studies have indicated that the determination to completely remove carious dentin is difficult with the Carisolv treatment and the possibility of remaining caries following the Carisolv treatment is a major concern; caries removal with Carisolv leaves up to a mean of 50 µm more carious dentin than round burrs (Splieth & others, 2001). Some clinical guidelines are, therefore, necessary to identify residual carious dentin. Splieth and others (2001) and Cederlund and others (1999a) verified caries removal according to the color and hardness of the lesion with a sharp explorer; the hardness of the dentin was checked with a dental explorer until a leather-hard texture was reached or a sharp scratching sound was heard. As these methods are operator-dependent, the authors recommend a combination of hardness testing by explorer and DIAGNOdent. In this study, initially, gross caries removal was verified according to the color and hardness of the lesion with a sharp explorer as suggested by Splieth and others (2001) and Cederlund and others (1999a). Subsequently, the treated cavity was carefully assessed

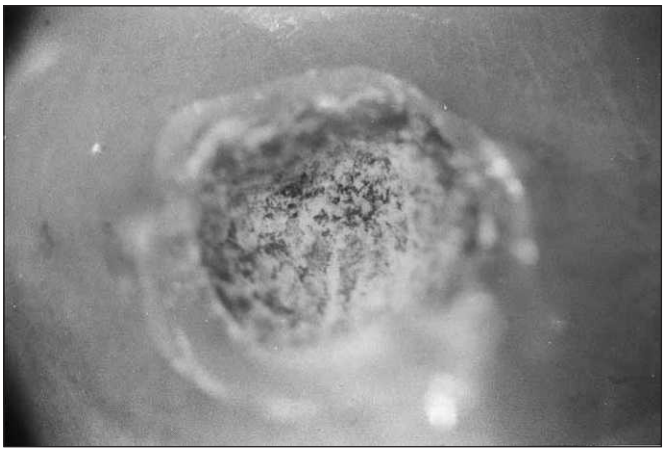


Figure 2. *Postoperative view of remaining dentin surface following carious dentin removal with Carisolv. Cavity surface revealed an irregular pattern.*

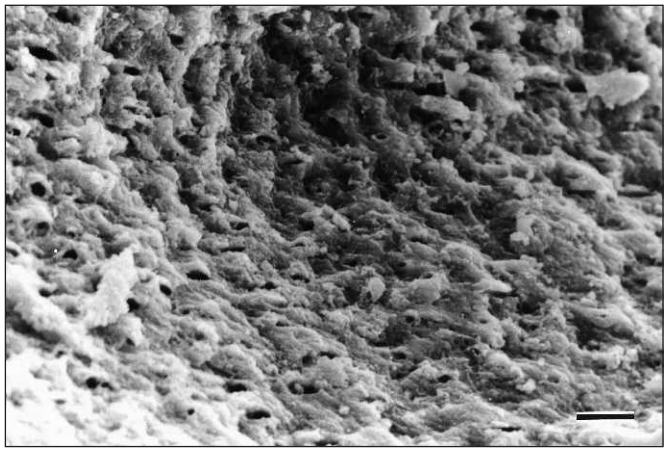


Figure 3. *Representative scanning electron microscopy (SEM) photographs of dentin surface following carious dentin removal with Carisolv. There remained a minimal debris-like smear layer remain and most of the dentinal tubules were opened (original magnification 1000x, and bar represents 10 µm).*

by means of DIAGNOdent. The usefulness of this device for the assessment of carious dentin removal has previously been reported by Ross (1999). Further research on the ability of DIAGNOdent to detect dental caries should be performed.

The degree of softened dentin removal was determined by KHN measurements of the cavity floor and the adjacent sound dentin as suggested by Aoki and others (1998). The results of Knoop Hardness measurements of the Carisolv cavity floor confirmed that the possibility of remaining residual, softened dentin was minimal in this study because no statistically significant differences were noted in microhardness of the Carisolv cavity floor dentin and the adjacent sound dentin (reference control). Since this study is the first to examine the KHN of the Carisolv cavity floor, the results of KHN of the adjacent sound dentin corresponded to a previous study by Banerjee and others (1999). The results further indicate that accurate evaluation of KHN is possible by methods used in this study, and the efficiency of complete carious dentin removal by the Carisolv chemo-mechanical system is no longer difficult when a proper clinical guide is used.

Surface Characteristics of the Prepared Cavity

The results of SEM observations of the current study demonstrated that the Carisolv cavity surface possessed some distinguishing features compared to the surfaces of the conventional mechanical burr in previous studies (Smith, 1982; Eick & others, 1970). The burr cavity surface showed a relatively flat appearance and exhibited a debris-like smear layer that may interfere with adhesion, wetting, penetration and hardness of the prepared cavity (Smith, 1982; Eick & others, 1970). Although applying some acidic conditioners could solve these problems, care should be taken not to damage the collagen matrix of the dentin. On the other hand, the dentinal surfaces after Carisolv treatment were irregular, and there remained a minimal debris-like smear layer and most of the dentinal tubules were opened. These features were similar to the structures of the Carisolv-treated dentin surface that has previously been described as flaky or as an irregular surface (Wennerberg & others, 1999; Banerjee, Kidd & Waston, 2000; Cederlund, Lindskog & Blomlöf, 1999b). The highly irregular surface of the dentinal floor is said to be an advantageous substrate for adhesive resin composite bonding. Carisolv systems induced a rougher surface with respect to the slope or curvature area when compared with conventional caries removal using burrs. The surface was affected in a similar way as that of acid etching (Wennerberg & others, 1999) and, in particular, when modern bonding systems are used, Carisolv has no adverse effect on bonding to caries-affected dentin (Haak & others, 2000). However, a loosely attached debris-like smear layer could also develop due to crushing and burnishing of the Carisolv applicator tip on the dentinal surface as seen in our SEM analysis; the loosely attached debris may also interfere with adhesion, as reported in mechanically-prepared cavities of previous studies (Smith, 1982; Eick & others, 1970). Anticipated differences between conventional and chemo-mechanical caries removal are omitted if the dentin surface is treated with phosphoric acid or a highly acidic self-etching primer for bonding (Haak & others, 2000). Further research for evaluating the bond strength or microleakage of the resin composite restoration in cavities prepared by Carisolv with or without acid etching is necessary to confirm these results.

CONCLUSIONS

The results indicate that Carisolv solution does not produce any adverse side effects on dentinal compositions of treated cavities. Furthermore, complete carious dentin removal by Carisolv is no longer difficult when a proper clinical guide is used. Therefore, cavity preparations with Carisolv, a less traumatic caries removal system, may also be favorable in clinical dentistry.

(Received 10 June 2002)

References

Aoki A, Ishikawa I, Yamada T, Otsuki M, Watanabe H, Tagami J, Ando Y & Yamamoto H (1998) Comparison between Er:YAG laser and conventional technique for root caries treatment *in vitro Journal of Dental Research* **77(6)** 1404-1414.

Banerjee A, Sherriff M, Kidd EAM & Waston TF (1999) A confocal microscopic study relating the autofluorescence of carious dentine to its microhardness *British Dental Journal* **187(4)** 206-210.

Banerjee A, Kidd EAM & Waston TF (2000) *In vitro* evaluation of five alternative methods of carious dentine excavation *Caries Research* **34(2)** 144-150.

Berggren U & Meynert G (1984) Dental fear and avoidance: Causes, symptoms, and consequences *Journal of the American Dental Association* **109(2)** 247-251.

Cederlund A, Lindskog S & Blomlöf J (1999a) Efficacy of Carisolv assisted caries excavation *The International Journal of Periodontics & Restorative Dentistry* **19(5)** 464-469.

Cederlund A, Lindskog S & Blomlöf J (1999b) Effect of a chemo-mechanical caries removal system (Carisolv) on dentin topography of non-carious dentin *Acta Odontologica Scandinavica* **57(4)** 185-189.

Dammaschk T, Stratmann U, Mokrys K, Kaup M & Ott KH (2001) Histocytological evaluation of the reaction of rat pulp tissue to Carisolv *Journal of Dentistry* **29(4)** 283-290.

Dammaschke T, Stratmann U, Mokrys K, Kaup M & Ott KH (2002) Reaction of sound and demineralised dentine to Carisolv *in vivo* and *in vitro Journal of Dentistry* **30(1)** 59-65.

Eick JD, Wilko RA, Anderson CH & Sorensen SE (1970) Scanning electron microscopy of cut tooth surfaces and identification of debris by use of the electron microprobe *Journal of Dental Research* **49(6)** 1359-1368.

Ericson D, Bornstein R, Götrick B, Raber H & Zimmerman M (1998) Clinical multi-centre evaluation of a new method for chemo-mechanical caries removal *Caries Research* **32** Abstract #119 p 308.

Ericson D (1999a) *In vitro* efficacy of a new gel for chemo-mechanical caries removal *Journal of Dental Research* **77** Abstract #360 p 1252.

Ericson D, Zimmerman M, Raber H, Götrick B, Bornstein R & Thorell J (1999b) Clinical evaluation of efficacy and safety of a new method for chemo-mechanical removal of caries. A multi-centre study *Caries Research* **33(3)** 171-177.

Fusayama T (1988) Clinical guide for removing caries using a caries-detecting solution *Quintessence International* **19(6)** 397-401.

Haak R, Wicht MJ & Noack MJ (2000) Does chemomechanical caries removal affect dentine adhesion *European Journal of Oral Science* **108(5)** 449-455.

Habib CM, Kronman J & Goldman M (1975) A chemical evaluation of collagen and hydroxyproline after treatment with GK-101 (N-chloroglycine) *Pharmacology and Therapeutics in Dentistry* **2(3-4)** 209-215.

Harnirattisai C, Inokoshi S, Hosoda H & Shimada Y (1992) Interfacial morphology of an adhesive composite resin and etched caries-affected dentin *Operative Dentistry* **17(6)** 222-228.

Kimura Y, Yu DG, Fujita A, Yamashita A, Murakami Y & Matsumoto K (2001) Effects of Erbium, Chromium:YSGG laser irradiation on canine mandibular bone *Journal of Periodontology* **72(9)** 1178-1182.

Nadanovsky P, Cohen Carneiro F & Souza de Mello F (2001) Removal of caries using only hand instruments: A comparison of mechanical and chemo-mechanical methods *Caries Research* **35(5)** 384-389.

Ross G (1999) Caries diagnosis with the DIAGNOdent laser: A user's product evaluation *Ontario Dentist* **76(2)** 21-24.

Schutzbank SG, Galaini J, Kronman JH, Goldman M & Clarke RE (1978) A comparative *in vitro* study of GK-101 and GK-101E in caries removal *Journal of Dental Research* **57(9)** 861-864.

Smith DC (1982) Buonocore Memorial Lecture: A milestone in dentistry *Operative Dentistry* **7(1)** 14-25.

Splieth C, Rosin M & Gellissen B (2001) Determination of residual dentine caries after conventional mechanical and chemo-mechanical caries removal with Carisolv *Clinical Oral Investigation* **5(4)** 250-253.

Wennerberg A, Sawase T & Kultje C (1999) The influence of Carisolv on enamel and dentin surface topography *European Journal of Oral Science* **107(4)** 297-306.

Young C & Bongenhielm U (2001) A randomized, controlled and blinded histological and immunohistochemical investigation of Carisolv on pulp tissue *Journal of Dentistry* **29(4)** 275-281.

Zinck JH, McInnes-Ledoux P, Capdeboscq C & Weinberg R (1988) Chemomechanical caries removal: A clinical evaluation *Journal of Oral Rehabilitation* **15(1)** 23-33.

©Operative Dentistry, 2003, **28-4,** 352-356

Increment Technique for Extended Class V Restorations: An Experimental Study

R Müllejans • H Lang • N Schüler
MOF Badawi • WHM Raab

Clinical Relevance

On the basis of our findings, the authors conclude that using the increment technique for restoring extended Class V cavities with polyacid-modified resin composite can result in significantly less microleakage; however, it entails a greater expenditure of time and effort.

SUMMARY

The application and polymerization of composite or polyacid-modified resin composites in thin layers (increment technique) for filling cavities might partially compensate for the stress associated with polymerization shrinkage. In this study, the effect of this technique on the marginal integrity of Class V polyacid-modified composite restorations was investigated.

In 30 extracted premolars, extended Class V cavities were prepared with the apical margin in root dentin and the coronal margin in enamel. They were then subjected to different treatments (10 teeth in each case): a) placement and polymerization of a thin resin composite layer (Dyract-Dyract PSA) in the coronal part of the cavity (plus one increment for the residual part) (Group 1), b) a thin layer at the apical part of the cavity (plus one increment for the residual part) (Group 2) and c) restoration of the entire cavity with one increment (bulk technique) (Group 3). Following three days' storage in water, the teeth were subjected to cyclic thermal loading (4°C <-> 55°C, 2,000 cycles).

The percentages of defective bonding detected along the total length of the restoration margin were assessed before and after thermocycling by scanning electron microscopy. Following loading and thermocycling, no significant differences were found at the restoration-enamel interface. Fewer gaps were found at the restoration-dentin interface in Group 1 (18.7 ± 25.7%) and Group 2 (20.5 ± 22.8%) compared to the reference Group 3 (42.2 ± 30.6%) (Mann-Whitney U test: $p<0.05$).

Moreover, there were no significant differences between the two increment groups (1 and 2). In a significantly larger number of cases, a completely intact dentin restoration margin was detected when a coronal increment (Group 1) (44.4%) was used instead of the bulk technique (Group 3) (13.6%) (Chi square test: $p<0.05$).

*Rolf Müllejans, MD DMD, Department of Restorative and Preventive Dentistry, Heinrich-Heine-University, Duesseldorf, Germany

H Lang, professor, dr med dent, Department of Restorative and Preventive Dentistry, Heinrich-Heine-University, Duesseldorf, Germany

N Schüler, dr med, University Dental Clinic, Bonn, Germany
MOF Badawi, Department of Restorative and Preventive Dentistry, Heinrich-Heine-University, Duesseldorf, Germany

WHM Raab, professor, dr med dent, Department of Restorative and Preventive Dentistry, Heinrich-Heine-University, Duesseldorf, Germany

*Reprint request: Moorenstrasse 5, 40225 Duesseldorf, Germany; e-mail: muellejr@uni-duesseldorf.de

These results indicate that gap formation can be significantly minimized by using an increment technique to restore extended Class V cavities with polyacid-modified composite materials.

INTRODUCTION

The creation of long-lasting restorations in extended Class V cavities is a major challenge faced by dental practitioners (Matis, Cochran & Carlson, 1996; Prati & others, 1995; Prati & Pashley, 1992; Hickel, 1992).

Several studies have proven that the cervical margin of a Class V cavity is the most critical factor affecting restoration lifetime (Ferrari, Cagidiaco & Davidson, 1997; Sjodin, Uusitalo & van-Dijken 1996; Yap, Lim & Neo, 1995; Martin, Kingsford-Smith & Andrews, 1992; Mathis & others, 1990; Crim, 1989; Hembree & Andrews, 1978). Any lack of bonding literally paves the way for microbial invasion of dental restorations. In this situation, infection of dental pulp is inevitable.

One method commonly used to minimize polymerization shrinkage and, thus, gap formation in molars is the increment technique, which has proved its worth in several clinical and experimental studies (Ben-Amar & others, 1988; Crim, Swartz & Phillips, 1985; Full & Hollander, 1993a,b; Lutz, Krejci & Barbakow, 1992; Koenigsberg, Fuks & Grajower, 1989). In small cavities, the use of this technique is limited by tiny dimensions, especially since polymerization shrinkage is minimal in such restorations.

In extended cavities, by contrast, this technique produces good results.

This study investigated the effects, with respect to gap formation, of using an increment technique for polyacid-modified composite restorations in extended Class V cavities

METHODS AND MATERIALS

In this study, 30 extracted, caries-free upper and lower human premolars and molars were utilized. After extraction, the teeth were cleaned to remove all soft and hard tissue, then stored in saline solution for 3.5 months.

In all specimens, standardized Class V cavities were prepared using a conventional drill (Intra Lux 3, KaVo Dental GmbH, Biberach, Germany) and a medium-grain diamond bur (Figure 837XL, ISO 806 314 112 524 014, Hager & Meisinger, Dusseldorf, Germany). The dimensions were 3 mm in the mesio-distal direction, 5 mm in the occlusogingival direction and 2 mm in depth.

All the enamel surfaces were then etched with 36 wgt% phosphoric acid gel (Conditioner 36, Dentsply, Konstanz, Germany) for 20 seconds, rinsed with water (30 seconds) and dried with oil-free compressed air for 10 seconds. All treatments were carried out according to the manufacturer's instructions. The teeth were subsequently randomly assigned to the different experimental groups. All cavities were restored with Dyract AP (Dentsply DeTrey, Konstanz, Germany) and the adhesive Dyract-PSA (Dentsply DeTrey).

In Group 1, a thin (1-mm thick) layer of polyacid-modified composite was applied to the coronal part of the cavity and polymerized for 40 seconds (Figure 1). In all the specimens, a standard composite/polyacid-modified composite curing light (Spectrum 800, Model #703, Dentsply DeTrey) was used. All treatments were carried out according to the manufacturer's instructions. Then, the residual cavity was filled with an additional increment and polymerized for 40 seconds.

In Group 2, the cavities were restored in the same manner as Group 1; however, the thin first increment was placed in the *apical* part of the cavity (Figure 1).

A single-increment technique (bulk) was used in Group 3 (Figure 1).

After all the fillings were completed, the restoration surfaces were shaped with a flame-shaped medium-grain diamond bur (Figure 863, ISO 806 314 250 524 018, Hager & Meisinger, Dusseldorf, Germany) following the correct anatomical contours and polished with small, fine-grained discs (3M ESPE Dental, Seefeld,

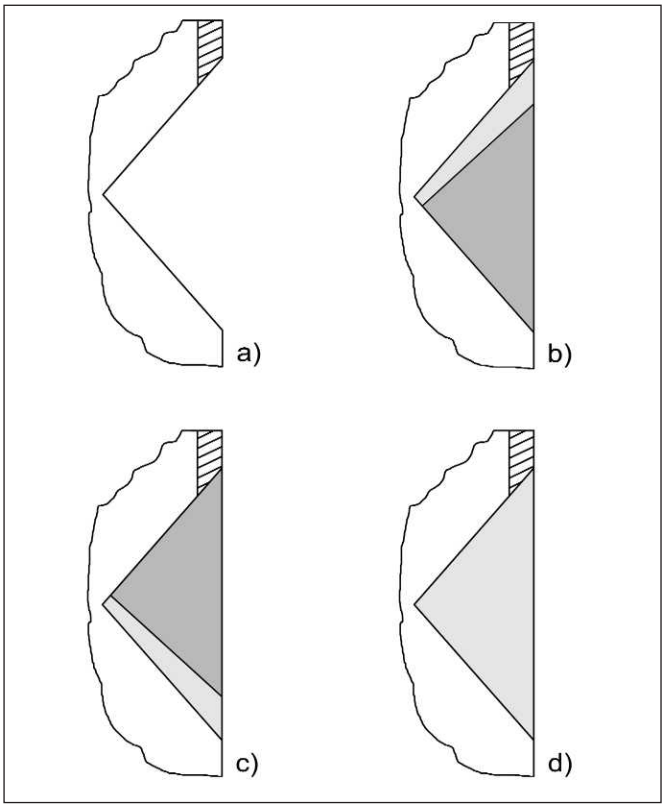


Figure 1. *Experimental design of the study. a) Class V defect (coronal part in enamel, apical part in dentin); b) Coronal increment (Group 1); c) Apical increment (Group 2); d) No increment / bulk technique (Group 3).*

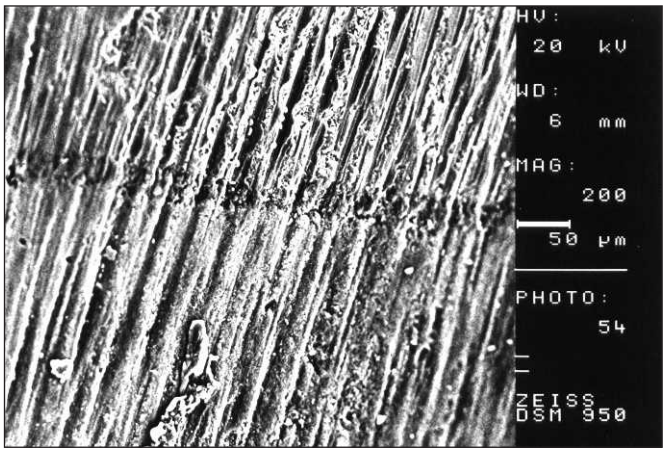


Figure 2. *Intact adhesive zone at the restoration margin.*

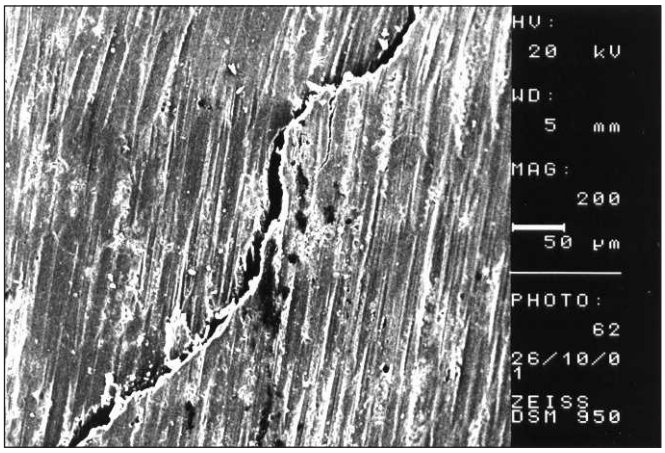


Figure 3. *Gap at the restoration margin.*

Table 1: *Gap Formation Depending on the Increment Technique (percentage of gap)*

Restoration Technique	Restoration-Enamel Interface	Restoration-Dentin Interface
Coronal increment (I)	17.9 ± 28.2%	18.7 ± 25.7%
Apical increment (II)	25.8 ± 30.0%	20.5 ± 22.8%
No increment/bulk (III)	20.1 ± 26.5%	42.2 ± 30.6%

Table 2: *Restoration Margins With and Without Microleakage Depending on the Increment Technique Used (% of samples)*

Dental Substance/Restoration Technique	Microleakage	No Microleakage
Enamel (bulk)	59.1%	40.9%
Enamel (coronal increment)	44.4%	55.6%
Enamel (apical increment)	55.6%	44.4%
Dentin (bulk)	86.4%	13.6%
Dentin (coronal increment)	55.6%	44.4%
Dentin (apical increment)	66.7%	33.3%

Germany). The specimens underwent three days' storage in saline solution. All specimens were then subjected to 2000 thermal cycles between 4°C and 55°C, with a dwell time of 60 seconds in each bath.

To record the effects on dental bonding, the complete restoration margins of all samples were systematically examined by SEM at a magnification of 200x (Figures 1 and 2). Therefore, all specimens were fixed to aluminum stubs with a fast-curing epoxy resin, sputter-coated with a 15-nm thick layer of platinum (MED-010 sputter device, Bal-Tec, Liechtenstein) and examined under a DSM 950 electron microscope (Zeiss, Jena, Germany).

The length of the total margin and the parts with defective bonding were measured for each tissue, enamel and dentin, and the fraction was then calculated in percent. The significance of the results was tested by the non-parametric Mann-Whitney U test. Furthermore, the authors determined whether any defective bonding occurred at the restoration margin. Here, the significance of the results was assessed with the Chi square test. The authors presumed a statistically significant difference of $p<0.05$. Statistical analysis was carried out with SPSS for Windows (V. 10.0, SPSS Inc, Chicago, IL, USA).

RESULTS

The scanning electron microscopic investigation revealed obvious differences between the study groups regarding the loss of bonding (Table 1): At the restoration-enamel margins of restorations created with the bulk technique (20.1 ± 26.5%), no statistical differences were noted between the group using the coronal (17.9 ± 28.2%) and the apical increment technique (25.8 ± 30.0%), respectively.

At the restoration-dentin margins, microleakage was significantly reduced by both the coronal (18.7 ± 25.7%) and the apical increment technique (20.5 ± 22.8%) compared with the bulk technique (42.2 ± 30.6%). No statistical differences were found between the two increment techniques.

In addition, the authors compared the number of specimens (in each group) in which no microleakage occurred after thermal cycling (Table 2). For the enamel portion of the cavities, no statistical differences were found. For the dentin segment, significantly more fillings (44.4%) with no microleakage (p=0.030) were noted when the coronal increment technique was applied than when no increment technique was used (13.6%).

DISCUSSION

Restoring dentin-bordered Class V cavities is a problem that has not yet been satisfactorily solved by the science of dentistry (Matis & others, 1996; Prati & others, 1995; Prati & Pashley, 1992; Hickel, 1992). This observation applies, in particular, to extended cavities at the enamel-cement junction (Ferrari & others, 1997; Sjodin & others, 1996; Yap & others, 1995; Martin & others, 1992; Mathis & others, 1990; Crim, 1989; Hembree & Andrews, 1978).

One of the most important causes of this problem is polymerization shrinkage, which is due to the different bond strengths exhibited by restoration materials at the enamel and dentin junctions, respectively. Moreover, the deformability of teeth is very high in these areas (Lang, Schwan & Nolden, 1996). As a result, Class V restorations run the risk of developing gaps at the filling margin, predisposing the dental pulp to microbiological invasion and infection.

The increment technique is commonly used on molars to reduce polymerization shrinkage (Ben-Amar & others, 1988; Crim & others, 1985; Full & Hollander, 1993a; Full & Hollander, 1993b; Lutz & others, 1992; Koenigsberg & others, 1989). It was of great interest to investigate whether such techniques could be useful in filling Class V cavities with extended dimensions so that practicability is given.

The results of this study showed that the loss of restoration bonding at dentin dental surfaces can be significantly reduced by employing a coronal or apical increment technique. Furthermore, a completely intact restoration margin was observed in a larger number of cases when a coronal increment was placed first in contrast to the bulk technique.

Although the superiority of this increment technique is obvious, consideration should be given to its practicability under clinical conditions. This technique can be used when the dental defect has attained certain minimal dimensions. At this size, polymerization shrinkage plays such an important role in gap formation that it should not be neglected.

Obviously, the coronal increment technique should be used on an even greater scale. For clinical applications it is necessary to bear in mind the proximity of the gingiva to the restoration margin. Compared to the experimental specimens, restoration work in clinical situations apparently places a higher demand on the dentist's time and manual ingenuity.

CONCLUSIONS

Gap formation in extended Class V cavities can be significantly reduced in clinical practice by using an increment technique. With the coronal increment technique, in particular, the chances of achieving a restoration margin without any microleakage after thermal cycling is significantly higher compared to the bulk technique. It should still be kept in mind, however, that this technique is associated with an increased expenditure of time and manual effort.

(Received 13 June 2002)

References

Ben-Amar A, Liberman R, Nordenberg D & Metzger Z (1988) The effect of retention grooves on gingival marginal leakage in Class II posterior composite resin restorations *Journal of Oral Rehabilitation* **15(4)** 325-331.

Crim GA (1989) Influence of bonding agents and composites on microleakage *Journal of Prosthetic Dentistry* **61(5)** 571-574.

Crim GA, Swartz ML & Phillips RW (1985) Comparison of four thermocycling techniques *Journal of Prosthetic Dentistry* **53(1)** 50-53.

Ferrari M, Cagidiaco MC & Davidson CL (1997) Resistance of cementum in Class II and V cavities to penetration by an adhesive system *Dental Materials* **13(3)** 157-162.

Full CA & Hollander WR (1993a) The composite resin restoration: A literature review Part I Proper cavity preparation and placement techniques *Journal of Dentistry for Children* **60(1)** 48-51.

Full CA & Hollander WR (1993b) The composite resin restoration: A literature review Part II Comparisons between composite and alloy restorations *Journal of Dentistry for Children* **60(1)** 52-56.

Hembree JH & Andrews JT (1978) Microleakage of several Class V anterior restorative materials: A laboratory study *Journal of the American Dental Association* **97(2)** 179-183.

Hickel R (1992) Cervical caries *Deutsche Zahnärztliche Zeitschrift* **10(1)** 654-658.

Koenigsberg S, Fuks A & Grajower R (1989) The effect of three filling techniques on marginal leakage around Class II composite resin restorations *in vitro Quintessence International* **20(2)** 117-121.

Lang H, Schwan R & Nolden R (1996) Deformation of Class V restorations under loading *Deutsche Zahnärztliche Zeitschrift* **10(1)** 613-616.

Lutz F, Krejci I & Barbakow F (1992) The importance of proximal curing in posterior composite resin restorations *Quintessence International* **23(9)** 605-607.

Martin FE, Kingsford-Smith ED & Andrews N (1992) Microleakage of glass ionomer/composite resin restorations: A laboratory study Part 2 The influence of bonding systems *Australian Dental Journal* **37(3)** 172-177.

Mathis RS, DeWald JP, Moody CR & Ferracane JL (1990) Marginal leakage in Class V composite resin restorations with glass ionomer liners *in vitro Journal of Prosthetic Dentistry* **63(5)** 522-525.

Matis BA, Cochran M & Carlson T (1996) Longevity of glass-ionomer restorative materials: Results of a 10-year evaluation *Quintessence-International* **27(6)** 373-382.

Prati C, Ferrieri P, Galloni C, Mongiorgi R & Davidson CL (1995) Dentin permeability and bond quality as affected by new bonding systems *Journal of Dentistry* **23(4)** 217-226.

Prati C & Pashley DH (1992) Dentin wetness, permeability and thickness and bond strength of adhesive systems *American Journal of Dentistry* **5(1)** 33-38.

Sjodin L, Uusitalo M & van-Dijken J (1996) Resin-modified glass ionomer cements. *In vitro* microleakage in direct Class V and Class II sandwich restorations *Swedish Dental Journal* **20(3)** 77-86.

Yap AU, Lim CC & Neo JC (1995) Marginal sealing ability of three cervical restorative systems *Quintessence International* **26(11)** 817-820.

©Operative Dentistry, 2003, **28-4,** 357-364

Composite Cure and Shrinkage Associated with High Intensity Curing Light

AUJ Yap • NY Wong • KS Siow

Clinical Relevance

Composite cure and shrinkage associated with the use of very high intensity lights is material-dependent.

SUMMARY

This study investigated the effectiveness of cure and post-gel shrinkage of three visible light-cured composite resins (In Ten-S [IT], Ivoclar Vivadent; Z100 [ZO], 3M-ESPE; Tetric Ceram [TC], Ivoclar Vivadent) when polymerized with a very high intensity (1296 ± 2 mW/cm²) halogen light (Astralis 10, Ivoclar Vivadent) for 10 seconds. Irradiation with a conventional (494 ± 3 mW/cm²) halogen light (Spectrum, Dentsply) for 40 seconds was used for comparison. The effectiveness of cure was assessed by computing the hardness gradient between the top and bottom surfaces of 2-mm composite specimens after curing. A strain-monitoring device was used to measure the linear polymerization shrinkage associated with the various composites and curing lights. A sample size of five was used for both experiments. Data was analyzed using ANOVA/Scheffe's post-hoc and Independent Samples *t*-tests at significance level 0.05. Results showed that the effect of the curing method on the effectiveness of cure and shrinkage was material-dependent. Polymerization of IT and TC with Spectrum for 40 seconds resulted in significantly more effective cure than polymerization with Astralis for 10 seconds. Polymerization of ZO with Spectrum for 40 seconds resulted in significantly more shrinkage than polymerization with Astralis for 10 seconds. In view of the substantial time saving, using high intensity lights may be a viable method to polymerize composites.

*Adrian UJ Yap, BDS, MSc, PhD, FAMS, FADM, FRSH, associate professor, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

NY Wong, BSc (Hons), Department of Chemistry, Faculty of Science, National University of Singapore, Republic of Singapore

KS Siow, BSc, MSc, PhD, associate professor, Department of Chemistry, Faculty of Science, National University of Singapore, Republic of Singapore

*Reprint request: 5 Lower Kent Ridge Road, Singapore 119074, Republic of Singapore; e-mail: rsdyapuj@nus.edu.sg

INTRODUCTION

Light-cured composites have become almost universal in modern clinical dentistry. They have revolutionized the practice of dentistry by maximizing work time and reduce setting time. The ongoing interest in curing lights for composite restorations has been fueled by the need for a light that works reliably and conveniently for many years and the desire of dentists to place restorations with good physical stability in less clinical time. Curing lights have been developed with varying outputs and curing cycles to speed up and/or reduce marginal gaps in composite restorations. The technology utilized for curing lights ranges from conventional halogen bulbs to more exotic (and expensive) systems

using lasers, plasma arc and LEDs (light-emitting diodes). Regardless of the type of light and curing cycles employed, composite polymerization shrinkage still remains a problem (Sakaguchi & others, 1991; Peutzfeldt, Sahafi & Asmussen, 2000; Yap, Ng & Siow, 2001; Yap, Soh & Siow, 2002). The shrinkage of composites, which is about 1%-to-5% volume (Davidson & Feilzer, 1997), can be divided into two phases: pre-gel and post-gel. During pre-gel polymerization, the composite is able to flow and stress within the structure relieved (Davidson & de Gee, 1984). After gelation, flow ceases and cannot compensate for shrinkage stresses. Post-gel polymerization, consequently, results in significant stresses in the surrounding tooth structure and composite-tooth bond (Feilzer, de Gee & Davidson, 1987). These stresses can cause clinical problems such as postoperative pain, tooth fracture and marginal debonding that can result in microleakage and secondary caries (Eick & Welch, 1986; Torstenson & Oden, 1989; Asmussen, 1975).

Methods used to reduce the effects of polymerization shrinkage include developing stronger bonding agents and non-shrinking composites, using bases and liners that act as shock absorbers (Kemp-Scholte & Davidson, 1990), strategically placing composites in appropriate cavity configurations (Lutz, Krejci & Barbakow, 1991; Bertolotti, 1991) and using "soft-start" and low-intensity curing units (Uno & Asmussen, 1991; Unterbrink & Muessner, 1995; Feilzer & others, 1995; Davidson & Feilzer, 1997; Yap & others, 2001; Yap & others, 2002). In spite of the positive effects of the latter, the trend in curing lights has been to increase light intensity. High intensity light sources have reportedly improved the immediate depth of cure, physical and mechanical properties of composite restoratives (Rueggeberg & Jordan, 1993; Rueggeberg & others, 1993; Rueggeberg, Caughman & Curtis, 1994). They have, however, been reported to result in high polymerization shrinkage stresses (Uno & Asmussen, 1991; Unterbrink & Muessner, 1995; Feilzer & others, 1995). It is important to note that the polymerization process depends on total light energy (intensity x time) rather than light intensity, alone (Miyazaki & others, 1996). As such, the equivalent degree of cure to conventional lights may be achieved by applying a high intensity light for a shorter time.

Recently, a high intensity halogen curing light (Astralis 10, Ivoclar Vivadent, Schaan, Liechtenstein) and a low shrinkage fast-set composite formulation (In-Ten S, Ivoclar Vivadent) have been introduced to the dental profession. No known independent literature is available regarding this new curing light and material. The effectiveness of the cure and post-gel shrinkage associated with the use of Astralis 10 for 10 seconds and a conventional halogen light (Spectrum, Dentsply, Milford, DE, USA) for 40 seconds was compared in this study. The cure and shrinkage of In-Ten S was also contrasted to that of two other commercially available visible light-cured composites (Z100, 3M-ESPE, St Paul, MN, USA; Tetric Ceram, Ivoclar Vivadent).

METHODS AND MATERIALS

Table 1 shows the technical profiles of the materials evaluated. Prior to beginning the experiments, the light intensities of the two light curing units were assessed with a commercial radiometer (Cure Rite, EFOS Inc, Ontario, Canada). Readings were repeated five times and the mean light intensities were as follows: Astralis 10 - 1296 ± 2 mW/cm^2 and Spectrum - 494 ± 3 mW/cm^2. The diameter of the light tips were 8 and 9 mm for Astralis and Spectrum, respectively.

Table 1: *Technical Profiles of the Materials Evaluated*

Material/ (Shade/Lot #)	Manufacturer	Recommended Curing Parameters	Composition
In Ten-S (A2/D51682)	Ivoclar Vivadent, Schaan, Liechtenstein	1200 mW/cm^2 for 10 seconds	*Resins:* BIS-GMA, UDMA and ethoxylated BIS-GMA *Fillers*: Barium glass, ytterbium trifluoride *Filler* volume: 51%
Z100 (A2/20010404)	3M-ESPE, St Paul, MN, USA	400 mW/cm^2 for 40 seconds	*Resins*: BIS-GMA, TEGDMA *Fillers*: Zirconia, silica *Filler* volume: 66%
Tetric Ceram (A2/D54267)	Ivoclar Vivadent, Schaan, Liechtenstein	400 mW/cm^2 for 40 seconds	*Resins*: BIS-GMA, UDMA and TEGDMA *Fillers*: Barium glass, ytterbium trifluoride, barium alumino-fluorosilicate glass, silica *Filler* volume: 60%

BIS-GMA = Bisphenol A-glycidyl methacrylate
TEGDMA = Triethylene glycol dimethacrylate
UDMA = Urethane dimethacrylate

Protocols employed to evaluate the effectiveness of cure and post-gel shrinkage were based on that used by Yap and others (2001). The sample size for curing light and materials combination was five.

To investigate the effectiveness of cure, the composites were placed in black delrin molds with square cavities 2-mm deep and 4-mm wide/long and confined between two opposing acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide (1-mm thick) was then placed on the molds and excess material was extruded by pressure application. The composites were then irradiated from the top for 10 and 40 seconds with Astralis and Spectrum, respectively. Immediately after light polymerization, the acetate strips were removed, and the specimens in their molds were positioned centrally beneath the indenter of a digital microhardness tester (FM7, Future-Tech Corp, Tokyo, Japan) to assess Knoop's hardness (KHN) of the top and bottom surfaces. A 500g load was applied through the indenter with a dwell time of 15 seconds. The mean KHN and hardness ratios were then calculated and tabulated using the formula: Hardness ratio = KHN of bottom surface/KHN of top surface.

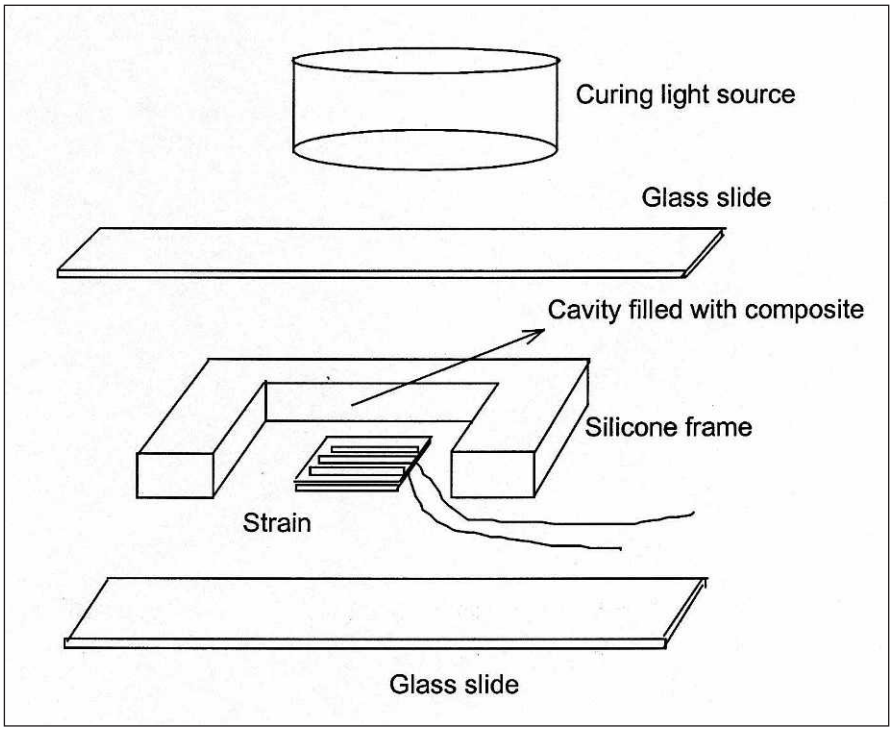


Figure 1. *Diagrammatic representation of the experimental set-up for the assessment of polymerization shrinkage.*

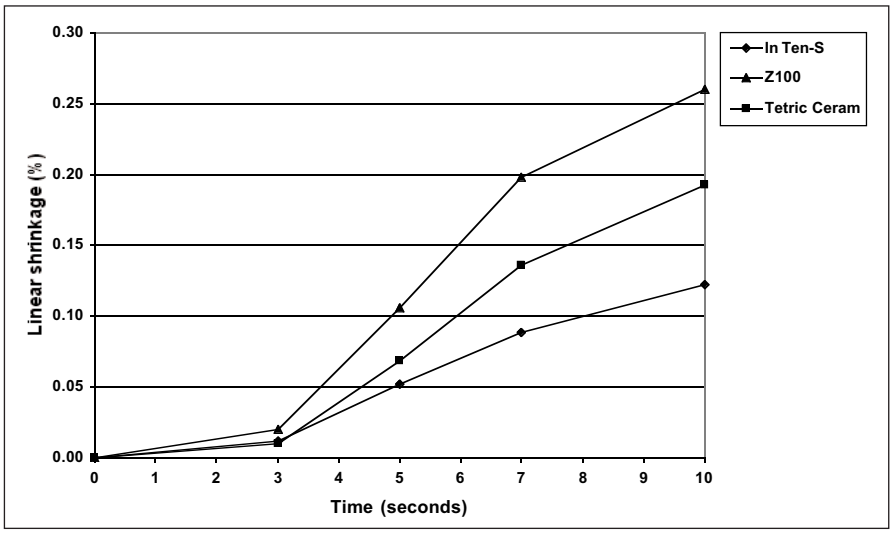


Figure 2. *Mean linear percent shrinkage during light curing with Astralis 10.*

Post-gel polymerization shrinkage was measured with foil electrical resistance strain gauges (Foil Strain Gauge, RS Components Ltd, Singapore). The gauges were 2 mm in length and had an electrical resistance 120 Ω and gauge factor 2.00. A diagrammatic representation of the test configuration for measuring polymerization shrinkage is shown in Figure 1. A glass slide served as the base of the set-up and a stiff, black silicone frame (inner length 7.0 mm, width 4.0 mm and height 2.0 mm) was used to circumscribe the composite sample with the exception of a window for the strain gauge leads. The strain gauges were attached onto the flat glass surface and resin composites were placed in the cavity of the silicone frame with the strain gauge in place. Care was taken to ensure complete filling of the frame and the excess composite material was extruded using pressure applied through a second glass slide and removed. The surface tack of the composite was adequate to ensure adhesion between the strain gauge and the composite materials. The leads from the strain gauge were connected to a strain-monitoring device (Strain Gauge Recorder, Cole Parmer Instruments, IL, USA) initially balanced at zero. The strain-monitoring device consisted of a chart recorder that functions by rationing sense voltage to signal voltage and converting it to analog output. Dimensional changes are, thus, effectively transferred to the gauges and measured in terms of resistance.

The composite specimens were light-polymerized using the two light curing units and the dimensional change dur-

ing and post light polymerization was monitored in air at room temperature (25 ± 1°C). Polymerization shrinkage measurements during light polymerization were taken at 1, 3, 5, 7 and 10 seconds for Astralis and every 10 seconds up to 40 seconds for Spectrum. Post-light polymerization shrinkage measurements were taken at 0 (immediately after light polymerization), 1, 10, 30 and 60 minutes after removing the curing lights. Percentage linear shrinkage was derived from the following equation: Percentage linear shrinkage ($\Delta L/L \times 100$) = ($\Delta R/R)/K \times 100$, where ΔL = Change in length, L = Original length, ΔR = Change of resistance, R = Original resistance and K = Gauge factor (2). Data was subjected to MANOVA followed by one-way ANOVA/Scheffe's post-hoc tests and Independent Samples *t*-tests at significance level 0.05.

RESULTS

Table 2 shows the KHN and hardness ratio of the various materials after polymerization with the two light cure units. Results of statistical analysis for KHN of the top/bottom surfaces and hardness ratio are shown in Table 3 and 4. The mean percentage linear shrinkage during light polymerization is shown in Figures 2 and 3, while up to 60 minutes post-light polymerization is shown in Table 5 and Figure 4. Results of statistical analysis for mean percentage linear shrinkage at the various post-light polymerization time intervals are shown in Tables 6 and 7.

Mean top KHN ranged from 29.96 to 69.99* and 32.34 to 72.02 for Astralis and Spectrum, respectively. Values for mean bottom KHN were lower and ranged from 24.28 to 64.98 and 27.86** to 67.98 for the two light curing methods. With the exception of curing InTen-S with Astralis for 10 seconds, the hardness ratios were around 0.8 and above. Two-way ANOVA showed significant interaction between the materials and curing lights for hardness ratio. The effect of the curing method on hardness ratio was therefore material-dependent. For InTen-S and Tetric, polymerizing with Spectrum for 40 seconds resulted in significantly greater bottom KHN and hardness ratio than polymerizing with Astralis for 10 seconds (Table 3). For all materials, no significant difference in top KHN was observed between the two curing methods. The top and bottom KHN of Z100 was significantly greater than InTen-S and Tetric with both curing methods (Table 4). With Astralis, the hardness ratio of Z100 was significantly greater than Tetric, which was, in turn, significantly greater than InTen-S. With Spectrum, the hardness ratios of Z100 and Tetric were significantly greater than InTen-S.

Regardless of the curing method, all composites continued to shrink after removing the light source. For both curing lights, the highest mean post-light poly-

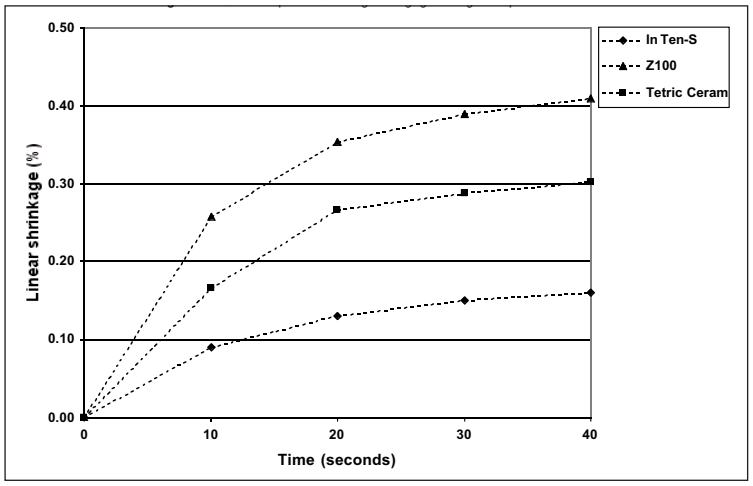


Figure 3. *Mean linear percent shrinkage during light curing with Spectrurum.*

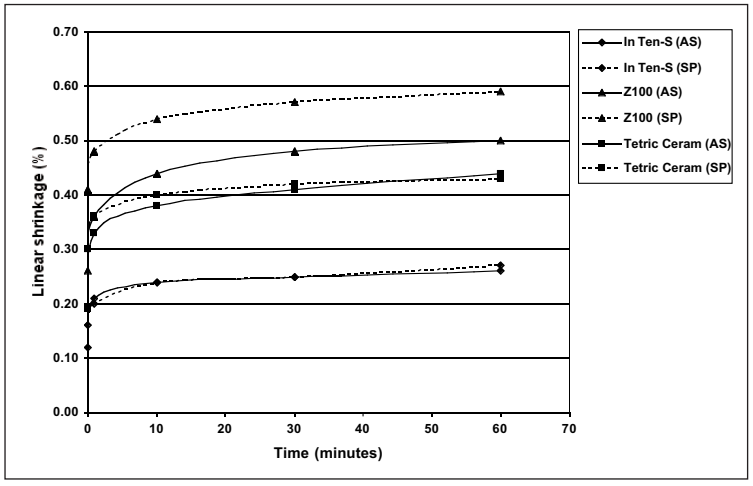


Figure 4. *Mean linear percent shrinkage after light curing.*

Table 2: *KHN and Hardness Ratio of the Various Materials After Polymerization with the Two Light Cure Units*

Light Cure Unit	Material	Top KHN	Bottom KHN	Hardness Ratio
Astralis 10 (10 seconds)	InTen-S	35.58 (2.21)	24.56 (2.61)	0.69 (0.05)
	Z100	69.99 (0.74)*	64.98 (2.59)	0.93 (0.04)
	Tetric Ceram	29.96 (2.20)	24.28 (2.08)	0.81 (0.08)
Spectrum (40 seconds)	InTen-S	35.70 (2.39)	27.86 (1.95)**	0.78 (0.01)
	Z100	72.02 (5.08)	67.98 (8.45)	0.94 (0.07)
	Tetric Ceram	32.34 (1.96)	31.40 (1.36)	0.97 (0.03)

Standard deviations in parentheses.

Table 3: *Comparison of Top KHN, Bottom KHN and Hardness Ratio Between the Two Curing Methods*

Material	KHN	Significance
In Ten-S	Top	NS
	Bottom	Spectrum > Astralis 10
	Ratio	Spectrum > Astralis 10
Z100	Top	NS
	Bottom	NS
	Ratio	NS
Tetric Ceram	Top	NS
	Bottom	Spectrum > Astralis 10
	Ratio	Spectrum > Astralis 10

NS denotes no statistically significant difference while > denotes statistically significant differences in KHN or hardness ratio (Results of Independent Samples t-tests [p<0.05]).

Table 4: *Comparison of Top KHN, Bottom KHN and Hardness Ratio Between Materials*

Curing Light	KHN	Significance
Astralis 10 (10 seconds)	Top	Z100 > In-Ten S > Tetric
	Bottom	Z100 > Tetric & InTen-S
	Ratio	Z100 > Tetric > InTen S
Spectrum (40 seconds)	Top	Z100 > Tetric & InTen-S
	Bottom	Z100 > Tetric & InTen-S
	Ratio	Z100 & Tetric > InTen-S

> denotes statistically significant differences in KHN or hardness ratio (Results of one-way ANOVA/Scheffe's post-hoc test [p<0.05]).

merization shrinkage was observed with Z100. For Z100, shrinkage at 60 minutes was 0.50% and 0.59% for Astralis and Spectrum, respectively. At this time interval, the shrinkage of Z100 was significantly greater than Tetric, which was, in turn, significantly greater than InTen-S (Table 7). MANOVA revealed that the effect of curing methods on shrinkage was both material- and time-dependent. For all materials, shrinkage associated with Spectrum was significantly greater than Astralis immediately after removing the light source (time=0 minutes). For InTen-S and Tetric, no significant difference in shrinkage was observed between the two curing methods from one minute post-light polymerization onwards. Significant differences in shrinkage between the two curing methods were, however, observed for Z100 at all post-light polymerization light intervals.

DISCUSSION

The effectiveness of composite cure may be assessed by direct and indirect methods. Direct methods such as laser Raman and infrared spectroscopy are not used routinely as they are complex, expensive and time consuming. Indirect methods, which include scraping, visual and surface hardness, are more commonly employed (Yap & others, 2001). Knoops hardness testing was selected for this study due to its relative simplicity and good correlation to the degree of conversion using infrared spectroscopy (Asmussen, 1982; DeWald & Ferracane, 1987). A2 shade was selected to minimize the effects of colorants on light polymerization (Bayne, Heymann & Swift, 1994) and 2-mm thick composite specimens were used to ensure uniform and maximum polymerization (Yap, 2000). Since a minimum intensity of 400 mW/cm^2 has been suggested for routine polymerization (Rueggeberg & others, 1994), a curing light (Spectrum) greater than this intensity, together with the manufacturers' recommended cure time of 40 seconds for Z100 and Tetric, was used for comparison.

At the top surface, no significant difference in KHN was observed between the two curing methods. This is in agreement with previous studies that found the top surface hardness of composites was less dependent on light intensity than the bottom surfaces (Denehy & others, 1993; Rueggeberg & Jordan, 1993). At the bottom surfaces, significant differences in hardness between the two curing methods were observed for InTen-S and Spectrum. For both materials, polymerization with Spectrum resulted in significantly greater bottom KHN and hardness ratio compared to polymerization with Astralis. These findings are not surprising in view of the light energy densities (intensity x time) of the two light curing methods (Astralis - 12.96 J/cm^2 [1296W/cm^2 for 10 seconds]; Spectrum - 19.76 J/cm^2 [494 W/cm^2 for 40 seconds]). The higher light energy density associated with using Spectrum leads to greater cure of the bottom surfaces and, hence, hardness ratio. It is, however, important to note that the effect of curing methods on hardness ratio is material-dependent and no significant difference in bottom KHN and hardness ratio was observed for Z100. The explanation for this observation is not known and warrants further investigation. Possible hypotheses include differences in resin formulation and content. As the hardness ratio was almost identical for both curing methods, it can be inferred that cure of Z100 is highly effective and less dependent on light energy density. The significantly lower top/bottom hardness and hardness ratio of InTen-S and Tetric compared to Z100 may be attributed to their lower filler loading and use of UDMA. One study showed depth of cure to be less in certain UDMA composites due to a greater mismatch in the refractive index between monomer and filler (Söderholm, Achanta & Olsson, 1993).

Hardness ratio should be "1" if polymerization is completely effective as the hardness of the bottom surface should be the same as the top surface. However, as light passes through the bulk of the composite, light intensity is reduced due to light scattering by filler particles and the resin matrix (Ruyter & Øysæd,

1982). This accounts for the slight difference in hardness between the top and bottom surfaces of the composite specimens. The hardness gradient should, however, not exceed 10 to 20% (hardness ratio should be around 0.8 or greater) for light-activated composites to be adequately polymerized (Pilo & Cardash, 1992). Most material-curing method combinations fulfilled this criterion with the exception of polymerization of InTen-S with Astralis for 10 seconds. The hardness ratio for the latter was only 0.69. The poor cure may lead to decreased clinical longevity due to less than ideal physical properties (Ferracane, 1995) and clinical problems arising from cytotoxicity of inadequately polymerized materials (Caughman & others, 1991). InTen-S should, therefore, be cured with conventional lights of greater than 400 mW/cm^2 for 40 seconds instead of manufacturer's recommended use of Astralis for 10 seconds. The use of the latter for curing Z100 and Tetric is, however, feasible.

Table 5: *Mean Linear Percentage Shrinkage of the Various Materials Post Polymerization*

Curing Light	Time (minutes)	In Ten-S	Z100	Tetric Ceram
Astralis 10 (10 seconds)	0	0.12 (0.02)	0.26 (0.01)	0.19 (0.02)
	1	0.21 (0.03)	0.36 (0.02)	0.33 (0.02)
	10	0.24 (0.03)	0.44 (0.02)	0.38 (0.01)
	30	0.25 (0.03)	0.48 (0.02)	0.41 (0.01)
	60	0.26 (0.03)	0.50 (0.03)	0.44 (0.02)
Spectrum (40 seconds)	0	0.16 (0.03)	0.41 (0.02)	0.30 (0.02)
	1	0.21 (0.03)	0.48 (0.04)	0.36 (0.03)
	10	0.24 (0.03)	0.54 (0.03)	0.40 (0.03)
	30	0.25 (0.03)	0.57 (0.03)	0.42 (0.04)
	60	0.27 (0.03)	0.59 (0.02)	0.43 (0.04)

Standard deviation in parentheses.

Table 6: *Comparison of Mean Linear Percentage Shrinkage (Post Polymerization) Between the Two Curing Methods*

Material	Time (minutes)	Significance
In Ten-S	0	Spectrum > Astralis 10
	1	
	10	NS
	30	
	60	
Z100	0	
	1	
	10	Spectrum > Astralis 10
	30	
	60	
Tetric Ceram	0	Spectrum > Astralis 10
	1	
	10	
	30	NS
	60	

NS denotes no statistically significant difference while > denotes statistically significant differences in mean linear percentage shrinkage (Results of Independent Samples t-tests [p<0.05]).

Table 7: *Comparison of Mean Linear Percentage Shrinkage (Post Polymerization) Between Materials*

Curing Light	Time (Minutes)	Significance
AS10	0	Z100 > Tetric > InTen-S
	1	Z100 & Tetric >InTen-S
	10	
	30	Z100 > Tetric > InTen-S
	60	
SP40	0	
	1	
	10	Z100 > Tetric > InTen-S
	30	
	60	

> denotes statistically significant differences in mean linear percentage shrinkage (Results of one-way ANOVA/Scheffe's post-hoc test [p<0.05]).

From Figures 2 and 3, the rate of shrinkage during light polymerization appears to be the greatest during the middle of the curing cycle with both curing methods. All composites continued to shrink after removing the light source. This phenomenon had been reported in previous studies involving a variety of light curing regimens and composite materials (Yap & others, 2002; 2001; 2000). Shrinkage is caused by the monomers becoming covalently bonded by the polymerization reaction, thus, exchanging van der Waals' distances for covalent bond distances. The degree of shrinkage is dictated by the number of covalent bonds formed (extent of polymerization or effectiveness of cure) and the type of monomers used (Ferracane,

1995). A more thorough cure may explain the significantly higher shrinkage of Z100 despite its higher filler loading (Venhoven, de Gee & Davidson, 1993). Increased filler loading should theoretically lead to decreased polymerization shrinkage (Ferracane, 1995). For all materials, the significant amount of shrinkage observed after removing the light sources may be partially attributed to thermal contraction due to the loss of radiant heat (Yap & others, 2000). The effect of light curing method on post-gel polymerization shrinkage is both material- and time-dependent. At time 0 (immediately after light polymerization), irradiation with Spectrum resulted in significantly more shrinkage than Astralis for all materials. From one minute onward, no significant difference in shrinkage was observed between the two curing methods for InTen-S and Tetric. This could be caused by the longer period of heating (four times longer) associated with Spectrum, the low thermal conductivity and high coefficient of expansion of composites (O'Brien, 1997). It may also be partially contributed to the lower cure and viscosity of the UDMA monomer used (Ferracance, 1995). This might permit some degree of stress relaxation after post-light polymerization. Differences between the two curing methods, however, remained significant for up to 60 minutes for Z100. This correlated well with the highly effective cure of Z100 with both curing methods as evidenced by similar and high hardness ratios. The higher shrinkage associated with Spectrum is probably due to its higher light energy density compared to Astralis. At all time intervals, the shrinkage of Z100 was generally significantly greater than Tetric and InTen-S. This corroborated the results of Versluis, Sakaguchi and Douglas (1993), who found that Z100 had the greatest contraction stress among different composite materials.

The results of this experiment do not support the view that using high intensity lights results in greater polymerization shrinkage (Uno & Asmussen, 1991; Unterbrink & Muessner, 1995; Burgess & others, 1999; Dennison & others, 2000). The central issue is not the intensity of the curing light but the light energy density (intensity x time). Greater light energy densities result in more effective cure, which leads to higher polymerization shrinkage (Sakaguchi, Douglas & Peters, 1992). The current work also showed that the effect of curing methods on composite cure and shrinkage was material-dependent and confirmed the work of Christensen and others (1999). They implicated resin formulation, rather than light type or curing mode, as the important factor in polymerization problems. In view of the substantial time saving, the use of high intensity lights may be a viable method for polymerizing composites clinically.

CONCLUSIONS

Under the conditions of this *in-vitro* study:

1. The effect of curing methods on composite cure and shrinkage is material-dependent.
2. Polymerization of InTen-S and Tetric Ceram with Spectrum for 40 seconds resulted in significantly more effective cure than with Astralis 10 for 10 seconds.
3. For Z100, no significant difference in cure was observed between the two curing methods.
4. Polymerization of Z100 with Spectrum for 40 seconds resulted in significantly more shrinkage than with Astralis 10 for 10 seconds.
5. For InTen-S and Tetric Ceram, no significant difference in shrinkage was observed between the two curing methods.
6. Irradiation of composites with high intensity lights for short curing times may be a viable method of polymerizing composites clinically.

(Received 14 June 2002)

References

Asmussen E (1975) Composite restorative resins–composition versus wall-to-wall polymerization construction *Acta Odontologica Scandinavia* **33(3)** 337-343.

Asmussen E (1982) Factors affecting the quantity of remaining double bonds in restorative resin polymers *Scandinavian Journal of Dental Research* **90(6)** 90-496.

Bayne SC, Heymann HO & Swift EJ Jr (1994) Update on dental composite restorations *Journal of the American Dental Association* **125(6)** 687-701.

Bertolotti R (1991) Posterior composite technique utilizing directed polymerization shrinkage and a novel matrix *Practical Periodontics and Aesthetic Dentistry* **3(1)** 53-58.

Burgess JO, DeGoes M, Walker R & Ripps AH (1999) An evaluation of four light-curing units comparing soft and hard curing *Practical Periodontics and Aesthetic Dentistry* **11(1)** 125-132.

Caughman WF, Caughman GB, Shiflett RA, Rueggeberg F & Schuster GS (1991) Correlation of cytotoxicity, filler loading and curing time of dental composites *Biomaterials* **12(18)** 737-740.

Christensen RP, Palmer TM, Ploeger BJ & Yost MP (1999) Resin polymerization problems–are they caused by resin curing lights, resin formulation or both *Compendium of Continuing Education in Dentistry* **25(Supplement)** S42-54.

Davidson CL & de Gee AJ (1984) Relaxation of polymerization contraction stresses by flow in dental composites *Journal of Dental Research* **63(2)** 146-148.

Davidson CL & Feilzer AJ (1997) Polymerization shrinkage and polymerization shrinkage stress in polymer-based restorations *Journal of Dentistry* **25(6)** 435-440.

Denehy GE, Pires JAF, Cuitko E & Swift EJ (1993) Effects of curing tip distance on light intensity and composite resin microhardness *Quintessence International* **24(7)** 517-521.

Dennison JB, Yaman P, Seir R & Hamilton JC (2000) Effect of variable light intensity on composite shrinkage *Journal of Prosthetic Dentistry* **84(5)** 499-505.

DeWald JP & Ferracance JL (1987) A comparison of four modes of evaluating depth of cure of light-activated composites *Journal of Dental Research* **66(3)** 727-730.

Eick JD & Welch FH (1986) Polymerization shrinkage of posterior composite resins and its possible influence on postoperative sensitivity *Quintessence International* **17(2)** 103-111.

Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.

Feilzer AJ, Dooren LH, de Gee AJ & Davidson CL (1995) Influence of light intensity on polymerization shrinkage and integrity of restoration-cavity interface *European Journal of Oral Science* **103(5)** 322-326.

Ferracane JL (1995) Current trends in dental composites *Critical Review in Oral Biology and Medicine* **6(4)** 302-318.

Kemp-Scholte CM & Davidson CL (1990) Complete marginal seal of Class V resin composite restorations effected by increased flexibility *Journal of Dental Research* **69(6)** 1240-1243.

Lutz F, Krejci I & Barbakow F (1991) Quality and durability of marginal adaptation in bonded composite restorations *Dental Materials* **7(2)** 107-113.

Miyazaki M, Oshida Y, Moore BK & Onose H (1996) Effect of light exposure on fracture toughness and flexural strength of light-cured composites *Dental Materials* **12(6)** 328-332.

O'Brien WJ (1997) *Dental Materials and Their Selection* 2nd ed Illinois Quintessence Publishing Co p 97-114.

Peutzfeldt A, Sahafi A & Asmussen E (2000) Characterization of resin composites polymerized with plasma arch curing units *Dental Materials* **16(5)** 330-336.

Pilo R & Cardash HS (1992) Post-irradiation polymerization of different anterior and posterior visible light-activated resin composites *Dental Materials* **8(5)** 299-304.

Rueggeberg FA, Caughman WF, Curtis JW Jr & Davis HC (1993) Factors affecting cure at depths within light-activated resin composites *American Journal of Dentistry* **6(2)** 91-95.

Rueggeberg FA, Caughman WF & Curtis JW Jr (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19(1)** 26-32.

Rueggeberg FA & Jordan DM (1993) Effect of light-tip distance on polymerization of resin composite *International Journal of Prosthodontics* **6(4)** 364-370.

Ruyter IE & Øysæd H (1982) Conversion in different depths of ultraviolet and visible light activated composite materials *Acta Odontologica Scandinavia* **40(3)** 179-192.

Sakaguchi RL, Douglas WH & Peters MCRB (1992) Curing light performance and polymerization contraction of composite restorative materials *Journal of Dentistry* **20(3)** 183-188.

Sakaguchi RL, Sasik CT, Bunezak MA & Douglas WH (1991) Strain gauge method for measuring polymerization contraction of composite restoratives *Journal of Dentistry* **19(5)** 312-316.

Söderholm KJM, Achanta S & Olsson S (1993) Variables affecting the depth of cure of composites *Journal of Dental Research* **72(Special Issue)** 138.

Torstenson B & Oden A (1989) Effects of bonding agent types and incremental techniques on minimizing contraction gaps around resin composites *Dental Materials* **5(4)** 218-223.

Uno S & Asmussen E (1991) Marginal adaptation of a restorative resin polymerized at reduced rate *Scandinavian Journal of Dental Research* **99(5)** 440-444.

Unterbrink GL & Muessner R (1995) Influence of light intensity on two restorative systems *Journal of Dentistry* **23(3)** 183-189.

Venhoven BA, de Gee AJ & Davidson CL (1993) Polymerization contraction and conversion of light-curing BIS-GMA-based methacrylate resins *Biomaterials* **14(11)** 871-875.

Versluis A, Sakaguchi R & Douglas W (1993) Post-gel shrinkage measurements by means of strain gauges *Journal of Dental Research* **72(Special Issue)** 386.

Yap AUJ (2000) Effectiveness of polymerization in composite restoratives claiming bulk placement: Impact of cavity depth and exposure time *Operative Dentistry* **25(2)** 113-120.

Yap AUJ, Wang HB, Siow KS & Gan LM (2000) Polymerization shrinkage of visible-light-cured composites *Operative Dentistry* **25(2)** 98-103.

Yap AUJ, Ng SC & Siow KS (2001) Soft-start polymerization: Influence on effectiveness of cure and post-gel shrinkage *Operative Dentistry* **26(3)** 260-266.

Yap AUJ, Soh MS & Siow KS (2002) Post-gel shrinkage with pulse activation and soft-start polymerization *Operative Dentistry* **27(1)** 81-87.

©Operative Dentistry, 2003, **28-4,** 365-370

Effects of Light-Curing Time on the Cytotoxicity of a Restorative Resin Composite Applied to an Immortalized Odontoblast-Cell Line

CA de Souza Costa • J Hebling • CT Hanks

Clinical Relevance

Many resin composites used in restorative dentistry have excellent physical/mechanical properties. However, when these resinous materials are irradiated for a short time or with low intensity light, monomeric materials may be poorly polymerized, interfering negatively with their mechanical properties and making the resinous materials more cytotoxic.

SUMMARY

This *in vitro* study evaluated the cytotoxic effects of a restorative resin composite applied to an immortalized odontoblast-cell line (MDPC-23). Seventy-two round resin discs (2-mm thick and 4 mm in diameter) were light-cured for 20 or 40 seconds and rinsed, or not, with PBS and culture medium. The resin discs were divided into four experimental groups: Group 1: Z-100/20 seconds; Group 2: Z-100/20 seconds/rinsed; Group 3: Z-100/40 seconds; Group 4: Z-100/40 seconds/rinsed. Circular filter paper was used as a control material (Group 5). The round resin discs and filter papers were placed in the bottom of wells of four 24-well dishes (18 wells for each experimental and control group). MDPC-23 cells (30,000 cells/cm²) were plated in the wells and allowed to incubate for 72 hours. The zone of inhibition around the resin discs was measured under inverted light microscopy; the MTT assay was carried out for mitochondrial respiration and cell morphology was measured under SEM. The scores obtained from inhibition zone and MTT assay were analyzed with the Kruskal-Wallis followed by Dunnett tests. In Groups 1, 2, 3 and 4, the thickness of the inhibition zone was 1,593 ± 12.82 µm, 403 ± 15.49 µm, 1,516 ± 9.81 µm and 313 ± 13.56 µm, respectively. There was statistically significant difference among the experimental and control groups at the 0.05 level of significance. The MTT assay demonstrated that the resin discs of the experimental groups 1, 2, 3 and 4 reduced the cell metabolism by 83%, 40.1%, 75.5% and 24.5%. Only between the Groups 2 and 4 was there no statistically significant difference for mitochondrial respiration. Close to the resin discs, the MDPC-23 cells exhibited rounded shapes, with only a few cellular processes keeping the cells attached to the substrate or, even

*Carlos Alberto de Souza Costa, DDS, MS, PhD, University of São Paulo State/UNESP, School of Dentistry, Araraquara, Brazil

Josimeri Hebling, DDS, MS, PhD, University of São Paulo State/UNESP, School of Dentistry, Araraquara, Brazil

Carl Thomas Hanks, DDS, PhD, University of Michigan, School of Dentistry, Ann Arbor, MI, USA

*Reprint request: Rua Humaitá, 1680 CEP: 14.801-903, Centro, CP: 331, Araraquara, SP, Brazil; e-mail: casouzac@foar.unesp.br

disruption of plasma membrane. Adjacent to the inhibition zone, the cultured cells exhibited multiple fine cellular processes on the cytoplasmic membrane organized in epithelioid nodules, similar to the morphology observed to the control group. Based on the results, the authors may conclude that the Z-100 resin composite light cured for 20 seconds was more cytopathic to MDPC-23 cells than Z-100 light cured for 40 seconds. The cytotoxic effects of the resin discs decreased after rinsing them with PBS and culture medium. This was confirmed by MTT assay and upon evaluation of the inhibition zone, which was narrower following rinsing of the resin discs.

INTRODUCTION

Recently, several investigations (Sakaguchi, Douglas & Peters, 1992; Kanca, 1986; Rueggeberg & others, 1993) have described the association between time of light curing and light intensity and the use of resin composites which are thicker or thinner than 2 mm to restore cavities. An appropriate polymerization of the resin composite seems to be important to maintain or provide the excellent physical and mechanical properties of restorative materials and ensure the dimensional stability of cavity restoration (Pilo, Oelgiesser & Cardash, 1999). Consequently, less thorough polymerization of a resin composite results in free methacrylates inside the material. According to Watts (1996), these methacrylates act as either open chains or residual free monomers, weakening the mechanical/physical properties of the dental material. In this situation, free monomers may be leachable in saliva, causing secondary caries, increasing the water sorption and, consequently, interfering with the color stability of the dental material (Pearson & Longman, 1989).

Gerzina and Hume (1996) reported that unconverted components of resin composite located in the base of a composite restoration can diffuse through the cured resin bonding agent layer and dentinal tubules to reach the pulpal space. In this way, clinical procedures that allow for complete (deep) polymerization of resin composite during the restorative procedures will certainly favor clinical success that is characterized by maintenance of the excellent physical/chemical properties of the composites. However, several factors may interfere with adequate polymerization of the resin composite. These factors include chemical composition, the amount and size of particulates, shade and refraction rate of the resin. In addition, there are many factors related to light intensity and length of time in which the resin-based material is exposed to the irradiation. Low intensity of light or reduced time of light curing may result in appropriate superficial polymerization but may also result in deficient monomer-polymer conversion in deep areas of the resin-based dental material. Consequently, a number of free monomers in the base of a resin composite restoration (in direct contact with dentin substrate) seem to play an important role in the cytopathic effects of a restorative resin composite by causing damage to the pulpal tissue (Stanley & others, 1972). Therefore, this *in vitro* study evaluated the cytotoxicity of the resin composite Z-100 (3M/ESPE) light-cured for 20 or 40 seconds and rinsed, or not, to remove unreacted components from their surface.

METHODS AND MATERIALS

Immortalized odontoblast cell line—MDPC-23 (Hanks, Sun & Fang, 1998) were grown in T-75 sterilized flasks (Costar Corp, Cambridge, MA, USA). Cell passage ($1x10^5$cells/cm^2) was carried out every three days until they were used in the current experiment. Complete medium used for cell culture was the α-modification of Minimum Essential Medium (α-MEM–SIGMA Chemical Co, St Louis, MO, USA) supplemented with 10% fetal bovine serum (FBS) glutamine, penicillin/streptomycin and 50 µl/ml ascorbic acid.

Ninety round, cover-glasses (Fisher Scientific, Pittsburgh, PA, USA), 12 mm in diameter were kept in 70% ethanol for six hours, then rinsed with phosphate buffered saline (PBS) for 15 minutes with gentle shaking. These glass discs were placed in the bottom of wells of 24-well dishes (Costar Corp, Cambridge, MA, USA). Seventy-two circle samples (18 for each treatment of the experimental material), 2-mm thick and 4 mm in diameter, were prepared with the resin composite Z-100 (3M Dental Division, St Paul, MN, USA). Another 18 samples consisted of simple filter paper used as controls (Table 1). Half of the Z-100 resin composite samples (36) were light cured for 20 seconds (Optilux 500, Demetron/Kerr, Danbury, CT, USA). The other half of the samples was irradiated for 40 seconds. The light intensity generated by the unit was 530 mW/cm^2, which

Table 1: *Relationship Among Experimental and Control Groups and the Number of Samples According to the Methods Used to Evaluate the Cytotoxicity of the Resin Composite*

			Evaluation		
Groups	Description	Samples	MTT	Inhibition Zone	SEM
1	Z-100 20 seconds	18	16	16	2
2	Z-100 20 seconds/rinsed	18	16	16	2
3	Z-100 40 seconds	18	16	16	2
4	Z-100 40 seconds/rinsed	18	16	16	2
5	Control (filter paper)	18	16	16	2

was monitored with a radiometer (Demetron/Kerr). During light-curing, the light source was kept 2 mm away from the surface of the resin samples. Half of the samples irradiated for 20 seconds (18) or 40 seconds (18) were rinsed three times with PBS and α-MEM in order to remove unreacted components on the surface of the experimental material. All samples were placed on the circular cover glasses positioned on the bottom of wells. In the control group, sterilized round filter paper, 4 mm in diameter (Matheson Scientific Inc, Detroit, MI, USA), was placed in the bottom of the wells in the same manner as the experimental resin composite. MDPC-23 cells were plated at 30,000 cells/cm^2 in the wells (18 wells for each experimental and control groups) and maintained for 72 hours in a humidified incubator with 5% CO_2 and 95% air at 37°C. Cell metabolic activity was assessed by succinic dehydrogenase (SDH) activity, which is a measure of the mitochondrial respiration of the cells (Costa & others, 1999). The MTT (Methyltetrazolium Test) assay was chosen in this *in vitro* study because previous investigations had demonstrated the effectiveness of this technique to estimate the cytotoxicity of a specific dental material (Wataha, Hanks & Craig, 1991; Wataha, Hanks & Craig, 1992; Hashieh & others, 1999; Costa, Edwards & Hanks, 2001). The MTT assay is a colorimetric analysis used to detect cell metabolic activity (mitochondrial respiration). A pale yellow substrate 3-(4,5-cimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide is converted to a dark blue-purple product in the presence of active mitochondria. The color produced is quantified by spectrophotometric means. The inhibition zone around the experimental resin composite and the filter paper was measured by inverted light microscopy. Two representative samples of each experimental and control group were selected. The cell morphology was evaluated in these samples by scanning electron microscopy after fixation of the cells for 24 hours in 2.5% glutaraldehyde in PBS, post-fixation for one hour in osmium tetroxide and critical-point drying. The scores obtained from MTT assays and thickness of inhibition zones were statistically analyzed by the Kruskal-Wallis method complemented by Dunnett's tests, in which all groups were compared to determine whether the effects of the resin composite in different experimental conditions were significant at the 0.05 level of significance.

RESULTS

In Groups 2 (Z-100, 20 seconds, rinsed) and 4 (Z-100, 40 seconds, rinsed), the authors observed inhibition zones with 403 ± 15.49 µm and 313 ± 13.56 µm in thickness, respectively. However, in Groups 1 and 3, in which the resin discs were not rinsed before applying the MDPC-23 cells, the inhibition zones were 1,593 ± 12.82 µm and 1,516 ± 9.81 µm thick, respectively. The Kruskal-Wallis statistical analysis complemented with Dunnett's test demonstrated that the inhibition zones were not sta-

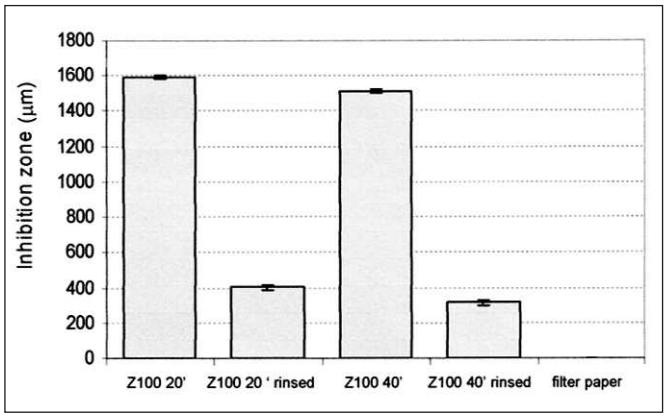


Figure 1. *Interaction between the inhibition zone and the experimental control groups.*

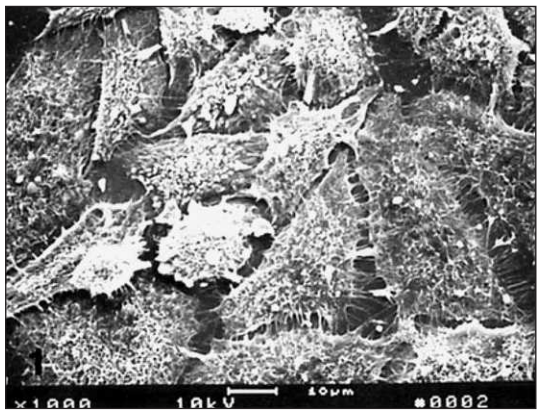


Figure 2. *Group 5—Control. Note that the MDPC-23 cells exhibit many fine cellular processes, which seem to attach them to the glass substrate. SEM, original magnification 1,000x.*

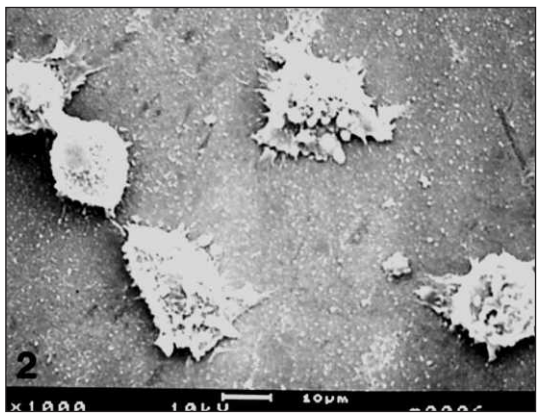


Figure 3. *Group 1—Z-100 irradiated for 20 seconds. Only a few cells remained attached to the glass substrate. These cells close to the thick inhibition zone exhibit round shape and a few short cytoplasmatic processes on their cytoplasmic membrane. SEM, original magnification 1,000x.*

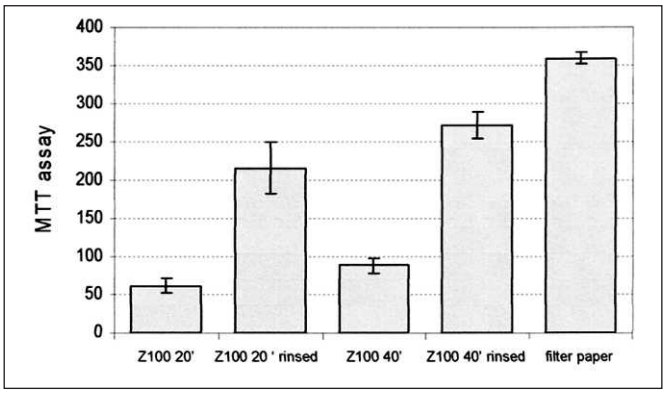


Figure 4. *Cell metabolic activity assessed for succinic dehydrogenase activity.*

tistically different among the experimental groups. In addition, no statistically significant difference occurred when the experimental groups were compared to the control group. In the control group, there was no inhibition zone formation. The interaction between the inhibition zone and the experimental and control groups is shown in Figure 1.

Cell morphology was demonstrated in the control group where MDPC-23 cells were organized into epithelioid nodules. The MDPC-23 cells exhibited many fine cellular processes that seemed to attach themselves to the glass substrate (Figure 2). Similar cell morphology was observed in Groups 2 and 4. In Groups 1 and 3, the cells close to the thick inhibition zone exhibited a round shape with a few short cytoplasmic processes or dramatic disruption of the plasma membrane (Figure 3). Adjacent cells exhibited morphology similar to the control group.

Cell metabolic activity that was assessed for succinic dehydrogenase (SDH) activity by means of the MTT assay is shown in Figure 4. Considering the mitochondrial respiration was 100% for Group 5 (control), a reduction of 83%, 40.1%, 75.5% and 24.5%, for Groups 1, 2, 3 and 4, respectively, were estimated. The statistical analysis demonstrated that there was no statistically significant difference between Groups 2 and 4, in which the dentin discs were rinsed for 20 or 40 seconds, respectively. However, comparisons conducted among the experimental groups and the control group demonstrated statistically significant differences regarding cell metabolism. Non-rinsed resin discs irradiated for 20 seconds were more cytotoxic, whereas, those irradiated for 20 or 40 seconds, then rinsed, resulted in less cytotoxic effects on MDPC-23 cells.

According to the statistical analysis of correlation (Pearson correlation test), a high negative correlation (r=-.96) was demonstrated between the metabolic activity of MDPC-23 cells and the thickness of the inhibition zone formed around the resin discs.

DISCUSSION

In this *in vitro* study, the period of light-curing was demonstrated to be inversely related to the cytotoxicity of the tested resin composite. In addition, the rinsing procedure to remove residual unreacted monomers from the resin composite surface following polymerization decreases the cytotoxicity of the experimental resin-based material. Consequently, it seems that 40 seconds of light-curing associated with the rinsing procedure (Group 4) was the best experimental condition evaluated in this study. However, there was no statistically significant difference when the resin discs were irradiated for 20 or 40 seconds and immediately rinsed. On the other hand, 20 seconds of light-curing time with no rinsing procedure was the most toxic experimental situation evaluated. In this condition (Group 1), the resin composite gave rise to a remarkably wide inhibition zone and significantly reduced the cellular mitochondrial respiration by 83%. These data were confirmed through Kruskal-Wallis and complementary Dunnett tests.

According to Ferracane (1995), restorative resin composite consists of organic polymerizable matrix (monomers, co-monomers, initiators, co-initiators, inhibitors of polymerization and photostabilizer-organic phase), reinforcing fillers (quartz, glasses, borosilicate and amorphous silica-inorganic phase) and silane coupling agent, which connects of organic matrix with inorganic fillers. Partially polymerized resin composites may have their components released when unbonded monomers and/or additives are eluted by solvents after setting or when leachable components are created by degradation or erosion with time (Geurtsen, 1998). In addition, it seems that a wide range of factors may be responsible for the release of unbonded compounds from cured dental composites. An important factor that determines the amount of leachable resin components is the monomer-polymer conversion mechanism. Consequently, curing time, thickness of resin increment and light intensity provided by light units plays a role in the amount of unconverted monomers that may, in turn, remain in the resin composite. In this study, resin discs 2 mm in thickness were cured using a light unit that exhibited light intensity of 530mW/cm^2. However, the samples received different time lengths of light-curing, including 20 seconds (Groups 1 and 3) or 40 seconds (Groups 2 and 4). In this situation, different cytopathic effects caused by the resin discs light cured for different periods of time were observed. This was demonstrated through the evaluation of widths of the zones of inhibition around the samples and the cell metabolism assessed by MTT assay. Probably, the short-time curing procedure (20 seconds) applied in Groups 1 and 3 was not enough to make an adequate monomer-polymer conversion. Consequently, in Groups 1 and 3, the

high amount of unconverted monomers was probably responsible for the wide zone of inhibition and the remarkable reduction of cell metabolism. A direct correlation between light-curing time and the degree of monomer-polymer conversion was previously reported by Tanaka and others (1991). The authors evaluated the residual monomers TEGDMA and BIS-GMA eluted by water from polymerized dental composites light cured for 30 or 50 seconds. They demonstrated that the longest light-curing time resulted in a significant decrease in the residual monomer content into water. While the current *in vitro* study did not evaluate the amount of residual monomers released in culture medium, it is probable that a poor monomer-polymer conversion plays an important role in the cytopathic effects caused by the resin discs irradiated for only 20 seconds (Group 1). Resin discs irradiated for 40 seconds (Group 3) demonstrated decreased cytotoxic effects on the odontoblast-cell line, MDPC-23.

According to Rueggeberg and Margeson (1990) oxygen plays a role in the formation of an inhibited layer on the surface of the resin composite in contact with room air. It seems that the thickness of the air-inhibited layer depends on the monomer composition and activating system. Consequently, short-time light curing may result in a thicker layer of unreacted monomers, that, in turn, may be removed from the resin surface mechanically or by applying solvents. In the current study, the resin discs were rinsed three times with PBS and culture medium (Groups 2 and 4) in order to remove superficial, unconverted monomers that had decreased the cytopathic effects on the MDPC-23 cells when compared with non-rinsed samples (Groups 1 and 3). This was confirmed by statistical analysis. Consequently, the authors suggest that PBS and culture medium were effective solutions for removing most of the unreacted monomers from the resin discs, even when the light-curing time was reduced from 40 to 20 seconds.

Spahl, Budzikiewicz and Geurtsen (1998) reported that basic monomers and various additives, such as coinitiators, photostabilizers and inhibitors can be extracted by methanol from resin composite without any problem. However, even highly water-soluble compounds were not extracted in cytotoxic concentrations. Consequently, the authors suggest that PBS and culture medium used to rinse the resin discs were not enough to remove all the unreacted resin monomers from the samples surface. For this reason, even in Groups 2 and 4, in which the resin discs were rinsed, there was a decrease in the cell metabolism by 40.1% and 24.5%, respectively. In addition, Spahl and others (1998) reported that the bulky and large monomers, such as BIS-GMA, cannot diffuse through the set resins and have very low solubility in water. Consequently, this basic monomer is probably not responsible for the high cytopathic effects. In contrast, small comonomers such as ethylene glycol compounds (TEGDMA) can diffuse easily within a polymerized resin composite because it is far more water-soluble. Since Z-100 resin composite contains BIS-GMA and TEGDMA in its composition, the authors speculate that TEGDMA, rather than BIS-GMA monomers, were responsible for the cytopathic effects observed, especially when the resin discs were light cured for 20 seconds.

In the last decade, many *in vitro* studies (Gerzina & others, 1991; Gerzina & Hume, 1994) reported that unreacted monomers from resin composite can be released into an adjacent aqueous phase and diffuse across dentinal tubules to reach the pulpal space. Consequently, Gerzina and Hume (1996) evaluated the effect of bonding resins on the release and diffusion through dentin of monomer components of composites. In that paper, the authors evaluated Z-100 resin composite in combination or not with a bonding agent. It was demonstrated that a specific monomer (TEGDMA) from the resin composite can be released and diffused through dentinal tubules. Higher diffusion of resin components was observed when Z-100 was applied on dentin previously conditioned with phosphoric acid and hybridized with a bonding agent. Consequently, TEGDMA diffusion from Z-100 was not prevented by the presence of a hybrid layer on dentin. Based on these data, the authors suggest that the higher the level of unconverted monomers on the surface of Z-100, the higher the amount of monomer that may diffuse across dentinal tubules. Several *in vitro* studies (Geurtsen, 1998; Hanks & others, 1991; Geurtsen, Spahl & Leyhausen, 1998) have demonstrated that TEGDMA is a cytotoxic dental resin compound.

The authors suggest that the amount of unconverted monomers on dentin should be reduced to avoid diffusion of the residual monomers to the pulpal space, resulting in chemical damage to the pulp tissue. This may be accomplished by treating appropriate increments of restorative resin with adequate light intensity and the time of light curing, which will play an important role in converting uncured monomers into polymers. In addition, TEGDMA eluted from a polymerized resin composite restoration may also serve as a substrate for polyethylene glycol degrading microorganisms that proliferate within the marginal gap between a filling and the cavity wall, causing secondary caries as well as pulpal irritation due to the production of microbial cytotoxic substances (Qvist, 1993).

CONCLUSIONS

Based upon the experimental conditions, it was concluded that the period of light curing is inversely related to the cytotoxic effects of the experimental resin composite. In addition, the rising procedure to remove

residual unreacted monomers from the surface of the resin composite following its polymerization decreases the cytotoxicity of this resin-based material.

Acknowledgements

This study was partially supported by the Fundação de Amparo à Pesquisa do Estado de São Paulo/FAPESP (grant #00/7893-0) and Conselho Nacional de Desenvolvimento Científico e Tecnológico/CNPq (grant #520027/00-0).

(Received 17 June 2002)

References

Costa CA, Vaerten MA, Edwards CA & Hanks CT (1999) Cytotoxic effects of current dental adhesive systems on immortalized odontoblast cell line MDPC-23 *Dental Materials* **15(6)** 434-441.

Costa CA, Edwards CA & Hanks CT (2001) Cytotoxic effects of cleansing solutions recommended for chemical lavage of pulp exposures *American Journal of Dentistry* **14(1)** 25-30.

Ferracane JL (1995) Current trends in dental composites *Critical Review in Oral Biology and Medicine* **6(4)** 302-318.

Gerzina TM, Picker K, Hood A & Hume W (1991) Toxicity and quantitative analysis of TEGDMA and composite resin eluates *Journal of Dental Research* **70** Abstract #1263 p 424.

Gerzina TM & Hume WR (1994) Effect of dentine on release of TEGDMA composite from resin *in vitro Journal of Oral Rehabilitation* **21(4)** 463-468.

Gerzina TM & Hume WR (1996) Diffusion of monomers from bonding composite-resin combinations through dentine *in vitro Journal of Dentistry* **24(1-2)** 125-128.

Geurtsen W (1998) Substances released from dental resin composites and glass ionomer cements *European Journal of Oral Science* **106(2 pt 2)** 687-695.

Geurtsen W, Spahl W & Leyhausen G (1998) Residual monomer/additive release and variability in cytotoxicity of light-curing glass-ionomer cements and compomers *Journal of Dental Research* **77(12)** 2012-2019.

Hanks CT, Strawn SE, Wataha JC & Craig RG (1991) Cytotoxic effects of resin components on cultured mammalian fibroblasts *Journal of Dental Research* **70(11)** 1450-1455.

Hanks CT, Sun ZL, Fang DN, Edwards CA, Wataha JC, Ritchie HH & Butler WT (1998) Cloned 3T6 cell line from CD-1 mouse fetal molar dental papillae *Connective Tissue Research* **37(3-4)** 233-249.

Hashieh IA, Cosset A, Franquin JC & Camps JC (1999) *In vitro* cytotoxicity of one-step dentin bonding systems *Journal of Endodontics* **25(2)** 89-92.

Kanca J 3rd (1986) The effect of thickness and shade on the polymerization of light-activated posterior composite resins *Quintessence International* **17(12)** 809-811.

Pearson GJ & Longman CM (1989) Water sorption and solubility of resin-based materials following inadequate polymerization by a visible-light curing system *Journal of Oral Rehabilitation* **16(1)** 57-61.

Pilo R, Oelgiesser D & Cardash HS (1999) A survey of output intensity and potential for depth of cure among light-curing units in clinical use *Journal of Dentistry* **27(3)** 235-241.

Qvist, V (1993) Resin restorations: Leakage, bacteria, pulp *Endodontic Dental Traumatology* **9(4)** 127-152.

Rueggeberg FA & Margeson DH (1990) The effect of oxygen inhibition on an unfilled/filled composite system *Journal of Dental Research* **69(10)** 1652-1658.

Rueggeberg FA, Caughman WF, Curtis JW Jr & Davis HC (1993) Factors affecting cure at depths within light-activated resin composites *American Journal of Dentistry* **6(2)** 91-95.

Sakaguchi RL, Douglas WH & Peters MC (1992) Curing light performance and polymerization of composite restorative materials *Journal of Dentistry* **20(3)** 183-188.

Spahl W, Budzikiewicz H & Geurtsen W (1998) Determination of leachable components from four commercial dental composites by gas and liquid chromatography/mass spectrometry *Journal of Dentistry* **26(2)** 137-145.

Stanley HR, Myers CL, Heyde JB & Chamberlain J (1972) Primate pulp response to an ultraviolet light cured restorative material *Journal of Oral Pathology* **1(3)** 108-114.

Tanaka K, Taira M, Shintani H, Wakasa K & Yamaki M (1991) Residual monomers (TEG DMA and BIS-GMA) of a set visible-light-cured dental composite resin when immersed in water *Journal of Oral Rehabilitation* **18(4)** 353-362.

Wataha JC, Hanks CT & Craig RG (1991) The *in vitro* effects of metal cations on eukaryotic cell metabolism *Journal Biomedical Materials Research* **25(9)** 1133-1149.

Wataha JC, Hanks CT & Craig RG (1992) *In vitro* synergistic, antagonistic, and duration of exposure effects of metal cations on eukaryotic cells *Journal Biomedical Materials Research* **26(10)** 1297-1309.

Watts DC (1996) The structural scope of biomaterial as amalgam alternatives Transactions of Academy *Dental Materials* **9** 51-67.

©Operative Dentistry, 2003, **28-4,** 371-377

Effectiveness of Composite Cure Associated with Different Curing Modes of LED Lights

MS Soh • AUJ Yap • KS Siow

Clinical Relevance

The effectiveness of cure associated with LED curing lights is product dependent.

SUMMARY

This study compared the effectiveness of cure of two LED (light-emitting diodes) lights (Elipar FreeLight [FL], 3M-ESPE; GC e-Light [EL], GC) to conventional (Max [MX], Dentsply-Caulk [control]), high intensity (Elipar TriLight [TL], 3M-ESPE) and very high intensity (Astralis 10 [AS], Ivoclar Vivadent) halogen lights. The 10 light-curing regimens investigated were: FL1 400 mW/cm² [40 seconds], FL2 0-400 mW/cm² [12 seconds] → 400 mW/cm² [28 seconds], EL1 750 mW/cm² [10 pulses x 2 seconds], EL2 350 mW/cm² [40 seconds], EL3 600 mW/cm² [20 seconds], EL4 0-600 mW/cm² [20 seconds] → 600 mW/cm² [20 seconds], TL1 800 mW/cm² [40 seconds], TL2 100-800 mW/cm² [15 seconds] → 800 mW/cm² [25 seconds], AS1 1200 mW/cm² [10 seconds], MX 400 mW/cm² [40 seconds].

Effectiveness of cure with the different modes was determined by measuring the top and bottom surface hardness (KHN) of 2-mm thick composite (Z100, [3M-ESPE]) specimens using a digital microhardness tester (n=5, load=500 g; dwell time=15 seconds).

Results were analyzed using one-way ANOVA/Scheffe's post-hoc test and Independent Samples *t*-test ($p<0.05$). At the top surface, the mean KHN observed with LED lights ranged from 55.42 ± 1.47 to 68.54 ± 1.46, while that of halogen lights was 62.64 ± 1.87 to 73.14 ± 0.97. At the bottom surface, the mean KHN observed with LED and halogen lights ranged from 46.90 ± 1.73 to 66.46 ± 1.18 and 62.26 ± 1.93 to 70.50 ± 0.87, respectively. Significant differences in top and bottom KHN values were observed between different curing regimens for the same light, and between LED and halogen lights. Although curing with most modes of EL resulted in significantly lower top and bottom KHN values than the control, no significant difference was observed for the different modes of FL. Hence, the effectiveness of composite cure with LED LCUs is product dependent.

MS Soh, BSc (Hons), research scholar, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

*Adrian UJ Yap, BDS, MSc, PhD, FAMS, FADM, FRSH, associate professor, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

KS Siow, BSc, MSc, PhD, associate professor, Department of Chemistry, Faculty of Science, National University of Singapore, Republic of Singapore

*Reprint request: 5 Lower Kent Ridge Road, Singapore 119074, Republic of Singapore; e-mail: rsdyapuj@nus.edu.sg

INTRODUCTION

Light-activated resin composite restoratives have been widely applied in clinical dentistry since their introduction in the late 1970s. With the dramatic rise in the use of composite restoratives, there is also a rapid increase in the number of light-activation units. Halogen-based light curing units (LCUs) are the most commonly employed light-activation units in dentistry. Photocuring of dental composite by halogen-based LCUs is initiated by electromagnetic wavelengths between 400 and 500 nm. The blue light region of the visible spectrum is produced from a halogen bulb combined with a filter. Light in this wavelength region activates camphoroquinone (CQ), the photoinitiator present in most light-activated resin composite restoratives, most effectively. The absorption spectrum of CQ lies in the 450-500 nm wavelength range, with peak absorption at 470 nm (Lee & others, 1993; Denehy & others, 1993). It was also suggested that a minimum intensity of 400 mW/cm² in the proper spectral distribution is necessary for complete polymerization of light-activated composite of 2 mm in depth (Rueggeberg, Caughman & Curtis, 1994; Tate, Porter & Dosch, 1999).

The use of halogen curing light units to polymerize dental composite has several drawbacks despite their popularity. The halogen bulbs (which have a limited effective lifetime of about 40 to 100 hours), reflector and filter degrade over time due to high operating temperatures and the large quantity of heat produced during the curing cycles (Jandt & others, 2000a). The aforementioned will reduce the effectiveness of polymerization in composite restoratives (Barghi, Berry & Hatton, 1994). Adequate polymerization of light-activated composites is important not only to ensure optimum physico-mechanical properties (Asmussen, 1982) but also to ensure that clinical problems do not arise due to the cytotoxicity of inadequately polymerized material (Caughman & others, 1991). The effectiveness of polymerization is dependent not only upon the chemistry of the material and concentration of the initiator, but also upon the filler particle type, size and quantity. In addition, polymerization is dependent on the effectiveness of the radiation sources, including spectral distribution, intensity, exposure time and alignment of the light-tip guide (Harrington, Wilson & Shortall, 1996).

To overcome the several drawbacks of halogen curing light units, blue LED (light-emitting diodes) LCUs have been developed for polymerization of light-activated dental materials. LEDs have lifetimes of more than 10,000 hours and undergo little degradation of light output over time. They use junctions of doped semiconductors (p-n junctions) to generate light and, hence, require no filters to produce blue light and are resistant to shock and vibration. Their relatively low power consumption makes them suitable for portable use. The narrower spectral output of these blue LEDs of 440 to 490 nm fall within the CQ absorption spectrum (Mills, Jandt & Ashworth, 1999). Previous studies (Mills & others, 1999; Jandt & others, 2000; Stahl & others, 2000) have shown that blue LED LCUs have the potential to polymerize dental composites without having the drawbacks of halogen LCUs. Nomura and others (2002) reported that dental resins cured with blue LED have a higher degree of polymerization and more stable three-dimensional structures than those cured with halogen lamps. Kurachi and others (2001) and Dunn and Bush (2002) have, however, found that LED LCUs resulted in a lower effectiveness of cure. The number of studies on the effectiveness of cure of LED lights are still limited and differences in findings have yet to be explained.

Another current trend in composite polymerization is the use of high intensity lights for short durations (St Georges & Miguez, 2001). High intensity lights provide higher values for degree of conversion, but they also produce higher contraction strains during composite polymerization (Unterbrink & Muessner, 1995; Sakaguchi & Berge, 1998; Dennison & others, 2000). Clinically, contraction stresses may contribute to post-operative sensitivity, microleakage, recurrent caries and marginal discoloration. One way to minimize polymerization shrinkage without affecting the degree of conversion of light-activated composites is to allow flow during setting by means of controlled polymerization. Applying short pulses of energy or pre-polymerization with low-intensity light, followed by a final cure with high intensity (soft-start techniques), may minimize polymerization shrinkage. Studies have shown that these polymerization modes result in smaller marginal gap, increased marginal integrity and improved material properties (Kanca & Suh, 1999; Uno & Asmussen, 1991; Mehl, Hickel & Kunzelmann, 1997). Yap, Soh and Siow (2002) have, however, found that the effectiveness of cure at the bottom surface of composites was significantly affected by pulse activation and soft-start regimens. It was found that resin polymerization associated with the initial cure of pulse activation and soft-start polymerization regimens may interfere with light transmission during the final cure. However, the effectiveness of cure associated with the soft-start and pulse cure modes of LED LCUs has not been investigated.

This study compared the effectiveness of composite cure of two LED lights to conventional, high intensity and very high intensity halogen lights. The effectiveness of cure of soft-start and pulse activation regimens in LED LCUs was also investigated.

METHODS AND MATERIALS

A mini-filled resin composite (Z100; 3M-ESPE, St Paul, MN, USA) of A2 shade and five LCUs were selected for this study. The five LCUs were two blue

Table 1: *Details of the Light Curing Units (LCU) and the Various Curing Modes Evaluated*

LCU	Curing Modes	Curing Profiles
Elipar FreeLight (LED)	Standard (FL1)	400 mW/cm² (40 seconds)
3M-ESPE, Seefeld, Germany	Exponential (FL2)	0-400 mW/cm² → 400 mW/cm² (12 seconds) (28 seconds)
GC e-Light (LED)	Pulse Curing (EL1)	750 mW/cm² (10 pulses x 2 seconds)
GC Europe, Leuven, Belgium	Standard (EL2)	350 mW/cm² (40 seconds)
	Turbo (EL3)	600 mW/cm² (20 seconds)
	Soft-start curing A (EL4)	0-600 mW/cm² → 600 mW/cm² (20 seconds) (20 seconds)
Max (Halogen)	Standard (MX)	400 mW/cm² (40 seconds)
Dentsply-Caulk, Milford, DE, USA		
Elipar TriLight (Halogen)	Standard (TL1)	800 mW/cm² (40 seconds)
3M-ESPE, Seefeld, Germany	Exponential (TL2)	100-800 mW/cm² → 800 mW/cm² (15 seconds) (25 seconds)
Astralis 10 (Halogen)	High Power (AS1)	1200 mW/cm² (10 seconds)
Ivoclar-Vivadent, Liechtenstein, Austria		

Curing profiles are based on manufacturers' information.

LED (Elipar FreeLight, 3M-ESPE, Seefeld, Germany; GC e-Light, GC Europe, Leuven, Belgium, Europe), a high intensity halogen (Elipar TriLight, 3M-ESPE, Seefeld, Germany), a very high intensity halogen (Astralis 10, Ivoclar Vivadent, Schaan Liechtenstein, Austria) and a conventional halogen (Max, Dentsply-Caulk, Milford DE, USA) lights. The 10 light-curing regimens examined from these five LCUs are detailed in Table 1. The intensity of all the curing lights was checked with a radiometer (Cure Rite, EFOS INC, Ontario, Canada) prior to use to ensure consistency in intensity output from the light source. Standard deviations ranging from 2.17 to 5.34 mW/cm² were obtained for the various lights.

Elipar FreeLight contains 19 LEDs aligned on three consecutive planes that emit light mainly in the wavelength range of 440 to 490 nm, while GC e-Light, which contains 64 LEDs, produces a visible blue light between 440 and 490 nm wavelengths for photopolymerization of dental materials.

The hardness testing methodology used to assess the effectiveness of cure was based on that used by Yap (2000). The composite was placed in black delrin molds with square cavities 2-mm deep and 4-mm wide/long confined between two opposing acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide (1-mm thick) was then placed on the molds and excess material was extruded by pressure application. The composite was then irradiated from the top through the glass slide and acetate strip using the different light-curing modes. Immediately after light polymerization, the acetate strips were removed, and the specimens in their molds were positioned centrally beneath the indenter of a digital microhardness tester (FM7, Future-Tech Corp, Tokyo, Japan) to assess the Knoop's Hardness Number (KHN) of the top and bottom surfaces. A 500g load was then applied through the indenter with a dwell time of 15 seconds. The KHN corresponding to each indentation was computed by measuring the dimensions of the indentations and using the formula KHN=1.451 x (F/d^2), where F is the test load in Newtons and d is the longer diagonal length of an indentation in millimeters. Five specimens were made for each light-curing mode. Three readings were taken for each specimen and averaged to form a single value for that specimen. The mean KHN and hardness ratio of the five specimens were then calculated and tabulated using the following formula: Hardness ratio = KHN of bottom surface/KHN of top surface. The data obtained were subjected to one-way analyses of variance (ANOVA)/Scheffe's post-hoc test and Independent Samples *t*-test at a significance level of 0.05.

RESULTS

Table 2 shows the mean KHN and hardness ratio of the different light curing regimens for the different LCUs. The results of statistical analysis are shown in Tables 3 and 4, while Table 3 compares the mean KHN and hardness ratio of various LCUs or modes to the control light source (MX) and Table 4 compares the curing regimens of the same LCUs.

At the top surface of the composite, KHN after polymerization with MX (control) was found to be significantly higher than EL1, EL3 and EL4 and significantly

Table 2: *Mean KHN and Hardness Ratio Observed for the Different LCU and Their Respective Curing Modes*

LCU	Light-Curing Modes	Top KHN	Bottom KHN Ratio	Hardness
Elipar FreeLight	FL1	68.54 (1.46)	66.46 (1.18)	0.97 (0.02)
	FL2	68.20 (1.80)	64.66 (1.18)	0.95 (0.02)
GC e-Light	EL1	58.70 (1.32)	53.14 (1.36)	0.91 (0.02)
	EL2	65.62 (0.11)	54.86 (1.88)	0.84 (0.03)
	EL3	55.42 (1.47)	46.90 (1.73)	0.85 (0.03)
	EL4	61.34 (0.95)	52.68 (0.45)	0.86 (0.01)
Max	MX	65.44 (0.17)	65.30 (0.17)	1.00 (0.00)
Elipar TriLight	TL1	69.90 (1.34)	68.70 (1.34)	0.98 (0.01)
	TL2	73.14 (0.97)	70.50 (0.87)	0.96 (0.01)
Astralis 10	AS1	62.64 (1.87)	62.26 (1.93)	0.99 (0.00)

Standard deviations in parentheses

Table 3: *Results of Mean KHN and Hardness Ratio of Various LCU and Modes to Conventional Halogen LCU*

Variable	Significance
KHN Top	EL1, EL3, EL4 < MX < TL1, TL2
KHN Bottom	EL1, EL2, EL3, EL4 < MX < TL2
Hardness Ratio	EL1, EL2, EL3, EL4, FL2 < MX

Results of One-way ANOVA/Scheffe's post-hoc test ($p<0.05$). < indicates statistical significance.

lower than TL1 and TL2. No significant difference in top KHN was observed between MX and EL2, AS1, FL1 and FL2. When the different modes of the same light were compared, mean KHN after polymerization with EL2 was found to be significantly higher than EL1, EL3 and EL4. KHN after polymerization with TL2 was found to be significantly higher than TL1. No significant difference was found between FL1 and FL2.

At the bottom surface of the composite, KHN after polymerization with MX was found to be significantly higher than EL1, EL2, EL3 and EL4 and significantly lower than TL2. No significant difference was observed for MX and AS1, FL1, FL2 and TL1. For the comparison of the mean KHN among the different modes of the same LCU, FL1 was found to be significantly higher than FL2. EL1, EL2 and EL4 were found to be significantly higher than EL3. TL2 was found to be significantly higher than TL1.

The hardness ratio associated with EL1, EL2, EL3, EL4 and FL2 was found to be significantly lower than MX. No significant difference in hardness ratio was observed between MX and AS1, FL1, TL1 and TL2. The hardness ratio of EL1 was significantly higher than EL2 and EL3. TL1 was significantly higher than TL2. No significant difference was observed between FL1 and FL2.

DISCUSSION

The effectiveness of composite cure may be assessed directly and indirectly. Direct methods that assess the degree of conversion, such as infrared spectroscopy and laser Raman spectroscopy, have not been accepted for routine use as they are complex, expensive and time-consuming (Rueggeberg & Craig, 1988). Indirect methods have included visual, scraping and hardness testing. Incremental surface hardness has been shown to be an indicator of the degree of conversion (Asmussen, 1982) and a good correlation between Knoops hardness and infrared spectroscopy has been reported (DeWald & Ferracane, 1987). Effectiveness of polymerization of both top and bottom surfaces of specimens were assessed by a digital microhardness tester in this study due to its relative simplicity.

Denehy and others (1993) have found that light intensity measurements decreased with increasing distance from curing tip, and the reduced light intensity will result in a softer bottom of a 2-mm thick specimen. Hence, to assess the effectiveness of cure by the different curing light units, the composite material investigated and the distance of light-cure tip from composite (1 mm via usage of glass slide) were standardized in this study. Shade A2 was selected to minimize the effects of colorants on light polymerization (Bayne, Heymann & Swift, 1994) and 2-mm thick composite specimens were evaluated as they gave uniform and maximum polymerization (Yap, 2000). As a minimum intensity of 400 mW/cm² has been suggested for routine polymerization (Rueggeberg & others, 1994; Tate & others, 1999), this light intensity, together with the manufacturer's recommended cure time of 40 seconds was used as control in this study.

One inherent drawback to light-activated composites was that the degree to which these materials cure is proportional to the amount of light to which they are exposed (Rueggeberg & others, 1994). For resin composites 2 mm in depth, source intensity and exposure duration are the two important factors that influenced resin cure (Rueggeberg & others, 1993). The top surface hardness of a resin composite was less dependent on light intensity than the bottom surface hardness (Denehy & others, 1993; Fowler, Swartz & Moore, 1994). This was attributed to the fact that at the top surface, sufficient light energy reaches and activates the photoinitiator camphoroquinone, initiating poly-

Table 4: *Comparison of Mean KHN of the Various Curing Modes for the Same Lights*

Variable	LCU	Differences
KHN Top	**Elipar FreeLight**	NS
	GC e-Light	EL2 > EL1, EL3, EL4
		EL4 > EL1, EL3
		EL1 > EL3
	Elipar TriLight	TL2 > TL1
KHN Bottom	**Elipar FreeLight**	FL1 > FL2
	GC e-Light	EL1, EL2, EL4 > EL3
	Elipar TriLight	TL2 > TL1
Hardness Ratio	**Elipar FreeLight**	NS
	GC e-Light	EL1 > EL2, EL3
	Elipar TriLight	TL1 > TL2

Results of One-way ANOVA/Scheffe's post-hoc test or Independent Samples t-test ($p<0.05$). > indicates statistical significance, while NS denotes no statistical significance.

merization. Continuing exposure sustains the activation of photoinitiator molecules near the surface. Hence, duration of exposure is the more important factor in polymerization of surface resin composites. This phenomenon accounted for the significant difference in the top KHN of EL1 and EL3. The total exposure duration for these two regimens was 20 seconds. At the top surface of the composite, curing with both TL1 and TL2 was found to be significantly harder than MX. This was due to the use of higher light intensity that caused an increase in the total light energy density. Light energy density is defined as the product of intensity and exposure duration. The light energy density for MX, FL1, EL2, AS1 and TL1 was found to be 16000, 16000, 14000, 12000 and 32000 mJ/cm², respectively. The total light energy density of TL1 was found to double the light energy density of MX. This phenomenon accounted for the significantly harder top obtained with TL1 compared to MX. No significant difference in KHN of the top surface was observed between MX and FL1, EL2 and AS1. This was due to the slight variation in the total light energy density produced. Light energy density for all the other light-curing regimens was not calculated, as the rate of increased light intensity for soft-start and pulse curing regimens could not be obtained.

At the upper surface of a restoration, where no overlying composite interfered with light transmission, it has been found that even a curing source with relatively low intensity can cure the resin matrix to an extent almost equal to when high intensity lights are used (Rueggeberg & Jordan, 1993). Hansen and Asmussen (1993) have also found that inferior curing units were able to polymerize the surface just as effectively as good light sources. Hence, the quality of a curing unit cannot be assessed by top surface hardness. As light passes through the bulk of the restorative material, its intensity is decreased greatly due to light absorption and scattering by resin composites, thus, decreasing the potential for cure (Ruyter & Øysæd, 1982). Therefore, intensity of the light source becomes the more critical factor in determining the effectiveness of polymerization for the bottom surface hardness. For all four curing modes of GC e-Light, hardness of the bottom surfaces was significantly lower than the control despite the similar light energy densities between the two lights. Reasons for this phenomenon are not known; however, possible hypotheses include differences in the determination of light intensity and the spectrum of the light source. The greatest bottom KHN was found with the TL2 curing regimen. This phenomenon can be explained by using higher light intensity. No significant difference in bottom KHN was observed between MX and AS1 (the very high intensity/low duration LCU). This may be due to the similar light energy density of the two LCUs. The use of very high intensity was coupled with a short duration. Heat produced by the light source may also play a part in the curing of the composites.

If polymerization was effective (that is, maximum cure of the specimens was achieved), an ideal ratio of 1:1 should be achieved. The hardness of the top surface should be similar to the bottom surface. However, as light passed through the bulk of the composite, the light intensity was greatly reduced due to light scattering and absorption, thus, decreasing the effectiveness of polymerization (Ruyter & Øysæd, 1982). This scattering of light accounted for the minor differences in hardness between the top and bottom surfaces of the 2-mm composites evaluated in this study. Studies had also suggested that the hardness gradient should not exceed 10% to 20% (hardness ratio should be greater than 0.8) for adequately photo-activated resin composites (Pilo & Cardash, 1992; Yearn, 1985). A hardness ratio of EL1, EL2, EL3, EL4 and FL2 was generally lower than MX and correlated well with the results of the KHN bottom. The hardness ratio obtained in this study for all light-curing modes was found to be greater than 0.8.

When comparing the different modes in the same LCU, a significant difference in the top and bottom KHN of the composite was observed using soft-start and pulse activation regimens of GC e-Light. Lower bottom KHN for FL2 compared to FL1 and TL1 compared to TL2, which was also observed when comparisons were made for the different modes in the same LCU. Hence, polymerization associated with the initial cure of pulse activation and soft-start polymerization regimens may interfere with light transmission during the final cure (Yap & others, 2002).

The use of blue LED LCU to polymerize light-activated dental materials was first proposed by Mills and others (1999) in 1995. They used an LED LCU that consisted of 25 LEDs for their study. Jandt and others (2000a) found that an LED LCU that consisted of 27 blue LEDs

gave adequate polymerization. Fujibayashi and others (1998) have also found that LCUs, which consisted of 61 blue LEDs, gave an effective curing depth and degree of conversion. Dunn and Bush (2002) have cited that the poor performance of LED LCU that was used in their study might result from only seven blue LEDs being used. The aforementioned studies appear to suggest that the effectiveness of cure of LED LCUs may depend on the number of LEDs. This was not corroborated in the current study. In the current study, the authors observed that the effectiveness of cure of composites with Elipar FreeLight, which consisted of 19 LEDs, was comparable to conventional halogen LCUs. However, the effectiveness of cure of composites with GC e-Light, which consisted of 64 LEDs, resulted in a softer bottom as compared to the conventional halogen LCUs. Based on the results obtained from this study, EL2 will be the recommended curing regimen employed if GC e-Light LCU is to be used. The hypothesis employed in this study was that that the number of LEDs present in an LCU is not the issue for optimum cure. The light output produced from an LED LCU is not dependent on the number of LEDs but on the design of the LED in the LCU or the size of LED used. Hence, while LED LCUs have great potential to achieve effective composite cure, more studies are warranted.

CONCLUSIONS

Under the conditions of this *in vitro* study:

1. The effectiveness of cure associated with LED curing lights is product dependent.
2. The effectiveness of cure at the top and bottom surface of composites with most modes of GC e-Light was significantly lower than the conventional halogen light.
3. The effectiveness of cure at the top and bottom surfaces of composites with Elipar FreeLight is comparable to the conventional halogen light.
4. Effectiveness of cure of both the top and bottom surfaces of composites with Elipar TriLight was significantly higher than the conventional halogen light.
5. The effectiveness of cure at the top and bottom surfaces of composites with Astralis 10 is comparable to the conventional halogen light.

(Received 18 June 2002)

References

Asmussen E (1982) Factors affecting the quantity of remaining double bonds in restorative resin polymers *Scandinavian Journal of Dental Research* **90(6)** 490-496.

Barghi N, Berry T & Hatton C (1994) Evaluating intensity output of curing lights in private dental offices *Journal of the American Dental Association* **125(7)** 992-996

Bayne SC, Heymann HO & Swift EJ Jr (1994) Update on dental composite restorations *Journal of the American Dental Association* **125(6)** 687-701.

Caughman WF, Caughman GB, Shiflett RA, Rueggeberg F & Schuster GS (1991) Correlation of cytotoxicity, filler loading and curing time of dental composites *Biomaterials* **12(8)** 737-740.

Denehy GE, Pires JA, Cvitko E & Swift EJ Jr (1993) Effects of curing tip distance on light intensity and composite resin microhardness *Quintessence International* **24(7)** 517-521.

Dennison JB, Yaman P, Seir R & Hamilton JC (2000) Effect of variable light intensity on composite shrinkage *Journal of Prosthetics Dentistry* **84(5)** 499-505.

DeWald JP & Ferracane JL (1987) A comparison of four modes of evaluating depth of cure of light-activated composites *Journal of Dental Research* **66(3)** 727-730.

Dunn WJ & Bush AC (2002) A comparison of polymerization by light-emitting diode and halogen-based light-curing units *Journal of the American Dental Association* **133(3)** 335-341.

Fowler CS, Swartz ML & Moore BK (1994) Efficacy testing of visible-light-curing units *Operative Dentistry* **19(2)** 47-52.

Fujibayashi K, Ishimaru K, Takahashi N & Kohno A (1998) Newly developed curing unit using blue light-emitting diodes *Dentistry Japan* **34** 49-53.

Hansen EK & Asmussen E (1993) Correlation between depth of cure and surface hardness of a light-activated resin *Scandinavian Journal of Dental Research* **101(1)** 62-64.

Harrington E, Wilson HJ & Shortall AC (1996) Light-activated restorative materials: A method of determining effective radiation times *Journal of Oral Rehabilitation* **23(3)** 210-218.

Jandt KD, Mills RW, Blackwell GB & Ashworth SH (2000) Depth of cure and compressive strength of dental composites cured with blue light emitting diodes (LEDs) *Dental Materials* **16(1)** 41-47.

Kanca J 3rd & Suh BI (1999) Pulse activation: Reducing resin-based composite contraction stresses at the enamel cavosurface margins *American Journal of Dentistry* **12(3)** 107-112.

Kurachi C, Tuboy AM, Magalhães DV & Bagnato VS (2001) Hardness evaluation of a dental composite polymerized with experimental LED-based devices *Dental Materials* **17(4)** 309-315.

Lee SY, Chiu CH, Boghosian A & Greener EH (1993) Radiometric and spectroradiometric comparison of power outputs of five visible light-curing units *Journal of Dentistry* **21(6)** 373-377.

Mehl A, Hickel R & Kunzelmann KH (1997) Physical properties and gap formation of light-cured composites with and without "softstart-polymerization" *Journal of Dentistry* **25(3-4)** 321-330.

Mills RW, Jandt KD & Ashworth SH (1999) Dental composite depth of cure with halogen and blue light emitting diode technology *British Dental Journal* **186(8)** 388-391.

Nomura Y, Teshima W, Tanaka N, Yoshida Y, Nahara Y & Okazaki M (2002) Thermal analysis of dental resins cured with blue light-emitting diodes (LEDs) *Journal of Biomedical Material Research* **63(2)** 209-213.

Pilo R & Cardash HS (1992) Post-irradiation polymerization of different anterior and posterior visible light-activated resin composites *Dental Materials* **8(5)** 299-304.

Rueggeberg FA, Caughman WF & Curtis JW Jr (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19(1)** 26-32.

Rueggeberg FA, Caughman WF, Curtis JW Jr & Davis HC (1993) Factors affecting cure at depths within light-activated resin composites *American Journal of Dentistry* **6(2)** 91-95.

Rueggeberg FA & Craig RG (1988) Correlation of parameters used to estimate monomer conversion in a light-cured composite *Journal of Dental Research* **67(6)** 932-937.

Rueggeberg FA & Jordan DM (1993) Effect of light-tip distance on polymerization of resin composite *International Journal of Prosthodontics* **6(4)** 364-370.

Ruyter IE & Øysæd H (1982) Conversion in different depths of ultraviolet and visible light activated composite materials *Acta Odontologica Scandinavica* **40(3)** 179-192.

Sakaguchi RL & Berge HX (1998) Reduced light energy density decreases post-gel contraction while maintaining degree of conversion in composites *Journal of Dentistry* **26(8)** 695-700.

St Georges AJ & Miguez PA (2001) Visible Light-Curing: Part I *Journal of Esthetic and Restorative Dentistry* **13(2)** 140-143.

Stahl F, Ashworth SH, Jandt KD & Mills RW (2000) Light-emitting diode (LED) polymerisation of dental composites: Flexural properties and polymerisation potential *Biomaterials* **21(13)** 1379-1385.

Tate WH, Porter KH & Dosch RO (1999) Successful photocuring: Don't restore without it *Operative Dentistry* **24(2)** 109-114.

Uno S & Asmussen E (1991) Marginal adaptation of a restorative resin polymerized at reduced rate S*candinavian Journal of Dental Research* **99(5)** 440-444.

Unterbrink GL & Muessner R (1995) Influence of light intensity on two restorative systems *Journal of Dentistry* **23(3)** 183-189.

Yap AU (2000) Effectiveness of polymerization in composite restoratives claiming bulk placement: Impact of cavity depth and exposure time *Operative Dentistry* **25(2)** 113-120.

Yap AU, Soh MS & Siow KS (2002) Effectiveness of composite cure with pulse action and soft-start polymerization *Operative Dentistry* **27(1)** 44-49.

Yearn JA (1985) Factors affecting cure of visible light activated composites *International Dental Journal* **35(3)** 218-225.

©Operative Dentistry, 2003, **28-4,** 378-387

The Effect of Collagen Removal and the Use of a Low-Viscosity Resin Liner on Marginal Adaptation of Resin Composite Restorations with Margins in Dentin

MAJR Montes • MF de Goes • GMB Ambrosano
RM Duarte • LC Sobrinho

Clinical Relevance

Marginal quality was significantly enhanced by collagen depletion with 10% sodium hypochlorite in an acetone-based adhesive system and by the use of an intermediate layer of low-viscosity composite in a self-etching adhesive system.

SUMMARY

This study evaluated the influence of collagen removal and the use of a low-viscosity liner on the marginal quality of composite restorations for the total-etch system, Prime & Bond 2.1 (PB) and the self-etching primer system, Clearfil SE Bond (CSEB), in high C-factor cavities with margins in dentin. High C-factor cavities were made on dentin exposed from ground labial surfaces of 100 bovine lower incisors, randomly assigned to 10 treatment groups and restored with composite Z 250, placed in bulk. In Group 1 (PB), control group, PB was applied according to the manufacturer's directions; in Group 2 (PB/PLF), an intermediate layer of low-viscosity composite Protect Liner F (PLF) was applied on the bonding resin surface; in Group 3 (PB/SH) following acid-etching, the surfaces were treated with 10% sodium hypochlorite (SH) for one minute and in Group 4 (PB/SH/PLF), the same procedure was conducted as for Group 3, plus an intermediate layer of PLF was applied as for Group 2. In Group 5 (CSEB), the control group, CSEB was applied according to the manufacturer's directions; in Group 6 (CSEB/PLF), an intermediate layer of PLF was applied; in Group 7 (SH/CSEB), the cavity surface was pre-treated with SH; in Group 8 (SH/CSEB/PLF), SH pre-treatment was conducted as for Group 7, then an intermediate layer of PLF was applied; in Group 9 (CSEB/SH), after CSEB-primer application, the surface was treated with SH, followed by CSEB-adhesive application and in Group 10 (CSEB/SH/PLF), the same was conducted as for Group 9, then an intermediate layer of PLF was applied. The specimens were stored at

Marcos Antonio Japiassú Resende Montes, MDS, PhD, assistant professor, School of Dentistry of Pernambuco, University of Pernambuco, UPE, Brazil

*Mario Fernando de Goes, MDS, PhD, titular professor, Faculdade de Odontologia de Piracicaba—UNICAMP, Departmento de Odontologia Restauradora—Materiais Dentários, Brazil

Gláucia Maria Bovi Ambrosano, MS, PhD, associate professor, Faculdade de Odontologia de Piracicaba—UNICAMP, Departmento de Odontologia Restauradora—Materiais Dentários, Brazil

Rosangela Marques Duarte, MDS, associate professor, School of Dentistry, Federal University of Paraiba, UFPB, Brazil

Lourenço Correr Sobrinho, MDS, PhD, associate professor, Faculdade de Odontologia de Piracicaba—UNICAMP, Departmento de Odontologia Restauradora—Materiais Dentários, Brazil

*Reprint request: Av Limeira, 901, Piracicaba—SP, Brazil; e-mail: degoes@fop.unicamp.br

37°C for 24 hours, polished, molded and replicas were obtained in epoxy resin. The replicas were gold-sputter coated and observed by SEM (300x) for marginal quality classification. The Kruskal-Wallis non-parametrical multi-comparison Test ($p<0.05$) was used to obtain statistical analysis of the data. Results demonstrated that both adhesive systems in the control groups presented low marginal quality and a high variability. The use of an intermediate layer of PLF significantly improved the marginal quality with the CSEB system but had no effect with the PB system. Collagen depletion with SH enhanced marginal quality for the PB system and did not influence the CSEB system results.

INTRODUCTION

A primary concern of composite restorations is the shrinkage that follows polymerization. This shrinkage can create forces that may disrupt the bond to cavity walls and form marginal gaps, leaving the tooth more susceptible to post-operative sensitivity and recurrent caries. In order to restore a tooth in such a way that it is leakproof, there must be no dimensional mismatch at the tooth-restoration interface. Unfortunately, resin composites do not meet these requirements (Davidson & Feilzer, 1997). As polymerization proceeds, the amount of stress generated within the composite depends on the extent of the reaction, the stiffness of the composite and its ability to flow (Davidson & de Gee, 1984). The correlation between stiffness and the filler volume of composites has been demonstrated, and more heavily filled materials have consequently higher elastic modulus and produce more polymerization stress (Braem & others, 1987); this is in accordance to Hooke's law, which states that stress is directly proportional to strain in elastic deformation of a body. Less rigid composites may resolve this problem; however, lower inorganic filler content in composites has resulted in lower mechanical properties that are critical to their performance (Ferracane & Berge, 1995; Condon & Ferracane, 1998; 2000).

Another important point is that the ratio between the bound to unbound surfaces, termed the "C-factor," relate to the shape of the prepared cavity (Feilzer, de Gee & Davidson, 1987; Carvalho & others, 1996). The relative amount of unbound resin surface determines the ability of the composite to relieve the developing stresses. The combination of the two factors and a high elastic modulus shrinking restorative material confined in a high "C-factor" cavity condition will challenge and frequently destroy the bond leading to post-operative sensitivity and poor marginal quality (Unterbrink & Liebenberg, 1999). Many efforts have been made to overcome this problem, and the concept of an "elastic cavity wall" (Kemp-Scholte & Davidson, 1990a,b; Van Meerbeek & others, 1993) has been suggested as a solution. This concept is based on an interposition of a lower elastic modulus intermediate layer between the restorative composite and the adhesive-dentin interface, with the ability to withstand "plastic flow" on its initial polymerization phase, allowing the material to absorb the strain and diminish the effect of a rigid contraction at the interface (Davidson, de Gee & Feilzer, 1984; Labella & others, 1999). Low-viscosity composites could be the most appropriate materials for this concept, acting as a stress-absorbing layer between the adhesive-dentin interface and the shrinkage of the composite by partially relieving the polymerization contraction stress (Swift & others, 1996; Bayne & others, 1998; Unterbrink & Liebenberg, 1999; Labella & others, 1999; Choi, Condon & Ferracane, 2000; Montes & others, 2001).

The first generation of low-viscosity composites was introduced in 1996. They retained the same small particle size as traditional hybrid composites, however, they reduced the filler content and, consequently, viscosity (Bayne & others, 1998). Although these so-called flowable composites present a higher polymerization shrinkage than the traditional hybrid composites, their significantly lower elastic modulus (30–50%) may be less detrimental to the bond interface area since they are less rigid (Swift & others, 1996; Bayne & others, 1998; Unterbrink & Liebenberg, 1999; Labella & others, 1999; Choi & others, 2000; Montes & others, 2001).

A current concept of bonding to dentin is based on diffusion, polymerization and, consequently, micromechanical retention of monomers within the network of collagen fibers exposed after acid application. This mixed structure, formed by collagen fibers enveloped by resin and residual hydroxyapatite crystals, was first described as the "hybrid layer" in 1982 (Nakabayashi, Kojima & Masuhara, 1982). The role performed by the hybrid layer in dentin bonding has been evaluated by many investigations and has been the subject of controversy, since it has been shown that the resin infiltration in the collagen network may be incomplete (Sano & others, 1995). Therefore, many attempts to simplify the bonding process and reduce the technique sensitivity have been developed in the last decade (Van Meerbeek & others, 1998; Harada & others, 2000). A simple method to prevent the incomplete resin infiltration into the collagen network is to avoid its unprotected exposure. This can be achieved by treating the enamel and dentin with acidic self-conditioning monomer solutions instead of employing a conventional total-etch procedure in which the acid and the primer are applied separately (Watanabe, Nakabayashi & Pashley, 1994). Thus, the self-etching primer systems are considered to be an easy, reliable procedure to achieve the best conditioned surface for bonding (Harada & others, 2000). The hybrid layers formed by these self-etching primer sys-

tems are thinner (about 1 µm thick) and more uniform when compared to the hybrid layers formed by the total etch systems, which are about 4 µm thick (Vargas, Cobb & Denehy, 1997; Prati & others, 2000; Montes & others, 2001). For some authors, the hybrid layer does not contribute significantly to dentin bonding (Finger, Inoue & Asmussen, 1994; Nakajima & others, 1995; Gwinnett & others, 1996; Yoshiyama & others, 1996). For other authors, the collagen removal had a beneficial effect in dentin bonding for acetone-based adhesives and an adverse effect in water and ethanol-based adhesives (Inai & others, 1998; Pioch & others, 1999; Prati, Chersoni & Pashley, 1999; Saboia, Rodrigues & Pimenta, 2000). Sodium hypochlorite is a non-specific proteolytic agent that effectively removes organic compounds at room temperature (Sakae, Mishima & Kozawa, 1988) and has been frequently applied for collagen removal after acid-etching with various and controversial results (Gwinnett, 1994; Uno & Finger, 1995; Vargas, Cobb & Armstrong, 1997; Inai & others, 1998; Saboia & others, 2000).

Dentin has an elastic modulus in the range of 11-19.3 GPa (Van Meerbeek & others, 1993; Armstrong & others, 1998). The hybrid layer has a relatively low elastic modulus, in the range of 4.8-9.6 GPa, significantly lower than that of unaltered dentin (Van Meerbeek & others, 1993). The presence of the collagen layer could possibly allow for the establishment of an elastic gradient at the interface that could absorb part of the stress generated by the shrinking restorative composite. However, this layer is thought not to be the necessary thickness to play an important role in relieving stresses as its nature suggests (Van Meerbeek & others, 1993). Theoretically, in order to compensate for this small, hybrid layer thickness, the interposition of a resilient layer provided by a material with an elastic modulus similar to that presented by the hybrid layer itself would be appropriate (Kemp-Scholte & Davidson, 1990a,b; Van Meerbeek & others, 1993). On the other hand, how the collagen removal and, consequently, the absence of a hybrid layer influences the marginal adaptation in dentin still remains uncertain, since it would put two rigid surfaces in direct contact with one suffering a rigid contraction. Moreover, if the hybrid layer has any role in relieving stresses generated by polymerization contraction, the differences in thickness between the hybrid layers formed by a total-etch and a self-etching primer adhesive systems should be considered.

This study measured and evaluated the influence of collagen removal in a total-etch bonding technique system in a self-etching primer system and the use of an intermediate layer of a low-viscosity composite in the marginal integrity of composite restorations in cavities with a high C-factor and margins in dentin.

METHODS AND MATERIALS

The materials, manufacturers, composition and batch numbers for this study are listed in Table 1. Freshly extracted bovine lower incisors (n=100) obtained from a local *abattoir* and stored frozen for no longer than one month were used to evaluate the marginal integrity of the resin composite restorations. The teeth were ground on their labial surfaces with a water-irrigated grinding wheel using 320-grit silicone carbide paper (SiC) to obtain a flat dentin surface. Each was finished with 600-grit SiC to produce standardized surfaces. The cavities were made using a diamond bur #2294 (KG Sorensen Ltda–Brazil), especially designed for this purpose, with an active part measuring 1.8 mm in diameter and 1.5-mm deep, producing cavities with an internal area of 11.02 mm^2 and a free surface area of 2.54 mm^2, giving, as a result, standardized cavities with a C-factor of approximately 4.3 with margins totally located in dentin.

Table 1: *Materials Used in This Study*

Materials	Composition	Batch #	Manufacturer
Prime & Bond 2.1	Etchant: 37%H_3PO_4	67510	Dentsply Caulk, Milford, DE, USA
	Adhesive: UDMA, PENTA, R5-62-1 resin, BIS-EMA, Butylated hydroxitoluene, 4-Ethyl dimethyl aminobenzoate, Cetilamine hydrofluoride, Acetone		
Clearfil SE Bond	Primer: MDP, HEMA, Hydrophilic dimethacrylate, Camphorquinone, N,N-Diethanol p-toluidine, Water, Ethanol	51208	Kuraray Co Ltd, Japan
	Adhesive: MDP, BIS-GMA, HEMA, Hydrophobic dimethacrylate, Camphorquinone, N,N-Diethanol p-toluidine, silanated colloidal silica	0039AY	Kuraray Co Ltd, Japan
Protect Liner F	BIS-GMA, TEGDMA, fluoride-methyl methacrylate, camphorquinone, silanised colloidal silica, prepolymerized organic filler		
Z 250	BIS-GMA, UEDMA, BIS-EMA, zirconia/silica filler	9AM	3M Dental Products, St Paul, MN, USA

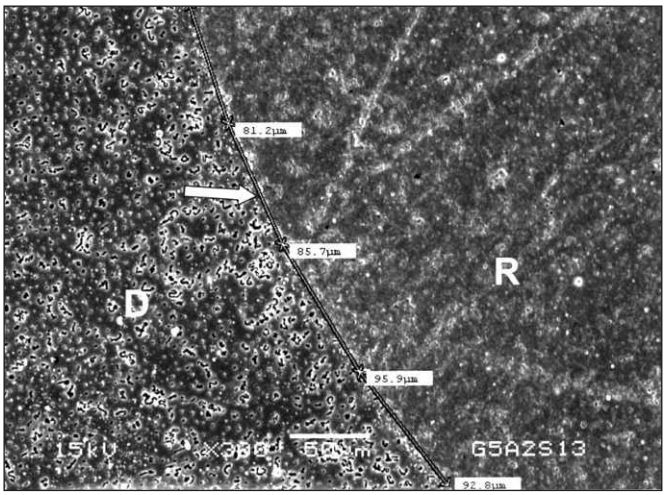


Figure 1. *Photomicrograph illustrating a section of the replica in epoxy resin with the measurement steps for classification of marginal quality. D=dentin; R=resin composite; Arrow=margin measurement. Original magnification 300x.*

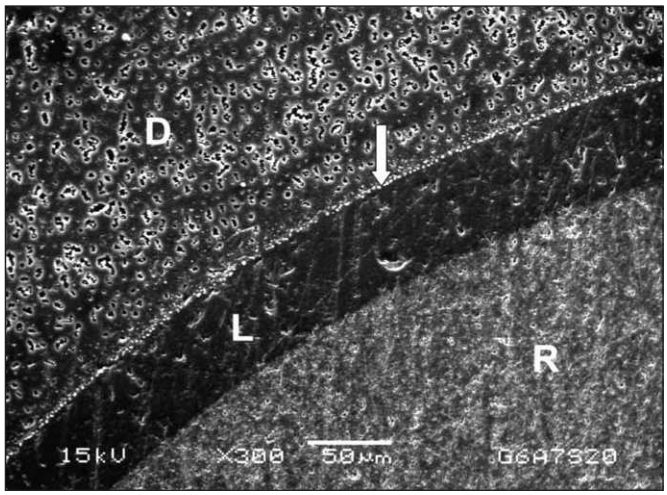


Figure 2. *Photomicrograph of a replica in epoxy resin illustrating a section of a specimen of Group 6 (CSEB/PLF) classified as "perfect margin." Note the continuous transition between filling and dentin. D=dentin; R=resin composite; L=low-viscosity resin intermediate layer; Arrow=margin line. Original magnification 300x.*

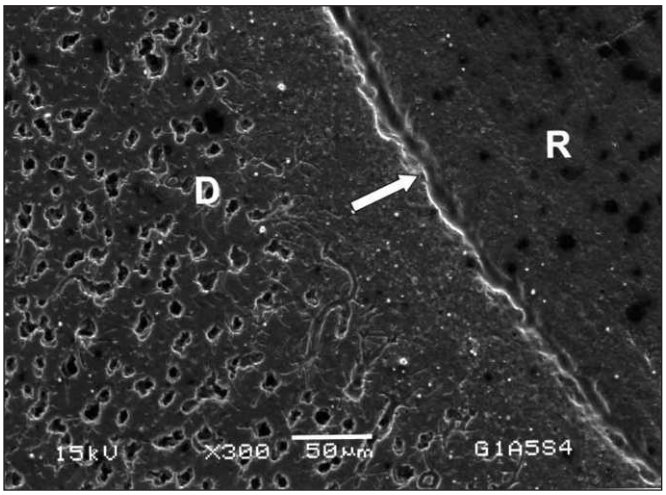


Figure 3. *Photomicrograph of a replica in epoxy resin illustrating a section of a specimen of control Group 1 (PB) classified as "marginal irregularity." Note there is not a continuous transition between filling and dentin, nevertheless it is a gap free margin. D=dentin; R=resin composite; Arrow=margin line. Original magnification 300x.*

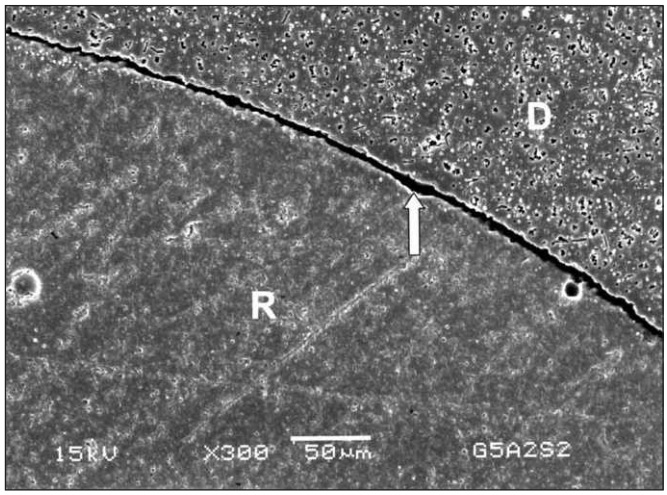


Figure 4. *Photomicrograph of a replica in epoxy resin illustrating a section of a specimen of control Group 5 (CSEB) classified as "marginal gap." Note the loss of interfacial adhesion between filling and dentin. D=dentin; R=resin composite; Arrow=marginal gap. Original magnification 300x.*

The teeth were randomly divided into 10 groups of 10 specimens each. For all the groups, the cavities were restored with resin composite Z 250 placed in bulk and light cured for 30 seconds.

For Groups 1 to 4, the dentin surfaces were etched with 37% phosphoric acid (Dentsply Caulk, Milford, DE, USA) for 15 seconds, rinsed with running water for 20 seconds and the excess water removed by blotting with tissue paper, leaving the dentin visibly moist. Adhesive Prime & Bond 2.1 (PB) was applied using a saturated disposable brush, gently air dried with oil-free compressed air from a syringe for five seconds, keeping the tip 2 cm from the surface and light cured for 20 seconds.

All light-curing procedures used a 3M XL3000 (3M Dental Products) curing unit with a light intensity of 500 mW/cm^2. The light intensity was measured with a radiometer (Curing Radiometer, model 100, Demetron/Kerr, Danbury, CT, USA).

Group 1-control (PB). PB + Z 250.

For Group 2 (PB/PLF), following PB application, the adhesive surface was covered with a layer of Protect Liner F (PLF) (Kuraray Co Ltd, Japan) and light cured for 20 seconds.

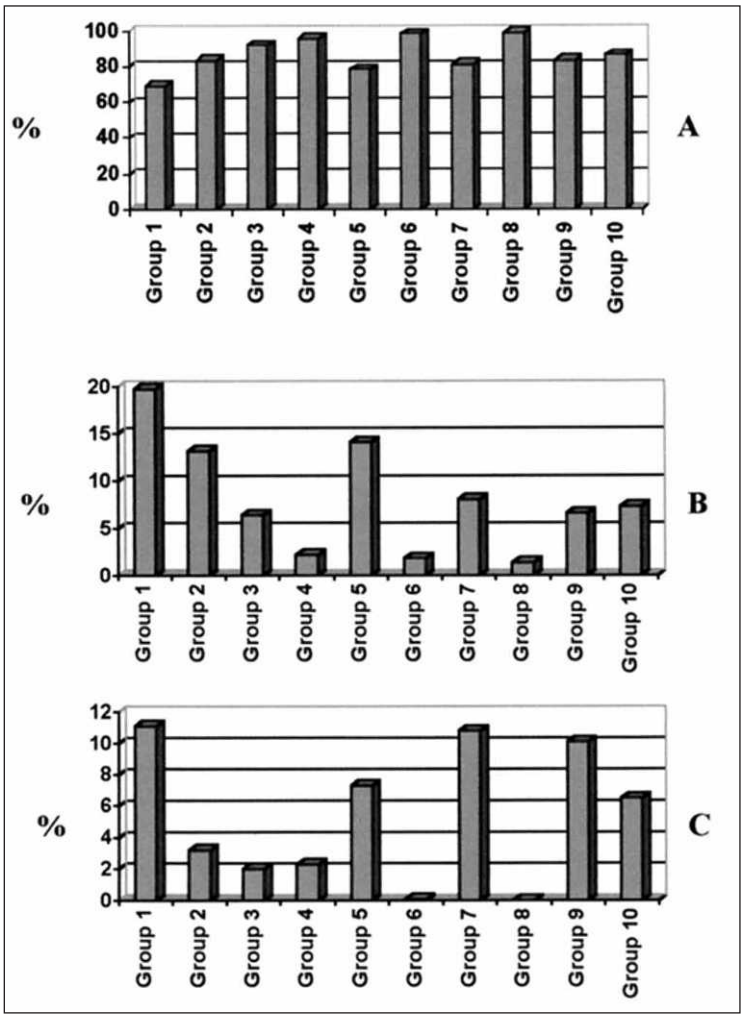


Figure 5. *Comparison of mean values of marginal quality among the groups: (A) perfect margin; (B) marginal irregularity; (C) marginal gap.*

For Group 3 (PB/SH), after acid conditioning as for Groups 1 and 2, a 10% sodium hypochlorite (SH) solution was lightly applied for one minute, then the teeth were rinsed with running water for 20 seconds and the excess water removed by blotting with tissue paper, leaving the dentin visibly moist.

For Group 4 (PB/SH/PLF), after acid conditioning, SH and adhesive application as for Group 3, a layer of PLF was applied as for Group 2.

For Groups 5 to 10, the adhesive system Clearfil SE Bond (CSEB) (Kuraray Co Ltd) was applied. The primer was applied with a saturated disposable brush for 20 seconds, dried with a mild air flow, followed by applying the adhesive resin, gentle air flow and light cured for 10 seconds.

Group 5-control (CSEB). CSEB + Z 250.

For Group 6 (CSEB/PLF), following CSEB application, a layer of PLF was applied.

For Group 7 (SH/CSEB), before applying CSEB, a SH solution was applied on the smear layer covered dentin surface as described for Groups 3 and 4.

For Group 8 (SH/CSEB/PLF), the same was done as for Group 7, and after applying CSEB, a layer of PLF was applied.

For Group 9 (CSEB/SH), the CSEB-primer was applied for 20 seconds and dried with mild air flow. The specimens were then immersed in a 50% acetone solution for 30 seconds to remove the primer monomers, rinsed with running water, dried with oil-free compressed air and a SH solution was applied as previously described. The CSEB-adhesive resin was applied and light cured for 10 seconds.

For Group 10 (CSEB/SH/PLF), the procedures were the same as for Group 9, except that after applying the CSEB-adhesive resin, a layer of PLF was applied.

The specimens were stored in distilled water at 37°C for 24 hours. The restorations were then finished with 1000-grit SiC under water and polished with 6, 3, 1 and 0.25 µm diamond paste using a polish cloth under water and ultrasonically cleaning for 10 minutes between the grits. The restorations were molded with a low-viscosity polyvinyl siloxane material (Aquasil, Dentsply DeTrey, Konstanz, Germany) and the molds were poured with epoxy resin (Buehler, Lake Buff, IL, USA), gold-sputter coated (Balzers–SCD 050 Sputter Coater, Liechtenstein) and observed by SEM (JEOL, JSM-5600LV, Scanning Electron Microscope, Japan) for evaluation, measurement and classification of the cavity margins. The measurements and classification were made with 300x magnification directly on the microscope monitor, using a multi-point measuring device, observing all the cavity perimeter in approximately 20 sections. The measurements were recorded and classified in steps of 50 to 150 µm (Figure 1), according to morphologically-defined parameters, into one of four types as described by Hannig, Reinhardt and Bott (2001): "perfect margin" defined as a continuous, gap-free transition between filling and dentin (Figure 2); "marginal irregularity," characterized as a non-continuous but gap-free transition between filling and dentin (Figure 3); "marginal gap," observed as a gap formation and loss of interfacial adhesion (Figure 4) and "overhang" that could not appear in more than 0.5% of the total perimeter sum. Marginal quality was calculated by adding each of the classified step measurements, and the total sum of each classification was expressed as a percentage of the cavity perimeter for each specimen.

The results of SEM-analysis were subjected to non-parametric multiple comparison Kruskal-Wallis test ($p<0.05$).

RESULTS

The Kruskal-Wallis test demonstrated significant differences among the groups. The results of the non-parametric multiple comparison test are summarized in Tables 2 to 4 and the mean values are represented in Figure 5. As the occurrence of "overhang" was considered a mistake in specimen preparation and could not exceed 0.5% of the restorations perimeters, the values obtained in this classification, when they appeared, were disregarded and, consequently, not considered for statistical analysis.

Group 8 (SH/CSEB/PLF) showed the highest average values (98.6%) and the lowest standard deviation (1.12%) for "perfect margin" occurrence, which were not statistically different in decreasing order from Group 6 (CSEB/PLF), 98.1% ± 2.03; Group 4 (PB/SH/PLF), 95.4% ± 5.14 and Group 3 (PB/HS), 91.7% ± 8.88. Two Groups from each adhesive system showed the highest average values. Three among these four Groups (8, 6 and 4) had the low-viscosity composite (PLF) as an intermediate layer, and SH (8, 4 and 3) as a surface pre-treatment agent. The lowest values for "perfect margin," with the highest standard deviations, were obtained for Control Group 1 (PB), 69.2% ± 16.02, which were not statistically different, in increasing order from Control Group 5 (CSEB), 78.6% ± 11.65; Group 7 (SH/CSEB), 81.1% ± 7.26; Group 9 (CSEB/SH), 83.3% ± 5.35; Group 2 (PB/PLF), 83.6% ± 15.47 and Group 10 (CSEB/SH/PLF), 86.1% ± 5.49. Thus, as observed above, Control Groups 1 and 5 presented the lowest values for "perfect margin." These results are shown in Table 2 and the mean values are represented in Figure 5A.

Table 3 and Figure 5B summarize the occurrence of "marginal irregularity" among the groups. The Control Groups 1 (PB) and 5 (CSEB) presented the highest values, 19.7% ± 8.53 and 14.1% ± 11.52, respectively, followed by Group 2 (PB/PLF), with 13.2% ± 12.39. The lowest values were obtained in increasing order by Group 8 (SH/CSEB/PLF), with 1.4% ± 1.12; Group 6 (CSEB/PLF), 1.8% ± 2.04 and Group 4 (PB/SH/PLF), 2.2% ± 2.47. For each separate adhesive system, the lowest values for "marginal irregularity" were present when the low-viscosity composite was used as an intermediate layer for CSEB and SH pre-treatment was used for PB.

Control Group 1 (PB) presented the highest values for "marginal gap," 11.1% ± 9.47, although no significant differences were noted for Group 7 (SH/CSEB), 10.8% ± 5.42; Group 9 (CSEB/SH), 10.1% ± 4.29; Control Group 5 (CSEB), 7.3% ± 5.53 and Group 10 (CSEB/SH/PLF), 6.5% ± 4.87. In Group 8 (SH/CSEB/PLF), "marginal gaps" were not observed, while for Group 6 (CSEB/PLF), they was present in 0.1% ± 0.26 of the restoration margin length. For Group 3 (PB/SH), Group 4 (PB/SH/PLF) and Group 2 (PB/PLF), the values for "marginal gaps" were 2.0% ± 3.26; 2.3% ± 3.8 and 3.2% ± 4.46, respectively. These results are summarized in Table 4 and are represented in Figure 5C. When compared to the control groups, a clear reduction in "marginal gap" occurrence could be noted when the low-viscosity composite PLF was used as the intermediate layer for both adhesive systems. The pre-treatment with SH significantly reduced the occurrence of "marginal gaps" for the PB Groups and did not influence occurrence in the CSEB Groups.

Table 2: *Occurrence of "Perfect Margin"*

Groups	Mean (%)	SD*
Group 1 (PB)	69.2	16.02 C
Group 2 (PB/PLF)	83.6	15.47 BC
Group 3 (PB/SH)	91.7	8.88 AB
Group 4 (PB/SH/PLF)	95.4	5.14 A
Group 5 (CSEB)	78.6	11.65 C
Group 6 (CSEB/PLF)	98.1	2.03 A
Group 7 (SH/CSEB)	81.1	7.26 C
Group 8 (SH/CSEB/PLF)	98.6	1.12 A
Group 9 (CSEB/SH)	83.3	5.35 C
Group 10 (CSEB/SH/PLF)	86.1	5.49 BC

**Same letters indicate no statistical difference (p<0.05).*

Table 3: *Occurrence of "Marginal Irregularity"*

Groups	Mean (%)	SD*
Group 1 (PB)	19.7	8.53 A
Group 2 (PB/PLF)	13.2	12.39 ABC
Group 3 (PB/SH)	6.4	7.14 CD
Group 4 (PB/SH/PLF)	2.2	2.47 D
Group 5 (CSEB)	14.1	11.52 AB
Group 6 (CSEB/PLF)	1.8	2.04 D
Group 7 (SH/CSEB)	8.1	5.36 BC
Group 8 (SH/CSEB/PLF)	1.4	1.12 D
Group 9 (CSEB/SH)	6.6	4.06 BC
Group 10 (CSEB/SH/PLF)	7.3	3.45 BC

**Same letters indicate no statistical difference (p<0.05).*

Table 4: *Occurrence of "Marginal Gap"*

Groups	Mean (%)	SD*
Group 1 (PB)	11.1	9.47 A
Group 2 (PB/PLF)	3.2	4.46 BCD
Group 3 (PB/SH)	2.0	3.26 DE
Group 4 (PB/SH/PLF)	2.3	3.80 CDE
Group 5 (CSEB)	7.3	5.53 AB
Group 6 (CSEB/PLF)	0.1	0.26 DE
Group 7 (SH/CSEB)	10.8	5.42 A
Group 8 (SH/CSEB/PLF)	0.0	0.00 E
Group 9 (CSEB/SH)	10.1	4.29 A
Group 10 (CSEB/SH/PLF)	6.5	4.87 ABC

**Same letters indicate no statistical difference (p<0.05).*

DISCUSSION

The two major difficulties incurred in achieving a reliable technique for composite restorations are the rigid polymerization shrinkage of the restorative composites that challenge the bonded interface and the dentin itself as a substrate for bonding.

Due to their inherent chemistry, current composites shrink upon polymerization, thus, introducing strain in the final restorations. The magnitude of the stress generated is dependent on the elastic modulus of the composite, that is, at a given shrinkage value, the most rigid material will cause the highest stress (Hooke's law). Unless the surrounding structures to which the composite is bonded supply enough elastic compliance, something will fracture in order to compensate for the reduced volume and, consequently, create gaps (Davidson & Feilzer, 1997). Thus, it has been suggested that if a low elastic modulus layer was interposed between the rigid shrinking composite and the adhesive layer, it would provide a decrease in this deleterious effect to the bonded walls, mainly in high C-factor cavities (Kemp-Scholte & Davidson, 1990a,b; Carvalho & others, 1996; Swift & others, 1996; Davidson & Feilzer, 1997; Rees, O'Dougherty & Pullin, 1999; Unterbrink & Liebenberg, 1999; Choi & others, 2000; Montes & others, 2001), such as the 4.3 C-factor cavities used in this study. The results of this study agree with the previous findings of Kemp-Scholte and Davidson (1990a,b) who reported an improvement in marginal quality and a reduction in polymerization contraction stresses of between 20% and 50% when an intermediate low modulus resin layer was used. Indeed, a tendency for better marginal quality was observed when PLF was applied, with a significant decrease in the occurrence of "marginal gaps" for both adhesive systems when compared to the control groups (Table 4 and Figure 5C). When these results are observed for each adhesive system separately, the significant effect on CSEB Groups, in which a remarkable improvement in marginal quality was obtained, is noticeable (Tables 2 to 4 and Figure 5). Swift and others (1996) previously reported similar results, that is, the bonding systems designed for use with low-viscosity intermediate resins, such as CSEB, generally had less microleakage than other systems, including PB, where the use of PLF had little effect on microleakage performance. PLF is a microfilled composite with 42% by weight of colloidal silica and pre-polymerized filler with a very low-viscosity that facilitates its application in thin layers. Moreover, its formulation indicates a possible and desirable elastic "buffer" behavior for use as an intermediate layer (Condon & Ferracane, 2000), thereby, reinforcing the "elastic cavity wall concept" (Montes & others, 2001).

The two adhesive systems used in this study represent the two currently acceptable ways to obtain micromechanical retention between resin and dentin. PB is a single bottle adhesive system with acetone as volatile solvent that demands a previous acid-etching procedure. Its action is based upon complete removal of the smear layer and demineralization of subsurface intact dentin, leaving a collagen rich, moist surface where adhesive resin must diffuse to form the hybrid layer. The major problem with this technique is its sensitivity due to its moisture dependence, mainly to acetone-based systems such as PB (Tay, Gwinnett & Wei, 1996). In fact, the high SD for "perfect margin" occurrence observed for Groups 1 and 2, 16.02 and 15.47, respectively (Table 2), directs attention to the high variability of these results, which demonstrates the difficulty of accomplishing this procedure successfully. CSEB uses a self-etching primer that partially demineralizes the smear layer and the underlying intact dentin, using them as bonding substrate (Pashley & Carvalho, 1997; Van Meerbeek & others, 1998). When the two Control Groups 1 (PB) and 5 (CSEB) were compared for marginal quality, no significant difference was observed at the level of significance by the multi comparison non-parametrical test ($p<0.05$). The results indicate that under the conditions of this study, the differences between these two bonding approaches were not significant when traditionally applied, that is, neither the thicker hybrid layer of PB nor the more uniform, thinner hybrid layer of CSEB appeared to have influenced the results. However, a slight tendency for a better performance could be noticed for CSEB. Indeed, although the hybrid layer has a low elastic modulus (Van Meerbeek & others, 1993), its thinness must have compromised the performance as a polymerization stress "breaker" in this high C-factor cavity condition, with the high elastic modulus composite Z 250 placed in bulk and a continuous, intense light-curing ($500 mW/cm^2$) for 30 seconds, which is an extremely unfavorable situation for the bonding procedures (Feilzer & others, 1987; Obici & others, 2002).

The presence of the collagen network resulting from acid-etching has been constantly questioned as an important structure for dentin bonding since it has been shown that resin monomers do not fully diffuse through it to reach intact dentin. This incomplete penetration produces a porous layer of exposed collagen subject to hydrolysis and degradation, resulting in microleakage and failure over time (Sano & others, 1994). The removal of collagen with SH has been suggested as a suitable method to overcome this problem since it alters the composition of the dentin surface as it becomes similar to etched enamel, that is, a more predictable and hydrophilic substrate for bonding (Sakae & others, 1988; Tanaka & Nakai, 1993; Inaba & others, 1995). When SH was applied after acid-etching for PB Groups 3 and 4, an improvement in marginal quality was evident and statistically significant (Tables 2 to 4

and Figure 5). The decrease in the SD of "perfect margin" occurrence also showed the consistency of these data (Table 2), indicating that the results became more constant. Some morphological studies focusing on the resultant dentin surface after collagen depletion with SH described a rough, porous and eroded surface that appeared more compatible with bonding resins than the collagen-rich surface produced by acid-etching (Inai & others, 1998; Perdigão & others, 1999). Indeed, after collagen depletion with SH treatment, an increase in wettability may be expected due to deproteinization, resulting in a hydrophilic surface, since hydroxyapatite is a high-energy substrate, while collagen has a low-energy surface. Thus, the interactions between adhesive resin and this rough mineral tissue are more likely to occur (Attal, Asmussen & Degrange, 1994). These results for PB agree with previous findings that suggest that collagen depletion enhanced the bond strength for acetone-based adhesive systems (Inai & others, 1998; Pioch & others, 1999; Prati & others, 1999; Saboia & others, 2000). These improved results have been attributed to the higher diffusibility and the higher capacity to displace acetone when compared with other solvents, such as ethanol or water (Jacobsen & Soderhold, 1995). This ability might have favored the diffusion of PB through this mineral-rich tissue and high-energy substrate, thereby, enhancing marginal quality. Furthermore, Inai and others (1998) and Prati and others (1999) suggested that the adhesive monomers in PB are acidic and may re-etch the mineral dentin surface. This could create a very thin hybrid layer (0.3 to 0.5 μm) not detected by SEM, which may ensure higher bond strengths.

For the CSEB Groups 7 and 8, the use of SH must be analyzed from another viewpoint since these systems do not require a previous acid-etching procedure and, therefore, do not expose the unprotected collagen network as PB. When SH was applied on the smear layer-covered dentin, no significant difference was noticed when these Groups were compared to Groups 5 and 6 regarding neither mean values nor SD (Tables 2 to 4). The composition of the smear layer is generally similar to the originating tissue, therefore, it is assumed that its mineral and collagen phases were similar in content to normal dentin where the cavities were performed, that is, about 50% vol mineral and 30% vol collagen (Pashley & others, 1993). Thus, the pre-treatment of the smear layer with SH may have eliminated this collagen phase; however, CSEB utilized an acidic primer solution (MDP-based) that can permeate through the water-filled channels between the particles of the smear layer, enlarging them and, thereby, reaching intact dentin (Harada & others, 2000; Tay & Pashley, 2001). Thus, this collagen removal influenced marginal quality since it did not seem to represent an obstacle to CSEB-primer action. Indeed, SEM studies show no significant morphological changes when SH is applied on smear layers, suggesting only superficial collagen removal (Tanaka & Nakai, 1993; Prati & others, 1999); however, a light reduction in smear layers thickness was observed after one minute of scrubbing action during SH application and, although this was attributed to a higher thickness of the smear layers negative influence on bond strength of self-etching primers to dentin (Koibuchi, Yasuda & Nakabayashi, 2001), this light reduction, if it occurred in this study, did not influence marginal quality.

For Groups 9 and 10, the thin, collagen-rich surface exposed by the action of CSEB acidic primer was depleted by SH, then, the adhesive resin was placed and polymerized. The results of these procedures indicated a significant decrease in marginal quality when compared to Groups 6 and 8 when PLF was used as an intermediate layer. However, data were not statistically different from Control Group 5 and Group 7 (Tables 2 to 4). According to Tay and Pashley (2001), CSEB-primer was not strong enough to dissolve the smear layer, but etched through it to demineralize the subsurface intact dentin to a depth of 0.5 μm. Indeed, Harada and others (2000) had previously described a surface after CSEB-primer action where this mild action was observed. Theoretically, a drastic decrease would be expected in marginal quality since the CSEB-primer had been removed and the bonding was merely based upon the interaction between the CSEB-adhesive resin and a more mineral dentin surface. One possible explanation for these results may rely on the composition of the CSEB-adhesive resin itself (Table 1), which also contains the acidic monomer MDP in its composition and could have re-etched the dentin surface, creating a shallow hybrid layer. The higher viscosity of the CSEB-adhesive resin, when compared to CSEB-primer, may be attributed to the presence of the high molecular weight monomer BIS-GMA, the colloidal silica filler and the lack of ethanol and water as solvents and must also have influenced its wettability on dentin surface. Nevertheless, the previous primer action, followed by SH collagen depletion, must have increased the surface energy of dentin, which somehow could have partially compensated the CSEB-adhesive resin interaction for this decreased wettability and, therefore, positively influenced marginal quality.

CONCLUSIONS

Based on the results of this study, both the adhesive systems, Prime & Bond 2.1 (PB) and Clearfil SE Bond (CSEB), presented the lowest occurrence of "perfect margin" and a high occurrence of "marginal gaps" in control groups. The high standard deviations observed for these control groups also demonstrated the low reliability of these procedures under test conditions. The use of the low-viscosity resin, Protect Liner F (PLF), remarkably improved the marginal quality for the CSEB Groups and diminished the standard deviations,

which appeared to make the procedure more reliable. PLF also had a slight, but not statistically significant influence on marginal quality for the PB Groups. Collagen depletion with SH improved the marginal quality for the PB Groups and reduced standard deviations, although it did not influence the results for the CSEB Groups. Thus, the hybrid layer appeared to be not the only paradigm for immediate or short-term bonding to dentin with the products tested.

(Received 18 June 2002)

References

Armstrong SR, Boyer DB, Keller JC & Park JB (1998) Effect of hybrid layer on fracture toughness of adhesively bonded dentin-resin composite joint *Dental Materials* **14(2)** 91-98.

Attal JP, Asmussen E & Degrange M (1994) Effects of surface treatment on the free surface energy of dentin *Dental Materials* **10(4)** 259-264.

Bayne SC, Thompson JY, Swift EJ Jr, Stamatiades P & Wilkerson M (1998) A characterization of first-generation flowable composites *Journal of the American Dental Association* **129(5)** 567-577.

Braem M, Van Doren VE, Lambrechts P & Vanherle G (1987) Determination of Young's modulus of dental composites: A phenomenological model *Journal of Materials Science* **22** 2037-2042.

Carvalho RM, Pereira JC, Yoshiyama M & Pashley DH (1996) A review of polymerization contraction: The influence of stress development versus stress relief *Operative Dentistry* **21(1)** 17-24.

Choi KK, Condon JR & Ferracane JL (2000) The effects of adhesive thickness on polymerization contraction stress of composite *Journal of Dental Research* **79(3)** 812-817.

Condon JR & Ferracane JL (1998) Reduction of composite contraction stress through non-bonded microfiller particles *Dental Materials* **14(4)** 256-260.

Condon JR & Ferracane JL (2000) Assessing the effect of composite formulation on polymerization stress *Journal of the American Dental Association* **131(4)** 497-503.

Davidson CL & de Gee AJ (1984) Relaxation of polymerization contraction stress by flow in dental composites *Journal of Dental Research* **63(2)** 146-148.

Davidson CL, de Gee AJ & Feilzer A (1984) The competition between the composite-dentin bond strength and the polymerization contraction stress *Journal of Dental Research* **63(12)** 1396-1399.

Davidson CL & Feilzer AJ (1997) Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives *Journal of Dentistry* **25(6)** 435-440.

Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.

Ferracane JL & Berge HX (1995) Fracture toughness of experimental dental composites aged in ethanol *Journal of Dental Research* **74(7)** 1418-1423.

Finger WJ, Inoue M & Asmussen E (1994) Effect of wettability of adhesive resins on bonding to dentin *American Journal of Dentistry* **7(1)** 35-38.

Gwinnett AJ (1994) Altered tissue contribution to interfacial bond strength with acid conditioned dentin *American Journal of Dentistry* **7(5)** 243-246.

Gwinnett AJ, Tay FR, Pang KM & Wei SH (1996) Quantitative contribution of the collagen network in dentin hybridization *American Journal of Dentistry* **9(4)** 140-144.

Hannig M, Reinhardt K-J & Bott B (2001) Composite-to-dentin bond strength, micromorphology of the bonded dentin interface and marginal adaptation of Class II composite resin restorations using self-etching primers *Operative Dentistry* **26(2)** 157-165.

Harada N, Nakajima M, Pereira PNR, Yamaguchi S, Ogata M & Tagami J (2000) Tensile bond strength of a newly developed one-bottle self-etching resin bonding system to various dental substrates *Dentistry in Japan* **36(1)** 47-53.

Inaba D, Duschner H, Jongebloed W, Odelius H, Takagi O & Arends J (1995) The effects of a sodium hypochlorite treatment on demineralized root dentin *European Journal of Oral Sciences* **103(6)** 368-374.

Inai N, Kanemura N, Tagami J, Watanabe LG, Marshall SJ & Marshall GW (1998) Adhesion between collagen depleted dentin and dentin adhesives *American Journal of Dentistry* **11(3)** 123-127.

Jacobsen T & Soderhold KJ (1995) Some effects of water on dentin bonding *Dental Materials* **11(2)** 132-136.

Kemp-Scholte CM & Davidson CL (1990a) Complete marginal seal of Class V resin composite restorations effected by increased flexibility *Journal of Dental Research* **69(6)** 1240-1243.

Kemp-Scholte CM & Davidson CL (1990b) Marginal integrity related to bond strength and strain capacity of composite resin restorative systems *The Journal of Prosthetic Dentistry* **64(6)** 658-664.

Koibuchi H, Yasuda N & Nakabayashi N (2001) Bonding to dentin with a self-etching primer: The effect of smear layers *Dental Materials* **17(2)** 122-126.

Labella R, Lambrechts P, Van Meerbeek B & Vanherle G (1999) Polymerization shrinkage and elasticity of flowable composites and filled adhesives *Dental Materials* **15(2)** 128-137.

Montes MA, de Goes MF, da Cunha MR & Soares AB (2001) A morphological and tensile bond strength evaluation of an unfilled adhesive with low-viscosity composites and a filled adhesive in one and two coats *Journal of Dentistry* **29(6)** 435-441.

Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials Research* **16(3)** 265-273.

Nakajima M, Sano H, Burrow MF, Tagami J, Yoshiyama M, Ebisu S, Ciucchi B, Russell CM & Pashley DH (1995) Tensile bond strength and SEM evaluation of caries-affected dentin using dentin adhesives *Journal of Dental Research* **74(10)** 1679-1688.

Obici AC, Sinhoreti MA, de Goes MF, Consani S & Sobrinho LC (2002) Effect of photo-activation method upon polymerization shrinkage of restorative composites *Operative Dentistry* **27(2)** 192-198.

Pashley DH & Carvalho RM (1997) Dentine permeability and dentine adhesion *Journal of Dentistry* **25(5)** 355-372.

Pashley DH, Ciucchi B, Sano H & Horner JA (1993) Permeability of dentin to adhesive agents *Quintessence International* **24(9)** 618-631.

Perdigão J, Thompson JY, Toledano M & Osorio R (1999) An ultra-morphological characterization of collagen-depleted etched dentin *American Journal of Dentistry* **12(5)** 250-255.

Pioch T, Kobaslija S, Schagen B & Götz H (1999) Interfacial micromorphology and tensile bond strength of dentin bonding systems after NaOCl treatment *The Journal of Adhesive Dentistry* **1(2)** 135-142.

Prati C, Chersoni S & Pashley DH (1999) Effect of removal of surface collagen fibrils on resin-dentin bonding *Dental Materials* **15(5)** 323-331.

Prati C, Pashley DH, Chersoni S & Mongiorgi R (2000) Marginal hybrid layer in Class V restorations *Operative Dentistry* **25(3)** 228-233.

Rees JS, O'Dougherty D & Pullin R (1999) The stress reducing capacity of unfilled resin in a Class V cavity *Journal of Oral Rehabilitation* **26(5)** 422-427.

Saboia VA, Rodrigues AL & Pimenta LA (2000) Effect of collagen removal on shear bond strength of two single-bottle adhesive systems *Operative Dentistry* **25(5)** 395-400.

Sakae T, Mishima H & Kozawa Y (1988) Changes in bovine dentin mineral with sodium hypochlorite treatment *Journal of Dental Research* **67(9)** 1229-1234.

Sano H, Shono T, Takatsu T & Hosoda H (1994) Microporous dentin zone beneath resin-impregnated layer *Operative Dentistry* **19(1)** 59-64.

Sano H, Takatsu T, Ciucchi B, Horner JA, Matthews WG & Pashley DH (1995) Nanoleakage: Leakage within the hybrid layer *Operative Dentistry* **20(1)** 18-25.

Swift EJ Jr, Triollo PT Jr, Barkmeier WW, Bird JL & Bounds SJ (1996) Effect of low-viscosity resins on the performance of dental adhesives *American Journal of Dentistry* **9(3)** 100-104.

Tanaka J & Nakai H (1993) Application of root canal cleaning agents having dissolving abilities of collagen to the surface treatment for enhanced bonding of resin to dentin *Dental Materials Journal* **12(2)** 196-208.

Tay FR, Gwinnett AJ & Wei SH (1996) The overwet phenomenon: A transmission electron microscopic study of surface moisture in the acid-conditioned, resin-dentin interface *American Journal of Dentistry* **9(4)** 161-166.

Tay FR & Pashley DH (2001) Aggressiveness of contemporary self-etching systems I: Depth of penetration beyond dentin smear layers *Dental Materials* **17(4)** 296-308.

Uno S & Finger WJ (1995) Function of the hybrid zone as a stress absorbing layer in resin-dentin bonding *Quintessence International* **26(10)** 733-738.

Unterbrink GL & Liebenberg WH (1999) Flowable resins composites as "filled adhesives": Literature review and clinical recommendations *Quintessence International* **30(4)** 249-257.

Van Meerbeek B, Perdigão J, Lambrechts P & Vanherle G (1998) The clinical performance of adhesives *Journal of Dentistry* **26(1)** 1-20.

Van Meerbeek B, Willems G, Celis JP, Roos JR, Braem M, Lambrechts P & Vanherle G (1993) Assessment by nano-indentation of the hardness and elasticity of the resin-dentin bonding area *Journal of Dental Research* **72(10)** 1434-1442.

Vargas MA, Cobb DS & Armstrong SR (1997) Resin-dentin shear bond strength and interfacial ultrastructure with and without a hybrid layer *Operative Dentistry* **22(4)** 159-166.

Vargas MA, Cobb DS & Denehy GE (1997) Interfacial micromorphology and shear bond strength of single-bottle primer/adhesives *Dental Materials* **13(5)** 316-324.

Watanabe I, Nakabayashi N & Pashley DH (1994) Bonding to ground dentin by a phenyl-P self-etching primer *Journal of Dental Research* **73(6)** 1212-1220.

Yoshiyama M, Sano H, Ebisu S, Tagami J, Ciucchi B, Carvalho RM, Johnson MH & Pashley DH (1996) Regional strengths of bonding agents to cervical sclerotic root dentin *Journal of Dental Research* **75(6)** 1404-1413.

©Operative Dentistry, 2003, **28-4,** 388-394

Influence of the Substrate and Load Application Method on the Shear Bond Strength of Two Adhesive Systems

PEC Cardoso • MA Meloncini • E Placido
JDO Lima • AU Tavares

Clinical Relevance

Because the results of this study found that substrate has a significant effect on bonding, clinicians should be aware that clinical performance of a dentin bonding product may vary from tooth to tooth, regardless of the specific bonding product being used.

SUMMARY

The difficulty with comparing data obtained from different research centers calls for the standardization of laboratory procedures. This *in vitro* study compared the shear bond strength (SBS) of two adhesive systems—a self-etching system, Etch&Prime 3.0 and a one-bottle total-etch system, Single Bond—using two methods of load application (orthodontic edge wire loop and knife-edge blade). The hypothesis of substrate influence on the results obtained for both tests was also investigated. Twenty-four recently extracted human teeth were embedded in PVC tubes using acrylic resin and divided into two groups (n=12). A proximal surface of each tooth was wet-ground flat to expose dentin. Etch&Prime 3.0 and Single Bond adhesive systems were applied according to the manufacturers' instructions, and cone-shaped restorations were then built using Z100 resin composite. After storage in distilled water at 37°C for 24 hours, the specimens were submitted to SBS testing using an orthodontic edge wire loop. The same teeth were again embedded in PVC tubes using acrylic resin and the other proximal surface was wet-ground flat to expose dentin. The specimens were prepared as explained above, and after storage in distilled water under the same previous conditions, they were submitted to SBS testing using a knife-edge blade. Two-way ANOVA and Tukey's tests were performed to determine any statistically significant differences among testing conditions. Results indicated that SBS values obtained for Etch&Prime 3.0 were significantly lower than Single Bond for both methods

*Paulo Eduardo Capel Cardoso, assistant professor, Department of Dental Materials, School of Dentistry, University of São Paulo, São Paulo, Brazil

Marco Antônio Meloncini, professor, Department of Dental Materials, Unip/Sorocoba—Paulista University of Sorocaba, São Paulo, Brazil

Eliane Placido, PhD student, Department of Dental Materials, School of Denistry, University of São Paulo, São Paulo, Brazil

Janaína De Oliveira Lima, dental practitioner, São Paulo, Brazil

Andréa Urbano Tavares, PhD student, Department of Dental Materials, School of Dentistry, University of São Paulo, São Paulo, Brazil

*Reprint request: Av Prof Lineu Prestes, 2227 Cidade Universitaria "Armando de Salles Oliveira," São Paulo, Brazil 05508-900; e-mail: paulocapel@uol.com.br

of load application ($p<0.001$). Regarding the load application method, statistically significant higher values ($p<0.01$) were obtained for Etch&Prime 3.0 when the knife-edge blade was used, whereas, no significant difference was observed between the two methods for Single Bond. There was a significant correlation between the bond strength values obtained on the same tooth ($p<0.05$). SEM examination found that Etch&Prime 3.0 demonstrated narrower tags than Single Bond. Moreover, the peritubular dentin was not adequately decalcified when Etch&Prime 3.0 was used.

INTRODUCTION

Adhesive materials have evolved, with new materials being introduced to the dental market at a rapid pace (Haller, 2000). This fact makes it impossible to obtain data on their performance by means of long-lasting clinical trials and, hence, laboratory investigations play an important role in providing information in a short period of time at low cost (Cardoso & others, 1999).

Traditional adhesive systems have involved substrate etching, applying a primer and applying a bonding resin (Burke & McCaughey, 1995). Modern adhesive systems are characterized by the search for a simplified mode of clinical application and their effects on the smear layer (Van Meerbeek & others, 1998). Multi-step bonding systems were followed by one-bottle bonding systems, in which the functions of primer and adhesive were combined. They were reported to produce similar results to those of multi-step products (Castelnuovo, Tjan & Liu, 1996; Tjan, Castelnuovo & Liu, 1996; Wilder & others, 1998; Yap, Ho & Wong, 1998; Swift & Bayne, 1997). In these systems, the smear layer can be either completely removed or modified. More recently, even the acid etching step was incorporated into the self-etching primer and conditioner/primer/adhesive systems (Haller, 2000). In the latter system, two components are mixed, which result in the formation of an acidic monomer that superficially etches dentin. The smear layer is dissolved and incorporated during hybrid layer formation, with the additional advantage of a less sensitive application technique (Koibuchi, Yasuda & Nakabayashi, 2001).

Although data obtained *in vitro* vary considerably, they can be clinically relevant (Øilo & Austrheim, 1993). Because of the significance of these differences, standardized tests, such as tensile (Nakabayashi & Saimi, 1996) and shear bond strength tests (Toledano & others, 2001), microleakage quantification and microscopic analysis of marginal gap formation on the restoration/dental substrate interface were established to evaluate new restorative materials, as can be found in the regulations of International Organization for Standardization (1993) ISO TR 11405 Dental Materials. Other assessment methodologies have also been proposed, including microtensile tests (Sano & others, 1994; Pashley & others, 1999), SEM and TEM analysis of the bonding interface (Perdigão & others, 1994; Kwong & others, 2000; Prati & others, 2000), wetta- bility and surface tension, chemical resistance and polymerization efficacy (Eliades, 1994). These methodologies complement existing data and provide additional information regarding the performance of a material. Moreover, more reliable assessments are obtained after a series of different, associated tests (for example, shear x SEM micromorphologic analysis), thus, providing more reliable results (Prati & others, 1998).

Different methods or modifications of the same test are also commonly found in the literature. Among the different bond strength tests, the shear bond strength test (SBS) is the most commonly used by different research centers (Inai & others, 1998; Nara & others, 1998; Pashley & others, 1993; Watanabe & others, 1998). There are many possibilities for applying this methodology, and several variations are commonly seen on the primary test (Dunn & Söderholm, 2001; Swift & Bayne, 1997).

Different values for the same evaluated material are found even when the same testing methodology is used. By varying the load application method in an SBS test, Sinhoreti (1997) demonstrated that significantly different results for a dental material are achieved under the same conditions. Cardoso, Braga and Carrilho (1998) compared the mean bond strength values obtained using microtensile, shear and tensile tests that use three adhesives systems. They concluded that the microtensile strength results were much higher than the tensile and SBS tests. On the other hand, Øilo and Austrheim (1993) demonstrated similar values on tensile and SBS tests using four adhesive systems. They pointed out that both tests were equally adequate in providing information on the bonding efficacy. Moreover, these authors also verified that the substrate was the main reason for the widely variable results observed in the literature since it cannot be standardized. Substrate, therefore, plays a relevant role in bond strength tests of adhesive systems.

This *in vitro* study evaluated the SBS of two adhesive systems (Etch&Prime 3.0 and Single Bond) under two load-application methods, orthodontic edge wire loop and knife-edge blade, and verified the hypothesis of substrate influence on the results, since both tests were performed on a same tooth. The morphologic aspects of the resin-dentin infiltrated zones were also analyzed using Scanning Electron Microscopy.

METHODS AND MATERIALS

Shear Bond Strength Testing

Twenty-four recently extracted, sound human molars stored in distilled water at 37°C were cleaned of debris with curettes and flour of pumice using a rubber cup in a slow-speed handpiece. They were mounted in PVC tubes using acrylic resin. A proximal surface was randomly chosen and ground flat in a polishing machine using 220-grit sandpaper under constant refrigeration (Struers A/S, DK-2610 Rodovre, Denmark) in order to obtain a dentin surface with approximately a 6-mm diameter. The molars were polished consecutively using 600-grit sandpaper for one minute in the same polishing machine to provide a standardized smear layer formation (Koibuchi & others, 2001).

Specimens were randomly assigned to two groups (n=12) according to the adhesive system—Etch&Prime 3.0 (Degussa, Hüls, AG, Germany) and Single Bond (3M ESPE, St Paul, MN, USA). Table 1 presents the composition of each adhesive system. For Single Bond, adhesive system surfaces were etched using 37.5% phosphoric acid (3M ESPE) for 15 seconds. The tooth was thoroughly rinsed and excessive water was removed from the dentin surface with an air blow, so that the surfaces remained visibly moist. Adhesive was applied with a disposable brush (KG Brush Sorensen, Alphaville, Barueri, São Paulo 06465-130, Brazil), gently air-dried and light-cured for 10 seconds using an Optilux 500

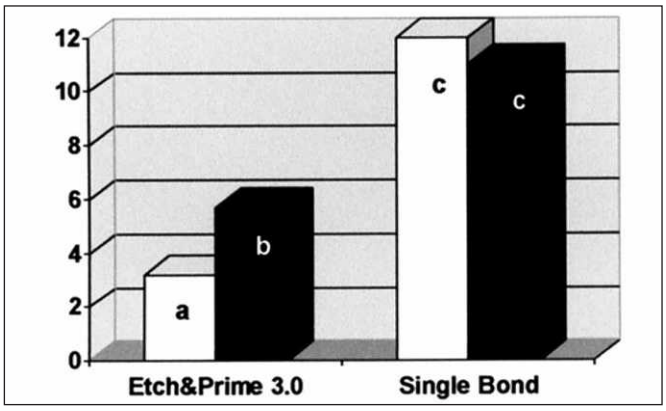


Figure 1. Mean shear bond strength values using orthodontic edge wire loop and knife-edge blade (MPa). Different letters indicate statistically significant differences between values (Tukey 5%).

Table 1: *Adhesive Systems Tested in This Study*

Adhesive System	Composition	Manufacturer
Etch&Prime 3.0 (EP)	Primer A. Tetra-methacryloxyethylpyrophosphate, 2-hydroxyethylmethacrylate, initiators, stabilizers Primer B. 2-hydroxyethylmethacrylate, ethanol, distilled water, stabilizers	Degussa Geschäftsbereich Dental, Hüls, AG, Germany
Single Bond (SB)	2-hydroxyethylmethacrylate, BIS-GMA, dimethacrylates, amines, methacrylate functional copolymer of polymer of polyacrylic and polyitaconic acids, ethanol, water	3M ESPE, St Paul, MN, USA

Table 2: *Individual Shear Bond Strength Values Obtained Using the Orthodontic Edge Wire Loop and Knife-Edge Blade Methods for Each Adhesive System (MPa)*

Adhesive System					
Etch&Prime 3.0			3M Single Bond		
Specimen	Orthodontic Edge Wire Loop	Knife-Edge Blade	Specimen	Orthodontic Edge Wire Loop	Knife-Edge Blade
1	5.6	9.8	1	18.3	12.1
2	3.4	6.9	2	5.9	6.1
3	4.0	3.6	3	18.3	14.9
4	1.0	3.4	4	4.6	7.7
5	4.7	6.4	5	15.8	9.4
6	1.8	5.5	6	26.0	15.0
7	2.5	4.4	7	3.9	10.1
8	3.0	4.4	8	11.3	17.4
9	1.0	4.4	9	10.5	10.5
10	5.4	4.9	10	8.1	11.8
11	0.8	4.9	11	11.7	11.3
12	5.7	9.4	12	9.1	5.7

Table 3: *Mean Shear Bond Strength Values (MPa), Standard Deviation (SD) and Coefficient of Variation (CV) (in percentage) for Etch&Prime 3.0 and Single Bond Adhesive Systems*

Loading Method	Adhesive System					
	Etch&Prime 3.0			Single Bond		
	Mean	SD	CV (%)	Mean	SD	CV (%)
Orthodontic Edge Wire Loop	3.2	1.9	58.0	12.0	6.6	54.8K
Knife-Edge Blade	5.7	2.1	37.0	11.0	3.6	32.6

light-curing unit (Demetron/Kerr Corp, Orange City, CA, USA). The light intensity was verified prior to performing the above procedures in order to maintain readings in the range of 600 mW/cm^2. Acid etching was not performed prior to applying Etch&Prime 3.0. The two components were mixed in an appropriate dish and applied on the dentin surface with a disposable brush. After 30 seconds the excessive material was removed with a slight blow of air and light cured for 10 seconds (Optilux 500 light-curing unit—Demetron/Kerr Corp). A second layer was applied and the above mentioned steps repeated.

A specially designed silicone mold with a cone-shaped perforation 3 mm in diameter and 3 mm in height was immediately fixed onto each treated dentin surface using cyanoacrylate glue (Loctite, São Paulo, Brazil). This mold was filled with Z100 resin composite (shade A2—3M ESPE) in three increments, and each was light-cured for 40 seconds (Optilux 500 light-curing unit—Demetron/Kerr). Care was taken to ensure that each cone was bonded to the dentin surfaces at the same angle. The specimens were stored in distilled water at 37°C for 24 hours, then the shear bond strength was determined using an orthodontic edge wire (0.5 x 0.5 mm) in a Universal Testing Machine (Instron Universal Testing Instrument, Model 1125, Canton, MA, USA) at a load rate of 0.5 mm/minute. The wire was placed as a loop around the composite, cone-shaped restoration as close as possible to the dentin surface during testing.

The same teeth used in the previous test were again mounted in PVC tubes using acrylic resin and another intact proximal surface was chosen for preparation. Previous labeling on each tooth was maintained. A flat dentin surface approximately 6 mm in diameter was wet-ground and polished as previously described. Adhesive systems were applied according to the manufacturers' recommendations, and cone-shaped restorations were built using Z100 resin composite following the same procedures as the other method. After storage in water for 24 hours at 37°C, the specimens were tested in shear using a knife-edge blade mounted in a Universal Testing Machine (Otto-Wolpert-Werke, Germany) at a crosshead speed of 0.5 mm/minute. Fractured surfaces were examined using a stereomicroscope (Bausch & Lomb, Bern, Switzerland) under 10x magnification to verify any evidence of dentin pullout. Data were analyzed using two-way ANOVA and Tukey's test.

Scanning Electron Microscopy

One recently extracted human molar was selected for SEM investigation. Two Class V cavities (5 mm in length x 3 mm wide x 2 mm deep) were cut into this same tooth using two proximal surfaces chosen at random in order to observe the dentin interface ultramorphologic aspects. After prophylaxis using pumice paste and rubber cup in a slow-speed handpiece, adhesive systems were applied according to manufacturers' instructions as presented above, and the cavities were restored using Z100 resin composite placed in two increments approximately 1 mm each, and light cured for 40 seconds (Optilux 500 light-curing unit, Demetron/Kerr Corp). The teeth were stored in distilled water at 37°C for 24 hours.

The tooth was then completely decalcified in 37% hydrochloric acid for 36 hours, so that the resin tags formed at the composite-adhesive-dentin interfaces could be examined. The restorations were then mounted on aluminum stubs using colloidal silver and sputter-coated with gold. Scanning electron micrographs were obtained with 3000x magnification using a Stereoscan 40 scanning electron microscope (LEO, England, 20.00 KV).

RESULTS

Table 2 presents the individual SBS values obtained for each adhesive system and mean values obtained for each experimental group are shown in Table 3 and Figure 1. According to statistical analysis using two-way ANOVA and Tukey's test, a statistically significant difference was observed between the two adhesive systems ($p<0.001$), so that higher mean SBS values were obtained using Single Bond when compared to Etch&Prime 3.0.

Regarding the two-load application methods, Etch&Prime 3.0 showed statistically significant higher values for the knife-edge blade method than the orthodontic edge wire loop ($p<0.01$). No statistically significant difference between the two load application methods was demonstrated for Single Bond. Although a difference was found between the two methods for Etch&Prime 3.0, the quantitative disproportion between the magnitudes of adhesion for each dentin bonding agent was maintained.

The substrate also had a significant influence on the results obtained in this study. This finding was confirmed by Spearman's correlation test, which showed a significant correlation ($p<0.05$) between the results obtained in the same tooth for the two different tests.

The scanning electron micrographs shown in Figures 2 and 3 represent the resin composite tags that penetrated dentin. Both systems dissolved the smear layer, thus, opening tubules and forming a hybrid layer. Resin tags obtained for Etch&Prime 3.0 were narrower than the other system and peritubular dentin appeared to be incompletely decalcified. On the other hand, Single Bond promoted longer, more consistent tag formation with greater decalcification of peritubular dentin by the conditioner solution and impregnation with resin. These results reflect the shear bond strength values obtained for the test groups.

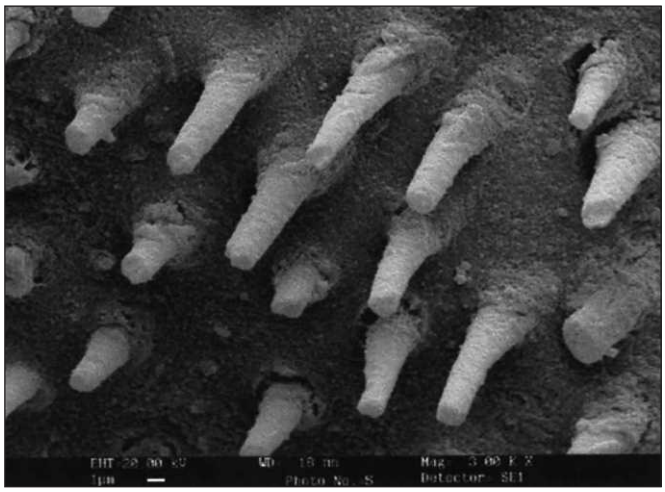


Figure 2. *Scanning electron micrograph (3000x) showing resin tags when Etch & Prime 3.0 was applied to dentin.*

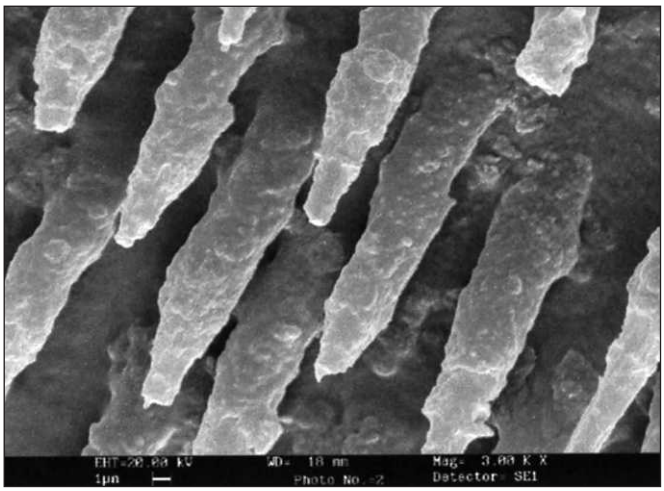


Figure 3. *Scanning electron micrograph (3000x) showing resin tags when Single Bond was applied to dentin.*

DISCUSSION

Although there is standardization in the SBS test according to ISO TR 11405 (1993) guidelines, it can be performed using varied methodologies, which can possibly lead to different results, as demonstrated by Sinhoreti (1997).

Studies performed by Van Noort and others (1989) and Della Bona and Van Noort (1995) demonstrated by means of finite elements that when the cone-shaped resin block was bonded to the dentin surface, it was subjected to a certain load and stress distribution was not uniform at the adhesion interface. Moreover, minor details in specimen geometry, shape and size, and the elastic modulus of some materials involved could present a significant variability in the results within a same test. This also explains the vast differences among values obtained by different authors for SBS tests using the same commercial product (Nara & others, 1998; Inai & others, 1998; Watanabe & others, 1998; Cardoso & others, 1998).

These particulars add to the difficulty in standardizing the dentinal substrate, thus, generating high average coefficients of variation. In this study, discrepant values ranging from 3.9 to 26.0 MPa for Single Bond and 0.8 to 5.7 MPa for Etch&Prime 3.0 were observed with the SBS methodology using orthodontic edge wire loop, so that the average coefficients of variation were higher than 50% for the latter. In the knife-edge blade methodology, the average coefficient of variation was below 40%, which was similar to that described in the guidelines of ISO-TR 11405 (1993).

Furthermore, in this study, the knife-edge blade load application method had significantly higher values for the Etch&Prime 3.0 adhesive system when compared to the orthodontic edge wire loop methodology. However, for the Single Bond adhesive system, the difference among mean values was not statistically significant. These observations do not agree with the results by Sinhoreti (1997), which showed significantly higher mean values for the orthodontic edge wire loop when compared to the knife-edge blade-load application method. As for the loading methods, there are still some doubts related to the significance of these differences and the repercussion of these results.

By analyzing the results of this study, it is possible to determine that both methodologies did not alter the quantitative disproportion between the two tested materials. Etch&Prime 3.0 had lower values when compared to Single Bond, which demonstrates that a possible advantage of reducing a clinical application step resulted in lower bond strength. Silva (1998) studied the substrate influence on dentin adhesion with the same bonding systems as this study, using the tensile strength test, and lower values were found for Etch&Prime 3.0. Cardoso and others (1998) also noticed superior bond strength for Single Bond in the shear, tensile and microtensile bond strength tests, thus, confirming once again that further development is still required regarding the formulation of self-etching systems.

According to the classification based on the effects on the smear layer, Etch&Prime 3.0 is described as a system wherein the acidic part dissolves the mineral components of the substrate as long as it penetrates the dentinal tubules. Today, with clinical time reduction and technique simplification, Etch&Prime 3.0 can be considered a one-step adhesive system. Similar products represented by alleged self-etching primers are usually complemented with another bonding agent as a second step. This extra step can explain the better results obtained with similar products such as Clearfil SE Bond (Kuraray Medical Inc, Osaka, Japan) in tensile and microtensile strength testing when compared

to Single Bond (Imai & others, 1998; Hannig, Reinharedt & Bott, 1999; Takahashi & others, 2002).

Other speculations suggest that hydrogen ions remain free for some time after self-etching adhesives are applied, thus, possibly weakening the bond strength (Söderholm, 1997) or producing less demineralization, resulting in thinner tags under the hybrid layer and, hence, in lower bond strength (Cardoso & others, 1998). Perdigão (1995) studied the ultramorphological aspects of the resin/dentin interface using 17 adhesive systems and observed that the thinnest inter-diffusion zones were associated with self-etching adhesive systems.

This study confirms these observations by means of Scanning Electron Microscope analysis (Figures 2 and 3). Dentin decalcification promoted by Etch&Prime 3.0 can be noted to be less effective than when Single Bond is applied following substrate etching using phosphoric acid. Peritubular dentin was also minimally affected, if at all, by the application of Etch&Prime 3.0 (Figure 2). These data agree with previous findings obtained by Perdigão (1995), which showed less decalcification of the peritubular dentin when self-etching primer adhesive systems were used.

The substrate was another factor that greatly influenced the results obtained. Values obtained in a same dental element maintained similar relative magnitude in spite of the testing methodology. This means that for those teeth with high values for the orthodontic edge wire loop, relatively high values were also obtained on the other surface prepared for the knife-edge blade test. This finding was confirmed statistically and suggests that bond strength may vary among different teeth, in addition to the known variability related to the dentin depth (Pashley & others, 1993), storage time (Silva, 1998) and age of the tooth structure (Tagami & others, 1993).

CONCLUSIONS

1. Single Bond one-bottle bonding system produced higher shear bond strength results for both tests. SEM observations reflect this finding, with long, consistent tag formation.
2. Shear bond strength results obtained for Etch&Prime 3.0 were significantly weaker than Single Bond adhesive system. This may be due to less complete infiltration of monomers into the demineralized dentin surface, thus, compromising adhesion.
3. Data obtained in this study demonstrated an interesting substrate influence on shear bond strength results. More studies must be conducted to verify this same influence on different test methodologies, including microtensile, microleakage and micromorphological observations.

Acknowledgements

The authors acknowledge NAPEM (Núcleo de Apoio à Pesquisa em Materiais Dentários), School of Dentistry, University of São Paulo, Brazil. This study was developed as part of a component of the graduate course in Dental Materials, School of Dentistry, University of São Paulo, Brazil (experimental design and laboratory research techniques in biomaterials), under the responsibility of professor Dr Paulo Eduardo Capel Cardoso.

(Received 19 June 2002)

References

Burke FJT & McCaughey AD (1995) The four generations of dentin bonding *American Journal of Dentistry* **8(2)** 88-92.

Cardoso PE, Braga RR & Carrilho MR (1998) Evaluation of micro-tensile, shear and tensile tests determining the bond strength of three adhesive systems *Dental Materials* **14(6)** 394-398.

Cardoso PE, Placido E, Francci CE & Perdigão J (1999) Microleakage of Class V resin-based composite restorations using five simplified adhesive systems *American Journal of Dentistry* **12(6)** 291-294.

Castelnuovo J, Tjan AH & Liu P (1996) Microleakage of multistep and simplified-step bonding systems *American Journal of Dentistry* **9(6)** 245-248.

Della Bona A & Van Noort R (1995) Shear vs tensile bond strength of resin composite bonded to ceramic *Journal of Dental Research* **74(9)** 1591-1596.

Dunn WJ & Söderholm KJ (2001) Comparison of shear and flexural bond strength tests versus failure modes of dentin bonding systems *American Journal of Dentistry* **14(5)** 297-303.

Eliades G (1994) Clinical relevance of the formulation and testing of dentine bonding systems *Journal of Dentistry* **22(2)** 73-81 Review.

Haller B (2000) Recent developments in dentin bonding *American Journal of Dentistry* **13(1)** 44-50.

Hannig M, Reinhardt KJ & Bott B (1999) Self-etching primer vs phosphoric acid: An alternative concept for composite-to-enamel bonding *Operative Dentistry* **24(3)** 172-180.

Imai T, Itoh K, Tani C, Manabe A, Yamashita T, Hisamitsu H & Wakumoto S (1998) Effectiveness of simplified dentin bonding systems *Dental Materials Journal* **17(1)** 1-10.

Inai N, Kanemura N, Tagami J, Watanabe LG, Marshall SJ & Marshall GW (1998) Adhesion between collagen depleted dentin and dentin adhesives *American Journal of Dentistry* **11(3)** 123-127.

International Organization for Standardization (1993) ISO TR 11405 Dental Materials—Guidance on testing of adhesion to tooth structure Geneva: WHO.

Koibuchi H, Yasuda N & Nakabayashi N (2001) Bonding to dentin with a self-etching primer: The effect of smear layers *Dental Materials* **17(2)** 122-126.

Kwong SM, Tay FR, Yip HK, Kei LH & Pashley DH (2000) An ultrastructural study of the application of dentine adhesives to acid-conditioned sclerotic dentine *Journal of Dentistry* **28(7)** 515-528.

Nakabayashi N & Saimi Y (1996) Bonding to intact dentin *Journal of Dental Research* **75(9)** 1706-1715.

Nara Y, Suda S, Tanaka H & Dogon IL (1998) *In vivo* bond strength of two adhesive systems to cervical abrasion *Journal of Dental Research* **77** Abstract #802 p 206.

Øilo G & Austrheim EK (1993) *In vitro* quality testing of dentin adhesives *Acta Odontologica Scandinvica* **51(4)** 263-269.

Pashley EL, Tao L, Mattews WG & Pashley DH (1993) Bond strength to superficial, intermediate and deep dentin *in vivo* with four dentin bonding systems *Dental Materials* **9(1)** 19-22.

Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA & Tay F (1999) The microtensile bond test: A review *Journal of Adhesive Dentistry* **1(4)** 299-309.

Perdigão J, Swift EJ Jr, Denehy GE, Wefel JS & Donly KJ (1994) *In vitro* bond strengths and SEM evaluation of dentin bonding systems to different dentin substrate *Journal of Dental Research* **73(1)** 44-55.

Perdigão J (1995) An ultra-morphological study of human dentine exposed to adhesive systems Leuven, 132 p PhD Thesis: Catholic University of Leuven, Belgium.

Prati C, Chersoni S, Mongiorgi R & Pashley DH (1998) Resin-infiltrated dentin layer formation of new bonding systems *Operative Dentistry* **23(4)** 185-194.

Prati C, Pashley DH, Chersoni S & Mongiorgi R (2000) Marginal hybrid layer formation in Class V restorations *Operative Dentistry* **25(3)** 228-233.

Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R & Pashley DH (1994) Relationship between surface area for adhesion and tensile bond strength—evaluation of a microtensile bond test *Dental Materials* **10(4)** 236-240.

Silva EM (1998) Substrate influence on dentin adhesion São Paulo 62p MDS Thesis: School of Dentistry, University of São Paulo, Brazil.

Sinhoreti MA (1997) [Influência do tipo de carregamento sobre a resistência da união ao cisalhamento da interface dentina-resina] Piracicaba, p 105 MDS Thesis: Piracicaba School of Dentistry, University of Campinas.

Söderholm KJ (1997) Technique sensitivity of dentin bonding *General Dentistry* **45(3)** 218-222.

Swift EJ Jr & Bayne SC (1997) Shear bond strength of a new one-bottle dentin adhesive *American Journal of Dentistry* **10(4)** 184-188.

Tagami J, Nakajima M, Shono T, Takatsu T & Hosoda H (1993) Effect of aging on dentin bonding *American Journal of Dentistry* **6(3)** 145-147.

Takahashi A, Sato Y, Uno S, Pereira PN & Sano H (2002) Effects of mechanical properties of adhesive resins on bond strength to dentin *Dental Materials* **18(3)** 263-268.

Tjan AHL, Castelnuovo J & Liu P (1996) Bond strength of multi-step and simplified-step systems *American Journal of Dentistry* **9(6)** 269-272.

Toledano M, Osorio R, de Leonardi G, Rosales-Leal JI, Ceballos L & Cabrerizo-Vilchez MA (2001) Influence of self-etching primer on the resin adhesion to enamel and dentin *American Journal of Dentistry* **14(4)** 205-210.

Van Meerbeek B, Perdigão J, Lambrechts P & Vanherle G (1998) The clinical performance of adhesives *Journal of Dentistry* **26(1)** 1-20 Review.

Van Noort R, Noroozi S, Howard IC & Cardew G (1989) A critique of bond strength measurements *Journal of Dentistry* **17(2)** 61-67.

Watanabe LG, Marshall GW, Bedford DT & Mashall SJ (1998) Influence of etching on shear bond strength of dentin adhesives *Journal of Dental Research* **77** Abstract #797 p 205.

Wilder AD Jr, Swift EJ Jr, May KN Jr & Waddell SL (1998) Bond strengths of conventional and simplified bonding systems *American Journal of Dentistry* **11(3)** 114-117.

Yap AUJ, Ho KS & Wong KM (1998) Comparison of marginal sealing ability of new generation bonding systems *Journal of Oral Rehabilitation* **25(9)** 666-671.

©Operative Dentistry, 2003, **28-4**, 395-402

Fluoride Release and Uptake Capacities of Fluoride-Releasing Restorative Materials

N Attar • MD Turgut

Clinical Relevance

All materials tested displayed a constant fluoride release property that gradually diminished with time during the first 30 days of the study. Ion-releasing composite, conventional and resin-modified glass ionomer cements can be re-charged with topical fluoride gel.

SUMMARY

Many fluoride-releasing dental materials are being sold on the basis of their cariostatic properties. However, the amount fluoride release of these materials is still uncertain. This study investigated the fluoride release and uptake characteristics of four flowable resin composites (Heliomolar Flow, Tetric Flow, Wave, Perma Flo), one flowable compomer (Dyract flow), one conventional glass ionomer cement mixed with two different powder/liquid ratios (ChemFlex Syringeable and ChemFlex Condensable), one packable resin composite (SureFil), one ion-releasing composite (Ariston pHc) and one resin-modified glass ionomer cement (Vitremer). Seven discs (6-mm diameter and 1.5-mm height) were prepared for each material. Each disc was immersed in 3.5 ml of deionized water within a plastic vial and stored at 37°C. The deionized water was changed every 24 hours and the release of fluoride was measured for 30 days. At the end of this period, the samples were recharged with 2 ml of 1.23% acidulated phosphate fluoride (APF) gel for four minutes. Then, all samples were reassessed for an additional 10 days. The fluoride release of all samples was measured with a specific fluoride electrode and an ionanalyzer. Statistical analyses were conducted using two-way repeated measure ANOVA and Duncan's multiple range tests. For all tested materials, the greatest fluoride release was observed after the first day of the study ($p<0.01$) but gradually diminished with time. During the test period, Tetric Flow released the lowest amount of fluoride; however, no statistically significant difference was found from Wave and Heliomolar Flow ($p>0.05$). Ariston pHc released the highest amount of fluoride, followed by ChemFlex Syringeable, Vitremer and ChemFlex Condensable. There were statistically significant differences among these materials ($p<0.05$). Fluoride release of all materials were significantly increased after the first day following refluoridation and Ariston pHc released the greatest among all materials ($p<0.01$). At the end of two days of refluoridation, the fluoride release rate for each material dropped quickly and stabilized within three days.

*Nuray Attar, DDS, PhD, research assistant, Department of Restorative Dentistry, Faculty of Dentistry, Hacettepe University, Ankara, Turkey

Melek D Turgut, DDS, PhD, research assistant, Department of Pediatric Dentistry, Faculty of Dentistry, Hacettepe University, Ankara, Turkey

*Reprint request: Emek 8 Cadde, Buket A Apartmanı, No: 62 A Daire No: 12, 06510, Emek, Ankara, Turkey; e-mail: nurayattar@hotmail.com

INTRODUCTION

One of the major problems with restorative dentistry is the replacement of restorations due to secondary caries (Mjör, 1981; Kidd, Toffenetti & Mjör 1992). Therefore, fluoride has been added to most restorative materials because of its effectiveness in secondary caries prevention. Several studies have found that different concentrations of fluoride are released and taken up by enamel from fluoride releasing materials (Arends & Van der Zee, 1990; Forss & Seppa, 1990; Skartveit & others, 1990; Kawai & others, 1998; Eronat, Kocatas & Alpoz, 1999).

Conventional glass ionomer cements offer the advantage of releasing significant levels of fluoride ions to the surrounding tooth structure and oral environment (Retief & others, 1984; Forss & Seppa, 1990; Skartveit & others, 1990). Moreover, *in vitro* studies demonstrated that glass ionomer cements could be recharged with fluoride treatment or daily toothpaste (Creanor & others, 1994; Suljak & Hatibovic-Kofman, 1996; Forsten, 1998; Rothwell, Anstice & Pearson, 1998; Strother & others, 1998; Vieira, De Souza & Modesto, 1999; Yip & Smales, 1999; Peng & others, 2000; Posada, Emilson & Birkhed, 2000; Gao & Smales, 2001; Attar & Onen, 2002a). In addition to being so advantageous in high caries-risk patients, short working time, long setting time and sensitivity to water during the early stages of setting limit their use.

Resin-modified glass ionomer cements and polyacid-modified resin composites (compomers) claim to improve the mechanical properties of, while retaining the esthetic, adhesive and fluoride releasing properties of conventional glass ionomer cements (Mathis & Ferracane, 1989; Attin, Vataschki & Hellwig, 1996). Both laboratory and clinical research have clearly demonstrated the ability of the resin-modified glass ionomer cements to release fluoride (Momoi & Mc Cabe, 1993; Forsten, 1995; Burgess & others, 1996; De Araujo & others, 1996; Tam, Chan & Yim, 1997; Rothwell & others, 1998; Yip & Smales, 1999; Mazzaoui, Burrow & Tyas, 2000; Gao & Smales, 2001). The fluoride release from and the uptake by resin modified cements was reported to be higher than or the same as conventional glass ionomer cements (Momoi & Mc Cabe, 1993; Forsten, 1995; Burgess & others, 1996; De Araujo & others, 1996; Tam & others, 1997; Rothwell & others, 1998; Gao & Smales, 2001).

Whether polyacid-modified resin composites possess a combination of the characteristics of both composites and glass ionomers, they, in fact, show minimal glass ionomer reactions (Suljak & Hatibovic-Kofman, 1996; Guggenberger, May & Stefan, 1998). It has been shown that these materials release less fluoride initially compared to conventional and resin-modified glass ionomer cements (Suljak & Hatibovic-Kofman, 1996; Attin & others, 1999; Yip & Smales, 1999; Attar & Onen 2002a).

Many new restorative materials continue to be introduced by manufacturers, claiming wider range of uses and improved clinical performance. Of these, resin composites may be the most refined materials. The progression of resin composite from macrofills to microfills and from hybrid to microhybrids has produced new materials with improved restorative characteristics. Flowable composites have low viscosity and have been created by retaining the same small particle size of traditional hybrid composites and by reducing the filler content (Bayne & others, 1998). Flowable composites could be microfilled, microhybrid or hybrid resin composites or compomers. They are highly polishable because of the small filler particle sizes, which may be as small as 0.7 μm with filler content by weight of 50% to 70%. The fillers may be radiopaque and may contain fluoride (Bouschlicher, Cobb & Boyer, 1999; Murchison, Charlton & Moore, 1999; Estefan & others, 2000). However, it is not reported whether or not these materials can be recharged.

Condensable or packable composites were introduced by various manufacturers of dental resin composites as improved restorative materials simulating the favorable handling properties of amalgam through variations in resin composition, particle distribution and content of the incorporated fillers. Condensable composites differ from conventionally used posterior composites as a result of variations in the amount and size of filler particles or modifications in resin formulation (Dentsply De Trey-SureFil, 1998; 3M Dental Products-Filtek P-60, 1999). A high filler load and filler distribution give packable composites a different consistency compared with hybrid composites and make possible establishing physiological interproximal contacts in Class II restorations by using metal matrix bands and wooden wedges (Leinfelder, 1997; Leinfelder, Radz & Nash, 1998).

Another new approach in restorative dentistry was the introduction of an ion-releasing composite material in 1998. Ariston pHc is presented by its manufacturers as an alkaline glass filled ion-releasing amalgam substitute capable of releasing fluoride ions and buffering acids by releasing hydroxyl ions when the pH drops due to active plaque. This phenomenon is based on a newly developed alkaline glass filler and is expected to reduce the formation of secondary caries at the margins of the restoration due to an inhibition of bacterial growth, a reduced demineralization and a buffering of acids produced by cariogenic microorganisms (Vivadent-Ariston pHc, 1998).

Although many new materials have been developed, very little information has been released regarding their fluoride releasing capacities. Therefore, this *in vitro* study investigated the fluoride release and uptake characteristics of an ion-releasing composite, a packable composite, five flowable materials and compared

them with a resin-modified glass ionomer and a conventional glass ionomer cement with two different powder/liquid ratios used as controls.

METHODS AND MATERIALS

The nine aesthetic restorative materials used in the study and their manufacturers and batch numbers are shown in Table 1.

Table 1: *Restorative Materials Tested in the Study*

Type	Commercial Name	Manufacturer	Batch Number
Flowable compomer	Dyract Flow	Dentsply-DeTrey, GmbH D 78467 Konstanz, Germany	#606.04.430
Flowable composite	Heliomolar Flow	Vivadent Ivoclar AG, FL-9494, Schaan, Liechtenstein	#557032BN
Flowable composite	Tetric Flow	Vivadent Ivoclar AG, FL-9494, Schaan, Liechtenstein	#546323AN
Flowable composite	Wave	Southern Dental Ind (SDI) Victoria 3153, Australia	#000836
Flowable composite	PermaFlo	Ultradent Products Inc South Jordan, UT, USA	#1023
Ion-releasing composite	Ariston pHc	Vivadent Ivoclar AG, FL-9494, Schaan, Liechtenstein	AO9636
Packable composite	SureFil	Dentsply-DeTrey, GmbH D 78467 Konstanz, Germany	606-05-750
Resin-modified glass ionomer cement	Vitremer	3M, Dental Products, St Paul, MN, USA	Liquid 3303L/ Powder 318
Conventional glass ionomer cement	ChemFlex	Dentsply-DeTrey, GmbH D 78467 Konstanz, Germany	606-07-100

Sample Preparation

Seven samples of each material were prepared according to the manufacturers' instructions and placed into disposable, cylindrical teflon molds with a diameter of 6 mm and a height of 1.5 mm. They were then sandwiched between two Mylar-covered glass slides. Vitremer, ChemFlex Syringeable (thinly mixed) and ChemFlex Condensable (thickly mixed) were hand mixed according to each manufacturer's recommended powder/liquid ratios using the scoops. ChemFlex Syringeable and ChemFlex Condensable were chemically set. The resin-containing materials (Wave, Tetric Flow, Heliomolar Flow, PermaFlo, Dyract Flow, Ariston pHc, SureFil, Vitremer) were light cured from the top and bottom surfaces of samples with a Hilux Dental Curing light source for 40 seconds (Benlioglu Dental Inc Ankara, Turkey) at 500 mW/cm^2. The discs were then removed from the disposable Teflon molds and weighed in order to verify standardization within each material test group (±0.01).

Initial Fluoride Release

Each sample was placed in a polyethylene test tube filled with 3.5 ml of deionized water and incubated at 37°C. The average fluoride concentration in the deionized water was <0.01 ppm. Following 24 hours of incubation, the samples were grasped with clean metal forceps coated with nail varnish and washed with 0.5 ml deionized water using a syringe over the original holding tube, thus, collecting the rinse water in that tube. Each sample was dried for two minutes on absorbent paper, then transferred to a new polyethylene test tube containing 3.5 ml deionized water and stored at 37°C. The water was changed every 24 hours.

Fluoride release was determined at the end of the first, second, third, tenth, twentieth and thirtieth day after buffering the solution with equal volumes of TISAB II (4 ml) (Total Ionic Strength Adjustment Buffer, Orion Research, Inc, Beverly, MA, USA). Fluoride release was measured with a combination of fluoride electrode (Orion 9609BN, Orion Research Inc) and an ionanalyzer (Orion EA 940, Orion Research Inc). Data concerning fluoride was recorded in parts per million (ppm). The electrode was previously calibrated with a standard whose molarity spanned the actual concentrations of fluoride to be measured (0.01, 0.1,0.5, 1.0, 5.0, 10.0, 20.0 and 30.0 ppm). The fluoride concentration was determined by adding 4 ml TISAB II to each 4 ml sample solution. A small magnetic stirring bar was used immediately prior to the measurements.

Fluoride Ion Release After Exposure to APF Gel

Following 30 days of initial fluoride release, all samples were coated with 2 ml of 1.23% APF gel (Sultan Topex Thixotropic APF Sultan Dental Products Englewood, NJ, USA) and left in place for four minutes. After immersion, the sample was wiped clean with a tissue, then thoroughly rinsed by exposing each side to a stream of deionized water for 20 seconds. Each sample was dried for two minutes on absorbent paper, then transferred to a new polyethylene test tube containing 3.5 ml of deionized water and stored at 37°C. Fluoride release was measured at the end of the first, second, third, and tenth days.

Analysis of Data

Two-way repeated measure ANOVAs were performed to compare types of materials for each time point ($p<0.01$). The differences between materials was determined with Duncan's multiple range test ($p<0.05$). Two-way repeated measure ANOVA on restorative material ver-

Table 2: *Mean Fluoride Ion Levels Before and After APF Gel Application (ppm) (mean ± standard deviation)*

Groups	Day 1	Day 2	Day 3	Day 10	Day 20	Day 30	Day 1	Day 2	Day 3	Day 10
Aris.pHc	12.93 ± 0.51	6.84 ± 0.36	4.28 ± 0.34	3.40 ± 0.34	2.77 ± 0.12	2.46 ± 0.31	10.03 ± 0.69	1.64 ± 0.14	1.11 ± 0.25	0.57 ± 0.14
ChemF.Sy	18.67 ± 0.62	7.28 ± 0.91	2.02 ± 0.49	1.18 ± 0.30	1.72 ± 0.26	0.97 ± 0.25	3.10 ± 0.31	1.59 ± 0.07	0.88 ± 0.12	0.29 ± 0.01
Vitremer	15.75 ± 1.40	5.13 ± 0.44	1.78 ± 0.23	1.32 ± 0.08	1.38 ± 0.16	0.94 ± 0.10	3.28 ± 0.25	1.02 ± 0.10	0.70 ± 0.12	0.80 ± 0.20
ChemF.Co	12.34 ± 0.98	4.39 ± 0.45	1.40 ± 0.06	1.12 ± 0.25	1.05 ± 0.14	0.95 ± 0.18	4.11 ± 0.36	1.56 ± 0.34	0.60 ± 0.10	0.43 ± 0.01
Dyract flo[a]	2.78 ± 0.27	0.51 ± 0.04	0.22 ± 0.04	0.16 ± 0.02	0.18 ± 0.02	0.13 ± 0.02	1.5 ± 0.20	1.07 ± 0.17	0.75 ± 0.07	0.30 ± 0.06
SureFil[a]	2.07 ± 0.15	0.92 ± 0.06	0.72 ± 0.09	0.33 ± 0.03	0.29 ± 0.03	0.15 ± 0.02	1.94 ± 0.18	0.39 ± 0.09	0.31 ± 0.05	0.12 ± 0.04
PermaFlo[b]	1.47 ± 0.08	0.52 ± 0.05	0.25 ± 0.05	0.23 ± 0.02	0.16 ± 0.04	0.09 ± 0.02	1.37 ± 0.08	0.52 ± 0.10	0.29 ± 0.04	0.10 ± 0.05
Wave[b,c]	1.60 ± 0.33	0.27 ± 0.07	0.15 ± 0.04	0.10 ± 0.03	0.07 ± 0.02	0.09 ± 0.03	1.36 ± 0.10	0.26 ± 0.08	0.11 ± 0.03	0.07 ± 0.0
HelioFlo[b,c]	1.13 ± 0.03	0.26 ± 0.37	0.12 ± 0.02	0.12 ± 0.03	0.06 ± 0.02	0.06 ± 0.02	1.26 ± 0.10	.52 ± 0.07	0.63 ± 0.05	0.09 ± 0.0
Tetric Flo[c]	1.36 ± 0.03	0.18 ± 0.03	0.12 ± 0.02	0.08 ± 0.02	0.06 ± 0.02	0.06 ± 0.02	1.28 ± 0.05	0.26 ± 0.04	0.23 ± 0.03	0.07 ± 0.0

Groups with the same superscript letter are not significantly different (p>0.05)

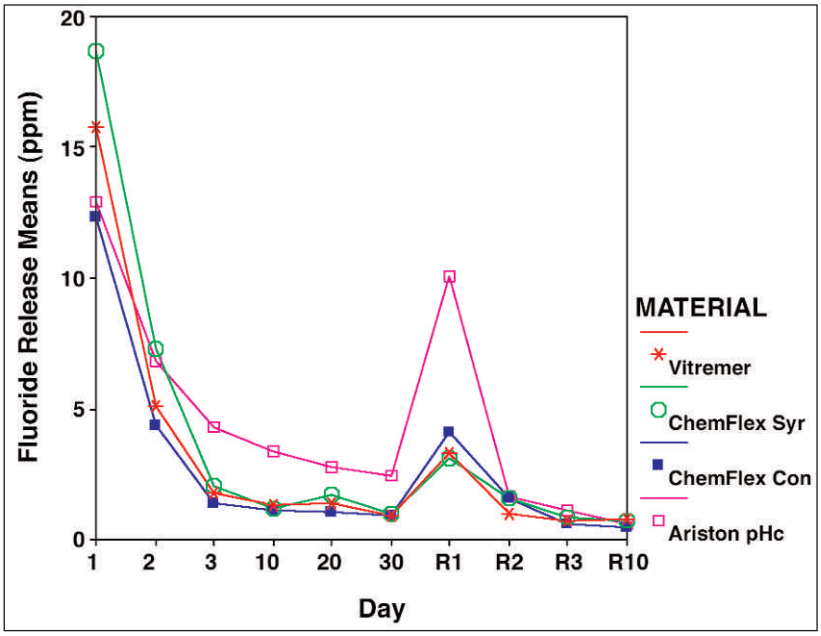


Figure 1. *Fluoride release of Vitremer, ChemFlex Syringeable, ChemFlex Condensable, Ariston pHc both before and after refluoridation (R), on the basis of average of the seven samples.*

sus time revealed statistically significant differences. As a result, the authors examined the differences between the mean values and the differences between the mean values when compared to the first 24 hours ($p<0.01$). One-way ANOVA was performed to analyze the differences between fluoride release of the 30th day and the first, second, third and tenth days after refluoridation ($p<0.01$).

RESULTS

The mean values and standard deviations of the fluoride releases (ppm) of samples both before and after refluoridation are listed in Table 2.

An analysis of two-way repeated measure ANOVA indicated significant differences in fluoride release among materials ($p<0.01$).

Duncan's multiple range test showed statistically significant differences between the groups (Table 2). Tetric Flow released the lowest amount of fluoride but showed no significant differences from Wave and Heliomolar Flow. Ariston pHc released the highest amount of fluoride followed by ChemFlex Syringeable, Vitremer and ChemFlex Condensable and significant differences were found among these materials ($p<0.05$). Significant differences were found in the amounts of daily fluoride release between ChemFlex Syringeable and ChemFlex Condensable. ChemFlex Syringeable released more fluoride than ChemFlex Condensable during the 30-day testing period ($p<0.05$).

Using two-way repeated measure ANOVA to compare the time revealed a significant difference ($p<0.01$). The concentration of fluoride release was higher during the first 24 hours, declined on the second day, then gradually diminished with time. Fluoride release of all materials was significantly increased after the first day following refluoridation. However, the levels of fluoride release dropped rapidly soon afterwards.

Statistical analysis of two-way repeated measure ANOVA on restorative material versus time revealed statistically significant differences ($p<0.01$). All materials released the highest amount of fluoride after the first 24 hours, with fluoride release continuing over the entire 30-day testing period. The amount of fluoride release on the first day ranged from 18.67 to 1.13; the greatest was from ChemFlex Syringeable (18.67), followed by Vitremer (15.75), Ariston pHc (12.93), ChemFlex Condensable (12.34), Dyract flow (2.78), SureFil (2.07), Wave (1.60), PermaFlo (1.47), Tetric Flow (1.36) and Heliomolar Flow (1.13). Initial fluoride release decreased with time. ChemFlex Syringeable, Vitremer, Ariston pHc and ChemFlex Condensable showed significantly higher initial fluoride release rates during the first 24 hours ($p<0.01$).

Table 2 and Figures 1 and 2 show the effects of refluoridation on fluoride release. After the first day, there was an increase in fluoride release of all materials ($p<0.01$). In particular, Ariston pHc released relatively more fluoride (Figure 1). ChemFlex Condensable displayed higher refluoridation-release property than did the ChemFlex Syringeable. The patterns of fluoride release before and after APF gel application were similar, so that after the first 24 hours high levels decreased with time. After two days the fluoride release rates of each material usually dropped quickly and became stabilized within three days ($p<0.01$).

Figure 1 shows the fluoride release of Ariston pHc, ChemFlex Syringeable, Vitremer, ChemFlex Condensable from the first to the 30th day and after refluoridation on the basis of the average of the seven samples.

Figure 2 shows the fluoride release from the other test materials from the first to the 30th day and after refluoridation.

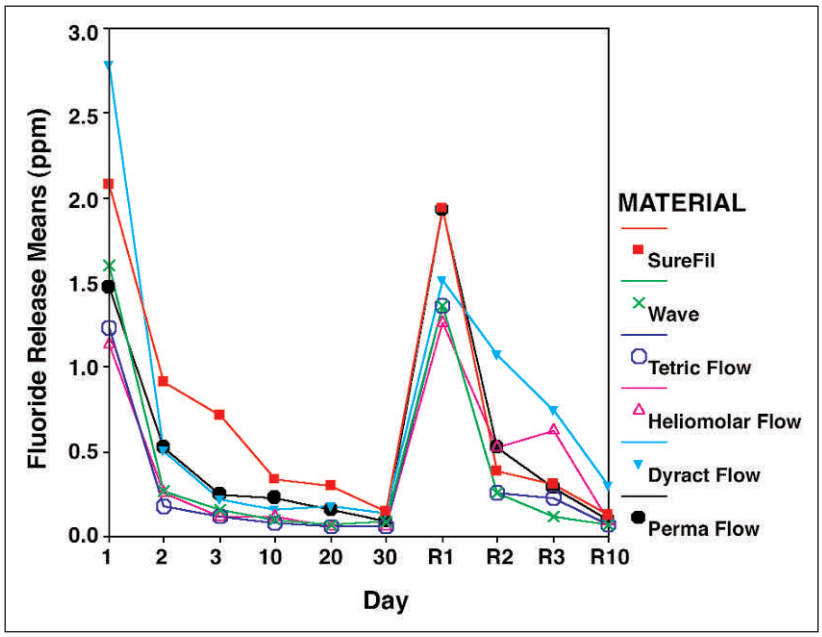


Figure 2. *Fluoride release of SureFil, Wave, Tetric Flow, Heliomolar Flow, Dyract Flow and Perma Flow, both before and after refluoridation (R).*

DISCUSSION

In vitro studies have shown that fluoride released from dental restoratives effectively inhibits tooth demineralization in artificial carious solutions or gels (Hicks, Flaitz & Silverstone, 1986; Tam & others, 1997; Hicks & Flaitz, 2000; Attar & Onen, 2002b). This is one important reason why these kinds of materials are widely used in dentistry. There are numerous *in vitro* studies that examined the release of fluoride from different restorative materials; however, the precise levels of fluoride ion release required for various clinical situations still remain to be determined.

In this study, fluoride release patterns of conventional glass ionomer cement, resin-modified glass ionomer cement and composites were found to agree with previous studies (El-Mallakh & Sarkar, 1990; De Schepper & others, 1991; Perrin, Persin & Sarrazin, 1994; Young & others, 1996; Suljak & Hatibovic-Kofman, 1996; Forsten, 1998; Carvalho & Cury, 1999). The highest release of fluoride occurred during the first 24 hours, decreased with time, but continuous release of fluoride was measured during the entire period. While ChemFlex Syringeable, Vitremer, ChemFlex Condensable and Ariston pHc demonstrated a surge in fluoride release in the first 24 hours, the flowable composites Tetric Flow, Wave, Heliomolar Flow, Perma Flo, packable composite SureFill and flowable compomer Dyract flow did not show high initial fluoride release rates. However, SureFil and Dyract flow released relatively more fluoride than all the flowable composites ($p<0.05$).

All materials evaluated in this study released fluoride ions but the patterns were variable. The ion-releasing composite conventional glass ionomer cement mixed with two different powder/liquid ratios, and resin-modified glass ionomer cement maintained a relatively high fluoride release rate. The fact that fluoride releases from conventional and resin-modified glass ionomer cements was greatest during the first 24 hours agrees with previous studies (El Mallakh & Sarkar, 1990; De Schepper & others, 1991; Creanor & others, 1994; Perrin & others, 1994; Young & others, 1996). The high concentration observed in the first day is called as the "burst effect" of fluoride. The reason for the rapid fall of fluoride release during subsequent days is likely to result from the initial burst of fluoride released from the glass particles as they dissolve in the polyalkenoate acid during the setting reaction. The later slow release occurs as the glass dissolves in the acidified water of the hydrogel matrix (De Moor, Verbeeck & De Maeyer, 1996). The mean fluoride releases of aesthetic restorative materials shown as graphs in Figures 1 and Figure 2 reveal the initial "burst" effect of ChemFlex Syringeable, Vitremer, Ariston pHc, ChemFlex Condensable and the remaining constant release level for 30 days. However, packable composite SureFil, flowable composites Tetric Flow, Wave, Heliomolar Flow, Perma Flo and flowable compomer Dyract flow did not show an initial fluoride "burst effect."

Significant differences were found in the amounts of daily fluoride release between ChemFlex Syringeable and ChemFlex Condensable ($p<0.05$). The results of this study show that low powder/liquid ratios of ChemFlex Syringeable lead to more fluoride release than high ratios of ChemFlex Condensable during the first 30

days. This result agreed with the findings of a previous study that examined ChemFil and Fuji II with different powder/liquid ratios (Perrin & others, 1994). In contrast, ChemFlex Condensable released more fluoride than Chem Flex Syringeable after the first 24 hours following refluoridation ($p<0.05$).

Fluoride releases of all materials were significantly increased after the first day following refluoridation, and Ariston pHc released relatively more among all materials. After two days of refluoridation, the fluoride release rate of each material usually dropped quickly and stabilized within three days. Flowable composites, flowable compomer and packable composite exhibited a significantly lower refluoridation-release property than Ariston pHc, ChemFlex Condensable, Vitremer and ChemFlex Syringeable. Only the ion-releasing resin composite Ariston pHc could release significantly greater fluoride for three days after refluoridation and demonstrate that it could function in the same or a better role of fluoride reservoirs compared to conventional (with two different powder/liquid ratios) and resin-modified glass ionomer cements.

Previous studies have shown that the amount of fluoride released from fluoride containing resin composites was lower than those of conventional and resin-modified glass ionomer cements (Swift, 1989; Young & others, 1996; Forsten, 1998; Vieira & others, 1999; Carvalho & Cury, 1999; Mazzaoui & others, 2000; Attar & Onen, 2002a). In this study, flowable composites, flowable compomer and packable composite were observed to release low levels throughout the period of the study. Also, these materials exhibited a significantly lower refluoridation-release property than ion-releasing composite, conventional (with two different powder/liquid ratios) and resin-modified glass ionomer cements. However, the most interesting finding in this study was that ion-releasing resin composite released more fluoride than the other materials. The reason for this high release is unknown. Ariston pHc contains an alkaline glass filler, barium-aluminum-fluorosilicate and ytterbium trifluoride, although the manufacturers did not disclose the actual amounts. Another reason could relate to erosion or degradation of the material. Ariston pHc releases increased amounts of fluoride, calcium and hydroxyl ions in response to a local decrease in pH (Vivadent- Ariston pHc, 1998; Mazzaoui & others, 2000). In this study, relatively high daily levels of fluoride ions were released from Ariston pHc. Peng and others (2000) reported that the relatively high cumulative levels of fluoride ions released from freshly mixed Ariston pHc and four compomers demonstrated that some resin-containing materials release amounts comparable to more viscous conventional glass ionomer cements.

The fluoride values of packable resin composite (SureFil), flowable resin composites (Heliomolar Flow, Tetric Flow, Wave, Perma Flo) and flowable compomer (Dyract flow) decreased to almost zero at the end of the second day after refluoridation. Laboratory studies have shown that although conventional and resin-modified glass ionomer cements can be recharged, resin composites cannot (Hatibovic-Kofman & Koch, 1991; Suljak & Hatibovic-Kofman, 1996; Burgess & others, 1996; Attar & Onen, 2002a). In this study, Tetric Flow released the least amount of fluoride; however, no statistically significant difference was found from Wave and Heliomolar Flow. SureFil (packable resin composite) and Dyract flow (flowable compomer) released relatively more fluoride compared to the other flowable composites ($p<0.05$). The increased fluoride release by SureFil, Heliomolar Flow, Tetric Flow, Wave, Perma Flo and Dyract flow after exposure to APF fluoride were thought to result from surface-retained fluoride.

In this study, *in vitro* samples were placed in deionized water. Glockman and others (1997) showed that glass ionomer cements released more fluoride in water than in artificial saliva. El-Mallakh and Sarkar (1990) also reported that fluoride release into artificial saliva is less than deionized water. However, in this study, the presence of plaque or pellicle that may concentrate fluoride levels was not taken into account. The amount of fluoride release in deionized water could be different from that found in the oral cavity, because saliva is a constantly changing medium with respect to temperature, pH, protein content and many other factors. A number of studies have shown that the release of fluoride is greater in acid mediums (Forsten, 1991a; Vieira & others, 1999; Carvalho & Cury, 1999). In the oral environment, this could be the case, especially with a plaque-induced acidojenic challenge. It has been postulated that the increased fluoride release at pH 5, as compared with neutral pH, may be caused by the undesirable, faster dissolution of the filling material (Forsten, 1991b).

The ultimate goal of correlating fluoride release with actual caries reduction is an objective that can only be met by completing controlled clinical studies on materials with well-characterized kinetics of fluoride release. Apparently, such conflicting results increase the demand for even more clinical data on the anticariogenity of fluoride containing materials. Further studies are needed to clinically corroborate these findings.

CONCLUSIONS

1. All materials tested in the study demonstrated the ability to release fluoride initially. During the test period Ariston pHc released significantly more fluoride than other materials. ChemFlex Syringeable followed by Vitremer and ChemFlex Condensable released significantly more fluoride than the balance of the materials. Tetric Flow released the lowest amount of fluoride.

2. The concentration of fluoride release was higher during the first 24 hours, declined on the second day, then gradually diminished with time.
3. Fluoride release of all materials significantly increased after the first day following refluoridation, and Ariston pHc released relatively more among all materials. After two days of refluoridation, the fluoride release rates of each material usually dropped quickly and stabilized within three days.

Acknowledgements

Erdem Karabulut MSc from the Department of Biostatistics, Hacettepe University is gratefully acknowledged for the statistical analysis performed.

(Received 19 June 2002)

References

3M Dental Products (1999) *Filtek P-60 Posterior Restorative System Technical Manual* St Paul MN 55144.

Arends J & Van Der Zee Y (1990) Fluoride uptake in bovine enamel and dentin from a fluoride-releasing composite resin *Quintessence International* **21** 541-544.

Attin T, Vataschki M & Hellwig E (1996) Properties of resin-modified glass-ionomer restorative materials and two polyacid-modified resin composite materials *Quintessence International* **27** 203-209.

Attin T, Buchalla W, Siewert C & Hellwig E (1999) Fluoride release/uptake of polyacid-modified resin composites (compomers) in neutral and acidic buffer solutions *Journal of Oral Rehabilitation* **26** 388-393.

Attar N & Onen A (2002a) Fluoride release and uptake characteristics of aesthetic restorative materials *Journal of Oral Rehabilitation* **29** 791-798.

Attar N & Onen A (2002b) Artificial formed caries-like lesions around esthetic restorative materials *The Journal of Clinical Pediatric Dentistry* **26** 289-296.

Bayne SC, Thompson JY, Swift EJ, Stamatiades P & Wilkerson M (1998) A characterization of first-generation flowable composites *Journal of the American Dental Association* **129** 567-577.

Bouschlicher MR, Cobb DS & Boyer DB (1999) Radiopacity of compomers, flowable and conventional resin composites for posterior restorations *Operative Dentistry* **24(1)** 20-25.

Burgess JO, Norling BK, Ralph Rawls H & Ong JL (1996) Directly placed esthetic restorative materials—The Continuum *Compendium of Continuing Education in Dentistry* **17** 731-732, 734.

Carvalho AS & Cury JA (1999) Fluoride release from some dental materials in different solutions *Operative Dentistry* **24(1)** 14-19.

Creanor SL, Carruthers LMC, Saunders WP, Strang R & Foye RH (1994) Fluoride uptake and release characteristics of glass ionomer cements *Caries Research* **28** 322-328.

Dentsply De Trey (1998) *SureFil Technical Manual High Density Posterior Restorative* Dentsply De Trey Konstanz.

De Araujo FB, García-Godoy F, Cury JA & Conceiçao EN (1996) Fluoride release from fluoride-containing materials *Operative Dentistry* **21** 185-190.

De Moor RJ, Verbeeck RM & De Maeyer EA (1996) Fluoride release profiles of restorative glass ionomer formulations *Dental Materials* **12** 88-95.

De Schepper EJ, Berr EA, Cailleteau JG & Tate WH (1991) A comparative study of fluoride release from glass ionomer cements *Quintessence International* **22** 215-219.

El-Mallakh BF & Sarkar NK (1990) Fluoride release from glass ionomer cements in de-ionized water and artificial saliva *Dental Materials* **6** 118-122.

Eronat N, Kocatas N & Alpoz AR (1999) A comparative study of fluoride uptake from dentin bonding agents and glass-ionomer cements in permanent and primary tooth enamel *Quintessence International* **30** 496-500.

Estafan D, Dussetschleger FL, Miuo LE & Kondamani J (2000) Class V lesions restored with flowable composite and added surface sealing resin *General Dentistry* **48** 78-80.

Forss H & Seppa L (1990) Prevention of enamel demineralization adjacent to glass ionomer filling materials *Scandinavian Journal of Dental Research* **98** 173-178.

Forsten L (1991a) Short and long term fluoride release from glass ionomer based liners *Scandinavian Journal of Dental Research* **99** 340-342.

Forsten L (1991b) Fluoride release and uptake by glass ionomers *Scandinavian Journal of Dental Research* **99** 241-245.

Forsten L (1995) Resin-modified glass ionomer cements: Fluoride release and uptake *Acta Odontologica Scandinavica* **53** 222-225.

Forsten L (1998) Fluoride release and uptake by glass ionomers and related materials and its clinical effect *Biomaterials* **19** 503-508.

Gao W & Smales RJ (2001) Fluoride release/uptake of conventional and resin-modified glass ionomers and compomers *Journal of Dentistry* **29** 301-306.

Glockman E, Sigusch B, Gehroldt C & Triemer K (1997) Fluoride release of different types of glass ionomer cements *Journal of Dental Research* **76** Abstract #2421 p 316.

Guggenberger R, May R & Stefan KP (1998) New trends in glass-ionomer chemistry *Biomaterials* **19** 479-483.

Hatibovic-Kofman S & Koch G (1991) Fluoride release from glass ionomer cement *in vivo* and *in vitro Swedish Dental Journal* **15** 253-258.

Hicks MJ, Flaitz CM & Silverstone LM (1986) Secondary caries formation *in vitro* around glass ionomer restorations *Quintessence International* **17** 527-532.

Hicks MJ & Flaitz CM (2000) Resin-modified glass-ionomer restorations and *in vitro* secondary caries formation in coronal enamel *Quintessence International* **31** 570-578.

Kawai K, Tantbirojn D, Kamalawat AS, Hasegawa T & Retief DH (1998) *In vitro* enamel and cementum fluoride uptake from three fluoride-containing composites *Caries Research* **32** 463-469.

Kidd EA, Toffenetti F & Mjör IA (1992) Secondary caries *International Dental Journal* **42** 127-138.

Leinfelder KF (1997) New developments in resin restorative systems *Journal of the American Dental Association* **128** 573-581.

Leinfelder KF, Radz GM & Nash RW (1998) A report on a new condensable composite resin *Compendium of Continuing Education in Dentistry* **19** 230-237.

Mazzaoui SA, Burrow MF & Tyas MJ (2000) Fluoride release from glass ionomer cements and resin composites coated with a dentin adhesive *Dental Materials* **16** 166-171.

Mathis RS & Ferracane JL (1989) Properties of glass-ionomer/resin-composite hybrid material *Dental Materials* **5** 355-358.

Mjör IA (1981) Placement and replacement of restorations *Operative Dentistry* **6(2)** 49-54.

Momoi Y & Mc Cabe JF (1993) Fluoride release from light activated glass ionomer restorative cements *Dental Materials* **9** 151-154.

Murchison DF, Charlton DG & Moore WS (1999) Comparative radiopacity of flowable resin composites *Quintessence International* **30** 179-184.

Perrin C, Persin M & Sarrazin J (1994) A comparison of fluoride release from four glass-ionomer cements *Quintessence International* **25** 603-608.

Peng D, Smales RJ, Yip HK & Shu M (2000) *In vitro* fluoride release from aesthetic restorative materials following recharging with APF gel *Australian Dental Journal* **45** 198-203.

Posada A, Emilson CG & Birkhed D (2000) Fluoride release *in vitro* from a resin-modified glass ionomer after exposure to NaF solutions and toothpastes *Swedish Dental Journal* **24** 117-125.

Retief DH, Bradley EL, Denton JC & Switzer P (1984) Enamel and cementum fluoride uptake from a glass ionomer cement *Caries Research* **18** 250-257.

Rothwell M, Anstice HM & Pearson GJ (1998) The uptake and release of fluoride by ion-leaching cements after exposure to toothpaste *Journal of Dentistry* **26** 591-597.

Skartveit L, Tveit AB, Totdal B, Ovrebo R & Raadal M (1990) *In vivo* fluoride uptake in enamel and dentine from fluoride containing material *Journal of Dentistry for Children* **57** 97-100.

Suljak JP & Hatibovic-Kofman S (1996) A fluoride release-adsorption-release system applied to fluoride releasing restorative materials *Quintessence International* **27** 635-638.

Swift EJ (1989) Fluoride release from two composite resins *Quintessence International* **20** 895-897.

Strother JM, Kohn DH, Dennison JB & Clarkson BH (1998) Fluoride release and re-uptake in direct tooth colored restorative materials *Dental Materials* **14** 129-136.

Tam LE, Chan GPL & Yim D (1997) *In vitro* caries inhibition effects by conventional and resin-modified glass-ionomer restorations *Operative Dentistry* **22(1)** 4-14.

Vieira AR, De Souza IP & Modesto A (1999) Fluoride uptake and release by composites and glass ionomers in a high caries challenge situation *American Journal of Dentistry* **12** 14-18.

Vivadent (1998) Ariston pHc Scientific Documentation Vivadent Research and Development Scientific Service Vivadent Ivoclar Schaan Liechtenstein.

Yip HK & Smales RJ (1999) Fluoride release and uptake by aged resin-modified glass ionomers and a polyacid-modified resin composite *International Dental Journal* **49** 217-225.

Young A, Von Der Fehr FR, Sonju T & Nordbo H (1996) Fluoride release and uptake *in vitro* from a composite resin and two orthodontic adhesives *Acta Odontologica Scandinavica* **54** 223-228.

©Operative Dentistry, 2003, **28-4**, 403-414

Marginal Integrity of Pit and Fissure Sealants. Qualitative and Quantitative Evaluation of the Marginal Adaptation Before and After *In Vitro* Thermal and Mechanical Stressing

MM Stavridakis • V Favez • EA Campos • I Krejci

Clinical Relevance

The self-etching adhesive system used in this study proved to be as effective as phosphoric acid etching in the pretreatment of air-abraded enamel surface prior to sealant application. The high viscosity material performed equally well only when used in combination with the self-etching primer adhesive system as an intermediate layer. The halogen-curing unit led to better marginal adaptation than the plasma arc-curing unit, especially after thermal and mechanical stressing.

SUMMARY

This research quantitatively evaluated the marginal adaptation of pit and fissure sealants. The occlusal surfaces of 48 intact, caries-free human molars were cleaned with an air-abrasion unit. The teeth were then randomly divided into eight groups of six teeth each according to the type of enamel conditioning, sealant material applied and curing unit used. After applying either 40% phosphoric acid gel (K-etch, Kuraray Co) or a self-etching primer adhesive system (Clearfil SE Bond, Kuraray Co), sealant materials of two viscosities were applied (Teethmate F-1 and Protect-Liner-F, Kuraray Co) and cured with halogen (Optilux 500, Demetron) or plasma arc (Apollo-95E, Dental & Medical Diagnostic Systems, Ltd) curing units. The marginal adaptation of the pit and fissure sealant restorations was evaluated by using a computer-assisted quantitative margin analysis in a scanning electron microscope (SEM) on epoxy replicas before and after thermal and mechanical stressing of the teeth. The results were statistically analyzed with one-way analysis of variance (ANOVA) at a confidence level of 95% (p=0.05). A post-hoc Tukey HSD-test was used for multiple pairwise comparisons between groups. The null hypothesis was that there was no statistically significant difference between the groups that were tested in this study.

*Minos M Stavridakis, DDS, MS, dr med dent, visiting professor, Division of Cariology and Endodontology, School of Dentistry, University of Geneva, Geneva, Switzerland

Valerie Favez, DDS, teaching assistant, Division of Cariology and Endodontology, School of Dentistry, University of Geneva, Geneva, Switzerland

Edson A Campos, DDS, MS, dr med dent, professor of Operative Dentistry, Department of Restorative Dentistry, Faculty of Dentistry of Barretos, Barretos, Brazil and visiting professor, Division of Cariology and Endodontology, School of Dentistry, University of Geneva, Geneva, Switzerland

Ivo Krejci, DDS, dr med dent, PD, professor and chairman, Division of Cariology, Endodontology and Pediatric Dentistry, School of Dentistry, University of Geneva, Geneva, Switzerland

*Reprint request: 19, Rue Barthélemy-Menn, 1205 Geneva, Switzerland; e-mail: minosstauridakis@yahoo.com

The statistically significant differences between groups were more pronounced after loading. In most cases, the self-etching adhesive system (SE Bond) proved as effective as phosphoric acid etching (K-etch). The low viscosity sealant material (Teethmate F-1), in most cases, exhibited better marginal adaptation than the high viscosity material (Protect-Liner F). The high viscosity material performed equally well only when used in combination with the self-etching primer adhesive system as an intermediate layer. The halogen curing unit (Optilux 500) led to better marginal adaptation than the plasma arc curing unit (Apollo 95E), especially after thermal and mechanical stressing.

INTRODUCTION

Dentistry's primary objective today is one of preventing dental disease rather than curing it (Tandon, Kumari, & Udupa, 1989). Pit and fissure sealants were introduced almost 35 years ago as an individual preventive method for controlling caries (Cueto & Buonocore, 1967), and their success is based on the adhesion between the sealant material and enamel due to the mechanical interlock created by the acid-etch technique. Buonocore (1955) developed this technique, which consisted of etching the enamel surface with orthophosphoric acid. Etching enamel removes surface contaminants and increases surface energy, making it easily wettable and creates an irregular surface topography of micropores and microprojections (Lee, 1969). Resin then penetrates and polymerizes in the enamel micropores, thus forming a mechanical bond with the tooth (Buonocore, 1955). Cueto (Cueto & Buonocore, 1965) presented the first report of a clinical trial using an occlusal sealing technique. Thereafter, several researchers have investigated the effectiveness of pit and fissure sealants in preventing occlusal caries in observation periods ranging from one to 15 years after application (Mertz-Fairhurst & others, 1991; Mertz-Fairhurst & others, 1995).

Conditioning enamel with phosphoric acid is the standard method for preparing the enamel surface prior to bonding sealant materials (Buonocore, Matsui & Gwinnett, 1968; Buonocore, 1955). The goals of enamel conditioning are to clean enamel, remove the enamel smear layer (when rotary instrumentation is being used), increase microscopic roughness by removing prismatic and interprismatic mineral crystals and increase the surface energy of enamel in order to produce enough monomer infiltration to guarantee the retention of resin material (Busscher, Retief & Arends, 1987; Retief, 1975). During the last five decades, many materials and different approaches have been investigated as alternatives to phosphoric acid, which was first introduced as a conditioner for hard dental tissues in a quest to achieve perfect bonding to both enamel and dentin. In order to simplify the application of resin bonding systems to both enamel and dentin, the conditioning and priming steps were combined and self-etching primers developed. They are mainly dentin conditioners combined with a hydrophylic primer (Hasegawa & others, 1989; Chigira & others, 1989). On instrumented enamel, these self-etching primers proved to be an effective alternative to conventional phosphoric acid etchants in conditioning the enamel surface in order to secure a durable bonding and marginal seal of composite resin restorations (Yoshiyama & others, 1998; Perdigão & others, 1997).

The first objective of this research was to investigate the efficacy of a self-etching adhesive system in preparing the air-abraded enamel surface prior to sealant material application. Even though most of the research with self-etching primers has been targeted on dentin adhesion, there are many indications that they can provide an effective alternative to conventional phosphoric acid etchants in conditioning the enamel surface and securing a durable bonding and marginal seal of resin restorations (Inagaki & others, 1989; Hasegawa & others, 1989). These simplified systems, by decreasing the time and steps required for placement, are convenient to use, but their efficacy has not been investigated in pit and fissure sealants.

The second objective of this research was to investigate the effect of the viscosity of the sealant material on its ability to perfectly seal pits and fissures, since contradictory reports exist in the literature. Filled sealants are preferred by some clinicians as they are more wear-resistant than unfilled sealants (Strang & others, 1986), even though a three-year clinical study showed that an unfilled light-cured resin was significantly better retained than a filled light-cured resin (Brockmann, Scott & Eick, 1989). Viscosity and flow characteristics have been reported to have no effect on the sealing ability of sealants (Low, Lee & von Fraunhofer, 1978; Barnes & others, 2000). On the other hand, Irinoda and others (2000) reported that a low viscosity sealant penetrated fully into etched enamel, whereas, the high viscosity sealants did not penetrate enough to ensure that the acid-etched enamel was sufficiently infiltrated by the sealant.

The third objective of this research was to compare the efficacy of conventional halogen curing units and plasma arc curing units when used to cure pit and fissure sealants. Within the last few years, several new polymerization concepts ("softstart" polymerization, two-step and ramped/exponential polymerization modes) and curing units (high intensity halogen curing units, plasma arc curing units, blue LED curing units and argon lasers) were introduced to the dental profession. Plasma arc curing units with high intensities and

short exposure times (one-to-three seconds), if proved efficient, would help to reduce chairtime. This would be very useful in pediatric patients, especially when multiple pit and fissure sealant applications need to be performed in one appointment, as often occurs in school-based dental public health preventive programs.

This research quantitatively evaluated the marginal adaptation of pit and fissure sealants. Human enamel cleaned by air abrasion was conditioned with phosphoric acid or a self-etching primer adhesive system and sealant materials of two viscosities were applied, then cured with halogen or plasma arc curing units. The null hypothesis was that there was no statistically significant difference between the groups tested in this study.

METHODS AND MATERIALS

Forty-eight intact, caries-free human molars with completed root formation were used in this study. The teeth had been stored in 0.1% thymol solution for the time between extraction and use in this *in vitro* test. The absence of caries was determined according to clinical parameters, using visual inspection. Only teeth without white or brown lesions that, in addition, did not exhibit cavitated lesions, including microcavities and cavities exposing dentin, were selected. After scaling and pumicing the root surface, the apices of the teeth were sealed with two coats of nail varnish and mounted on custom made specimen holders, with their roots in the center, using a cold-polymerizing resin (Paladur, Kulzer & Co, Wehrheim, Germany). The occlusal surface of all teeth was then cleaned with the help of an air-abrasion unit (AirFlow prep K1, Electro Medical Systems SA, Nyon, Switzerland) that used a high-speed stream of 25 microns aluminum oxide particles that were propelled at 4.5 bars pressure. The aim was only to clean the occlusal surface of contaminants without removing a significant amount of enamel as in the case when air abrasion is used for microcavity preparation. The use of air abrasion was chosen to clean the occlusal surface since pumice slurry and prophylactic pastes, which are often used to clean the occlusal surfaces prior to acid etching, do not completely and consistently remove debris from pits and fissures. It was of upmost importance that all debris be removed not only from the cuspal inclines (as often is the case when pumice slurry and prophylactic pastes are used), but from the complete depth of the occlusal pit and fissures, as the internal adaptation of the pit and fissure sealants would also be investigated in a subsequent part of this research

Table 1: *Ingredients, Lot Numbers and Expiration Dates of the Materials Used*

Material	Ingredients	LOT #	Expiration Date
K-etch	Phosphoric acid (40%) Colloidal silica Water	213	2000-11
Clearfil SE Bond[a]		41120	2000-09
Primer	MDP HEMA Hydrophilic dimethacrylate CQ N, N-Diethanop p-toluidine Water	00112B	2001-10
Bond	MDP BIS-GMA HEMA Hydrophobic dimethacrylate CQ N, Diethanop p-toluidine Silanized colloidal silica	00049A	2001-10
Teethmate F-1	Hydrophobic dimethacrylate TEG-DMA MDP HEMA CQ	00809	2000-11
Protect-Liner F	Silanized colloidal silica Prepolymerized organic filler containing colloidal silica BIS-GMA TEG-DMA Methacryloyl fluoride-methyl methacrylate copolymer CQ	0040C	2001-02

[a]*Also marketed as Clearfil Mega Bond in Japan.*
Abbreviations: BIS-GMA; BIS-phenol-A-glycidylmethacrylate; CQ: di-Camphoroquinone; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; TEG-DMA: Triethylene glycol dimethacrylate.

Table 2: *Group Parameters Used in the Study of Marginal Adaptation of Pit and Fissure Sealants*

Group	Conditioning	Material	Polymerization
1 (PLH)	**P:** K-etch	**L:** Teethmate F1	**H:** Optilux 500
2 (SLH)	**S:** SE Bond	**L:** Teethmate F1	**H:** Optilux 500
3 (PHH)	**P:** K-etch	**H:** Protect-Liner F	**H:** Optilux 500
4 (SHH)	**S:** SE Bond	**H:** Protect-Liner F	**H:** Optilux 500
5 (PLP)	**P:** K-etch	**L:** Teethmate F1	**P:** Apollo 95E
6 (SLP)	**S:** SE Bond	**L:** Teethmate F1	**P:** Apollo 95E
7 (PHP)	**P:** K-etch	**H:** Protect-Liner F	**P:** Apollo 95E
8 (SHP)	**S:** SE Bond	**H:** Protect-Liner F	**P:** Apollo 95E

project. The occlusal surfaces were then examined under a stereomicroscope (Leica MZ6, Leica Microsystems AG, Wetzlar, Germany) at 4x magnification in order to check that no part of the occlusal surface was left without being air abraded and that all pits and fissures were clean of visible debris. The teeth were then randomly divided into eight groups of six teeth each based on the type of enamel conditioning, sealant material applied and curing unit used. The ingredients, lot numbers and expiration dates of the materials used in this study are reported in Table 1. The group parameters of the marginal adaptation of pit and fissure sealants used in the study are shown in Table 2.

Group 1 (PLH): The first letter (**P**) in the group's acronym stands for Phosphoric acid and identified the treatment of enamel prior to sealant material application. A 40% phosphoric acid gel (K-etch, Kuraray Co, Osaka, Japan) was applied with a disposable microbrush for 40 seconds in order to condition the enamel surface. The teeth were rinsed with water and air spray, using a dental syringe and thoroughly dried with oil-free compressed air. The second letter (**L**) in the group's acronym stands for Low viscosity, which identifies the viscosity of the sealant material. A low viscosity sealant material (Teethmate F-1, Kuraray Co) was applied with the manufacturer's disposable applicator nozzle, and the tip of an explorer was used to ensure that all pit and fissures were properly sealed. The sealant material was left intact for 20 seconds prior to polymerization to allow for a proper capillary action of resin infiltration. The third letter (**H**) in the group's acronym stands for Halogen curing unit, which identifies the type of curing unit used. The sealant material was light cured for 20 seconds with a halogen curing unit (Optilux 500, Demetron Research Corp, Danbury, CT, USA). The 11-mm curing tip was used to cover the entire occlusal surface in the majority of the teeth. Occasionally, when the occlusal surface of the tooth was too large, polymerization was performed for 20 seconds twice, once on the mesial and once on the distal side of the occlusal surface.

Group 2 (SLH): The first letter (**S**) in the group's acronym stands for Self-etching primer and identifies the treatment of enamel prior to applying the sealant material. A self-etching primer adhesive system (Clearfil SE Bond, Kuraray Co) was used to prepare the enamel surface. Clearfil SE Bond primer was applied to the entire occlusal surface with a disposable microbrush and left in place for 40 seconds before the volatile ingredients were evaporated with a mild oil-free air stream. Then, the Clearfil SE Bond adhesive resin (bond) was applied into the occlusal pit and fissures, also with a disposable microbrush, and left to penetrate for 10 seconds. With the help of a gentle oil-free air stream, the bond film was thinned before it was light-cured for 10 seconds (Optilux 500, Demetron). The same low viscosity sealant material (Teethmate F-1, Kuraray Co) was applied, and polymerization with the halogen-curing unit was performed, as previously described.

Group 3 (PHH): The 40% phosphoric acid gel (K-etch, Kuraray Co) was used to treat the enamel prior to applying the sealant material, as described in Group 1. The second letter (**H**) in the group's acronym stands for High viscosity, which identifies the viscosity of the sealant material. A high viscosity sealant material (Protect-Liner F, Kuraray Co) was applied with the same disposable applicator nozzle that was provided by the manufacturer of the low viscosity sealant material. The tip of an explorer was also used to ensure that all pits and fissures were properly sealed. The sealant material was left to penetrate for 20 seconds, then it was light cured in the same manner as the two previous groups.

Group 4 (SHH): The self-etching primer adhesive system (Clearfil SE Bond) was used to treat the enamel prior to applying the sealant material, as described in Group 2. The same high viscosity sealant material (Protect-Liner F, Kuraray Co) was applied as in Group 3, and polymerization with the halogen curing unit was performed in the same way as the last three groups.

Group 5 (PLP): The 40% phosphoric acid gel (K-etch, Kuraray Co) was used to treat the enamel prior to applying the sealant material, and the low viscosity sealant material (Teethmate F-1, Kuraray Co) was applied in the sane way as Group 1. The sealant material was left to penetrate for 20 seconds prior to polymerization. The third letter (**P**) in the group's acronym stands for Plasma arc curing unit; it identified the type of curing unit used. The sealant material was light cured for three seconds with a plasma arc curing unit (Apollo 95E, Dental & Medical Diagnostic Systems, Ltd, Deurle, Belgium). The 7.6-mm curing tip rather infrequently covered the entire occlusal surface of the teeth. In a majority of the cases, polymerization was performed for three seconds twice, once on the mesial and once on the distal side of the occlusal surface.

Group 6 (SLP): The self-etching primer adhesive system (Clearfil SE Bond) was used to treat the enamel prior to applying the sealant material, and the low viscosity sealant material (Teethmate F-1, Kuraray Co) was applied in the same way as described in Group 2. Polymerization was performed with the plasma arc-curing unit, as previously described.

Group 7 (PHP): The 40% phosphoric acid gel (K-etch, Kuraray Co) was used to treat the enamel prior to applying the sealant material, and the high viscosity sealant material (Protect-Liner F, Kuraray Co) was applied in the same way as in Group 3. Polymerization was performed with the plasma-arc curing unit in the same manner as described in the previous two groups.

Group 8 (SHP): The self-etching primer adhesive system (Clearfil SE Bond, Kuraray Co) was used to treat the enamel prior to applying the sealant material, and the high viscosity sealant material (Protect-Liner F, Kuraray Co) was applied in the same way as Group 4. Polymerization was performed with the plasma arc-curing unit, as in the last three groups.

After dark storage in 0.9% NaCl normal saline solution at 37°C for one week, the sealed teeth were simultaneously loaded with repeated thermal and mechanical stresses in a chewing machine (Krejci, Albertoni & Lutz, 1990; Krejci & others, 1990). Thermal cycling was carried out by flushing water with temperatures changing 3000x from 5°C to 50°C and vice versa, with a dwelling time of two minutes each. The mechanical stress comprised 1,200,000 load cycles and was transferred on the center of the occlusal surface with a frequency of 1.67 Hz and a maximal load of 49 N. It was applied by using a natural lingual cusp in contact with the sealant's surface. The cusps that were used were taken from extracted human third molars.

Immediately upon completing the sealing procedure (from this point on referred to as *initial* stage) and after the thermal and mechanical stressing procedure (from this point on referred to as *terminal* stage), impressions of the occlusal surfaces of the teeth were made with a polyvinylsiloxane impression material (President light body, Coltène AG, Altstätten, Switzerland). Prior to the initial impression, in order to remove the unpolymerized surface layers of the sealing material and bond of the self-etching primer adhesive system due to the oxygen inhibition of the polymerization process, the occlusal surface of the tooth was cleaned for approximately five seconds with pumice slurry and a nylon bristle brush (miniature prophy brushes, Hawe Neos Dental, Bioggio, Switzerland). Subsequently, epoxy replicas (Epofix Kit, Struers, Rødovre, Denmark) were prepared for the computer-assisted quantitative margin analysis in a scanning electron microscope (Philips XL20, Eidhoven, Netherlands) at 200x magnification (Krejci, Kuster & Lutz, 1993; Krejci & others, 1990). The marginal micromorphology was evaluated for the following qualities: "continuous margin" and "non-continuous margin." The quality criterion "non-continuous margin" was further characterized with the criteria "marginal fissure," "enamel fracture" and "sealant fracture." The different marginal qualities were assessed in percent of the total length of margins analyzed, and the values were statistically analyzed with one-way analysis of variance (ANOVA) at a confidence level of 95% (p=0.05). A post-hoc Tukey HSD-test was used for multiple pairwise comparisons between groups.

RESULTS

The qualitative evaluation of the marginal adaptation of the pit and fissure sealants of this study revealed

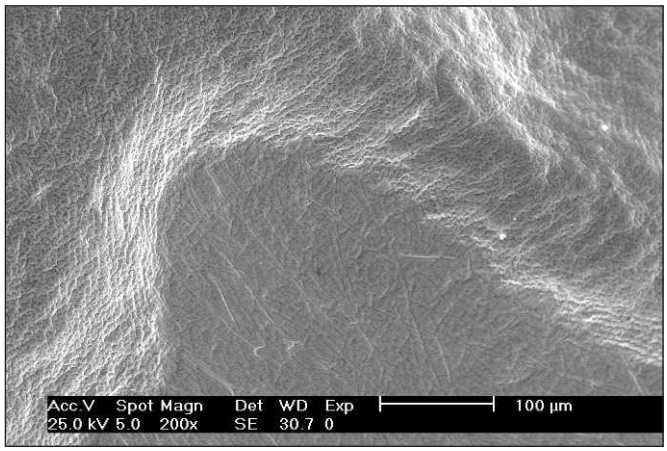


Figure 1. *A representative SEM photomicrograph of a "continuous margin," the sealing material exhibits excellent continuity with the enamel surface (200x magnification).*

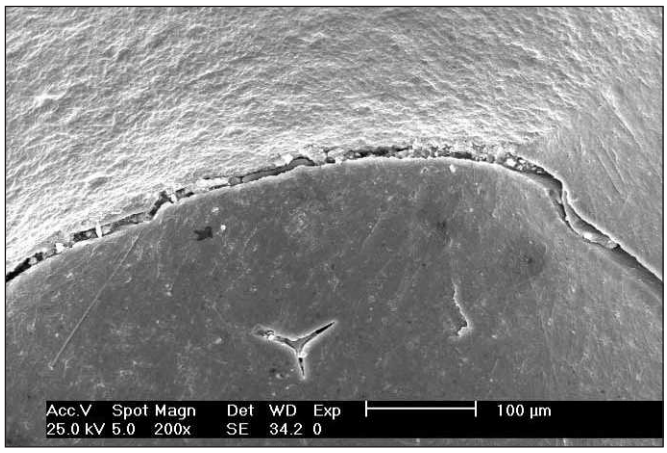


Figure 2. *A representative SEM micrograph of a "non-continuous margin" further characterized as "marginal fissure," the gap is localized at the enamel/sealant interface (200x magnification).*

that excellent marginal adaptation was feasible before and after thermal and mechanical stressing. A representative Scanning Electron Microscopy (SEM) micrograph of "continuous margin" is presented in Figure 1, where the sealing material exhibited excellent continuity with the enamel surface. Before thermal and mechanical stressing, only "marginal fissures" were encountered as "non-continuous" margins. After stressing, when discontinuity of the marginal adaptation was noted, a mixture of "marginal fissures," "sealant fractures" and "enamel fissures" was observed. A representative SEM micrograph of "marginal fissure" is presented in Figure 2, where the gap is localized at the enamel/sealant interface. The "sealant fractures" that were encountered were, in most instances, of a small magnitude and localized at the periphery of the margins of the sealants as illustrated in Figure 3. In the high viscosity sealant material groups that were polymerized with the plasma arc curing unit (Group 7

(PHP) and Group 8 (SHP) "sealant fractures" of greater magnitude, as illustrated in Figure 4, that extended over all the occlusal surface were encountered. "Enamel fractures" were scarcely observed, primarily around the occlusal contact points.

Table 3 summarizes the quantitative evaluation results of the marginal adaptation of the different groups of pit and fissure sealants tested in this study, where the mean percentage of "continuous margin" of each group before (initial) and after (terminal) thermal and mechanical stressing, and the statistical significance between groups are reported. Statistical analysis of the "non-continuous margin" results were not performed, as these percentages were complimentary to the percentages of "continuous margin." The results of the different types of "non-continuous margin" only at the terminal stage are reported in Table 4, since at the initial stage, only "marginal fissures" were encountered.

Table 3: *Marginal Adaptation Results of Pit and Fissure Sealants. Percentage of "Continuous Margin" Before (initial) and After (terminal) Thermal and Mechanical Stressing of the Sealants, and the Statistically Significant Differences Between Groups at Each Stage and Between Stages*

Group	Initial Mean ± SD		Terminal Mean ± SD		Statistical Significance (initial vs terminal)
1 (PLH)	99.8 ± 0.49	(A)	99.2 ± 0.53	(A)	NO
2 (SLH)	100.0 ± 0.00	(A)	95.1 ± 4.76	(A)	NO
3 (PHH)	95.7 ± 2.23	(A)	72.5 ± 10.05	(B)	YES
4 (SHH)	99.9 ± 0.36	(A)	94.5 ± 1.75	(A)	NO
5 (PLP)	83.1 ± 8.75	(A)	62.3 ± 7.52	(B)	YES
6 (SLP)	91.8 ± 4.45	(A)	47.5 ± 15.74	(C)	YES
7 (PHP)	88.8 ± 8.38	(A)	5.1 ± 4.92	(D)	YES
8 (SHP)	59.9 ± 29.9	(B)	5.9 ± 4.66	(D)	YES

In parenthesis, in addition to the mean and standard deviation values, the homogeneous subsets are marked at each stage (initial or terminal). Groups belonging to the same subset (have the same letter) do not have a statistically significant difference between them at the 0.05 level. The column on the right illustrates (when YES is noted) whether there was a statistically significant difference between the initial and terminal stage of each group..

Table 4: *Marginal Adaptation Results of Pit and Fissure Sealants. Percentage of Different Types of "Non-Continuous Margin" After (terminal) Thermal and Mechanical Stressing of the Sealants*

Group	"Marginal Fissure" Mean ± SD	"Sealant Fracture" Mean ± SD	"Enamel Fracture" Mean ± SD
1 (PLH)	0.8 ± 0.53		
2 (SLH)	1.7 ± 1.11	3.2 ± 4.35	0.1 ± 0.20
3 (PHH)	9.0 ± 5.99	18.3 ± 6.76	0.6 ± 0.65
4 (SHH)	3.3 ± 2.14	2.2 ± 3.14	0.2 ± 0.60
5 (PLP)	29.5 ± 5.46	8.2 ± 3.11	
6 (SLP)	39.7 ± 16.62	12.3 ± 5.31	4.2 ± 4.13
7 (PHP)	6.0 ± 5.14	89.0 ± 7.76	1.9 ± 2.11
8 (SHP)	15.5 ± 10.90	78.7 ± 14.73	0.8 ± 1.40

Occasionally, different types of "non-continuous margin" may have co-existed in the same region. This is why the sum of the percentages of different types of "non-continuous margin" is sometimes greater than the complimentary number of the "continuous margin" percentages reported in Table 3.

In most cases, the self-etching adhesive system (Clearfil SE Bond) proved to be as effective as phosphoric acid etching (K-etch) in the pretreatment of the enamel surface prior to sealant application. Statistically significant differences in pairwise comparisons with phosphoric acid etching were observed in three situations. In two of them (initial: Group 7 (PHP) versus Group 8 (SHP) and terminal: Group 5 (PLP) versus Group 6 (SLP)), where the plasma arc curing unit was used, the phosphoric acid etching groups exhibited better marginal adaptation. In both self-etching primer adhesive system groups (initial: Group 8 (SHP) and terminal: Group 6 (SLP)), there was one sample in each group that behaved very poorly, which increased the standard deviations of these groups. In the third situation, (terminal: Group 3 (PHH) versus Group 4 (SHH)), when the halogen-curing unit was used, it was the self-etching primer adhesive system group that produced better marginal adaptation.

The low viscosity sealant material (Teethmate F1), in most cases, exhibited better marginal adaptation than when the high viscosity material was used (Protect-Liner F). The statistically significant differences in the pairwise comparisons between the low and high viscosity sealant materials were in favor of the low viscosity sealant material in all four situations where they were observed (initial: Group 6 (SLP) versus Group 8 (SHP); terminal: Group 1 (PLH) versus Group 3 (PHH), Group 5 (PLP) versus Group 7 (PHP), and Group 6 (SLP) versus Group 8 (SHP). In only one case did the high-viscosity sealant material exhibit better marginal adaptation (initial: Group 5 (PLP) versus Group 7 (PHP)), however, the difference was not statistically significant.

The halogen-curing unit (Optilux 500) generally led to better marginal adaptation than the plasma-arc curing unit (Apollo 95E). The differences were statistically significant in all except three pairwise comparisons (initial: Group 1 (PLH)) versus Group 5 (PLP), Group 2 (SLH) versus Group 6 (SLP) and Group 3 (PHH) versus Group 7 (PHP).

The statistically significant differences between groups were more pronounced in the terminal stage. Prior to stressing, there were just two subsets with statistically significant differences between them, and seven out of eight groups had percentages that exceeded 80% of excellent marginal adaptation. After stressing, there were four subsets with statistically significant differences between them. The variations in the percentage of excellent marginal adaptation were large, as the best subset exceeded 90% of "continuous margin," while the worst exhibited less than 10% of "continuous margin."

Three out of four groups in which the halogen curing unit was used (Group 1 (PLH)), Group 2 (SLH) and Group 4 (SHH) did not exhibit statistically significant differences between the initial and terminal situation. Thermal and mechanical stressing caused a statistically significant decrease in the marginal adaptation only in one of the halogen curing unit groups (Group 3 (PHH)).

All groups in which the plasma arc curing unit was used (Group 5 (PLP)), Group 6 (SLP), Group 7 (PHP) and Group 8 (SHP) exhibited statistically significant differences for the criterion "continuous margin" between initial and terminal situation. In all of these groups, after thermal and mechanical stressing, there was a statistically significant decrease in the percentage of "continuous margin."

DISCUSSION

The results of this study support the use of self-etching primers as effective means for preparing air-abraded enamel for bonding of a fissure sealant. Air abrasion removes the outer enamel layer of a few microns thickness and induces an additional micro-etching pattern (Figure 5) that could enhance the efficacy of the self-etching primers. Removing this small layer of external enamel may have contributed to the efficacy of the self-etching adhesive system in conditioning the enamel surface prior to applying the sealant material. The acidic, self-etching primer was capable of creating an etching pattern on the enamel surface (Figure 6) which was less profound than when phosphoric acid was used (Figure 7). The results of this research cannot support the efficacy of the self-etching primer that was studied on non-prepared intact enamel, since the surface on which it was applied had been previously air abraded. This does not diminish the value of the results of this research project since air abrasion was performed in order to thoroughly clean the enamel surface from plaque, pellicle and other contaminants (Figure 8) and not to facilitate the action of self-etching primer, such as prophylactic pastes and pumice slurry, which are often used to clean the occlusal surfaces prior to acid etching and do not completely and consistently remove debris from pits and fissures (Figure 9).

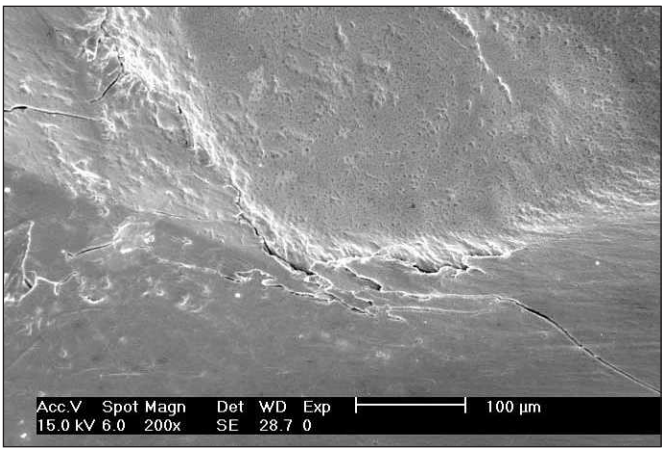


Figure 3. *A representative SEM micrograph of a "non-continuous margin" further characterized as "sealant fracture," this small magnitude gap is localized at the periphery of the margin inside the mass of the sealing material (200x magnification).*

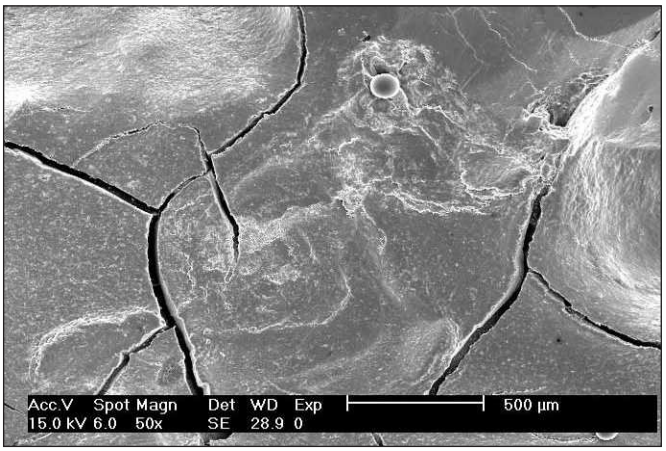


Figure 4. *A representative SEM micrograph of a "non-continuous margin" further characterized as "sealant fracture," these larger magnitude gaps are located over the whole occlusal surface inside the mass of the sealing material (50x magnification).*

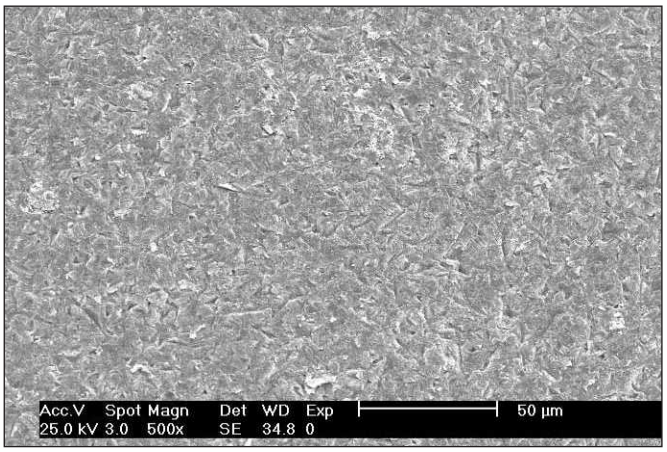


Figure 5. *A SEM micrograph of the occlusal surface that was cleaned with air abrasion. A micro-etching pattern is evident (500x magnification).*

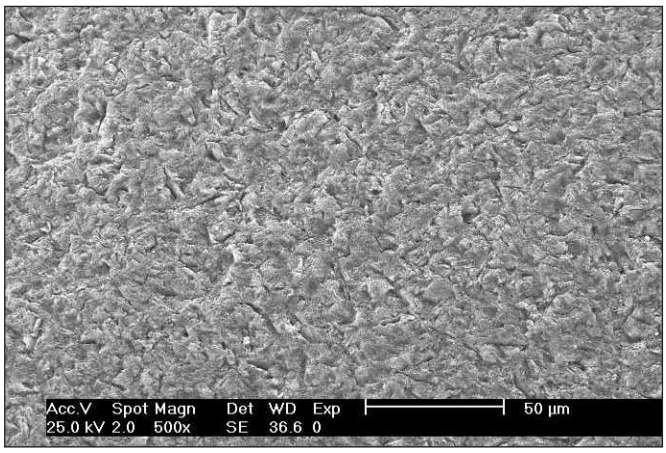


Figure 6. *A SEM micrograph of etched enamel of the occlusal surface, where the surface contaminants were removed with air abrasion prior to acidic monomer solution application (SE Primer) for 40 seconds. A less profound etching pattern, when compared to that of phosphoric acid, is evident after the application of the self-etching primer (500x magnification).*

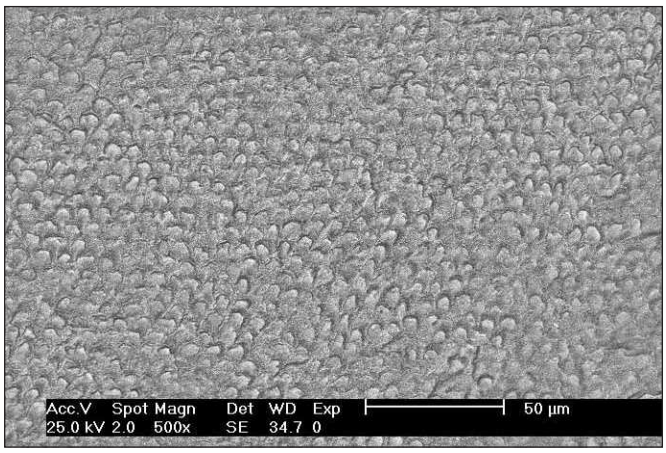


Figure 7. *A SEM micrograph of etched enamel of the occlusal surface, where the surface contaminants were removed with air abrasion prior to 40% phoshoric acid application for 40 seconds. The classic etching pattern of phosphoric acid etching is evident (500x magnification).*

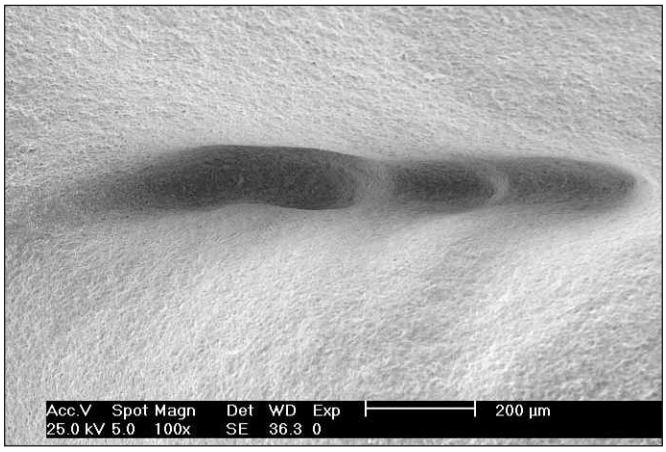


Figure 8. *A SEM micrograph of the occlusal surface that was cleaned with air abrasion. The surface contaminants were thoroughly removed from the entrance of the occlusal fissure (100x magnification).*

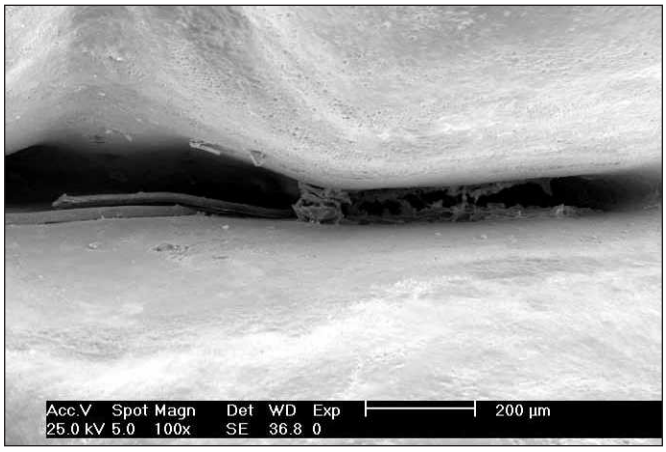


Figure 9. *A SEM micrograph of the occlusal surface after the application of prophlylactic paste with a rotary bristle brush. The deepest part of the fissure is not perfectly cleaned and bristles and debris are entrapped inside the fissure (100x magnification).*

It is also important to note that the self-etching primer was not applied alone, but the bond of the adhesive system was also applied according to the manufacturer's recommendations. Therefore, the true comparison between phosphoric acid and self-etching primers was not actually performed, even though there are strong indications to support the results of this research that self-etching primers may perform equally well. Currently, there are ongoing investigations where the self-etching primer was solely applied (without the bond) in order to compare phosphoric acid etching versus self-etching primer conditioning. Another issue is the fact that these positive results cannot be extrapolated to other invasive techniques, such as mechanical widening of the fissures with rotary instrumentation or when lasers are applied, unless the necessary research is performed.

The clinical consequences of etching enamel with weaker acids, and the presence of dissolved calcium phosphates and/or aluminum oxide particles that cannot be removed with rinsing which are then entrapped in the bonding resin layer are issues that certainly need to be thoroughly investigated. A recent, long-term *in vivo* study of the durability of self-etching primer bonded restorations suggested that the bond failure of the aged interfaces occurred through the loss of resinous material that probably occurred through water sorption and hydrolysis of the hydrophylic resin components (Sano & others, 1999). Therefore, long-term clinical longitudinal studies, where retention rates and mar-

ginal adaptation evaluation are examined, are needed in order to further investigate the positive results of this *in vitro* study, in which the self-etching adhesive system that was used proved to be as effective as phosphoric acid in conditioning air-abraded enamel prior to applying sealant.

The low viscosity sealant material exhibited better marginal adaptation than its high viscosity counterpart. Busscher and others (1987) suggested that a time-delay be introduced between the time of application and light activation when curing light-cured sealants to allow for a proper capillary action of resin infiltration. This is not related to viscosity but rather to diffusion kinetics as driven by the 2nd Fick's law. Nevertheless, the highly filled viscous sealant material performed equally well when used in combination with the self-etching primer adhesive system as an intermediate layer. The use of an intermediate bonding agent has been proposed in order to provide better flow of thick and viscous highly filled sealants which are difficult to spread into small fissures. Simonsen (1982) proposed using an unfilled resin as an intermediate layer under a filled sealant when placing a small preventive resin restoration. The bonding agent serves as a low-viscosity, flowable wetting agent for the interface between etched enamel and filled sealant, so that the viscous sealant spreads better and properly wets the surface of the fissures (Symons, Chu & Meyers, 1996; Davenport & Feigal, 1997). The results of this research seem to agree with this concept, since the high viscosity sealant material, when used in combination with the adhesive system and cured with the halogen curing unit (Group 4 (SHH)), resisted the thermal and mechanical stressing in a more favorable manner than when applied in etched enamel without the intermediate bonding layer (Group 3 (PHH)). Nevertheless, studies on marginal adaptation, alone, are not enough to substantiate the claim that an intermediate bonding agent provides better flow for highly filled sealants with a higher viscosity. Internal adaptation studies currently underway are definitely required before such a claim can be supported.

The plasma arc-curing unit did not prove its efficacy in providing excellent marginal adaptation after thermal and mechanical stressing. The efficacy of plasma arc curing units has been investigated in orthodontic bonding, where saving time is of major importance since numerous brackets need to be bonded in one appointment. There are reports that support the use of plasma arc curing units as advantageous alternatives to halogen curing units for orthodontic bonding (Ishikawa & others, 2001; Sfondrini & others, 2001). Even though one- and two-seconds exposure periods of plasma arc light were found incapable to produce adequate bonding, three-second exposure periods produced results similar to 20 seconds of halogen light (Pettemerides, Ireland & Sherriff, 2001). The results of this research suggest that one three-second exposure was not sufficient to cure the sealing materials and the adhesive system that were used. Six or nine second exposures have been reported as necessary to create bond strengths equal to those produced by exposure to 40 seconds of tungsten-quartz halogen light (Oesterle, Newman & Shellhart, 2001). If several polymerization cycles were used, the results of this study might have been different. Nevertheless, even if a great number of cycles were proven to be necessary, keeping in mind that most often two polymerization cycles were needed due to the limited diameter of the curing tip, this would make the main advantage of plasma-arc curing units, which is short time curing periods, obsolete and their higher cost would not be justified.

One explanation for the poor results might be the narrow spectrum of the emitted wavelength that might have been incompatible with the materials used in this study. Most composites are activated by the camphoroquinone/amine system for which maximum absorption occurs at 468 nm. Materials using photoinitiators that have absorption maxima lower than camphoroquinone (468 nm) may not be properly activated by the plasma-arc curing unit used (Stansbury, 2000). Apollo 96E produces considerably more intensity over a rather narrow wavelength range around this value and is more specific to the activation of camphoroquinone. However, the very short exposure period used offers low total irradiation energy to the system, which leads to incomplete conversion, especially at indepth regions (Nomoto, 1997).

Another explanation for the poor results may lie in the kinetics of polymerization for the camphoroquinone/amine system. It has been reported that relatively high exposure intensities and short curing times may result in compromised properties in the cured resin, as they may result in an increased early termination of polymer chains in the pre-gel polymerization period (Koliniotou-Kubia & Jacobsen, 1990; Kelsey & others, 1992). Munksgaard, Peutzfeldt and Asmussen (2000) reported that the elution of BISGMA and TEGDMA from experimental resin specimens and commercially available resin composite specimens cured with a plasma arc light unit (three seconds) was seven and four times higher, respectively, compared to elution from specimens cured with a halogen unit (40 seconds). This high amount of leachable monomers in a resinous material may indicate poor conversion of monomer to polymer and, consequently, poor mechanical properties. Plasma arc curing units have been reported to result in resin composite materials with inferior mechanical properties (surface hardness, flexural strength and modulus of elasticity [Sharkey & others, 2001; Hofmann & others, 2001]). Nevertheless, there is one report in the literature where a plasma arc curing unit sufficiently polymerized pit and fissure sealants and

produced restorations without microleakage (Stritikus & Owens, 2001). In that study, a different sealing material was applied, then cured with a different plasma arc curing unit for a longer curing period, and the sealant restorations were thermocycled for only 200 cycles, while no mechanical load was applied. It is possible that the poor results obtained in this research may be due to incompatibility between the materials used and the plasma-arc curing unit and should not be generalized to all materials and all plasma-arc curing units. As newer generations of plasma-arc curing units are introduced into the dental profession, they need to be evaluated with different pit and fissure sealants.

Due to the "coastline" periphery of the margins of pit and fissure sealants, it is nearly impossible or of little scientific value to conduct marginal adaptation evaluation with the help of microphotographs where the margin is simply counted as open or closed in certain predetermined areas, and the magnitude of the marginal discrepancy is recorded in terms of microns. In this study, the quality of the marginal adaptation of the pit and fissure sealants was evaluated with a computerized quantitative technique that was first described by Krejci, Lutz and Loher (1991) in the entire enamel-sealant external interface and not in a few locations of the periphery. Another advantage of this evaluation method is that it was a non-destructive technique. By working with epoxy replicas, evaluation of the marginal adaptation, both before and after mechanical and thermal stressing, was performed on the same pit and fissure sealant restoration.

In other destructive kinds of evaluation techniques, such as microleakage studies, often, each specimen has to be dissected in order to be evaluated. Because of that, most often, evaluation is performed only after the stressing (most often only thermocycling) procedure in order to reduce the number of specimens. In the case of poor results, it is not possible to differentiate whether the failure was caused by the stressing procedure or if the sealing procedure did not perform adequately from the beginning. With this research methodology, if the same specimens were evaluated before and after thermal and mechanical stressing, any deterioration of the marginal adaptation could only be the consequence of the stressing procedure.

Another point that needs to be emphasized is the usefulness of the stressing procedure as it helped to depict which groups performed favorably. Before stressing, the majority of the groups performed equally well, while after stressing, there were only a few groups that exhibited excellent marginal adaptation. After stressing, there were four, instead of two, subgroups that exhibited statistically significant differences between them, while the comparison between the initial and terminal stage also revealed some interesting points. Although fissure sealants are not recommended for occlusal contact (Heinrich-Weltzien & Kühnisch, 1999), in many clinical cases, this is difficult to realize. The stressing conditions applied in this *in vitro* study did not exceed the physiological range. The temperature of the water used for thermocycling (Palmer, Barco & Billy, 1992) and the chewing forces (Bates, Stafford & Harrison, 1975) were chosen according to physiologic data. The high number of load cycles represent a clinical service time of about five years (Krejci & Lutz, 1990). Therefore, the results of this study may be clinically more relevant in predicting long-term behavior than shear bond strength on flat enamel surfaces where no attempt is made to simulate cavity geometry or investigations where only thermal cycling or only short mechanical loading with extremely high chewing forces were applied (Krejci & others, 1994).

CONCLUSIONS

The self-etching primer adhesive system used in this study proved to be as effective as phosphoric acid in preparing air-abraded enamel for fissure sealing. The low viscosity sealant material exhibited better marginal adaptation than its high viscosity counterpart. The highly filled viscous sealant material performed equally well only when used in combination with the self-etching primer adhesive system as an intermediate layer, which probably played the role of a low-viscosity, flowable wetting agent. The halogen-curing unit resulted in better marginal adaptation than the plasma-arc curing unit, especially after thermal and mechanical stressing. Ongoing *in vitro* research that is currently underway will try to address some of the issues raised by this research project, while at the same time, long-term clinical studies are certainly needed in order to confirm the results of this *in vitro* study.

Acknowledgements

The authors express their gratitude to Marie-Claude Reymond for her most valuable technical assistance. Product support for this study was kindly provided by Kuraray Co (Osaka, Japan).

(Received 12 July 2002)

References

Barnes DM, Kihn P, von Fraunhofer JA & Elsabach A (2000) Flow characteristics and sealing ability of fissure sealants *Operative Dentistry* **25** 306-310.

Bates JF, Stafford GD & Harrison A (1975) Masticatory function: A review of the literature. II. Speed of movement of the mandible, rate of chewing and forces developed in chewing *Journal of Oral Rehabilitation* **2** 349-361.

Brockmann SL, Scott RL & Eick JD (1989) The effect of an air-polishing device on tensile bond strength of a dental sealant *Quintessence International* **20** 211-217.

Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34** 849-853.

Buonocore MG, Matsui A & Gwinnett AJ (1968) Penetration of resin dental materials into enamel surfaces with reference to bonding *Archives of Oral Biology* **13(1)** 61-70.

Busscher HJ, Retief DH & Arends J (1987) Relationship between surface-free energies of dental resins and bond strengths to etched enamel *Dental Materials* **3** 60-63.

Chigira H, Koike T, Hasegawa T, Itoh K, Wakumoto S & Hayakawa T (1989) Effect of the self etching dentin primers on the bonding efficacy of a dentin adhesive *Dental Materials Journal* **8** 86-92.

Cueto EI & Buonocore MG (1965) Adhesive sealing of pits and fissures for caries prevention *Journal of Dental Research* **43** Abstract #400 p 137.

Cueto EI & Buonocore MG (1967) Sealing of pits and fissures with an adhesive resin: Its use in caries prevention *Journal of the American Dental Association* **75(1)** 121-128.

Davenport S & Feigal RJ (1997) Contact angle and penetrativity of bonding agents effective in wet-bonding *Journal of Dental Research* **76** Abstract #425 p 67.

Hasegawa T, Manabe A, Itoh K & Wakumoto S (1989) Investigation of self-etching dentin primers *Dental Materials* **5** 408-410.

Heinrich-Weltzien R & Kühnisch J (1999) [Häufigkeit und Qualität der Fissurenversiegelung bei 8- und 14jöhrigen] *Prophylaxe Impuls* **1** 6-14.

Hofmann N, Hugo B, Schubert K & Klaiber B (2001) Comparison between a plasma arc light source and conventional halogen curing units regarding flexural strength, modulus, and hardness of photoactivated resin composites *Clinical Oral Investigations* **4** 140-147.

Inagaki A, Chigira H, Itoh K & Wakumoto S (1989) Effects of self-etching primers on dentin *Dental Materials* **5** 403-407.

Irinoda Y, Matsumura Y, Kito H, Nakano T, Toyama T, Nakagaki H & Tsuchiya T (2000) Effect of sealant viscosity on the penetration of resin into etched human enamel *Operative Dentistry* **25** 274-282.

Ishikawa H, Komori A, Kojima I & Ando F (2001) Orthodontic bracket bonding with a plasma-arc light and resin-reinforced glass ionomer cement *American Journal of Orthodontics and Dentofacial Orthopedics* **120** 58-63.

Kelsey WP, Blankenau RJ, Powell GL, Barkmeier WW & Stormberg EF (1992) Power and time requirements for use of the argon laser to polymerize composite resins *Journal of Clinical Laser Medicine & Surgery* **10** 273-278.

Koliniotou-Kubia E & Jacobsen PH (1990) The effect of irradiation time on the physical properties of light-cured resins *Clinical Materials* **6** 21-28.

Krejci I, Reich T, Lutz F & Albertoni M (1990) An *in-vitro* test procedure for evaluating dental restoration systems 1 A computer-controlled mastication simulator *Schweizerische Monatsschrift für Zahnahilkunde* **100** 953-960.

Krejci I, Hausler T, Sagesser & Lutz F (1994) New adhesives in Class V restorations under combined load and simulated dentinal fluid *Dental Materials* **10** 331-335.

Krejci I, Albertoni M & Lutz F (1990) An *in-vitro* test procedure for evaluating dental restoration systems 2 Toothbrush/toothpaste abrasion and chemical degradation *Schweizerische Monatsschrift für Zahnahilkunde* **100** 1164-1168.

Krejci I, Kuster M & Lutz F (1993) Influence of dentinal fluid and stress on marginal adaptation of resin composites *Journal of Dental Research* **72** 490-494.

Krejci I & Lutz F (1990) *In-vitro* test results of the evaluation of dental restoration systems. Correlation with *in-vivo* results *Schweizerische Monatsschrift für Zahnahilkunde* **100** 1445-1449.

Krejci I, Lutz F & Loher CE (1991) Quantitative *in-vivo* evaluation of four restorative concepts for mixed Class V restorations *Quintessence International* **22** 455-465.

Lee HL Jr (1969) Adhesion between living tissue and plastics I Adhesion of epoxy and polyurethane resins to dentin and enamel *Journal of Biomedical Materials Research* **3(2)** 349-367.

Low T, Lee KW & von Fraunhofer JA (1978) The adaptation of composite materials to etched enamel surfaces *Journal of Oral Rehabilitation* **5** 349-355.

Mertz-Fairhurst EJ, Williams JE, Pierce KL, Smith CD, Schuster GS, Mackert JR Jr, Sherrer JD, Wenner KK, Richards EE & Davis QB (1991) Sealed restorations: 4-year results *American Journal of Dentistry* **4** 43-49.

Mertz-Fairhurst EJ, Adair SM, Sams DR, Curtis JW Jr, Ergle JW, Hawkins KI, Mackert JR Jr, O'Dell NL, Richards EE & Rueggeberg F (1995) Cariostatic and ultraconservative sealed restorations: Nine-year results among children and adults *ASDC Journal of Dentistry Child* **62** 97-107.

Munksgaard EC, Peutzfeldt A & Asmussen E (2000) Elution of TEGDMA and BISGMA from a resin and a resin composite cured with halogen or plasma light *European Journal of Oral Sciences* **108** 341-345.

Nomoto R (1997) Effect of light wavelength on polymerization of light-cured resins *Dental Materials Journal* **16** 60-73.

Oesterle LJ, Newman SM & Shellhart WC (2001) Rapid curing of bonding composite with a xenon plasma arc light *American Journal of Orthodontics and Dentofacial Orthopedics* **119** 610-616.

Palmer DS, Barco MT & Billy EJ (1992) Temperature extremes produced orally by hot and cold liquids *Journal of Prosthetic Dentistry* **67** 325-327.

Perdigão J, Lopes L, Lambrechts P, Leitao J, Van Meerbeek B & Vanherle G (1997) Effects of a self-etching primer on enamel shear bond strengths and SEM morphology *American Journal of Dentistry* **10** 141-146.

Pettemerides AP, Ireland AJ & Sherriff M (2001) An *ex-vivo* investigation into the use of a plasma arc lamp when using a visible light-cured composite and a resin-modified glass poly(alkenoate) cement in orthodontic bonding *Journal of Orthodontics* **28** 237-244.

Retief DH (1975) The use of 50 percent phosphoric acid as an etching agent in orthodontics: A rational approach *American Journal of Orthodontics* **68** 165-178.

Sano H, Yoshikawa T, Pereira PN, Kanemura N, Morigami M, Tagami J & Pashley DH (1999) Long-term durability of dentin bonds made with a self-etching primer, *in vivo Journal of Dental Research* **78** 906-911.

Sfondrini MF, Cacciafesta V, Pistorio A & Sfondrini G (2001) Effects of conventional and high-intensity light-curing on enamel shear bond strength of composite resin and resin-modified glass-ionomer *American Journal of Orthodontics and Dentofacial Orthopedics* **119** 30-53.

Sharkey S, Ray N, Burke F, Ziada H & Hannigan A (2001) Surface hardness of light-activated resin composites cured by two different visible-light sources: An *in vitro* study *Quintessence International* **32** 401-405.

Simonsen RJ (1982) Preventive resin restoration. Innovative use of sealants in restorative dentistry *Clinical Preventive Dentistry* **4** 27-29.

Stansbury JW (2000) Curing dental resins and composites by photopolymerization *Journal of Esthetic Dentistry* **12** 300-308.

Strang R, Cummings A, Stephen KW & McMenemy P (1986) Further abrasion resistance and bond strength studies of fissure sealants *Journal of Oral Rehabilitation* **13** 257-262.

Stritikus J & Owens B (2001) An *in vitro* study of microleakage of occlusal composite restorations polymerized by a conventional curing light and a PAC curing light *Journal of Clinical Pediatric Dentistry* **24** 221-227.

Symons AL, Chu CY & Meyers IA (1996) The effect of fissure morphology and pretreatment of the enamel surface on penetration and adhesion of fissure sealants *Journal of Oral Rehabilitation* **23** 791-798.

Tandon S, Kumari R & Udupa S (1989) The effect of etch-time on the bond strength of a sealant and on the etch-pattern in primary and permanent enamel: An evaluation *ASDC Journal of Dentistry Child* **56** 186-190.

Yoshiyama M, Matsuo T, Ebisu S & Pashley D (1998) Regional bond strengths of self-etching/self-priming adhesive systems *Journal of Dentistry* **26** 609-616.

©Operative Dentistry, 2003, **28-4,** 415-422

Early Detection of Secondary Caries Using Quantitative, Light-Induced Fluorescence

C González-Cabezas • M Fontana
D Gomes-Moosbauer • GK Stookey

Clinical Relevance

The development of more effective diagnostic approaches to secondary caries will allow the dentist to detect and monitor secondary carious lesions at a very early stage. This, in turn, would allow for the use and testing of enhanced methods to observe, arrest and remineralize these lesions without having to replace the restoration, thus, extending the lifespan of restorations. In this investigation, the authors found that the QLF system has the potential to be used for this purpose.

SUMMARY

The authors hypothesize that the arrestment and remineralization of these lesions could be improved if secondary caries could be detected and monitored at earlier stages. Traditional diagnostic techniques detect secondary caries when it is relatively advanced and when significant tissue has been lost. This *in vitro* study evaluated the Quantitative Light-induced Fluorescence (QLF) system for detecting and monitoring demineralization surrounding tooth-colored restorations. This investigation was divided into three studies. The first study evaluated QLF for the detection and measurement of chemically induced lesions surrounding resin composite restorations. The second study evaluated QLF for the detection of demineralization around different tooth-colored restorations (glass ionomer, resin composite, compomer and smart material) created in a microbial caries model. Finally, the third study tested whether QLF was effective at detecting early wall lesions adjacent to resin composite restorations. Data from Study 1 demonstrated the potential for QLF to detect very early secondary caries and to distinguish between the different stages of early demineralization. Study 2 confirmed the potential for QLF to detect early secondary caries created by cariogenic bacteria and concluded that the four types of materials differed in their ability to prevent secondary caries in this model, with the glass ionomer being the most effective and the non-fluoride releasing composite performing the worst, which allowed for the development of larger lesions. The results of Study 3 suggest a potential application of the QLF system to detect early wall lesions. Data from this investigation

Carlos González-Cabezas, DDS, PhD, assistant professor, director of the MS/MSD Programs in Preventive, Oral Health Research Institute, Indiana University School of Dentistry, Indianapolis, IN

Margherita Fontana, DDS, PhD, assistant professor, director of the Microbial Caries Laboratory, Oral Health Research Institute, Indiana University School of Dentistry, Indianapolis, IN

Daniel Gomes-Moosbauer, DDS, clinical instructor, Oral Health Research Institute, Indiana University School of Dentistry, Indianapolis, IN

George K Stookey, MSD, PhD, professor emeritus, Oral Health Research Institute Indiana University School of Dentistry, Indianapolis, IN

*Reprint request: 415 Lansing Street, Indianapolis, IN 46202; e-mail: cgonzale@iupui.edu

strongly suggest that QLF is a potentially viable technology to detect and monitor early secondary caries.

INTRODUCTION

Replacing failed restorations is one of the major expenses in dental health care, despite the declining prevalence of dental caries during the past two decades (Blinkhorn & Davies, 1996). Studies have indicated that dental clinicians performed an average of 71% of all restorative treatments on previously restored teeth (Mjör, 1981; Maryniuk & Kaplan, 1986). There are a number of factors that can contribute to the failure of a restoration; however, regardless of the inaccuracies in its diagnosis, secondary caries is one of the most common reasons for replacing amalgam and resin composite restorations (Fontana & González-Cabezas, 2000).

The secondary caries lesion usually consists of two carious regions. The first is an outer lesion formed in the enamel or cementum of the tooth surface, similar in histology to a primary lesion. The second region is a wall lesion, which is a narrower defect in the enamel or dentin along the cavity wall-restoration interface (Hals, Höyer Andreassen & Bie, 1974; Kidd, Tofenetti & Mjör, 1992). Outer and wall lesions can occur together or separately (Kidd & O'Hara, 1990), so that the outer enamel can appear clinically sound but have undermined dentin underneath (Kidd & others, 1992). However, most secondary caries (88%) around amalgam restorations were found to have an outer lesion (Hals & others, 1974). Furthermore, in most cases, outer lesions were found to be in a more advanced state of demineralization than wall lesions (Hals & others, 1974). Because of the constant improvements in dental materials and the lack of current clinically derived histological analysis of secondary caries, it is not known whether the presence of outer and wall lesions has changed since Hals and others' studies in the 1970s. *In vitro* data suggest that secondary caries initiates at the outer surface, and the presence and progression of a wall lesion depends on the progression of the outer lesion. When no outer lesion is allowed to form *in vitro*, development of wall lesions is extremely difficult (González-Cabezas & Fontana, unpublished data). In a recent review, Mjör and Toffenetti (2000) concluded that "since the spread of primary and secondary caries in enamel follows enamel rods," it is likely that the orientation of the enamel rods in relation to the tooth-restoration interface will determine whether a wall lesion will be present and to what extent. Thus, if the rods from a surface lesion reach the tooth-restoration interface, they would give the appearance of a wall lesion. The question remains whether we need the presence of a wall lesion to be able to refer to the new outer lesion as recurrent caries.

Development of more effective diagnostic approaches will allow the dentist to detect and monitor outer secondary carious lesions at a very early stage; this, in turn, would allow for the use of enhanced methods to observe, arrest and remineralize these lesions without having to replace the restoration. A series of new methodologies are being developed to detect carious lesions at very early stages. Direct digital radiography, fiber-optic transillumination, optical coherence tomography, electrical conductance measurement, ultrasound and measurement of changes of light scattering are some of the techniques that have shown potential for detecting early caries. However, the authors are not aware whether these techniques have been evaluated for their potential to detect and quantify secondary caries.

Other methodologies that are being developed and evaluated for the detection of early caries are based on measuring changes of fluorescence of the carious lesion when compared to the sound tissue surrounding it, or on detecting penetration of a fluorescent dye into the carious lesion. Miles and Duncan (1996) used an argon laser to detect very early interproximal lesions adjacent to Class II amalgam restorations *in vitro*. Before the analysis, they applied a solution of fluorescein dye for 15 seconds to stain the lesion. They reported a specificity and sensitivity of 0.64 and 0.76, respectively. A relatively new system (DIAGNOdent, Kavo, Biberach, Germany) was designed to measure the fluorescence difference between carious and sound tissues using a portable diode laser-based system. Lussi and others (1999) compared this new laser system with electrical conductance measurements (ECM) using extracted teeth with histological analysis for validation and demonstrated that the laser system showed higher sensitivity and specificity than the ECM approach. The system has also shown a high inter-examiner agreement and correlation compared to ECM (Lussi & others, 1998).

The Quantitative Laser-induced Fluorescence system (QLF, Inspektor Research Systems, Amsterdam, The Netherlands) was designed to measure the loss of fluorescence of carious lesions when they are illuminated with an argon-ion laser (488-nm). This system has been evaluated in several *in vitro* and *in vivo* studies, showing a clear potential for its use in detecting and monitoring early carious lesions (Hafstrom-Bjorkman & others, 1992; Al-Khateeb & others, 1997; Angmar-Månsson, Al-Khateeb & Tranaeus, 1996). In some of the studies, a fluorescent dye was applied to the lesion to enhance the difference between the sound and carious tissues (Ando & others, 1997; Hall & others, 1997). In this case, the carious area was more fluorescent than the sound area. QLF was evaluated *in vitro* for the detection of secondary caries around amalgam and resin composite restorations and was found to be useful for this purpose (DeSchepper & others, 1996; Blackman & others, 1997). A pilot study by Tranæus and others

(1997) used the system to detect natural lesions around fillings and indicated a good correlation (r=0.81) between QLF and longitudinal microradiography. The QLF system has been modified and currently uses an arc lamp filtered to a small band (370 ± 80 nm). This system is integrated into a hand-held intra-oral camera by which an image of the area of analysis is saved. This modified system is called Quantitative Light-induced Fluorescence (QLF). System integration and software are similar to the laser-based version of QLF. This investigation, divided into three different studies, evaluated the potential of the current QLF system to detect and measure early demineralization around tooth-colored dental restorations.

METHODS AND MATERIALS

Study 1: Purpose

This study evaluated QLF for the detection and measurement of chemically-induced demineralization around resin composite restorations.

Specimen Preparation

Twenty-four clinically sound molars were selected from extracted human teeth and kept in buffered formalin (pH 6.7) for at least three weeks. The buccal sides were ground and polished gently (Gamma alumina 0.05 µm) to remove the outer layer of enamel, and the lingual side was flattened so that about one-half of the buccal-lingual width of the tooth remained. The roots were removed and the flattened tooth surface was affixed with a cyanoacrylate adhesive to one end of a 2-cm long Plexiglas rod. On each specimen, one rectangular box-shaped cavity, 2-mm wide, 3-mm long and 2-mm deep was prepared according to existing clinical standards using a #557 bur in a high-speed drill under water-cooling. The long axes of the cavities were oriented from mesial to distal. Then, specimens were restored with a resin composite (Silux, 3M, St Paul, MN, USA) using standard clinical techniques but without etching, dentin conditioning or bonding agent, to promote the formation of a gap between the restorative material and the tooth structure. Enamel was painted with a transparent acid resistant nail varnish leaving 0.5 to 1 mm of enamel surrounding half of the restoration without painting. The remaining area exposed for demineralization was the experimental side; the area around the other half of the restoration (covered with varnish) served as the control side (Figure 1).

Demineralization

The specimens were distributed randomly into two groups of 12 specimens each and incubated (37°C) in pairs in 28 ml of a demineralizing solution: 0.1 mol/L lactic acid, 0.2% carbopol and 50% were saturated with hydroxyapatite and adjusted to pH 5.0 (White, 1987) for 48 (Group 1) and 120 hours (solution was replaced after 72 hours; Group 2). After incubation, the specimens were rinsed with deionized water and stored in humid conditions at 4°C until QLF analysis was performed.

QLF Analysis

The nail varnish was removed with acetone and cotton swabs. QLF images were taken from the experimental (demineralized) and control (sound) sides. In order to calculate the fluorescence loss in the experimental and control sides, a computer-generated rectangle (inner patch) was placed in the area of analysis. The size of the rectangle was kept constant for all the analyzed areas of all specimens. The fluorescence values of the pixels within the rectangle were compared to the fluorescence values of the pixels surrounding the rectangle (Figure 1). The lesion threshold was set at 95%, indicating that pixels with fluorescence values of less than 95% of the reference fluorescence value were considered demineralized. Two different sets of values were obtained. The first was the average change in fluorescence (fluorescence loss) of the pixels under the threshold. The second value was ΔQ, which was obtained by multiplying the average change in fluorescence by the number of pixels (area) under the threshold value.

Confocal Laser Scanning Microscope (CLSM) Analysis

Each specimen was then sectioned perpendicular to the longitudinal side of the restoration by using a water-cooled, hard tissue microtome. Two sections were made per specimen: one in the control side and one in the experimental side. The sections were stained overnight with a freshly prepared solution of 0.1 mM Rhodamine B (Aldrich Chem Co, Milwaukee, WI, USA), and without further rinsing, they were air dried. After drying,

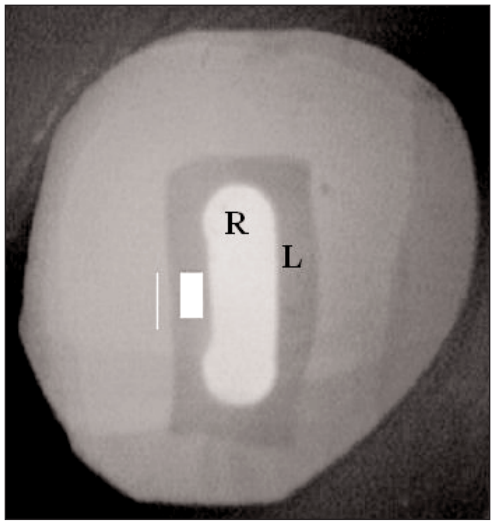


Figure 1. *Diagram showing the QLF analysis technique. Fluorescence values of the pixels in the white rectangular area were compared with the fluorescence values of pixels in the surrounding areas (white line). (R= restoration; L= lesion).*

the specimens were analyzed with a CLSM (Odyssey, Noran Instruments, Inc, Middleton, WI, USA; Fontana & others, 1996a). Images were obtained by using an argon-ion laser at a 488-nm excitation wavelength, a 10 µm confocal slit, a 550-nm long pass barrier filter and a 10x objective lens (final magnification for analysis, 290x). Areas were scanned plano-parallel to the transversal cut surface of the specimen and perpendicular to the natural surface of the tooth. Images were analyzed using Image-1 (version 4.0.3.C) software (Universal Images Corp, West Chester, PA, USA) to obtain lesion depth.

Data Analysis

Data were analyzed using Student *t*-test or Mann-Whitney Rank Sum Test (used when the data did not pass the normality test). Pearson's correlation coefficient was also calculated between CLSM lesion depth and the QLF results.

Study 2

Purpose

This study was designed to evaluate QLF for the detection of demineralization around different tooth-colored restorations created in an artificial mouth model (microbial model).

Specimen Preparation

Sixty-six sound, human, extracted, lower permanent incisors were selected. The labial enamel surface was pumiced and the lingual side flattened so that about half the labial-lingual width of the tooth remained. The roots were removed and the flattened tooth surfaces were affixed with a cyanoacrylate adhesive to one end of 1.5-cm long Plexiglas rods. On each specimen, one rectangular, box-shaped cavity, 1-mm wide, 4-mm long and 1.5-mm deep was prepared according to existing clinical standards using a #245 tungsten bur in a high-speed drill under air water cooling. The long axes of the cavities were oriented in a cervico-incisal direction. The specimens were divided into four groups and restored with PhotacFil (resin-modified glass ionomer, 3M ESPE, St Paul, MN, USA), Dyract (compomer, Dentsply Caulk, Milford, DE, USA), Ariston pHc (fluoride containing "smart" composite, Vivadent, Liechtenstein) or Silux (resin composite, 3M). There were 14 specimens prepared per group: 12 used for *in vitro* demineralization and two used as sound baselines for analytical techniques. All the restorative materials released fluoride except for Silux. The cavities were restored without etching or bonding agent to ensure the presence of a gap. The restored specimens were painted with a clear acid-resistant varnish, except for a 1-mm band around the restoration margins and they were mounted into acrylic plates made to fit tightly on the stirring magnet of the caries-forming vessels.

Demineralization

All the specimens were ethylene oxide gas sterilized prior to incubation. Experimental groups were inoculated once at the beginning of the experiment with a mid-log phase culture of a mix of *Streptococcus mutans* TH16 and *Lactobacillus casei* ATCC 7469. The specimens were exposed for four days in a microbial caries model at 37°C to circulating trypticase soy broth supplemented with 5% sucrose (TSBS) for 30 minutes three times a day and to a mineral washing solution (MW) for a total of 22.5 hours per day (Fontana & others, 1996b). The circulating fluids were delivered to and removed from the treatment vessels by peristaltic pumps regulated by timers. The TSBS was intended to reproduce nutrient intake three times/day, while the MW represented an artificial saliva buffer solution. After two days of treatment and at the end of the study, the drainage containers for all four groups were changed, and the drainage fluid was monitored for pH, bacteria viability and lack of contamination. At the end of the experiment, plaque was harvested from three treated specimens in each of the four groups and monitored for bacterial counts. Plaque samples from all groups were diluted 1:1000 and 1:10,000, plated and viable bacteria were recovered.

QLF Analysis

The varnish was removed from all the specimens using acetone. The QLF analysis was similar to that performed in Study 1. The demineralized area was compared to the areas of sound enamel that were originally under the varnish.

CLSM Analysis

Each specimen was sectioned perpendicular to the longitudinal side of the restoration by using a water-cooled, hard tissue microtome and analyzed using CLSM as described in Study 1.

Data Analysis

Data were divided by type of restoration and analyzed using a single factor analysis of variance model (ANOVA). Where significant ($p<0.05$) effects were detected, multiple comparisons were conducted using Tukey's procedure.

Study 3

Purpose

This study was designed to test whether the QLF methodology is effective for detecting early demineralization located only in the walls of cavities of resin composite restorations.

Specimen Preparation

Twenty-four clinically sound molars were selected from extracted human teeth that had been obtained from oral surgeons and kept in buffered formalin (pH 6.7) for at least three weeks. The lingual sides were flattened so that about one-half of the labial-lingual width of the teeth remained. The roots were removed and the flattened tooth surfaces were affixed with a cyanoacrylate

adhesive to one end of 2-cm long Plexiglas rods. On each specimen, one oval-shaped cavity, 2-mm wide, 4-mm long and 2-mm deep was prepared according to existing clinical standards using a #329 bur in a high-speed drill under air/water cooling. Specimen surfaces were coated with an acid resistant transparent varnish, being careful not to place varnish inside the cavity. Then, specimens were randomly distributed into two groups, the experimental group and the control.

Demineralization

The experimental group was incubated in a demineralizing solution, 0.1 mol/L lactic acid, 0.2% carbopol and 50% saturated with hydroxyapatite and adjusted to pH 5.0 (White, 1987) for four days (96 hours) at 37°C. The specimens were incubated in pairs in 28 ml of the demineralizing solution. After the respective incubation time, the specimens were rinsed with deionized water. Control group specimens were kept in humid conditions at 4°C.

Restoration Placement

All the specimens were restored with resin using standard clinical techniques but without etching, dentin preparation or bonding agent. The buccal sides were ground and gently polished (Gamma alumina 0.05 µm) to remove the varnish and any overhang of the restorations.

QLF Analysis

QLF images were taken from the experimental and control groups. The images were not analyzed with the QLF software to obtain quantitative data because it was very difficult to determine where to make the analyses. The QLF images were subjectively analyzed (single blind approach) and classified as having a lesion or not. The ones classified as having lesions were further classified by counting the number of sides surrounding the restoration with a lesion (up to 8).

CLSM Analysis

Each specimen was sectioned perpendicular to the longitudinal side of the restoration by using a water-cooled, hard tissue microtome and analyzed with CLSM. The images were saved as described in Study 1 and classified as positive on both sides of the restoration (2), positive on one side of the restoration (1) or negative (0 no lesions observed).

RESULTS

Study 1

The QLF analysis revealed that lesions were detected in demineralized sites of both experimental groups. The demineralized sites were statistically different ($p<0.05$) from the control sides for both groups (Table 1). When data from the experimental groups were compared, statistically significant differences were found in both QLF parameters between these groups. The average depth of the lesions, as measured by confocal microscopy, were 20 µm for the 48-hour group and 52 µm for the 120-hour group (Figure 2). This difference was statistically significant. No lesions were observed in the control sides in the confocal microscopy analysis. Pearson's correlation coefficient between lesion depth and QLF ΔQ was –0.64 ($p=0.003$).

Study 2

The pH values for all groups were similar at either two or four days of the experiment, indicating that the acidic challenge was similar among the four groups. There was no significant difference ($p>0.05$) in the amount of *lactobacilli* adhered to the teeth among any of the four groups, indicating that the challenge from these bacteria had been similar among the four groups. However, the Silux group had significantly more *S mutans* adhered to its surface than any of the other three groups.

Confocal microscopy and QLF data are illustrated in Table 2. CLSM data showed that the PhotacFil group had a significantly smaller mean lesion depth than the Silux group. Numerically, the group restored with Silux always had the deepest lesions, while the group restored with PhotacFil always had the shallowest lesions. Both the Dyract and Ariston pHc materials performed similarly. Regarding QLF analysis, no significant difference was found among the experimental

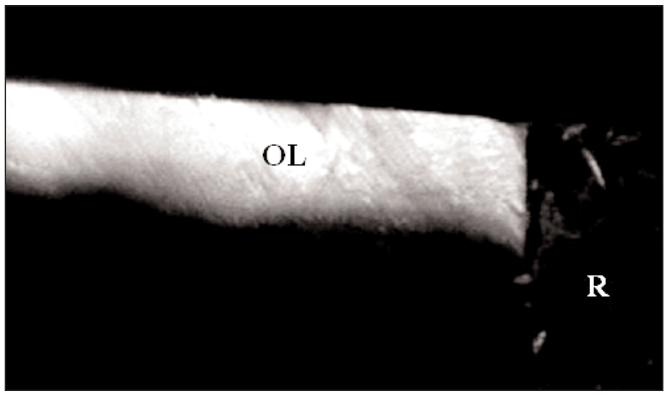


Figure 2. *Confocal image of secondary caries obtained from an experimental side (study 1). OL = outer lesion. R = Restoration.*

Table 1: *QLF and CLSM Caries Data-Study 1*[a]

	Group 48 Hours	Group 120 Hours	Control
QLF ΔQ (pixels)	-1770.9 ± 1079.4	-4023.2 ± 2411.2	-27.3 ± 61.8
QLF % Average Fluorescence Change	-9.2 ± 3.1	-19.1 ± 9.0	-7.9 ± 8.5
CLSM Lesion Depth (µm)	20.2 ± 7.4	52.3 ± 10.3	0 ± 0

[a]*Mean ± Standard Deviation*

Table 2: *QLF and CLSM Caries Data-Study 2*[a]

	Silux	Dyract	Ariston pHc	Photacfil	Baselines
QLF ΔQ (pixels)	-3284 ± 1803	-2863 ± 970	-3077 ± 1342	-2619 ± 935	-392 ± 720
QLF % Average Fluorescence Change	-21 ± 10	-18 ± 6.2	-19 ± 8.2	-17 ± 5.3	-7 ± 2.2
CLSM Lesion Depth (μm)	62 ± 17.9	50 ± 10.1	47 ± 13.6	39 ± 4.9	0 ± 0

[a]*Mean ± Standard Deviation*

groups; however, a significant difference was found between the experimental groups and the control-baseline specimens.

Study 3

In the QLF analysis, lesions were found in eight of the 10 specimens of the experimental group (sides involved per specimen ranged from two to eight). However, lesions were also found in four of the 10 specimens of the control group (the sides involved ranged from three to four). In the confocal analysis, no lesions were found in the control group. Lesions were found in eight out of 10 specimens of the experimental group, and six were located in both sides of the restoration. The two specimens that showed no lesions in the confocal analysis were the same two that were found sound in the QLF analysis. The reason for this is unclear.

DISCUSSION

Diagnosis of secondary caries is difficult. Conventional techniques are very limited in the detection and measurement of early secondary caries. These clinical techniques have focused primarily on the detection of ditching and color change around restorations. Color change around restorations is difficult to measure and subjective to interpret and, alone, is not a reliable indicator of secondary caries. For example, a gray-blue discoloration next to an amalgam could indicate corrosion, metal translucency or dental caries. Kidd, Joyston-Bechal & Beighton (1995) studied 175 teeth restored with amalgam to relate the level of marginal color change and ditching to the level of infection of dentin. The results suggested that narrow ditches and color change alone did not correlate with the level of infection. In composite or glass ionomer restorations, color changes are easier to interpret as being white, brown or gray spots, or just a line of stain between the restoration and the tooth.

Although it has been shown histologically that color change usually indicates secondary caries, the marginal color change could be caused by the leakage of exogenous pigments from food, drinks or medications, such as tea or chlorhexidine, which could stain, at least *in vitro*, residual arrested caries (Kidd, Joyston-Bechal & Smith, 1990; Kidd & others, 1992). Since 80% of the soft sites had stained margins but also 56% of the hard sites had stained margins, Kidd and others (1995) found that neither staining at the margins of a tooth-colored filling nor undermining stain adjacent to the filling would reliably predict soft dentin. Therefore, according to Kidd (1996), if color change were routinely used to trigger operative intervention, many fillings would be replaced unnecessarily.

A possible correlation between the size of the microgap and secondary caries has been suggested (Hodges, Mangum & Ward, 1995), and it has been estimated that the size of the marginal defect necessary for the production of secondary caries is approximately 50 μm (Jørgensen & Wakumoto, 1968). However, a more recent study found no such correlation (Özer & Thylstrup, 1995). Moreover, Kidd and O'Hara (1990) found no correlation between ditch presence and secondary caries. In addition, Kidd, Joyston-Bechal and Beighton (1994) found no correlation between either ditching or staining around amalgams or resin composites and secondary caries. These studies illustrate the difficulty in clinically diagnosing secondary caries. Despite improvements in technology in recent years (Pitts, 1996), dental radiography does not allow for the detection of demineralization at very early stages.

If development of a true wall lesion, and not residual caries that is left over by the dentist during the cavity preparation, depends on the progression of the outer lesion, as suggested by some (Mjör & Toffenetti, 2000), then it would be reasonable to assume that if the outer secondary lesion could be monitored and eventually "treated," it could also affect the progression of the wall lesion. Data from Study 1 confirms the potential of QLF to detect very early demineralization (mostly outer secondary caries lesions) created by chemical means in the lab, surrounding composite restorations. In addition, these results show the capacity of the technique to distinguish among the different stages of early demineralization that would allow the dentist to monitor small lesion changes over time.

Results from Study 2 confirm the potential of QLF to detect early secondary caries created by cariogenic bacteria around different types of tooth-colored restorative materials. The confocal data suggest that although the four materials had a similar effect on pH, they differed in their effect on preventing enamel demineralization. Among the three fluoride-containing dental materials, PhotacFil (resin-modified glass ionomer) was more effective in preventing enamel secondary caries in the initial days of demineralization in this model. Silux, the non-fluoride releasing control, performed the worst,

allowing for larger lesions to develop, as expected. The QLF data also demonstrated a trend for three fluoride-releasing materials for being more effective than the control (Silux) in inhibiting secondary caries. Fontana and others (1997), using this microbial model, reported significantly smaller wall lesions *in vitro* surrounding glass ionomers than amalgam or composite restorations. Also, glass ionomers have been shown to reduce the number of *S mutans* at the margins of such restorations (Svanberg, Mjör & Ørstavik, 1990), and fluoride release from resin-modified glass ionomers and glass ionomers is initially much higher than compomers (Shaw, Carrick & McCabe, 1998). Other types of fluoride–releasing materials include "smart" materials. A recent study concluded that a fluoride, calcium and hydroxyl-releasing "smart" composite placed in the presence of a gap was better than a non-fluoride releasing composite in inhibiting the development of secondary carious wall lesions (Fontana & others, 1999). This study demonstrated that this technology is applicable to a variety of tooth-colored restorative materials and also demonstrated the clinical and research applications that a new technology such as QLF has to offer.

The results of Study 3 suggest a potential application for the QLF system to detect early demineralization located at the walls of resin composite restorations. However, it also demonstrated that by being such a sensitive technique, any defects at the margin of the restoration might also be considered as small lesions (such as in the case of the sound controls, in our experiment), thus, increasing the possibility of false positives. This raises the concern whether QLF should be used to detect very incipient lesions at one point in time or if changes should be monitored over time. This last approach would decrease the risk of false positives, since only defects that changed over time would be considered active and in need of preventive treatment.

For the three studies, during QLF analysis it was found that some experimental areas had a limited loss of fluorescence, which was just a few percentage points below the threshold limit (and sometimes did not include all the pixels in the area of analysis below the threshold limit), indicating a uniform but incipient demineralization in some areas. In some control sides, it was also observed that some small artifacts caused a few pixels to fall below the threshold limit, providing a % average change in fluorescence data similar in some cases to that obtained from the experimental side, even though the results reflected data from a very small portion of the area of analysis. To solve this potential problem, images were analyzed taking into consideration the number of pixels below the threshold level in addition to the average change in fluorescence (ΔQ = area x % average change in fluorescence). ΔQ was designed to analyze lesions over time, so that the size and change of fluorescence of the lesion could be monitored. For comparison purposes in these studies, the size of the area of analysis was fixed for all the analyses and always located within the window of exposure so that the number of pixels that could be below the 95% threshold level was held constant.

CONCLUSIONS

Replacing restorations continues to be one of the most important problems in dentistry today. Even though the longevity of the restorations depends on different factors, including the type of restoration, the materials used, the dentition, the age of the patient and the operator, secondary caries is still one of the main reasons for restoration failure. This problem is expected to increase, at least in the US, since edentulism has decreased significantly in the last three decades. Better diagnostic systems would allow dentists to detect these lesions at a very early stage, allowing for the development of enhanced methods to arrest and remineralize them without having to replace the restoration. In this investigation, the authors found that the QLF system has the potential to be used for this purpose. However, the studies described here were performed in *in-vitro* conditions. *In vivo* conditions are different and a more difficult diagnosis should be expected. A pilot clinical study and an *in vitro* study of natural secondary caries are currently being carried out in our facilities. Initial data have shown the difficulty in analyzing natural secondary caries. It seems that it is difficult to distinguish incipient secondary caries from other artifacts, such as staining or ditching. Monitoring (over time) of the potential carious lesions seems to be the most reasonable approach for a definitive diagnosis of active lesions in *in-vivo* conditions. This, in turn, will help the dentist decide on the appropriate intensity of the intervention strategy to be used. Therefore, diagnostic systems should not only detect early lesions but also measure them, so data can be followed over time to help determine lesion activity.

Acknowledgements

Some of the data reported in this paper were presented at the second Indiana Conference in early caries detection (1999).

(Received 16 July 2002)

References

Al-Khateeb S, ten Cate JM, Angmar-Månsson B, de Josselin de Jong E, Sundstrom G, Exterkate RA & Oliveby A (1997) Quantification of formation and remineralization of artificial enamel lesions with a new portable fluorescence device *Advanced Dental Research* **11(4)** 502-506.

Ando M, Hall AF, Eckert GJ, Schemehorn BR, Analoui M & Stookey GK (1997) Relative ability of laser fluorescence techniques to quantitate early mineral loss *in vitro Caries Research* **31(2)** 125-131.

Angmar-Månsson B, Al-Khateeb S & Tranaeus S (1996) Monitoring the caries process. Optical methods for clinical diagnosis and quantification of enamel caries *European Journal of Oral Science* **104** 480-485.

Blackman D, van der Veen M, Lagerweij M, Ando M & Stookey GK (1997) Laser fluorescence diagnosis of demineralized enamel adjacent to resin restorations *Journal of Dental Research* **76** Abstract #943 p 131.

Blinkhorn AS & Davies RM (1996) Caries prevention. A continued need worldwide *International Dental Journal* **46** 119-125.

DeSchepper EJ, Hall AF, de Josselin de Jong E, Benedict S, Analoui M, Ando M, Beiswanger BB, Dixon SE, Willis GP & Stookey GK (1996) Demineralization detection adjacent to amalgam using quantitative laser fluorescence and dye-enhanced laser fluorescence in Stookey GK ed *Early Detection of Dental Caries Proceedings of the 1st Annual Indiana Conference* Indiana University School of Dentistry 165-182.

Fontana M & González-Cabezas C (2000) Secondary caries and restoration replacement: An unresolved problem *Compedium* **21(1)** 15-30.

Fontana M, González-Cabezas C, Wilson M & Appert C (1999) *In vitro* evaluation of a "smart" dental material for its efficacy in preventing secondary caries using a microbial artificial mouth model *American Journal of Dentistry* **12** S8-S9.

Fontana M, Li Y, Dunipace AJ, Noblitt TW, Fischer GM, Katz BP & Stookey GK (1996a) Measurement of demineralization of enamel using microradiography and confocal microscopy. A correlational study *Caries Research* **30** 317-325.

Fontana M, Dunipace AJ, Gregory RL, Noblitt TW, Li Y, Park KK & Stookey GK (1996b) An *in-vitro* microbial model for studying secondary caries formation *Caries Research* **30** 112-118.

Fontana M, Dunipace AJ, Gregory RL, González-Cabezas C, Wilson ME & Stookey GK (1997) Secondary caries around different dental materials: Microbial model study *Journal of Dental Research* **77** 282.

Hafstrom-Bjorkman U, Sundstrom F, de Josselin de Jong E, Oliveby A & Angmar-Månsson B (1992) Comparison of laser fluorescence and longitudinal microradiography for quantitative assessment of *in vitro* enamel caries *Caries Research* **26(4)** 241-247.

Hall AF, DeSchepper E, Ando M & Stookey GK (1997) *In vitro* studies of laser fluorescence for detection and quantification of mineral loss from dental caries *Advanced Dental Research* **11(4)** 507-514.

Hals E, Höyer Andreassen B & Bie T (1974) Histopathology of natural caries around silver amalgam fillings *Caries Research* **8** 343-358.

Hodges DJ, Mangum FI & Ward MT (1995) Relationship between gap width and recurrent dental caries beneath occlusal margins of amalgam restorations *Community Dental Oral Epidemiology* **23** 200-204.

Kidd EA, Joyston-Bechal S & Smith MM (1990) Staining of residual caries under freshly-packed amalgam restorations exposed to tea/chlorhexidine *in-vitro International Dental Journal* **40** 219-224.

Kidd EA & O'Hara JW (1990) The caries status of occlusal amalgam restorations with marginal defects *Journal of Dental Research* **69** 1275-1277.

Kidd EA, Toffenetti F & Mjör IA (1992) Secondary caries *International Dental Journal* **42** 127-138.

Kidd EA, Joyston-Bechal S & Beighton D (1994) Diagnosis of secondary caries: A laboratory study *British Dental Journal* **176** 135-139.

Kidd EA, Joyston-Bechal S & Beighton D (1995) Marginal ditching and staining as a predictor of secondary caries around amalgam restorations: A clinical and microbiological study *Journal of Dental Research* **74** 1206-1211.

Kidd EA (1996) Diagnosis of Secondary Caries in Stookey GK, ed *Early Detection of Dental Caries Proceedings of the 1st Annual Indiana Conference* Indiana University School of Dentistry 157-163.

Lussi A, Imwinkelried S, Pitts N, Longbottom C & Reich E (1999) Performance and reproducibility of a laser fluorescence system for detection of occlusal caries *in vitro Caries Research* **33(4)** 261-266.

Lussi A, Pitts N, Hotz P & Reich E (1998) Reproducibility of a laser f fluorescence system for detection of occlusal caries *Caries Research* **32** Abstract #88 p 297.

Jørgensen KD & Wakumoto S (1968) Occlusal amalgam fillings: Marginal defects and secondary caries *Odontology Tidskr* **76** 43-54.

Maryniuk GA & Kaplan SH (1986) Longevity of restorations: Survey results of dentists' estimates and attitudes *Journal of the American Dental Association* **112** 39-45.

Miles DA & Duncan JE (1996) Early detection of simulated, secondary carious lesions using dye-enhanced laser fluorescence in Stookey GK, ed *Early Detection of Dental Caries Proceedings of the 1st Annual Indiana Conference* Indiana University School of Dentistry 243-251.

Mjör IA (1981) Placement and replacement of restorations *Operative Dentistry* **6** 49-54.

Mjör IA & Toffenetti F (2000) Secondary caries: A literature review with case reports *Quintessence International* **31** 165-179.

Özer L & Thylstrup A (1995) Caries adjacent to Class II amalgam restorations in relation to plaque and marginal gap width *Caries Research* **29** Abstract #98 p 320.

Pitts N (1996) Advances in radiographic detection methods and caries management rationale in Stookey GK ed *Early Detection of Dental Caries Proceedings of the 1st Annual Indiana Conference Proceedings of the 1st Annual Indiana Conference* Indiana University School of Dentistry 39-50.

Shaw AJ, Carrick T & McCabe JF (1998) Fluoride release from glass-ionomer and compomer restorative materials: 6-month data *Journal of Dentistry* **26** 355-359.

Svanberg M, Mjör IA & Ørstavik D (1990) Mutans streptococci in plaque from margins of amalgam, composite, and glass ionomer restorations *Journal of Dental Research* **69** 861-864.

Tranæus S, de Josselin de Jong E, Lussi A & Angmar-Månsson B (1997) Quantitative light-induced fluorescence for assessment of enamel caries around fillings: A pilot study *Caries Research* **31** Abstract #132 p 324.

White DJ (1987) Use of synthetic polymer gels for artificial carious lesion preparation *Caries Research* **21** 228-242.

©Operative Dentistry, 2003, **28-4,** 423-428

Shear Bond Strength of Porcelain Laminate Veneer Bonded with Flowable Composite

MO Barceleiro • MS Miranda
KRHC Dias • T Sekito, Jr

Clinical Relevance

Flowable composite can be a suitable alternative material for porcelain veneer bonding.

SUMMARY

This study evaluated the "*in vitro*" shear bond strength of two materials to bovine enamel when used as porcelain laminate veneer bonding systems. The authors used the dual-cured resin cement Variolink II (Vivadent) and the light-cured flowable composite Natural-Flow (DFL). Porcelain cylinders were bonded to bovine enamel following manufacturers' directions. After 24 hours of storage in distilled water and thermocycling, the specimens were tested on a universal testing machine to determine the shear bond strengths. Mean strengths found in Groups 1 (resin cement) and 2 (flowable composite) were not statistically different ($p \leq 0.05$). The authors concluded that the shear bond strength of Natural-Flow is not different from Variolink II and that flowable composites can be a suitable alternative when used as porcelain laminate veneer bonding systems.

*Marcos De Oliveira Barceleiro, MSD, assistant professor, Fundação Educacional Serra dos Órgãos, Dental School, Department of Dentistry, Teresópolis, Rio de Janeiro, Brazil

Mauro Sayão De Miranda, PhD, associate professor, Universidade do Estado do Rio de Janeiro, Dental School, Department of Dentistry, Rio de Janeiro, Brazil

Katia Regina Hostilio Cervantes Dias, PhD, chairwoman, Universidade do Estado do Rio de Janeiro, Dental School, Department of Dentistry, Rio de Janeiro, Brazil

Terumitsu Sekito, Jr, MSD

*Reprint request: Rua Fileuterpe, 568, cob 01 São Pedro, Teresópolis RJ Brazil, 25955-101; e-mail: marcosbarceleiro@bol.com.br

INTRODUCTION

Patients demand for aesthetic dentistry is growing, and for many years, the most durable, predictable and efficient aesthetic treatment was full crowns. This procedure is relatively invasive, can remove substantial amounts of sound teeth (Friedman, 1991) and can cause adverse pulpal and/or periodontal effects despite it being the best aesthetic treatment (Peumans & others, 2000). The use of resin composite veneers has grown as an alternative to full crowns in unaesthetic teeth, but this treatment was not durable due to material limitations (Peumans & others, 1997a,b).

In a search for more durable aesthetics, porcelain veneers have been introduced during the 1980s as a durable anterior restoration with superior aesthetics (Peumans & others, 2000). The initial idea of porcelain veneers was introduced by Charles Pincus (1938), who used a denture adhesive to retain porcelain veneers during cinematic filming but, as there were no adhesive systems at that time to permanently attach them, the fragile restoration had to be removed after cinematic filming. The concept of porcelain veneers was reactivated by Calamia and Simonsen (1984), who described a new technique for porcelain veneers bonding. Their technique improved veneer retention, and as clinical and laboratory techniques continued to be refined (Peumans & others, 2000), porcelain veneer became a routine procedure. In the United Kingdom, for example, some 113,582 veneers were placed in general dental practice under NHS regulation in 1994/1995 (Brunton & Wilson, 1998).

According to Castelnuovo and others (2000), porcelain veneers are indicated for teeth with moderate discol-

oration caused by tetracycline, fluoride, age, amelogenesis imperfecta and other external factors. Porcelain veneer can also be selected for the restoration of traumatized, fractured and worn dentition. Abnormal tooth anatomy or malposition can also be corrected with porcelain veneers.

There are some reports in literature showing different observation periods of porcelain veneers that range from 12 months up to 15 years (Strassler & Nathanson, 1989; Christensen & Christensen, 1991; Friedman, 1998; Kihn & Barnes, 1998). The clinical survival rate of porcelain veneers bonded to enamel has been predictable (Friedman, 1998). In fact, Friedman (1998) reported that 93% of 3,500 porcelain veneers placed over a 15-year period were rated as successful.

Nevertheless, porcelain veneers still have many limitations, and many authors (Christensen & Christensen, 1991; Garber, 1993; Nordbø, Rygh-Thoresen & Henaug, 1994; Walls, 1995; Strassler & Weiner, 1995; Friedman, 1998) have described clinical trials involving porcelain veneer as having high failure rates. One study reported a high number (25%) of fractures and/or loss of porcelain veneers after 10 years of clinical functioning (Garber, 1993). Failures associated with porcelain veneers are related to fracture, microleakage and debonding (Friedman, 1998). Fracture alone accounted for 67% of the total failures recorded for porcelain veneers during a clinical observation period up to 15 years (Friedman, 1998). Conversely, microleakage and debonding accounted for 33% of the total failures. Because of these high failure rates, many attempts have been made to discover a new porcelain bonding system that could produce a strong porcelain/luting material/tooth adhesion complex. In fact, great advances have been made; however, the long-term resistance of veneers to marginal failure has yet to be proven (Troedson & Dérand, 1999).

The highest numeric value of load (63 MPa) needed to debond porcelain from enamel was found with etched silane-treated porcelain discs bonded to etched enamel (Stacey, 1993). Many authors have already shown high bond strengths between porcelain and tooth (Calamia & others, 1985; Stangel, Nathanson & Hsu, 1987; Stacey, 1993; Kato & others, 1996; Troedson & Dérand, 1998). In all of these studies, the porcelain veneers were cemented with dual-cured resin cement, which is the first-choice material for porcelain veneer cementation. However, Troedson and Dérand (1999) have shown that the technique of luting a veneer with resin cements demands skill from the practitioner, where adequate moisture control and proper handling of the resin cement are of utmost importance.

According to Friedman (1991), a variety of resin composites can be alternatively used to successfully retain veneers. Composites with higher filler contents such as hybrid materials, recognized for their superior optics, also offer excellent physical properties. The same author has asserted that it was possible to use many restorative composites to cement porcelain veneers. Contrary to what is expected, high viscosity hybrid composites can eliminate some of the difficulties frequently found in the porcelain veneer bonding technique. For example, when these composites are properly handled, accidental breakage is unlikely. Furthermore, according to Peumans and others (2000), control during the seating process is increased when light-curing materials are used, compared with dual cure or chemical curing materials. However, most importantly, the physical and optical properties of these composite materials are far superior to resin cements. Based on this information, Peumans and others (2000) have also indicated the use of light-cured resin composites for cementation of porcelain veneers.

With the advent of flowable composites, a new class of composites with reduced viscosity has emerged, with many indications, among them is use as a porcelain veneer bonding system (Behle, 1998). According to Bayne & others (1998), some of these materials would seem acceptable for cementing porcelain veneers. These low viscosity materials facilitate pre-polymerization cleanup, eliminating the (technically dangerous) need for a partial or a tacking polymerization. After the excess is removed and the position verified, the entire "sandwich" is polymerized at the same time. In addition, since they are light curing materials, control during the seating process is increased and the clinician has a longer working time compared with dual cure or chemical curing materials (Peumans & others, 2000). According to Behle (1998), flowable composites have another advantage over resin cements, as they prevent the incorporation of air.

While some advantages have already been described related to the use of flowable composites as an alternative porcelain veneer bonding system, the authors do not know the bond strength of porcelain veneer cemented with flowable composite. This study evaluated the shear bond strength of porcelain veneers cemented with flowable composites and determined whether this composite can be a suitable material for porcelain veneer bonding.

METHODS AND MATERIALS

Twenty shade A1 feldspathic porcelain (VITA Porcelain—VITA Zahnfabrik, D-79704 Bad Sackingen, Germany) cylinders (5 mm in diameter and 2 mm in height) were fabricated by condensing VITA VMK68 powder mixed with modeling liquid into a stainless steel mold. They were extruded and fired according to the manufacturer's instructions in a large commercial laboratory that serves practitioners in Rio de Janeiro, Brazil. Each specimen was flattened by grinding with

600 grit silicon carbide paper and etched with 10% hydrofluoric acid gel (Dentsply Int, York, PA, USA) for two minutes, washed with distilled water and dried with warm, oil-free air immediately before the experiment.

A total of 20 freshly extracted bovine lower incisors were thoroughly cleaned and stored in distilled water at 37°C. The teeth were embedded in epoxy resin with their vestibular face exposed. They were hand-polished on wet 200, 400 and 600 grit silicon carbide paper to expose an area of flattened, polished enamel at least 5 mm in diameter. The teeth were then randomly divided into two groups.

Group 1 (Control)—Resin Cement

A piece of tape with a circular hole 5 mm in diameter was positioned on the flattened enamel. The teeth were cleaned with fine flour of pumice using a rubber cup in a low-speed hand piece for 30 seconds. The delimited areas in the enamel were then etched with 37% ortophosphoric acid gel (3M Dental Products, St Paul, MN, USA) for 30 seconds, then thoroughly rinsed with distilled water and gently dried with oil-free air. A thin layer of Scotchbond Multipurpose II adhesive (3M Dental Products) was then applied, gently blown with a jet of oil-free air and light cured for 10 seconds with Spectrum Curing Unit (Dentsply Inc). After preparing the teeth, three layers of Monobond-S silane (Ivoclar-Vivadent AG, FL-9494, Schaan, Liechtenstein) were applied to the etched porcelain and gently dried for three minutes with oil-free air. Immediately after silane treatment, a thin layer of Scotchbond Multi-Purpose II adhesive (3M Dental Products) was applied with a brush. The adhesive was air thinned by blowing the surface with a jet of oil-free air and light cured for 10 seconds. Then, equal parts of Variolink II (Ivoclar-Vivadent AG) catalyst and opaque base were mixed and applied to the prepared porcelain. The porcelain rod was placed onto the prepared enamel under a 155 g static load (Sheth, Jensen & Tolliver, 1988) and the resin-cement was light cured from four directions (Incisal, cervical, mesial and distal) for 15 seconds each (60 seconds total).

Group 2 (Experimental)—Flowable Composite

The teeth and the porcelain cylinders were prepared as in Group 1. For cementation, A1 shade Natural Flow flowable composite (DFL Ind e Com LTDA, 22713-001, Rio de Janeiro, RJ Brazil), a commercially available flowable composite in Brazil, was applied to the prepared porcelain in the same way as the resin cement in Group 1. After application, the porcelain rod was placed on the prepared enamel under a 155 g static load (Figure 1) and the flowable composite was also light-cured from four directions (Incisal, cervical, mesial and distal) for 15 seconds each (60 seconds total).

When the 20 porcelain cylinders were already bonded, the excess resin cement or flowable composite around the bond site was carefully removed with an end-cutting bur in a slow-speed handpiece. The 20 bonded specimens were then stored in distilled water at 37°C for 24 hours to allow the resin layer to partially saturate with water. A longer storage time would have allowed for complete hygroscopic expansion of the resin; however, this study examined the early bond strength of resin-luted etched porcelain to enamel as a result of thermal cycling (Sheth & others, 1988). The specimens were then thermocycled at 5°C and 55°C for 600 cycles with a dwell time of 28 seconds in each bath.

Following thermocycling, bonded assembles were mounted in an Instron Universal Testing Machine (Instron Corp, Canton, MA, USA) and loaded in shear at a crosshead speed of 0.5 mm/minute until bond failure occurred. The force was applied parallel to the enamel flattened surface close to the bonding area and a recording expressed in MPa was made of the shear load at the point of failure (Figure 2). Differences between the means of all groups were statistically analyzed using the Student's-*t* test ($p \leq 0.05$).

RESULTS

Mean bond strength data and standard deviations for each group are shown in Table 1. The analysis indicates that the bond strengths were not influenced by the type of bonding system ($p=0.045<0.05$).

In Group 1, where the porcelain cylinders were bonded with the resin cement, the mean bond strength was 9.66 (± 3.00) MPa. In Group 2, where the porcelain cylinders were bonded with the flowable composite, the mean bond strength was 10.53 (± 3.71) MPa. The mean result found in Group 1 was lower than the mean result in Group 2, but the statistical analysis showed no sig-

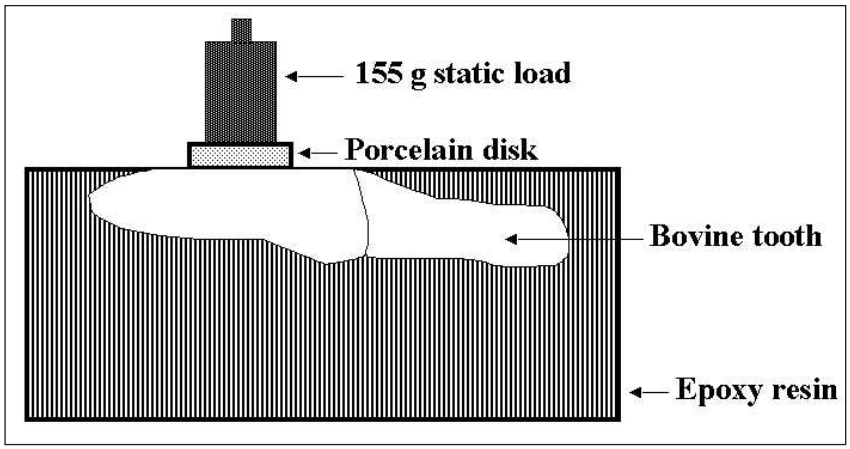


Figure 1. *Assembly used for cementation of porcelain disks.*

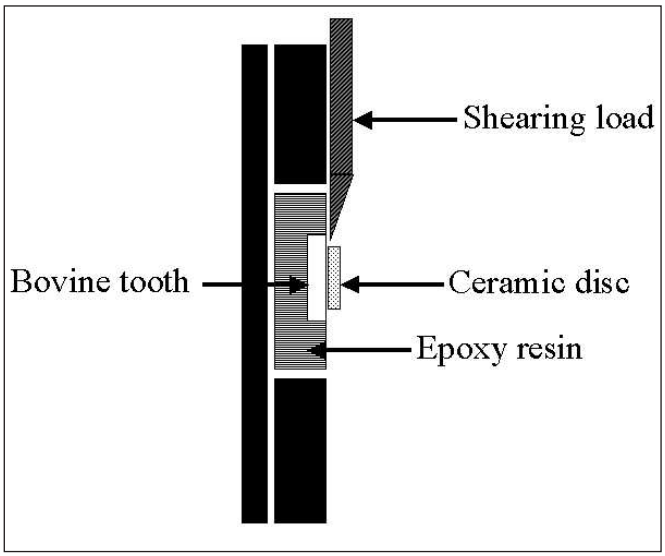


Figure 2. *Assembly used for determination of shear bond strength.*

nificant differences between the control group and the experimental group.

DISCUSSION

The use of porcelain veneer has grown since its introduction in the early 1980s partly because of the conservative nature of the technique. The ability to alter tooth form and color without conventional crown preparations distinguishes porcelain veneers from other methods (Friedman, 1991). Where indicated, the significant axial tooth reduction (1.5 to 2.0 mm), peculiar to full crown preparation, is not necessary. In fact, porcelain veneer preparation rarely requires more than a 0.5 to 1.0-mm reduction (Friedman, 1991; Peumans & others, 2000), which means a significant conservation of healthy tooth structure.

According to Friedman (1991), tooth optics, porcelain shade and opacity, composite shade and luting type, porcelain veneer thickness, existing restorations and (accidentally introduced) air spaces influence the result achieved with veneers. According to this author, the most common errors, however, are associated with the porcelain fabrication and the luting composite.

A growing number of resin-based luting agents are used to seat ceramic restorations, and it is indisputable that a strong, durable bond between the tooth and the ceramic restoration is indispensable for extending the life of the tooth and the restoration (Kato & others, 1996). Usually, a dual-cured luting composite is preferred for porcelain veneer bonding, but light cured resin cement can also be used.

This study evaluated the shear bond strength of porcelain laminate veneers cemented to enamel with flowable composite and test whether it would be a good alternative to resin cements. A dual-cured resin cement

Table 1: *Mean Shear Bond Strengths (MPa)*

Group	N	Mean Shear Bond	SD
1	10	9.66	3.00
2	10	10.53	3.71

was used as a control group, because a wide variety of studies (Calamia & others, 1985; Stangel & others, 1987; Stacey, 1993; Kato & others, 1996; Troedson & Dérand, 1998) have shown clinically acceptable bond strengths between porcelain, resin cement and teeth.

Bovine teeth were used in this study as a substitute for human teeth because human teeth are now scarce, and the objective was to compare shear bond strengths. Previous studies have shown little or no differences for bond strength tests comparing human and bovine teeth (Fowler & others, 1992; Nakamichi, Iwaku & Fusayama, 1983).

The statistical analysis showed that there were no statistically significant differences between the two groups in a level of significance of 95%. The mean shear bond strengths found in Group 1 (9.66 ± 3.00 MPa) and in Group 2 (10.53 ± 3.71 MPa) have shown that the bond strength was not influenced by the kind of luting agent, which means that flowable composite can be an alternative material for porcelain veneer bonding. Even the standard deviation was quite similar, showing that the two materials had almost the same characteristics. While these results were encouraging, the authors believe that flowable composite can be a good alternative to resin cements. However, some considerations should be made before they can indicate it in safety.

First, cementing porcelain veneer with flowable composite is a manufacturer's indication that has attracted little attention to date. According to Behle (1998), flowable composites can be indicated for bonding procedures, but the authors did not find any other report showing the same assertion.

Flowable composites may act as a light-cured resin cement. Some authors (Linden & others, 1991; Peumans & others, 2000) believe that a chemical catalyst might be desirable for porcelain veneer bonding, mainly in thicker (>0.7 mm) porcelain veneers. O'Keefe, Pease and Herrin (1991) have also asserted that the thickness of the veneers had a substantial effect on the light energy passing through the veneers. Based on this information, the authors recommend avoiding the use of light cured cements for porcelain veneer bonding and avoiding the use of flowable composites for the same purpose. Nevertheless, the same authors (Linden & others, 1991; O'Keefe & others, 1991; Peumans & others, 2000) agree that using a catalyst with anterior porcelain veneers is problematic because of the potential for discoloration (Linden & others, 1991), and they believe that an increase in exposure time is sufficient to

overtake the problem of unsatisfactory polymerization through thicker porcelain veneers. In addition, Cardash and others (1993) concluded that dual-cured composite luting resin should be preferred only for porcelain restorations that are 2-mm thick or greater. As porcelain veneer preparation rarely requires more than a 0.5 to 1.0 mm reduction (Friedman, 1991; Peumans & others, 2000), light-cured material can be reliably used for porcelain veneer bonding. In this study, the authors used a 2-mm height porcelain cylinder based on the study by Cardash and others (1993). Apparently, this porcelain thickness did not affect the transmittance of light, as the light-cured flowable composite showed the same bond strength from the dual-cured resin cement. The authors suggest that another shear bond strength study be conducted using the same materials, but using porcelain cylinders with smaller and larger thickness to evaluate whether flowable composites will show lower or higher bond strengths. They should also be compared to the dual-cured cement bond strengths under the same conditions.

Della Bona and Northeast (1994) related the importance of resin try-in paste included in some resin cement kits to assess the final appearance of cemented restorations. In fact, this is a negative point of flowable composite, as it cannot be tested prior to cementation. Nevertheless, Friedman (1991) has asserted that even when try-in techniques are used, the final optics can be realized only after the monomer resin has completed its conversion to polymer, which shows that the try-in paste may not be essential.

Peumans and others (2000) have asserted that a strong correlation was found between the consistency and the film thickness of luting agents, which shows that flowable composites might have the same film thickness as resin cements, as they have almost the same consistency. Based on this finding, the authors were not concerned about film thickness; however, they believe that other studies need to be conducted that compare flowable composite film thickness with resin cement film thickness.

The authors of this study agree with Bayne and others (1998) when they say that some flowable composites appear to be acceptable for veneer cementation. They further believe that studies evaluating bond strengths, microleakage, film thickness or even studies evaluating materials other than those used in this study, are necessary before flowable composite can be indicated as a secure alternative to resin cement. The advantages of flowable composites as a porcelain veneer bonding system were demonstrated in this study, which intended to draw the attention to this new use for flowable composites.

CONCLUSIONS

1. Porcelain veneer cemented to enamel with flowable composite showed shear bond strengths that are not statistically different from porcelain veneer cemented with resin cement.
2. Flowable composite can serve as an alternative to resin cement as a porcelain veneer bonding system, however, further studies are necessary to confirm this conclusion.

Acknowledgements

This study was financially supported by FAPERJ (Fundo de Amparo à Pesquisa do Estado do Rio de Janeiro).

The authors thank Joao Emidio, commercial laboratory, for producing the porcelain disks used in this study.

(Received 22 July 2002)

References

Bayne SC, Thompson JY, Swift EJ Jr, Stamatiades P & Wilkerson M (1998) A characterization of first-generation flowable composites *Journal of the American Dental Association* **129(5)** 567-577.

Behle C (1998) Flowable composites: Properties and applications *Practical Periodontics and Aesthetic Dentistry* **10(3)** 347-351.

Brunton PA & Wilson NHF (1998) Preparations for porcelain laminate veneers in general dental practice *British Dental Journal* **184(11)** 553-556.

Calamia JR & Simonsen RJ (1984) Effect of coupling agents on bond strength of etched porcelain *Journal of Dental Research* **63** Abstract #79 p 179.

Calamia JR, Vaidyanathan J, Vaidyanathan TK & Hirsch SM (1985) Shear bond strength of etched porcelains *Journal of Dental Research* **64** Abstract #1096 p 296.

Cardash HS, Baharav H, Pilo R & Ben-Amar A (1993) The effect of porcelain color on the hardness of luting composite resin cement *Journal of Prosthetic Dentistry* **69(6)** 620-623.

Castelnuovo J, Tjan AH, Phillips K, Nicholls JI & Kois JC (2000) Fracture load and mode of failure of ceramic veneers with different preparations *Journal of Prosthetic Dentistry* **83(2)** 171-180.

Christensen GJ & Christensen RP (1991) Clinical observations of porcelain laminate veneers: A three year report *Journal of Esthetic Dentistry* **3(5)** 174-179.

Della Bona A & Northeast SE (1994) Shear bond strength of resin bonded ceramic after different try-in procedures *Journal of Dentistry* **22(2)** 103-107.

Fowler CS, Swartz ML, Moore BK & Rhodes BF (1992) Influence of selected variables on adhesion testing *Dental Materials* **8(4)** 265-269.

Friedman MJ (1991) Augmenting restorative dentistry with porcelain veneers *Journal of the American Dental Association* **122(7)** 29-34.

Friedman MJ (1998) A 15-year review of porcelain veneer failure—a clinician's observations *Compendium of Continuing Education in Dentistry* **19(6)** 625-628.

Garber D (1993) Porcelain laminate veneers: Ten years later Part 1 Tooth preparation *Journal of Esthetic Dentistry* **5(5)** 56-62.

Kato H, Matsumura H, Tanaka T & Atsuta M (1996) Bond strength and durability of porcelain bonding systems *Journal of Prosthetic Dentistry* **75(2)** 163-168.

Kihn PW & Barnes DM (1998) The clinical longevity of porcelain veneers: A 48-month clinical evaluation *Journal of the American Dental Association* **129(6)** 747-752.

Linden JJ, Swift EJ Jr, Boyer DB & Davis BK (1991) Photo-activation of resin cements through porcelain veneers *Journal of Dental Research* **70(2)** 154-157.

Nakamichi J, Iwaku M & Fusayama T (1983) Bovine teeth as possible substitutes in the adhesion test *Journal of Dental Research* **62(10)** 1076-1081.

Nordbø H, Rygh-Thoresen N & Henaug T (1994) Clinical performance of porcelain laminate veneers without incisal overlapping: 3-year result *Journal of Dentistry* **22(6)** 342-345.

O'Keefe KL, Pease PL & Herrin HK (1991) Variables affecting the spectral transmittance of light through porcelain veneer samples *Journal of Prosthetic Dentistry* **66(4)** 434-438.

Peumans M, Van Meerbeek B, Lambrechts P & Vanherle G (1997a) The five-year clinical performance of direct composite additions to correct tooth form and position Part I: Aesthetic qualities *Clinical Oral Investigations* **1(1)** 12-18.

Peumans M, Van Meerbeek B, Lambrechts P & Vanherle G (1997b) The five-year clinical performance of direct composite additions to correct tooth form and position Part II: Marginal qualities *Clinical Oral Investigations* **1(1)** 19-26.

Peumans M, Van Meerbeek B, Lambrechts P & Vanherle G (2000) Porcelain veneers: A review of the literature *Journal of Dentistry* **28(3)** 163-177.

Pincus CL (1938) Building mouth personality *Journal of the California State Dental Association* **14** 125-129.

Sheth J, Jensen M & Tolliver D (1988) Effect of surface treatment on etched porcelain bond strength to enamel *Dental Materials* **4(6)** 328-337.

Stacey GD (1993) A shear stress analysis of the bonding of porcelain veneers to enamel *Journal of Prosthetic Dentistry* **70(5)** 395-402.

Stangel I, Nathanson D & Hsu CS (1987) Shear strength of the composite bond to etched porcelain *Journal of Dental Research* **66(9)** 1460-1465.

Strassler HE & Nathanson D (1989) Clinical evaluation of etched porcelain veneers over a period of 18 to 42 months *Journal of Esthetic Dentistry* **1(1)** 21-28.

Strassler HE & Weiner S (1995) Seven to ten year clinical evaluation of etched porcelain veneers *Journal of Dental Research* **74** Abstract #1316 p 176.

Troedson M & Dérand T (1998) Shear stresses in the adhesive layer under porcelain veneers *Acta Odontologica Scandinavica* **56(5)** 257-262.

Troedson M & Dérand T (1999) Effect of margin design, cement polymerization, and angle of loading on stress in porcelain veneers *Journal of Prosthetic Dentistry* **82(5)** 518-524.

Walls AW (1995) The use of adhesively retained all-porcelain veneers during the management of fractured and worn anterior teeth Part II: Clinical results after 5-years of follow-up *British Dental Journal* **178(9)** 337-340.

©Operative Dentistry, 2003, **28-4,** 429-439

Dentin Bonding: Effect of Degree of Mineralization and Acid Etching Time

GC Lopes • LCC Vieira • S Monteiro, Jr
M Caldeira de Andrada • CM Baratieri

Clinical Relevance

Bonding to sclerotic dentin resulted in lower bond strength to resin composite. Extending the phosphoric acid etching time can overcome this difficult factor with no detrimental effect to normal dentin considering the bond strength.

SUMMARY

This *in vitro* study verifies whether there are differences between bonding to hypermineralized dentin and normal dentin and if longer acid etching can improve the bond strength to this modified substrate without damaging the bond to normal dentin. Forty-two extracted human molars with chronic occlusal caries were transversally cut with a diamond saw under refrigeration. The occlusal surfaces were ground until the carious lesion was removed, exposing the sclerotic dentin in the center and polished to 600/grid. A 35% phosphoric acid (3M) was applied for 15 seconds in 15 specimens. SingleBond (3M) adhesive system was applied and a hybrid resin composite (Filtek Z250, 3M) was inserted in four 1-mm increments and light-cured. The remaining 15 molars were prepared in the same manner, but with an acid etching time of 30 seconds. After 24 hours in water, the specimens were cut in two perpendicular directions to obtain a cross section of approximately 0.7 mm² (n=25). A visual examination was conducted to select sticks between the two groups: sclerotic dentin (G15S or G30S) and normal dentin (G15N or G30N). Sticks without 100% sclerotic dentin (translucent area) or those with normal areas were not tested. Two-way ANOVA computed the µ-TBS data taking into consideration dentin type and acid etching time. The dentin Knoop hardness number (KHN) of the sticks was verified. A *t*-test compared the KHN data between sclerotic and normal dentin. Twelve additional molars (n=6) were prepared to observe the interface under a SEM. The mean (±SD) microtensile bond strengths (µ-TBS) were: G15S=56.4(±14.9), G15N=69.7(±17.2), G30S=63.2(±15.6) and G30N=67.7(±13.3). Two-way ANOVA showed higher µ-TBS to normal dentin than sclerotic dentin. Duncan's Post Hoc showed G15N had higher mean µ-TBS than G15S. Other comparisons were not significantly different. The *t*-test showed statistically higher microhard-

*Guilherme Carpena Lopes, DDS, MS, professor, Department of Operative Dentistry, Universidade do Sul de Santa Catarina, Tubarão, Brazil

Luiz Clovis Cardoso Vieira, DDS, MS, PhD, professor, Department of Operative Dentistry, Federal University of Santa Catarina, Florianópolis, Brazil

Sylvio Monteiro, Jr, DDS, MS, PhD, professor, Department of Operative Dentistry, Federal University of Santa Catarina, Florianópolis, Brazil

Mauro Amaral Caldeira de Andrada, DDS, MS, PhD, professor, Department of Operative Dentistry, Federal University of Santa Catarina, Florianópolis, Brazil

Carolina Mascarenhas Baratieri, DDS, student, School of Dentistry, Federal University of Santa Catarina, Florianópolis, Brazil

*Reprint request: Rua Laurindo Januário da Silveira nº 947, Apto 34A, Canto da Lagoa, Florianópolis, Santa Catarina, Brazil, 88062-200; e-mail: guilherme_lopes@ig.com.br

ness in sclerotic dentin than in normal dentin ($p<0.0001$). The hybrid layer (HL) formation was observed in all specimens without gap formation in any region. In sclerotic dentin (G15S), the HL was very thin, with minimal resin tags in the dentinal tubules and, when present, they were shorter. Doubling the etching time (G30S) resulted in more resin tags with an HL formation on peritubular dentin. The HL on normal dentin was thicker when it was acid etched for 30 seconds (G30N). Numerous resin tags were present with both etching times. The results suggest that the higher mineral amount in sclerotic dentin makes it difficult to bond to this substrate, resulting in a lower μ-TBS. However, doubling the etching time resulted in μ-TBS similar to normal dentin.

INTRODUCTION

Adhesion to dentin has historically been the target of various investigations in an effort to obtain adhesive systems capable of interacting in an efficient manner with this delicate substrate. Formation of a biofilm on the surface of instrumented dentin has been one reason for the difficulty with interaction of the adhesive system to dentin (Lopes & others, 2002). The smear layer obliterates the tubules and reduces dentinal permeability by 86% (Pashley, Livingstone & Greenhill, 1978). Dentin bonding systems can react with intertubular and peritubular dentin only when the smear layer is removed by acid conditioning or when the system is capable of diffusing through this layer of debris (Pashley & Carvalho, 1997). Thus, the smear layer, which served as a barrier to the first generation of adhesive agents bonding to dentin, might not affect some of the current adhesive systems, because this layer is completely removed by means of a previous treatment of acid etching. In this way, an effective dentin bond seems to be obtained with modern dentin bonding systems. A trend of adhesive dentistry has been to simplify procedures through using single-bottle systems, in spite of maintaining acid etching as a previous step.

The majority of laboratory tests for adhesion to dental tissues have been conducted on recently cut substrate with normal characteristics that have been polished. In spite of the significance these studies have for comparing different techniques and materials, this substrate is not always found in clinical situations. The same cavity may present different substrate conditions, such as instrumented and intact dentin, shallow and deep dentin, or sclerotic and normal dentin.

Recent investigations regarding the performance of single-bottle adhesive systems have used normal dentin as a test surface in spite of carious dentin and cervical sclerotic dentin frequently being the relevant adhesive substrates. In particular, the sclerotic zone of caries-affected dentin will be present on the dentinal surface of many cavity preparations. This substrate is bacteria-free, and for this reason should be preserved. This zone is shown to be harder (Craig, Gehring & Peyton 1959; Grajower, Araz & Bron-Levi, 1977), frequently darkened (Silverstone & Mjör, 1993) due to undermined carious lesions and presents an altered morphology as the tubules are obstructed with mineral crystals. In addition, the dental carious process is known to be multifactorial, that is, capable of being prevented, detained and even reverted, resulting in the arrest of carious lesions. Such chronic caries may represent an important substrate when adhesive restorations are conducted. Clinically, this altered tissue is smooth, bright and darkened by the dentinal sclerosis process.

Microtensile bond strength tests (Sano & others, 1994; Pashley & others, 1999) have allowed for the use of test specimens with a reduced area, making it possible to compare regional adhesion on various dentin sites. Investigations have shown that the efficacy of adhesive systems is reduced when applied to modified dentin substrates on non-carious cervical lesions (Yoshiyama & others, 1996) and the translucent carious zone (Nakajima & others, 1995; 1999; 2000). This fact is frequently attributed to difficulty in conditioning this substrate, which is caused by a tubule obliteration due to mineral deposits. Nevertheless, adhesion to the sclerotic dentin of hypermineralized chronic caries has not yet been tested.

This study evaluated the bond strength to sclerotic occlusal dentin using a single-bottle adhesive system. The hypotheses to be tested were (1) normal dentin provides higher bond strengths than sclerotic dentin, and (2) increased etching time increases the bond strength to sclerotic dentin.

METHODS AND MATERIALS

Tooth Type and Preparation

Forty-two extracted human molars with apparent chronic occlusal caries were selected and stored in physiologic serum for up to three months after extraction. The teeth were sectioned using a diamond disc under water coolant to remove occlusal enamel and the dentin surfaces were abraded with 60/grit silicon carbide (SiC) paper under running water until a flat surface was obtained. Under visual inspection, dark (usually dark brown) and bright areas, suggesting sclerotic dentin, occupied the central part of this flat dentinal surface (Figure 1).

A pilot project was devised to verify the hardness in this darkened dentinal area. Three molars with the same characteristics described above were similarly prepared, which resulted in the occlusal surfaces presenting a dark brown central region. The teeth were cut

longitudinally with a diamond disc at low speed. The two parts were embedded in epoxy resin (Epo-Thin, South Bay Technology Inc, San Clemente, CA, USA) and polished to 1200/grit SiC 24 hours later. The ensemble was rinsed under tap water to remove debris, and Knoop hardness was measured with an equal depth on the darkened region and the visually normal dentin. A Microhardness tester (Shimadzu hmv-2000, Shimadzu Corp, Kyoto 604-8511, Japan) was programmed with a 50g-load for 15 seconds (Craig & others, 1959). Four indentations were made on each specimen, separated by a distance of 50 µm (Harnirattisai & others, 1992). The results of this pilot project show this sclerotic region was hypermineralized and exhibited a dentin hardness 30% greater than normal dentin at the same depth (control). All 42 specimens were polished to 600/grit under constant water coolant to obtain a standard test surface and create a smear layer (Pashley & others, 1988); they were then stored in physiologic saline at room temperature for 24 hours.

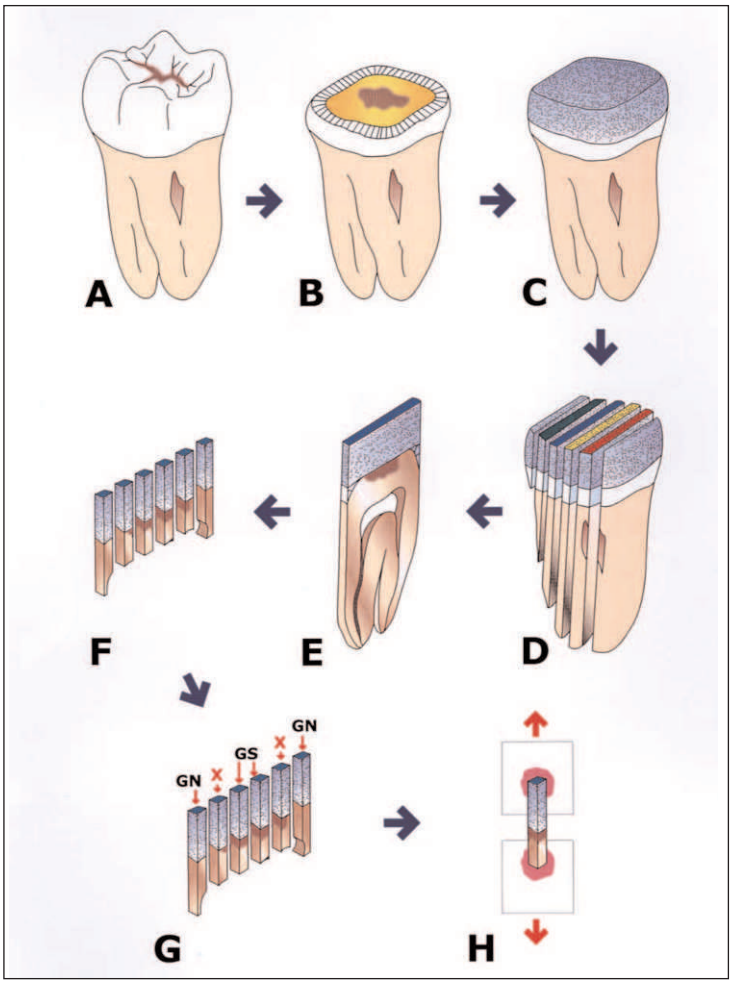


Figure 1. *Schematic representation of the preparation of resin composite-dentin beams for measuring microtensile bond strength in occlusal surfaces of normal vs sclerotic dentin. Adapted from Tay and others (2000)* Journal of Adhesive Dentistry **2(2)** *99-116, with permission.*

Bonding Procedure

Following preparation of the test surfaces and storage in physiologic saline, 15 specimens were rinsed under tap water and dried with air. The SingleBond adhesive system (3M Dental Products, St Paul, MN, USA) was applied per the manufacturer's instructions.

Acid etching with 35% phosphoric acid (Scotchbond Etching Gel, 3M) was performed for 15 seconds, rinsed for 15 seconds, wet dentin was maintained and two consecutive layers of Single Bond (3M) were applied with air drying for two to five seconds and light-cured for 10 seconds using an Optilux 401 (Demetron/Kerr, Danbury, CT, USA) with a previously set light intensity (550 mW/cm^2) using a curing radiometer (Demetron/Kerr). An additional 15 molars were prepared in the same manner but with an acid etching time of 30 seconds. A hybrid resin composite (Filtek Z-250, 3M) was applied to the dentin surface in four increments of 1 mm each with the layers being light cured for 40 seconds. The test specimens were stored in water at room temperature for 24 hours.

Scanning Electron Microscopy

The final 12 specimens were rinsed under tap water and dried with air. Areas of occlusal dentin not affected by the carious process and having a normal chromatic appearance were used as the control. Following the same adhesive procedures described above, six teeth were acid etched for 15 seconds and the other six were etched for 30 seconds.

After the adhesive procedures were completed, the specimens were attached to a cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) equipped with a diamond-impregnated copper disc (South Bay Technology) and cut at a low speed mesiodistally under water coolant. After the specimens were fixed for 12 hours in 2.5% glutaraldehyde in 0.1M sodium cacodilate (Electron Microscopy Sciences, Ft Washington, PA, USA), they were washed in distilled water for one minute and immersed in ethanol solutions of increasing concentrations (25, 50, 70, 90 and 100%). To assure full desiccation, the specimens were immersed in HMDS (Electron Microscopy Sciences) for 10 minutes and dried at room temperature for 24 hours (Perdigão, 1995).

The specimens were embedded in epoxy resin (Epo-Thin, South Bay Technology Inc) and polished to 2400/grit SiC with soft tissue disks that had increasingly fine aluminum-oxide suspensions (South Bay Technology). The specimens were then washed to remove residues.

The specimens were etched with 6N hydrochloric acid for 30 seconds and deproteinated with 2% sodium hypochlorite for 10 minutes. They were assembled on

Table 1: *Division of Each Group According to Dentin Characteristics and Etching Time*

Group	G15N	G15S	G30N	G30S
Dentin Type	Normal	Sclerotic	Normal	Sclerotic
Etching Time	15 seconds	15 seconds	30 seconds	30 seconds

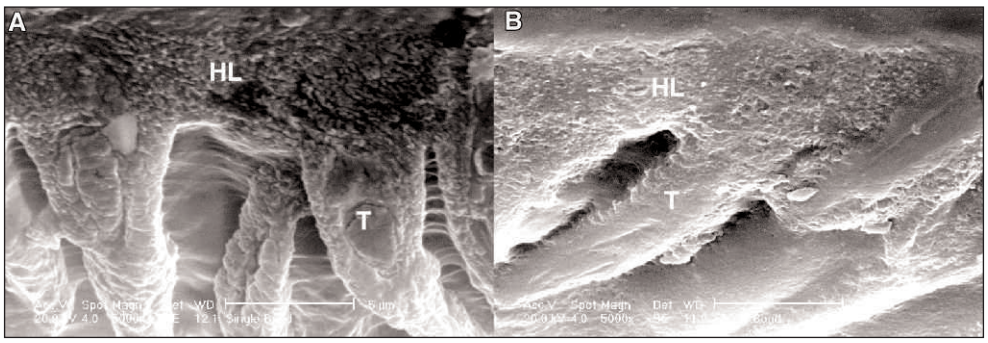


Figure 2. *SEM of the resin-dentin interface of a polished cross-section of normal dentin etched with 35% PA for 15 seconds (A) or 30 seconds (B), rinsed and bonded using SingleBond. The polished surface was exposed to HCl 6N, followed by 2%NaOCl to remove the subsurface dentin, exposing the morphology of the hybrid layer (HL), resin tags (T).*

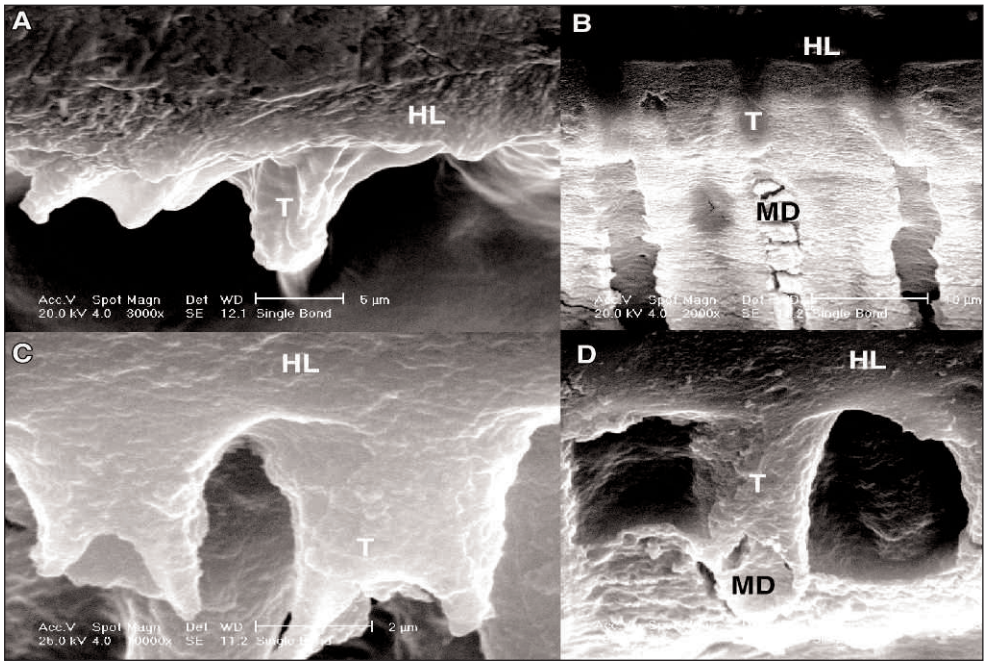


Figure 3. *SEM of the resin-dentin interface of a polished cross-section of sclerotic dentin etched with 35% PA for 15 seconds (A) or 30 seconds (B, C and D), rinsed and bonded using SingleBond. The polished surface was exposed to HCl 6N, followed by 2%NaOCl to remove the subsurface dentin. The hybrid layer (HL) in sclerotic dentin (A) was thinner than that of normal dentin (compare with Figure 2A). A minimal formation of resin tags (T) being observed within the tubules (A) when present, these resin tags (T) were shorter and without the formation of a hybrid layer on the peritubular dentin (A). With 30-seconds etching time, the resin tags (T) are more present but show a void in their inner part (C and D). This central portion was probably filled with mineral deposits (MD) which were subsequently dissolved during inclusion in HCl 6N (C or partially dissolved in other areas (B,D).*

sample-holders, covered with Palladium/Gold (Bal-Tec SCD 005, Balzers, Liechtenstein) and analyzed on a Philips XL 30 SEM (Philips Electronic Instruments Inc, Mahwah, NJ, USA) with a 20 to 25 kV current.

Microtensile Bond Test

The test specimens were bonded with a cyanoacrylate-based adhesive (Zapit, DVA, Corona, CA, USA) to acrylic cylinders in order to make handling easier during cutting. The restored teeth were attached to a cutting machine (Isomet) where a diamond disc (South Bay Technology) water coolant running at low speed cut along the first direction (mesiodistal), produced 0.8-mm thick slices. The restored tooth, after being cut into approximately eight slices, was rotated 90 degrees and cut once again in a bucco-lingual direction. Square-based beams (0.8 x 0.8 mm) resulted from these two cutting operations, leaving resin composite on one end and dentin on the other (Figure 1). Table 1 summarizes the characteristics of the four groups.

Visual examination was conducted to select those beams formed in sclerotic and normal dentin (Figure 1G). Only those beams having 100% sclerotic dentin or normal dentin area were selected. An equal number of beams for sclerotic and normal dentin coming from the same tooth were selected, thus reducing the dentin depth variation and the age difference of the teeth. A total of 25 beams was prepared for each group and stored in water for 24 hours at room temperature.

In preparation for the tensile test, the beams were bonded with a quick polymerization cyanoacrylate-based adhesive (Zapit) in a special jig for microtensile tests (Bencor Mult-T, Danville Engineering, San Ramon, CA, USA) in an Instron Universal testing machine (Model 4444, Instron Corp, Canton, MA, USA) at a speed of 0.5 mm/minute. Before the test, the area next to the adhesive interface of the beams was computed using the Series IX Software System (Instron Corp).

A two-way ANOVA test was used to analyze the data; the variables "presence of dentinal sclerosis" and "etching time" were considered. A Duncan's Post Hoc test was used to identify statistical differences between pairs of means of a confidence level of 95% for each data set.

Fractographic Analysis

To verify the type of fracture following the tensile test, the beams were mounted on sample-holders, covered with Gold/Palladium (Bal-Tec SCD 005) and analyzed on a Philips XL-30 SEM (Philips Electronic Instruments Inc) in a 20 kV current. Every beam was analyzed. According to Nikaido and others (2002), the failure modes were classified into groups.

Microhardness Measurements

Once the type of fracture was established, the dentin portion of the beams was embedded in epoxy resin (Epo-Thin, South Bay Technology) for polishing to 1200/grit after 24 hours. The ensemble was rinsed under tap water to remove residues, and the Knoop microhardness was measured 50 µm from the adhesive interface using a microhardness tester (Shimadzu hmv-2000) programmed with a 50g-load for 15 seconds (Craig & others, 1959). Four indentations were made in each specimen at a minimum distance of 50 µm between indentations (Harnirattsai & others, 1992). The results were given in Knoop Hardness Numbers (KHN). The Student *t*-test was used to compare the KHN data between sclerotic and normal dentin.

RESULTS

Scanning Electron Microscopy

A hybrid layer developed in every specimen and no fissures were visible at the interface in any site (Figures 2 and 3). On normal dentin, the hybrid layer was thicker and had a greater formation of resin tags inside the tubules (Figures 2A and 2B). On sclerotic dentin, the layer was not as thick, with a minimal formation of resin tags being observed within the tubules. When present, these tags were shorter (Figure 3). When comparing adhesion to sclerotic dentin where acid etching is made during the usually suggested time and with the time doubled, photomicrographs showed less resin tags were formed during the former without forming a hybrid layer on the peritubular dentin (Figures 3A). With an extended etching time, the tags were more prominent but showed a void in their inner layer (Figures 3B, 3C and 3D).

Microtensile Bond Test

The mean µ-TBS (±SD) are shown in Table 2. Duncan's post hoc test ranked the mean in two subsets at a confidence level of 95% (Table 2). G15N had the highest mean µ-TBS but was not statistically greater than those of G30N and G30S. G15S had the lowest mean µ-TBS; however, it was not significantly different from those of G30N and G30S. Table 3 shows the results of two-way ANOVA. Statistical significance existed for the type of dentin (p=0.03). Etching time and interaction of the two main factors were not significant.

Table 2: *Mean Microtensile Bond Strength to Dentin (MPa) and Fracture Mode*

Group	G15N	G15S	G30N	G30S
Mean(SD)	69.7(17.2)[a]	56.4(14.9)[b]	67.7(13.3)[ab]	64.2(15.6)[ab]
Fracture	C (95%)	C (85%)	C (53%)	C (61.5%)
Mode	B (5%)	B (7.5%) D (7.5%)	D (47%)	B (23%) D (15.5%)

**Means with the same letter are not significantly different at p≤ 0.05.*
A—cohesive failure within only resin composite; B—partial and complete cohesive failure in adhesive, including remnants of resin composite on the surface; C—mixed failure including cohesive failure in resin composite, cohesive failure in adhesive and failures at top/within/beneath the hybrid layer; D—cohesive failure within dentin.

Table 3: *Two-Way ANOVA of Microtensile Bond Strength Between the Groups*

	SQ	QM	GL	F	P	Significant
Dentin Type	1628.76	332.17	65	4.903	0.0303	Yes
Etching Time	187.64	332.17	65	0.565	0.4550	No
Interaction	550.03	332.17	65	1.658	0.2027	No

Table 4: *Mean of Knoop Hardness Number of Dentin (KHN)*

Group	G15N	G30N	G15S	G30S
Mean(SD)	59.1(2.9)	60.1(5.3)	75.6(5.0)	78.2(6.7)
Dentin	Normal Dentin		Sclerotic Dentin	
Mean(SD)	59.63(4.3)a		76.94(5.9)b	

**Means with different letter are significantly different at p≤ 0.05.*

Dentin Microhardness

Dentin microhardness results are shown in Table 4. The normal dentin group had mean Knoop hardness values of 59.1 (±2.9) KHN for G15N and 60.1 (±5.3) KHN for G30N group. Mean sclerotic dentin hardness was 75.6 (±5.0) KHN for G15S and 78.2 (±6.7) KHN for G30S.

Sclerotic and normal dentin hardness data were compared by means of a Student *t*-test. Sclerotic dentin exhibited greater hardness compared to normal dentin (p=0.0001).

Fractographic Analysis

Table 2 shows the percentage of each failure mode on the fractured surface of all specimens. The majority of specimens exhibited mixed failure of the fracture surface for all

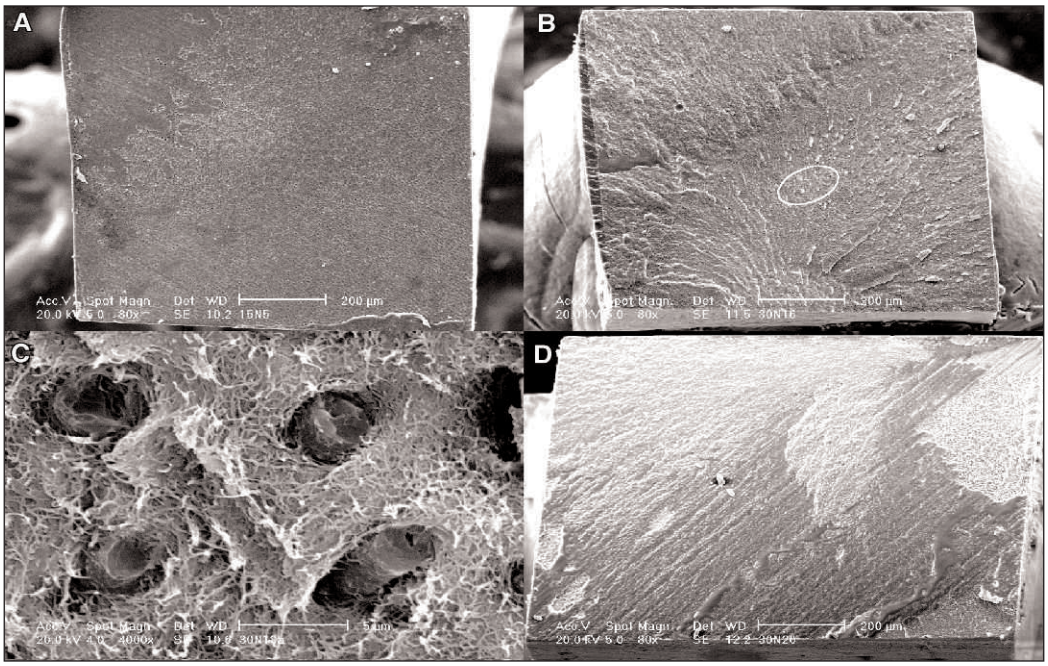


Figure 4. *SEM observations of the fractured surfaces of normal dentin. (A Mixed failure typically in G15N. (B) Cohesive failure within dentin in G30N. (C) Higher magnification of the area indicated by the circle in Figure 4B. The collagen fibrils are exposed at the fractured surface. (D) Mixed failure in G30N.*

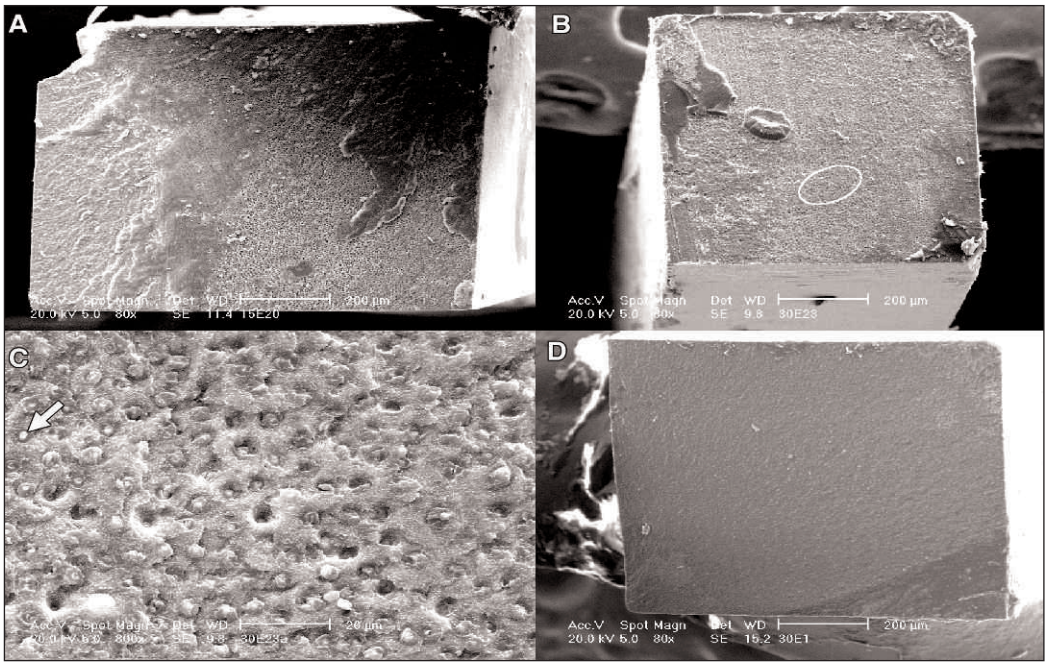


Figure 5. *SEM of the fractured surface sclerotic dentin. (A) Mixed failure typically in G15S. (B) Mixed failure in G30S. (C) Higher magnification of the area indicated by the circle in Figure 5B. Note the presence of hybridization next to the tubule walls (arrow), but having the central portion containing mineral deposits which were not dissolved during 35% PA etching for 30 seconds. (D) Complete cohesive failure in adhesive in G30S.*

groups. In normal dentin etched for 30 seconds (G30N), half the specimens exhibited cohesive failure within dentin (Figure 4B and 4C) and the other half exhibited mixed failure (Figure 4D).

In some of the sclerotic dentin with fractured surfaces etched for 30 seconds (G3OS), resin tags could be observed with hybridization next to the tubule walls. However, the central portion contained mineral deposits that were not dissolved during 35% phosphoric acid etching for 30 seconds (Figure 5C).

DISCUSSION

Researchers have shown that sclerotic dentin is different from normal dentin with respect to acid etching and adhesive procedures (Heymann & others, 1988, 1991; Duke & Lindemuth, 1990; 1991; Marshall & others, 2000). Van Meerbeek and others (1994) suggested that etching peri- and intertubular sclerotic dentin of non-carious lesions would be more difficult, resulting in a thinner hybrid layer and a lesser formation of resin tags within the dentin tubules. And, in some instances, it would be nonexistent at some sites. Such tubule obliteration in sclerotic dentin seems to remain even following acid-etching procedures (Kwong & others, 2000). Also, in select areas, the effective formation of resin tags is impeded by "sclerotic nuclei" extending beyond the mineralized dentin surface, projecting into the interdiffusion zone (Van Meerbeek & others, 1994). In occlusal sclerotic dentin, almost every tubule is occluded. In these situations, the bond between adhesive systems and this substrate seems to depend exclusively on the interdiffusion of the adhesive system into the intertubular dentin (Harnirattsai & others, 1992). Gwinnett (1993) has quantified the role played by infiltration of resin on dentin adhesion. In that

study, approximately two-thirds of the bond strength to occlusal dentin was determined to be attributed to irregular topography of the substrate together with surface physical and/or chemical interrelations, whereas, the remaining third was attributed to adhesive penetration (Gwinnett, 1993). Half of the latter was attributed to the formation of resin tags, and the rest to hybridization in the superficially demineralized intertubular dentin matrix. This aspect seemingly correlates to other investigations conducted on sclerotic tissue. Yoshiyama and others (1996) compared the bond strength to dentin of non-carious cervical lesions that have been naturally and artificially prepared using diamond burs. These authors found the bond strength to be smaller by 15% to 20% on dentin of natural cervical lesions (Yoshiyama & others, 1996). Similar levels of reduction were found in this investigation. Nakajima and others (2000) found analogous percentages of strength reduction when comparing bonding to caries-affected dentin.

When phosphoric acid etchant is applied to a dentin substrate to remove the smear layer, the dentin subsurface becomes demineralized, thus exposing the collagen fibers. To induce good resin-dentin adhesion, resin monomers must penetrate this demineralized sub-surface dentin so as to produce hybridization of dentin with the adhesive systems (Nakabayashi, Kojima & Masuhara, 1982). It is believed that the depth of penetration of monomers is partly dependent on the characteristics of the demineralized collagen fiber network before resin adhesives are applied. The structural characteristics of caries-affected collagen fibers exposed by acid etching can be different from those of collagen fibers in normal dentin. It is thought that the mineral occupying the interfibrillary spaces of caries-affected intertubular dentin can be different from that of the normal apatite due to the cyclic demineralization-remineralization process (Nakajima & others, 1999). The surface zone of etched sclerotic dentin might not have sufficient free functional fibers to establish a microporous network adequate for resin interdiffusion. However, the authors of this study feel that if the cause of bond strength reduction resulted from this alteration in the arrangement of collagen fibers, simply increasing the etching time would not restitute the strength levels found on normal dentin since this procedure would only increase the demineralization depth without altering the interfibrillary arrangement in the intertubular dentin.

Hardness results show caries-affected sclerotic dentin to be harder, pointing to greater mineralization. Induced hypermineralization makes sclerotic dentin more resistant to acid demineralization compared to normal dentin (Duke & Lindemuth, 1990; ten Cate, Jongebloed & Simons, 1987; Weber, 1994). Thus, the hybrid layer formed on sclerotic intertubular dentin was seen to be narrower (Figure 3A) when compared to normal dentin (Figure 2A). This same phenomenon was also observed on the sclerotic dentin of non-carious cervical lesions (Prati & others, 1998; Van Meerbeek & others, 1994). By microscopically evaluating the effect of acid etching on the dentin of non-carious lesions, Sakoolnamarka and others (2000) found a difference between the appearance of demineralization for the sclerotic dentin of non-carious cervical lesions and normal dentin. In this region, the intertubular dentin presented as being still saturated with mineral, with little exposure of the intertubular collagen fibers network in which the adhesive might penetrate (Sakoolnamarka & others, 2000). In this study, etching the sclerotic dentin for twice the time created a slightly thicker hybrid layer (Figure 3D). Despite several authors proving that the size of the hybrid layer has no influence on the bond strength to normal dentin (Finger, Inoue & Asmussen, 1994; Perdigão & others, 2000), the authors of this study believe that an increase in etching time of the sclerotic dentin would expose the intertubular collagen fibers more adequately. This would occur by removing greater mineral content, with an easier penetration of the adhesive, resulting in a bond strength similar to normal dentin. Another interesting aspect is whether the sclerotic peri- and intertubular dentin is more mineralized than unaltered normal dentin. If this is the case, the smear layer that had developed from this dentin might be less acid soluble than the normal smear layer (Van Meerbeek & others, 1993), and possibly removing this layer would not be as efficient if the usual 15-second acid etching was utilized.

The first hypothesis has been confirmed. In fact, it was reasonable to foresee dentin bonding systems with a bonding strategy of mechanical interlocking by forming an interdiffusion zone combined with the development of resin tags in the tubules as not having as much success when applied to sclerotic dentin as non-affected normal dentin. Clinically, Lambrechts, Braem and Vanherle (1987) found it difficult to obtain good quality adhesion to a surface of sclerotic dentin. Similarly, Duke, Robbins and Snyder (1991), in a three-year clinical evaluation, found that most restoration failures occurred on those cervical lesions classified as "more sclerotic." In addition, higher indexes of failure due to the loss of retention of restorations in cervical lesions, as reported by Heymann and others (1991) for older patients, might indirectly support this sclerotic process of the dentin, where an important cause for this adhesive failure would be the alteration of the microstructure and of the composition.

The second hypothesis was confirmed by a more adequate bond strength to the sclerotic dentin that was obtained by doubling the acid etching time. However, the fact that this bond reached strength levels similar to those of normal dentin came as a surprise.

Ultramorphological studies conducted on sclerotic dentin of non-carious cervical lesions had shown that, even after acid etching, mineral deposits remained in the interior of dentin tubules (Gwinnett & Jendressen, 1978; Harnirattisai & others, 1993; Kwong & others, 2000). In those studies, some photomicrographs showed these deposits to be more resistant to acid demineralization than the peritubular dentin itself, resulting in the projection of these intratubular deposits following acid etching (Gwinnett & Jendressen, 1978; Isokawa, Kubota & Kuwajima, 1973; Van Meerbeek & others, 1994; Kwong & others, 2000). Peritubular dentin is more easily dissolved with acid etching than intertubular dentin and intratubular crystal (Ogawa & others, 1983). When the authors of this study compared adhesion to sclerotic dentin performed in the usually suggested time, then in extended time, the photomicrographs showed a smaller number of resin tags formed in the first treatment (Figures 3A). With a doubled etching time, the resin tags became more apparent in spite of presenting a void in the central part (Figure 3B-3D). It is possible that the increased acid etching time produced greater demineralization in the peritubular dentin and in the surface of the intratubular deposits. This resulted in shorter resin tags having adequate hybridization near the peritubular dentin but, apparently, they also exhibited a void in their inner portion (Figures 3B-3D). This central portion was probably filled with these mineral deposits, which were subsequently dissolved during immersion in hydrochloric acid for the electron microscopy process (Figure 3C). The presence of resin tags within the tubules did not offer much retention unless these tags were adequately hybridized next to the lateral walls of the tubules (Swift, Perdigão & Heymann, 1995). Even the hydrophobic adhesives applied to etched dentin form these tags, albeit with a minimum improvement in bond strength (Fusayama & others, 1979). Thus, the increased etching time apparently enhanced the formation of resin tags, though short and with an empty interior, because they had adequate hybridization next to peritubular dentin, a fact that made the values of bond strength to sclerotic dentin similar to those found in normal dentin. The fractographic analysis showed that in sclerotic dentin etched for 30 seconds the major portion of fractured surfaces exhibited mixed failure (Figure 5B). In some of the surfaces seen in the G3OS group, resin tags could be observed with hybridization next to the tubule walls, but the central portion contained mineral deposits that were not dissolved during 35% phosphoric acid etching for 30 seconds (Figure 5C).

Demineralization depth varies according to acid type, concentration, pH, time and viscosity (Van Meerbeek & others, 1992). By employing an etchant with higher dissolution capacity but similar concentration, Nakajima and others (2000) found equal bond strength between caries-affected dentin and normal dentin using the One-Step (BISCO, Schaumberg, IL, USA) adhesive system. Further investigations must be conducted to compare the efficiency of etchants that cause deeper dentin demineralization on hypermineralized sclerotic dentin. It seems that 37.5% phosphoric acid (Kerr, Orange, CA, USA) is apparently the most effective commercially available etchant, resulting in the deepest dentin demineralization of normal dentin, with a depth average close to 5.8 µm (Perdigão, 1995).

By using etchants to remove the smear layer and surface demineralization of the dentin, there is a chance that resin monomers will not diffuse to the full depth of demineralized dentin. This would leave the collagen fibers exposed and susceptible to hydrolysis, possibly weakening the adhesive bond (Watanabe, Nakabayashi & Pashley, 1994; Nakabayashi & Pashley, 1998). Thus, it is speculated that minimally deep demineralization might give the adhesive system a better chance to diffuse across the whole net of collagen fibers. Thus, a minimum etching time of 15 seconds has been suggested by various authors, striving for an adequate bond (Van Meerbeek & others, 2001; Nakabayashi & Pashley, 1998). However, increasing conditioning to 30 seconds does not apparently affect adhesion of the present hydrophilic adhesive systems to normal dentin, with respect to bond strength. Similar to the results of this study, other recent studies do not point to a reduction in bond strength values (Pioch & others, 1998; Perdigão, Gereldeli & Gomes, 2001; Paul & others, 1999). Results obtained by Pioch and others (1998) showed none of the five tested adhesive systems had their bond strengths reduced when 15- and 30-second etching times were compared. This is possibly explained by the small difference in dentin demineralization depth following these two intervals of treatment with 35% phosphoric acid (from 1.9 to 2.7 µm) (Perdigão & Lopes, 2001). Both Perdigão and others (2001) and Paul and others (1999) have not found any difference in the dentin bond when the dentin was etched for longer then 30 seconds. In addition, a recent publication showed one-bottle adhesive systems to offer the same bond strength with different thicknesses of the hybrid layer (Perdigão & others, 2000). The mean intertubular dentin demineralization depth using 37.5% phosphoric acid (Kerr) is 5.8 µm (Perdigão, 1995), which is approximately twice the demineralization depth of 35% phosphoric acid etchant (3M) at about 3 µm (Perdigão & others, 1994). However, the same bond strength is found, regardless of the etching agent (Perdigão & others, 2000). Perdigão and Lopes (2001), measuring demineralization depth with various etching times using a 35% phosphoric acid (UltraEtch, Ultradent), proved that demineralization depth does not increase in the same

proportion as etching time. That is, doubling the acid etching time does not imply that the dentin will also be demineralized to a double depth. Thus, similar bond strength results under different etching times in normal dentin should be expected.

Nevertheless, by increasing demineralization depth, the authors increase the possibility of the adhesive system not penetrating this whole extension, which would leave an unprotected collagen zone. A discrepancy between the etching depth and the adhesive system penetrating capacity might make the collagen-rich zone underlying the hybrid layer susceptible to nanoinfiltration (Sano & others, 1994). Dörfer and others (2000) and Paul and others (1999) found a positive correlation between etching time and the nanoinfiltration level. However, the true clinical meaning that nanoinfiltration has on the longevity of a restoration has not yet been elucidated (Dorfer & others, 2000). The space between the collagen fibers that have not been involved by the adhesive system is too small to permit bacterial invasion (Dorfer & others, 2000). Further clinical evaluation, using different etching times, should be carried out to verify whether this greater nanoinfiltration might have clinical relevance. In addition, a correlation with the results of this study can be inferred because, with an increased etching time in normal dentin, a trend to have cohesive failure within dentin (Figure 4B) was involved, where the collagen fibers could be seen by the adhesive system and without mineral content (Figure 4C). These would possibly be acid-exposed collagen fibers that were not protected by the adhesive system. In spite of this resulting in reduced bond strength, future investigations that reproduced stresses in the oral medium might demonstrate whether these unprotected fibers suffer harm in a wet environment, thus jeopardizing this high bond strength over time.

Clinically, areas of sclerosis appearing in occlusal dentin after cavity preparation will reflect a tissue which, when exposed to acid etchant for the conventional period of time, will cut down the bond strength by approximately 20%. Applying the etching agent for twice as long will generate bond strengths similar to normal dentin without any loss in the bond to this unaltered substrate. Considering that dentin is modified during life (because calcified tissue deposition continues with functioning) (Mendis & Darling, 1979; Duke & Lindemuth, 1991), it is possible that, similar to what has been seen on caries-affected hypermineralized sclerotic dentin, an extended acid etch time might result in a more adequate bond strength to dentin of older patients. This would also lead to an increased longevity of cervical restorations of non-carious lesions. Nevertheless, this aspect calls for future scientific confirmation.

CONCLUSIONS

Based on the results of this study, the authors can conclude that:

1. Hardness of sclerotic dentin is approximately 30% higher than normal dentin with equal depth, showing this to be a more mineralized tissue;
2. The thickness of the hybrid layer formed on sclerotic dentin is less than normal dentin, thus, showing this tissue to be more resistant to demineralization caused by acid etching;
3. Bond strength to sclerotic dentin is not as high as normal dentin;
4. At the time of acid etching the sclerotic occlusal dentin, doubling the time of application of 35% phosphoric acid contributes to obtaining a bond strength similar to normal dentin; and
5. For normal occlusal dentin, no difference exists in bond strength when 35% phosphoric acid etchant is applied following the manufacturer's suggested time (15 seconds), or when the time is extended to 30 seconds.

Acknowledgements

The authors acknowledge Ms Amanda de Cerqueira for her assistance with the English version.

The authors have no financial interest in the materials discussed in this manuscript.

(Received 22 July 2002)

References

Craig RG, Gehring PE & Peyton FA (1959) Relation of structure to the microhardness of human dentin *Journal of Dental Research* **38(3)** 624-630.

Duke ES & Lindemuth JS (1991) Variability of clinical dentin substrates *American Journal of Dentistry* **4(5)** 241-246.

Duke ES & Lindemuth JS (1990) Polymeric adhesion of resin: Contrasting substrates *American Journal of Dentistry* **3(6)** 264-270.

Duke ES, Robbins JW & Snyder DE (1991) Clinical evaluation of a dentinal adhesive system: Three-year results *Quintessence International* **22(11)** 889-895.

Dörfer CE, Staehle HJ, Wurst MW, Duschner H & Pioch T (2000) The nanoleakage phenomenon: Influence of different dentin bonding agents, thermocycling and etching time *European Journal of Oral Sciences* **108(4)** 346-351.

Finger WJ, Inoue M & Asmussen F (1994) Effect of wetability of adhesive resins on bonding to dentin *American Journal of Dentistry* **7(1)** 35-38.

Fusayama T, Nakamura M, Kurosaki N & Iwaku M (1979) Nonpressure adhesion of a new adhesive restorative resin *Journal of Dental Research* **58(4)** 1364-1370.

Grajower R, Azaz B & Bron-Levi M (1977) Microhardness of sclerotic dentin *Journal of Dental Research* **56(4)** 446.

Gwinnett AJ & Jendressen MD (1978) Micromorphological features of cervical erosion after acid conditioning and its relation with composite resin *Journal of Dental Research* **57(4)** 543-549.

Gwinnett AJ (1993) Quantitative contribution of resin infiltration/hybridization to dentin bonding *American Journal of Dentistry* **6(1)** 7-9.

Harnirattisai C, Inokoshi S, Shimada Y & Hosoda H (1993) Adhesive interface between resin and dentin of cervical erosion/abrasion lesions *Operative Dentistry* **18(4)** 138-143.

Harnirattisai C, Inokoshi S, Shimada Y & Hosoda H (1992) Interfacial morphology of an adhesive composite resin and etched caries-affected dentin *Operative Dentistry* **17(6)** 222-228.

Heymann HO, Sturdevant JR, Brunson WD, Wilder AD, Sluder TB & Bayne SC (1988) Twelve-month clinical study of dentinal adhesives in Class V cervical lesions *Journal of the American Dental Association* **116(2)** 179-183.

Heymann HO, Sturdevant JR, Bayne SC, Wilder AD, Sluder TB & Brunson WD (1991) Examining tooth flexure effect on cervical restorations: A two-year clinical study *Journal of the American Dental Association* **122(6)** 41-47.

Isokawa S, Kubota K & Kuwajima K (1973) Scanning Electron Microscope study of dentine exposed by contact facets and cervical abrasion *Journal of Dental Research* **52(1)** 170-174.

Kwong SM, Tay FR, Yip HK, Kei LH & Pashley DH (2000) An ultrastructural study of the application of dentine adhesives to acid-conditioned sclerotic dentin *Journal of Dentistry* **28(7)** 515-528.

Lambrechts P, Braem M & Vanherle G (1987) Buonocore Memorial Lecture Evaluation of clinical performance for posterior composite resins and dentin adhesives *Operative Dentistry* **12(2)** 53-78.

Lopes GC, Baratieri LN, Andrada MAC & Vieira LCC (2002) Dental adhesion: Present state of the art and future perspectives *Quintessence International* **33(3)** 213-224.

Marshall GW, Chang YJ, Saeki K, Gansky SA & Marshall SJ (2000) Citric acid etching of cervical sclerotic dentin lesions: An AFM study *Journal of Biomedical Material Research* **49(3)** 338-344.

Mendis BR & Darling AI (1979) Distribution with age and attrition of peritubular dentine in the crowns of human teeth *Archives of Oral Biology* **24(2)** 131-139.

Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials Research* **16(3)** 265-273.

Nakabayashi N & Pashley DH (1998) Hybridization of dental hard tissues Tokyo Chicago Quintessence Publishing.

Nakajima M, Sano H, Burrow MF, Tagami J, Yoshiyama M, Ebisu S, Ciucchi B, Russell CM & Pashley DH (1995) Tensile bond strength and SEM evaluation of caries-effect dentin using dentin adhesives *Journal of Dental Research* **74(10)** 1679-1688.

Nakajima M, Sano H, Zheng L, Tagami J & Pashley DH (1999) Effect of moist vs Dry bonding to normal vs caries-affected dentin with Scotchbond Multi-Purpose Plus *Journal of Dental Research* **78(7)** 1298-1303.

Nakajima M, Sano H, Urabe I, Tagami J & Pashley DH (2000) Bond strength of single-bottle dentin adhesives to caries-effected dentin *Operative Dentistry* **25(1)** 2-10.

Nikaido T, Kunzelmann KH, Ogata M, Harada N, Yamaguchi S, Cox CF, Hickel R & Tagami J (2002) The *in vitro* dentin bond strengths of two adhesive systems in Class I cavities of human molars *Journal of Adhesive Dentistry* **4(1)** 31-39.

Ogawa K, Yamashita T, Ichijo T & Fusayama T (1983) The ultrastructure and hardness of the transparent layer of human carious dentin *Journal of Dental Research* **62(1)** 7-10.

Pashley DH & Carvalho RM (1997) Dentin permeability and dentin adhesion *Journal of Dentistry* **25(5)** 355-372.

Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA & Tay F (1999) The microtensile bond test: A review *Journal of Adhesive Dentistry* **1(4)** 299-309.

Pashley DH, Livingstone MJ & Greenhill JD (1978) Regional resistance to fluid flow in human dentin *in vitro Archives of Oral Biology* **23(9)** 807-810.

Pashley DH, Tao L, Boyd L, King GE & Horner JA (1988) Scanning electron microscopy of the substructure of smear layer in human dentine *Archives of Oral Biology* **33(4)** 265-270.

Paul SJ, Welter DA, Ghazi M & Pashley DH (1999) Nanoleakage at the dentin adhesive interface vs microtensile bond strength *Operative Dentistry* **24(3)** 181-188.

Perdigão J (1995) An ultra-morphological study of human dentine exposed to adhesive systems Thesis Leuven.

Perdigão J, Geraldeli S & Gomes G (2001) Effect of etching time on microtensile dentin bond strengths *Journal of Dental Research* **80** Abstract #539 p 103.

Perdigão J & Lopes M (2001) The effect of etching time on dentin demineralization *Quintessence International* **32(1)** 19-26.

Perdigão J, May KN, Wilder AD & Lopes M (2000) The effect of depth of dentin demineralization on bond strengths and morphology of the hybrid layer *Operative Dentistry* **25(3)** 186-194.

Perdigão J, Swift EJ, Denehy GE, Wefel JS & Donly KJ (1994) *In vitro* bond strengths and SEM evaluation of dentin bonding systems to different dentin substrates *Journal of Dental Research* **73(1)** 44-55.

Pioch T, Stontz S, Buff E, Duschner H & Staehle HJ (1998) Influence of different etching times on hybrid layer formation and tensile bond strength *American Journal of Dentistry* **11(5)** 202-206.

Prati C, Chersoni S, Mongiorgi R, Montanari G & Pashley DH (1999) Thickness and morphology of resin-infiltrated dentin layer in young, old, and sclerotic dentin *Operative Dentistry* 2**4(2)** 66-72.

Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R & Pashley DH (1994) Relationship between surface area for adhesion and tensile bond strengths-Evaluation of a micro-tensile bond test *Dental Materials* **10(4)** 236-240.

Silverstone LM & Mjör IA (1993) Dental caries in Hörsted-Bindslev P & Mjör IA (eds) *Modern Operative Dentistry* Chicago Quintessence Publishing 2nd edition.

Skoolnamarka R, Burrow MF, Prawer S & Tyas MJ (2000) Micromorphological investigation of non-carious lesions treated with demineralizing agents *Journal of Adhesive Dentistry* **2(4)** 279-287.

Swift EJ, Perdigão J & Heymann HO (1995) Bonding to enamel and dentin: A brief history of the art *Quintessence International* **26(2)** 95-110.

ten Cate JM, Jongebloed WL & Simons YM (1987) Adaptation of dentin to the oral environment in Thylstrup A, Leach SA & Qvist V (eds) *Dentin and Dentin Reactions in the Oral Cavity* Oxford IRL Press.

Van Meerbeek B, Braem M, Lambrechts P & Vanherle G (1994) Morphological characterization of the interface between resin and sclerotic dentine *Journal of Dentistry* **22(3)** 141-146.

Van Meerbeek B, Inokoshi S, Braem M, Lambrechts P & Vanherle G (1992) Morphological aspects of the resin-dentin interdiffusion zone with different dentin adhesive systems *Journal of Dental Research* **71(8)** 1530-1540.

Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry *Operative Dentistry* **26(Supplement 6)** 119-144.

Van Meerbeek B, Dhem A, Goret-Nicaise M, Braem M, Lambrechts P & Vanherle G (1993) Comparative SEM and TEM examination of the ultrastructure of the resin-dentin interdiffusion zone *Journal of Dental Research* **72(2)** 495-501.

Watanabe I, Nakabayashi N & Pashley DH (1994) Bonding to ground dentin by a Phenyl-P self-etching primer *Journal of Dental Research* **73(6)** 1212-1220.

Weber DF (1994) Human dentin sclerosis: A microradiographic survey *Archives of Oral Biology* **19(2)** 163-169.

Yoshiyama M, Sano H, Ebisu S, Tagami J, Ciucchi B, Carvalho RM, Johnson MH & Pashley DH (1996) Regional strengths of bonding agents to cervical sclerotic root dentin *Journal of Dental Research* **75(6)** 1404-1413.

©Operative Dentistry, 2003, **28-4,** 440-444

Cytotoxicity Evaluation of Single Component Dentin Bonding Agents

L Vajrabhaya • A Pasasuk • C Harnirattisai

Clinical Relevance

Applying a self-etching primer agent in clinical procedures where the remaining dentin is 0.5-mm thick or less after removing decay is safer to the subordinate pulpal tissue compared to using a total-etching bonding system. However, if the remaining dentin is more than 0.5-mm thick, the cytotoxic level may not be different for both types of agents.

SUMMARY

This study evaluated the cytotoxicity of four single component dentin bonding agents: Syntac Single Component, Prime & Bond 2.1, Single Bond and One Up Bond F. The test materials were applied on dentin discs of dentin barrier models in the same way as in the clinical procedures recommended by each manufacturer. Cell viability of L 929 after exposure with the bonding agents was determined by MTT assay. The results revealed that cell survival of the first three bonding agents was 60%, while the fourth was an impressive 93%. This study showed that a total-etching bonding system is more cytotoxic than a self-etching bonding system.

*La-ongthong Vajrabhaya, DDS, associate professor, Dept of Operative Dentistry, Faculty of Dentistry, Mahidol University, Bangkok, Thailand

Apaporn Pasasuk, DDS, MSc, instructor, Dept of Restorative Dentistry, Khonkaen University, Khonkaen, Thailand

Choltacha Harnirattisai, DDS, PhD, associate professor, Dept of Operative Dentistry, Faculty of Dentistry, Mahidol University, Bangkok, Thailand

*Reprint request: Yothi str, Bangkok 10400, Thailand; e-mail: dtlvj@mahidol.ac.th

INTRODUCTION

Adhesive dentistry was introduced into clinical dentistry in 1955 after developing the phosphoric acid etching technique on enamel (Buonocore, 1955). Acid etched dentin provides micro-mechanical retention for resin composite restoration material. Dentinal adhesion has been more difficult and less predictable because of its complex histology structure and the variable composition of dentin. In addition, the formation of a smear layer from dentin cutting decreases the permeability of resin into dentinal tubules. This means that the bond strength of the resin to the underlying dentin is limited by the cohesive strength of the smear layer.

Fusayama and others (1979) demonstrated an improvement in dentin bond strength by etching. After hydroxyapatite is removed by etching, the collapsed collagen network in dentin is raised to its original level by applying the primer agent. Unfilled adhesive resin is then applied and penetrates into the primed dentin copolymerizing with the primer to form a resin reinforced dentin called the "inter-diffusion zone" or "hybrid layer." This term was first described by Nakabayashi and Pashley (1998) and has become the primary bonding mechanism of most current adhesive systems. There

were originally three steps in the initial development of dentin primer of the clinical resin restorative system. Recently, various manufacturers have simplified the bonding procedures into two steps by introducing a combination of the primer solvent with the adhesive resin into a single bottle. These systems differ in their composition of the primer solvent that may contain ethanol/water, acetone or water only. They require etching as a step prior to application. More recently, in some systems, acid is included in the same bottle as the primer and bonding agent and becomes a single step component or a self-etching primer bonding system. The restorative procedure in this system, a modified smear layer, becomes less complicated than the previous one. The cytotoxicity of these bonding systems is what is being researched for clinical practice.

Dentin thickness influences the concentration and amount of bonding agents that penetrate through dentin into pulp space. So, the diffusion rate should be inversely proportional to the dentin thickness and directly proportional to the fraction of the cross-sectional area of dentin composed of dentinal tubules (Hamid & Hume, 1997). There is some controversy regarding whether pulp is damaged by diffusable hydrophilic resin, which is the main component of the bonding agent, especially if the remaining dentin above pulpal tissue is 0.5 mm or less. Also, it is questionable whether the new concept of applying adhesive resin directly onto exposed pulp tissue for pulp capping causes pulpal inflammation.

This study investigated the cytotoxicity of removal smear layer systems with their different primer compositions and a modified smear layer adhesive bonding system by using a dentin barrier test method.

METHODS AND MATERIALS

Cells

The target cells used in this experiment were L929 mouse fibroblast cell lines obtained from the Research Unit, Faculty of Dentistry, Mahidol University. Cells were maintained at 37°C under 5% CO_2 and 100% humidity in Dulbecco Modified Eagle's Medium supplemented with 10% fetal calf serum and antibiotic (penicillin G 200 µg/ml + streptomycin 200 µg/ml + fungizone 2 µg/ml). The medium was changed every other day, and when cells reached confluency, they were detached using 0.2% (w/v) trypsin and transferred to a new culture flask.

Dentin Disc Preparation

Intact non-occluded human third molars were extracted from patients between 19 and 25 years of age and stored in a physiological saline at 4°C prior to the experiment. They were then cut into cross sections 500 µm thick. The pulpal side of the dentin discs was etched with 50% citric acid to promote cell adhesion. They were soaked in physiological saline solution and disinfected by autoclaving at 121°C for 25 minutes. Before cell seeding, they were inserted into stainless steel inserts.

Seeding Cells on Dentin Disc

The sterile stainless steel inserts with dentin discs were put on agar medium in a 24-cell tissue culture plate. Then, 2 ml of L929 cell suspension at a concentration of 6.65×10^5 cells/ml was seeded on the pulpal side of the dentin disc and incubated at 37°C with 5% CO_2 at 100% humidity for 24 hours to obtain monolayer cell growth.

After the incubation period, the stainless steel inserts with the dentin disc were placed in the dentin barrier model. The pulpal side with monolayer cell attachment was put in the lower part of the model in contact with the culture medium. The model was then ready for testing with the bonding agents.

Test Materials

The test materials in this study were Syntac Single-Component, Prime & Bond 2.1, Single Bond and One Up Bond F. The first three materials are one-bottle bonding systems placed in different solvents of water, acetone and ethanol/water, respectively. The last material is a self-etching bonding system that is a modified smear layer primer. Table 1 lists the manufacturers and components of each test material.

Dentin Barrier Test Model

A cell culture chamber made of polycarbonate with a base of 40 x 40 mm and a height of 30 mm was used as the *in vitro* model (Schmalz & Schweikl, 1994). The chamber was separated into a pulpal side and cavity side by the dentin disc that was placed inside the stainless steel insert (as prepared in step 2). Cells were grown on the pulpal side (as described in step 3), while test material was placed in a silicone tube of the cavity side.

The Test Procedure

The test material was applied on the cavity side of the dentin disc according to the manufacturer's recommendation. The steps of testing on the dentin test model simulated actual clinical procedures. Briefly, the dentin discs were etched with 35% phosphoric acid before applying bonding agents of the Syntac Single-Component, Prime & Bond 2.1 and Single Bond groups. No acid was required on the dentin in the One Up Bond F group.

In the control group, a cotton pellet soaked with some culture media was put into the cavity side instead of bonding agents. Each test material and control group was conducted in five models and repeated in triplicate. The enzyme activity of the target cells was analyzed using MTT assay (dimethyl thiazol diphenyl tetrazolium bromide) after a 24-hour incubation period.

Table 1: *The Components in Each Bonding System*

Product	Components		Manufacturer
	Etchant	Adhesive	
Syntac Single Component	37%H_3PO_4	PAAMM, HEMA Maleic acid, modified Polyacrylic acid Methacrylate, water	Vivadent, Schaan, Liechtenstein
Prime & Bond 2.1	36% H_3PO_4	UDMA, PENTA Bisphenol A, Dimethacrylate, Acetone	Dentsply DeTray, Konstanz, Germany
Single Bond	35% H_3PO_4	BIS-GMA, HEMA Polyalkenoic acid Polymer, water Ethanol	3M Dental Products, St Paul, MN, USA
One Up Bond F	-	Bonding A: MAC 10, Methacrylate, Methacryloxyalkyl acid phosphate Bonding B: MMA, HEMA, water, F-deriverable, Micro filler	Tokuyama Corp Tokyo, Japan

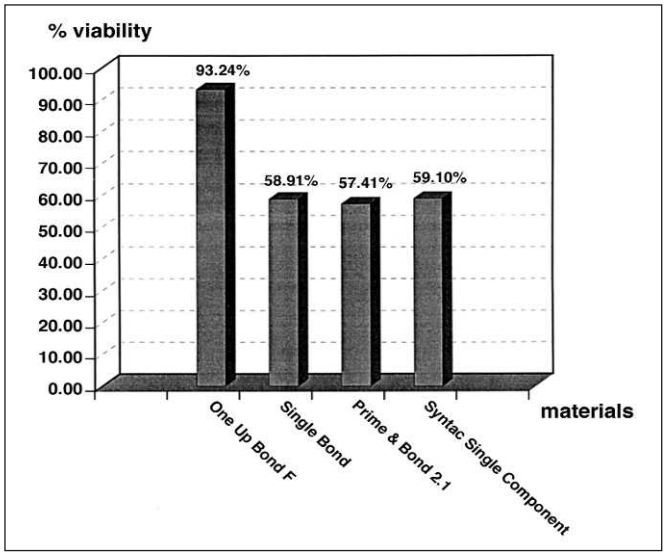


Figure 1. *Percentage of cell viability after diffusing of each dentin bonding system.*

MTT Assay

The dentin disc was removed from the stainless steel insert of the model and immediately inserted into freshly mixed 0.5 ml MTT solution (1 disc/well) in 48 tissue culture plate. The plate was incubated for three hours. Then, MTT solution was suctioned out and the disc washed twice with 0.5 ml PBS while still in the well. Two-hundred and fifty microlitres of DMSO (dimethyl sulfoxide) were put into each well. The plate was agitated for 30 minutes to enhance disbanding of the MTT formazan. Two hundred microlitres of aliquot from each well were drawn to a 96-well plate and the spectrophotometric absorbance (optical density) was measured at 540 nm with DMSO acting as a blank.

Statistical Analysis

The mean optical density of each dentin bonding agent was determined. Results of the experimental groups were expressed as percentages of the control. Statistical analysis was performed applying a Kruskal-Wallis test to compare differences among the groups. Then, each pair of test materials was compared using a two-sample Willcoxon rank sum (Mann-Whitney) test.

RESULTS

Figure 1 shows the percentages of cell viability after the diffusion of each dentin bonding system. After contact with Syntac Single Component, Prime & Bond 2.1 and Single Bond, the viability of the cells were the same in each case ($p>0.05$). The cell percentage in the One Up Bond F group showed a rather dramatic increase to 93.24%. This did not differ from the control group but was significantly different from the three bonding systems mentioned above ($p<0.05$).

DISCUSSION

Spangberg and Pascon (1988) showed that preparation of material for the experiments significantly altered the apparent cytotoxicity effect of a material. They stress the importance of material preparation, with International Organization for Standardization (ISO) 7405 (1994) encouraging the *in vitro* dentin barrier model for this particular type of experiment, especially Schmalz and Schweikl's (1994) model. Because dentin is an important barrier against diffusion of substances heading toward the pulp tissue (Hamid & Hume, 1997), their model closely simulates *in vivo* conditions where dentin interposes between dental materials and pulpal cells. This study simulated the clinical model and procedures in applying bonding agent on dentin. Also, the fittingly sensitive method of cytotoxicity test, MTT assay, was used.

The results of the percentage of cell viability in this study revealed that One Up Bond F (self-etching bonding system) had less cytotoxicity than the other three bonding agents. There are certain factors that contribute to this.

One Up Bond F is a smear layer modifying bonding agent. Smear layer is left on dentin so that less leachable toxic substances from the bonding agent can penetrate the dentin disc toward target cells underneath.

On the other hand, dentin etched with phosphoric acid and primed for the total-etching bonding system has a higher permeability of the dentinal tubules. Stanley, Going and Chauncey (1975) concluded from their experiment that the intensity of pulpal response was boosted as a result. When the remaining dentin thickness was 1 mm or less, regardless of whether it was entirely primary dentin or primary and reparative dentin, the percentage of abscess formation increased. In this way, total-etching bonding agents are more toxic than self-etching bonding agents.

White and others (1994) showed that acid etched dentin producing slight pulpal irritation is a transient phenomenon. After acid etching on dentin, two phenomenon occur when dentin bonding agent is applied. First, monomer diffuses at a higher rate into dentinal tubules. Second, since etching of dentin is performed in an aqueous environment that may interfere with the polymerization reaction of the bonding resin, the uncured monomer dissolves and diffuses through dentin to affect the cells underneath.

One Up Bond F has a fluoride component not found in the other three dentin bonding systems. Nakade and others (1999) suggested that the low concentration of fluoride could be a therapeutic agent. This might be one of the reasons that One Up Bond F revealed less cytotoxicity than the other three bonding systems.

Another reason for possible cytotoxicity may be that most current dentin bonding agents contain HEMA to enhance bond strength to dentin. The molecular weight of HEMA is 130.14 and is highly water-soluble. High concentration and early release of HEMA in the bonding agent may release free, unpolymerized HEMA within the dentinal tubules. Residual water penetration in the dentinal tubules and hybrid layer may result in poor polymerization and increased monomer release. The study by Bovillaquet and others (1998) suggested that decreased dentin thickness, particularly dentin with high permeability and unpolymerized monomer, pose a potential risk to the health of pulpal tissues. Therefore, dentin bonding systems that contain a higher concentration of HEMA may cause a greater diffusion rate of HEMA and other monomers through dentin.

Dentin permeability *in vivo* is not a constant. It can be changed when cavity preparation has been performed. It is a response of the pulpodentinal complex to irritation in a vital tooth. Large plasma protein in the pulpal fluid, such as fibrinogen, could be absorbed through the dentinal tubule wall. This will result in a decrease in dentin permeability (Vongsavan, Matthews & Matthews, 2000).

Every effort has been made to simulate *in vivo* conditions in the laboratory; however, it is impossible to create an environment that is a total replica. There are other factors that may help to decrease cytotoxicity as well, that is, pulpal blood flow, multilayer cells etc.

In cases of deep cavities, leaving only 0.5 mm of remaining dentin or in direct pulp capping, it is very important to choose the right type of bonding agents. The authors have seen quite an astounding difference in cell survival rates between the two systems. The recommended dentin barrier model experiment *in vitro* has shown that it is safer for pulpal cells to use bonding agents with self-etching primer bonding systems than total-etching systems with phosphoric acid.

CONCLUSIONS

Under the conditions of *in vitro* cytotoxicity testing, total and self-etching bonding systems were simulated in dentin barrier models. The study revealed that total-etching bonding systems were more cytotoxic to the target cells underneath dentin discs than self-etching bonding systems. Hence, other than the dangers of bacterial penetration, pulpal irritation should also be considered when choosing a system of bonding agent in clinical practice.

Acknowledgements

This study was supported by a Mahidol University Research grant. The authors thank Mrs Alka Gibbs for her critical reading of this manuscript.

(Received 2 August 2002)

References

Bovillaguet S, Virgillito, Wataha J, Ciucchi B & Holz J (1998) The influence of dentine permeability on cytotoxicity of four dentine bonding systems, *in vitro Journal of Oral Rehabilitation* **25(1)** 45–51.

Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34(6)** 849–853.

Fusayama T, Nakamura M, Kurosaki N & Iwaku M (1979) Non-pressure adhesion of a new adhesive restorative resin *Journal of Dental Research* **58(4)** 1364–1370.

Hamid A & Hume WR (1997) The effect of dentine thickness on diffusion of resin monomers *in vitro Journal of Oral Rehabilitation* **24(1)** 20–25.

International Organization for Standardization (1994) ISO 7405: Preclinical evaluation of biocompatibility of medical devices used in dentistry, test methods for dental material *ISO*.

Nakabayashi N & Pashley DH (1998) *Hybridization of Dental Hard Tissues* 1st ed Quintessence Publishing Co Ltd Tokyo p 57–83.

Nakade O, Koyama H, Arai J, Ariji H, Takada J & Kaku T (1999) Stimulation by low concentrations of fluoride of the proliferation and alkaline phosphatase activity of human dental pulp cells *in vitro Archives of Oral Biology* **44(1)** 89-92.

Schmalz G & Schweikl H (1994) Characterization of an *in vitro* dentine barrier test using a standard toxicant *Journal of Endodontics* **20(12)** 592–594.

Spangberg L & Pascon EA (1988) The importance of material preparation for the expression of cytotoxicity during *in vitro* evaluation of biomaterials *Journal of Endodontics* **14(5)** 247–250.

Stanley HR, Going RE & Chauncey HH (1975) Human pulp response to acid pretreatment of dentin and to composite restoration *Journal of the American Dental Association* **91(4)** 817–825.

Vongsavan N, Matthews RW & Matthews B (2000) The permeability of human dentine *in vitro* and *in vivo Archives of Oral Biology* **45(11)** 931-935.

White KC, Cox CF, Kanka J 3rd, Dixon DL, Farmer JB & Snuggs HM (1994) Pulpal response to adhesive resin systems applied to acid etched vital dentine: Damp versus dry primer application *Quintessence International* **25(4)** 259–268.

©Operative Dentistry, 2003, **28-4,** 445-452

Interfacial Gaps Following Ceramic Inlay Cementation vs Direct Composites

K Iida • S Inokoshi • N Kurosaki

Clinical Relevance

To prevent marginal enamel micro-fracture, an adhesive inlay restoration is preferable for a large Class II cavity.

SUMMARY

This study compared the interfacial integrity of Class II ceramic inlay restorations and direct resin composite restorations. The influence of a flowable resin composite liner was also evaluated. Class II DO cavities were prepared in 40 recently extracted mandibular molars and assigned to four treatment groups. Group A: direct composite restoration; Group B: Cerec inlays fabricated and cemented with a resin cement; Group C: adhesive lining with a flowable resin composite used prior to resin composite restoration; Group D: lining with a flowable resin composite prior to cementation of Cerec inlays. After finishing, polishing and thermocycling (4°C and 60°C x 500), the samples were cross-sectioned in a mesio-distal direction along the center of the fillings or inlays. The cross-sectioned surface was polished, and the adhesive interfaces between resin and enamel or dentin were observed under a scanning laser measurement microscope.

Ceramic inlay restorations showed no interfacial gaps in enamel, but direct resin composite restorations showed a significantly higher incidence of gaps at the interface or cracks in the interfacial enamel (p=0.0002). No differences were found in the resin–dentin interfaces for both the inlay and direct resin composite restorations. The use of a flowable resin composite as an adhesive liner produced a significantly greater gap-free resin-dentin interface in Cerec inlay and direct resin composite restorations (p=0.0233 & 0.0009), but it did not reduce gap formation at the resin-enamel interface.

*Koji Iida, DDS, assistant professor, Tokyo Medical and Dental University, Faculty of Dentistry, Section of General Dentistry, Department of Comprehensive Oral Health Care, Division of Comprehensive Patient Care, Graduate School, Tokyo, Japan

Shigehisa Inokoshi, DDS, PhD, private practitioner, Inokoshi Dental Clinic, Ueno, Tokyo, Japan

Norimasa Kurosaki, DDS, PhD, professor of General Dentistry, Tokyo Medical and Dental University, Faculty of Dentistry, Section of General Dentistry, Department of Comprehensive Oral Health Care, Division of Comprehensive Patient Care, Graduate School, Tokyo, Japan

*Reprint request: 5-45 Yushima 1-chome, Bunkyo-ku, Tokyo 113-8549, Japan; e-mail: iida.gend.@tmd.ac.jp

INTRODUCTION

Patient demands for aesthetic, tooth-colored restorations and environmental concerns about the use of mercury are driving down the use of amalgam for posterior restorations. The success of tooth-colored restorations, whether direct or indirect resin composites or ceramics, greatly depends on the effectiveness of the adhesive materials that must affix the restoration to tooth structure. They must also compensate for the fragility of the restoration and restored tooth and guarantee final marginal adaptation (Banks, 1990). Recent improvements in adhesive resin technology and resin compos-

ites have enabled the use of resin composite materials and brittle ceramics even for posterior teeth, which now provide several options for restoring large carious cavities.

However, resin materials have the drawback of shrinkage during polymerization (Feilzer, de Gee & Davidson, 1988). When shrinkage stress exceeds bond strength of the adhesive resin, separation at the adhesive interface occurs. When bond strength exceeds shrinkage stress and cohesive strength of the tooth, fracture of the tooth structure will occur (Davidson, 1997).

Enamel microfractures have been reported to occur due to a marked increase in the bond strength of resin to tooth structure (Jorgensen, Asmussen & Shimokobe, 1975; Han, Okamoto & Iwaku, 1992; Prati & other, 1992; Belli & others, 2001; Dietschi & others, 2002; Peutzfeldt & Asmussen, 2002; Malmström & others, 2002). This problem is enhanced in large, deep restorative cavities. Since incremental filling techniques (Crim, 1991; Beznos, 2001; Aguiar, Ajudarte & Lovadino, 2002) and the combined use of a flowable resin composite liner (Belli & others, 2001; Beznos, 2001; Chuang, Liu & Jin, 2001; Malmström & others, 2002) have failed to solve this problem, most clinicians have addressed it by placing indirect restorations (Shortall & others, 1989; Dietschi & Spreafico, 1997). The use of resin composites as luting cements for porcelain or resin composite inlay restorations reduces the adverse effects of bulk polymerization contraction of posterior resin composite restorations because the volume of resin used in bonding the inlay is reduced (Shortall & others, 1989; Crispin & others, 1994).

Indirect inlay restorations require temporary sealing of freshly cut dentin surfaces. Poor sealing may result in bacterial contamination of the cavity, possibly leading to pulp damage. Removal of the temporary seal, re-exposure of the prepared dentin surfaces and positive pressure during inlay insertion frequently induces discomfort in patients. Applying an adhesive resin liner to coat the freshly cut dentinal walls provides pulpal protection by minimizing irritation from mechanical, thermal and bacterial insults resulting from impression taking, temporary restoration placement and final cementation of the inlay (Inokoshi & others, 1995; 1998). Belli and others (2001) reported that a flowable resin composite lining produced more gap-free dentinal surfaces compared to direct resin composite restorations without lining. Flowable resin composite has also been suggested as a liner to fill irregular internal surfaces, proximal boxes and to block out undermined areas before taking an impression of the prepared cavity (Otsuki & others, 1993).

In this study, the internal adaptation of ceramic inlay restorations produced by the Cerec 2 system was compared to direct resin composite restorations by observing the interfacial gap between resin and the cavity enamel and dentin walls. This study tested the null hypotheses that the interfacial gaps of Cerec restorations were similar to direct resin composite restorations and that flowable resin composite lining does not influence the interfacial adaptation of direct composite and inlay restorations.

METHODS AND MATERIALS

Tooth Preparation

Forty recently extracted, non-carious human mandibular molars stored in 0.2% sodium azide water solution at 4°C were used. All the teeth were cleansed of dental plaque and calculus and stored in tap water following cleaning and throughout the experiment. Class II DO preparations were made with a high-speed handpiece using water spray and tapered diamond burs (regular grit bur: OC-H, fine grit bur: OC-I-1, Shofu, Kyoto, Japan). The occlusal part of the cavity was approximately 2.5-mm deep and 2.5-mm wide. The proximal box was 3.5-mm wide bucco-lingually, the mesio-distal width of the gingival wall was 1.5 mm and the gingival floor extended 1.5 mm above the cementoenamel junction (CEJ). The occluso-gingival dimension of the proximal box was approximately 3.0 to 3.5 mm, depending on the length of the crown. The taper of the cavity was approximately 1/10. All internal line angles were rounded (Figure 1).

The teeth were randomly assigned to one of four experimental groups of 10 teeth corresponding to the different restorative options: Group A (direct composite), Group B (ceramic inlay), Group C (direct composite with flowable composite liner) and Group D (ceramic inlay with flowable composite liner) (Figure 2).

Group A (Direct Composite)

The materials used in the study are listed in Table 1. Ten teeth were directly restored with a resin composite and a two-step bonding resin. After etching the cavity wall with 37% phosphoric acid (K-etchant, Kuraray Co, Osaka, Japan) for 40 seconds, a bonding resin (Clearfil Photo Bond, Kuraray Co) was applied to the cavity walls and light cured for 10 seconds. A metal matrix was then placed on the tooth, and the cavities were restored with a hybrid resin composite (Clearfil AP-X, Kuraray Co) by using the incremental insertion technique (two increments for each preparation). Every increment was light cured from the occlusal surface for 40 seconds.

Group B (Ceramic Inlay)

Ten teeth were restored with ceramic inlays fabricated by a CAD-CAM system (Cerec 2, SIRONA Dental System GmbH, Bensheim, Germany) (software version 4.2.2). The cavities were covered with a powder (Cerec powder, SIRONA Dental System GmbH) to acquire an optical impression. Inlays were designed and fabricat-

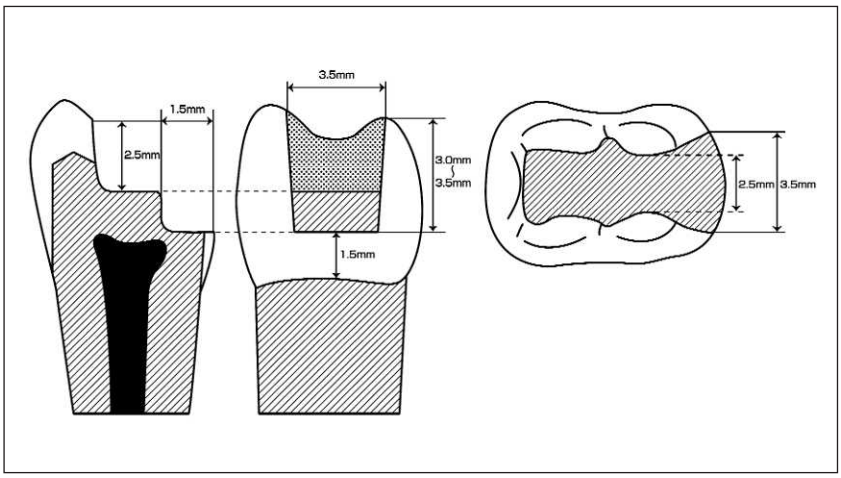


Figure 1. *Preparation design: Mesio-distal section view, proximal view and occlusal view.*

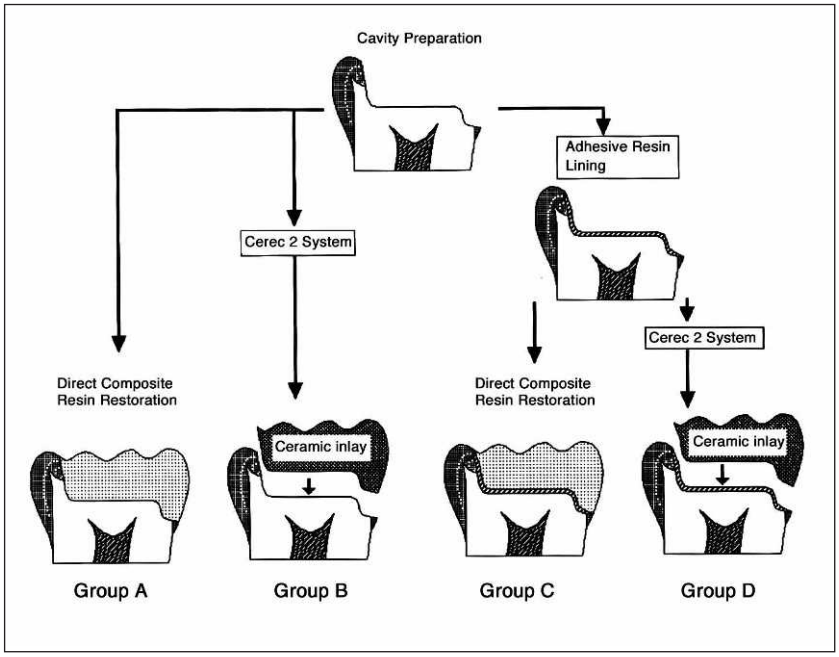


Figure 2. *Experimental procedures of the four groups.*

ed by the Cerec 2 system using porcelain blocks (VITA Blocks Mark II, VITA Zahnfabrik H Rauter GmbH & Co. KG, D-79713 Bad Sackingen, Germany) according to the manufacturer's directions. Minimal adjustment of the internal surfaces of the inlays was performed.

The inlays were conditioned with 37% phosphoric acid for five seconds, washed with water and air dried, because the manufacturer of the porcelain bonding agent recommended conditioning the porcelain surface with 37% phosphoric acid in order to accelerate the silane coupling reaction. The inner surface of the inlays was coated with a mixture of bonding resin (Clearfil Photo Bond) and silane coupling agent (Clearfil porcelain bond activator, Kuraray Co) and they were light cured for 10 seconds. The cavities were etched with 37% phosphoric acid for 40 seconds, then rinsed with water and dried. A mixture of the bonding resin (Clearfil Photo Bond) was applied, then light cured for 10 seconds. The inlays were cemented into the cavity with a dual cure resin cement (Clapearl DC, Kuraray Co). The cement was light cured from the occlusal surface for 40 seconds.

Group C (Direct Composite with Flowable Composite Liner)

A two-step self-etching primer bonding system and a flowable resin composite liner were used in this group. The cavity walls were primed with a mixture of LB primers A and B (Clearfil Liner Bond II, Kuraray Co) for 30 seconds and air dried for five seconds. A layer of bonding resin (Clearfil Liner Bond 2) was applied, thinned with a gentle stream of air and light cured for 20 seconds. After light curing the bonding resin, the cavity

Table 1: *The Components in Each Bonding System*

Material	Brand Name	Content	Batch #	Manufacturer
Etching gel	K-Etchant	37% Phosphoric acid	149	Kuraray Co
Adhesive resin	Clearfil Photo Bond	MDP, BIS-GMA, HEMA, EtOH	226, 382	Kuraray Co
Resin composite	Clearfil AP-X	BIS-GMA,TEGDMA,Barium glass	0318	Kuraray Co
Adhesive resin	Clearfil Liner Bond II	LB primer A (Phenyl-P, 5-NMSA,HEMA) LB primer B (HEMA,adhesive monomer) LB bond (MDP, HEMA, microfiller (10Wt%)	00018A 00008A 00031A	Kuraray Co
Low-viscosity liner composite	Protect Liner F	Microfilled resin (40w/w%)	0042	Kuraray Co
Dual-cured cement composite	Clapearl DC	A paste (Quartz (70%), BIS-GMA,HEMA) B paste (Quartz (70%), BIS-GMA,HEMA)	0005 0005	Kuraray Co
Silane coupling	Clearfil Porcelain Bond	5% r-MPTS Activator	0071	Kuraray Co
Porcelain Block	Cerec Vitablocks Mark II	SiO_2, AlO_3, NaO, KO, CaO_2	5353	Vita Zahnfabrik

surfaces were covered with a thin layer of a flowable resin composite liner (Protect Liner F, Kuraray Co) and light cured for 20 seconds. The cavities were restored with a hybrid resin composite (Clearfil AP-X) by using the incremental insertion technique (two increments for each preparation). Every increment was light-cured from the occlusal surface for 40 seconds.

Group D (Ceramic Inlay with Flowable Resin Composite Liner)

After cavity preparation, the cavity surfaces were covered with adhesive resin and flowable composite as described in Group C. Ceramic inlays were fabricated and placed in 10 teeth as described in Group B.

All the specimens were stored in tap water for one day to allow for sufficient cure of the dual cure resin cement of Groups C and D. The restorations were finished with super-fine diamond stones (V16ff, C16ff, GC Corp, Tokyo, Japan) and polished with silicone points (Type M2 & M3, #28, Shofu Inc, Kyoto, Japan) under water spray cooling. Immediately after polishing, all samples were thermocycled between 4°C and 60°C water 500 times.

The samples were then cross-sectioned mesio-distally along the center of the cavities using a diamond saw microtome (Leitz 1600 saw microtome, Leica Instrument GmbH, Nussloch, Germany) under copious water irrigation. The cross-sectioned surfaces were polished with waterproof silicon carbide papers (grits: #600, #1000, #1200), and diamond pastes (grits: 5 μm, 3 μm,1 μm).

Evaluation of the Adhesive Interface

Gaps or cracks at the resin-tooth interfaces were directly observed under a scanning laser microscope (LM1, Laser Tek, Tokyo, Japan). Images of the full interfaces were registered and printed. Any gaps or enamel cracks at three kinds of interfaces (occlusal enamel-resin, cervical enamel-resin, dentin-resin) were observed using high magnification and printed. The total length of the gaps or cracks was measured on the acquired images. The percentage of gaps or enamel cracks was calculated by dividing the total length of the gap or crack by the total length of each of the three interfaces. The percentage of gaps or cracks at each interface in the four groups was statistically analyzed with non-parametric Mann-Whitney U-test because distribution of data did not follow the normal distribution.

RESULTS

The percentage of interfacial gap in the enamel and dentin walls is shown in Tables 2-6.

The interfacial gaps in the enamel walls were mainly cohesive microfractures running parallel to the interface. Between 82.3% and 90.9% of the interfacial gaps were in enamel walls, and the rest ran along the enamel-resin interface. Interfacial gaps in the dentin walls involved separation from the dentin walls at the top of the dentin surface.

The direct resin composite restorations in Group A showed a high frequency of interfacial gaps at the occlusal and cervical enamel-resin interfaces. The Cerec inlays in Group B, however, showed no interfacial gaps in the enamel wall. Statistically significant differences were found between Groups A and B (p=0.0002).

There was no significant difference in gaps at the dentin-resin interface between Groups A and B. Separation of resin from the dentinal walls was observed in both groups in approximately 40% of the dentin-resin interfaces.

Although a flowable resin composite liner was used in Groups C and D, interfacial adaptation was similar to Groups A and B, respectively. Cohesive enamel microfractures at the occlusal and cervical enamel-resin interfaces were frequently observed in the direct restorations in Group C (Figure 3) but were significantly fewer in number in the Cerec inlay restorations in Group D (p=0.0002). No statistically significant difference was found in gaps at the dentin-resin interface between Groups C and D.

In Groups C and D, the thickness of flowable resin composite liner observed on the sectioned surfaces varied from 10 to 500 mm. The resin was thicker at the concaved line angle and at the bottom of the proximal box and thinnest at the occlusal walls.

Comparing gap formation in the occlusal and cervical enamel walls, the occlusal enamel walls exhibited significantly greater gap formation than the cervical walls in Groups A and C (p=0.0233 and 0.0009, respectively), whereas, no difference was found in Groups B and D.

Comparing the two different dentin bonding systems, Groups C and D showed significantly lower incidences of gap formation at the dentin-resin interface than Groups A and B (p=0.0065).

DISCUSSION

Sealing of cavity preparations to protect the exposed dental tissues against infiltration of harmful agents is very important for the longevity of the restored teeth. Although this can be attained by perfect adaptation of the restoration to the cavity walls, long-lasting, perfect sealing is difficult to achieve due to mismatches at the tooth/restoration interface. Adhesive resin is a powerful tool to correct imperfect marginal adaptations. However, dental resin materials have the drawback of shrinkage during polymerization (Feilzer & others, 1988). When shrinkage stress exceeds bond strength of the adhesive resin, separation at the adhesive interface occurs. When bond strength exceeds shrinkage stress

and cohesive strength of the tooth, fracture of the tooth structure will occur (Davidson, 1997).

Enamel microfractures that appear as cracks near the margin of the restoration and parallel to the interface reportedly occur immediately after polymerization of resin composite restorations that are strongly bonded to etched enamel (Jorgensen & others, 1975; Han & others, 1992; Prati & others, 1992; Belli & others, 2001; Dietschi & others, 2002; Peutzfeldt & Asmussen, 2002; Malmström & others, 2002). In this study, microfractures of occlusal and cervical enamel walls were a common occurrence with direct resin composite restorations. As the cross-sections of the specimens were directly observed using a scanning laser microscope to prevent crack formation that may have occurred if a scanning electron microscope was used (Belli & others, 2001), the enamel microfractures were not artifacts but were produced by polymerization shrinkage of the resin composite. The sectioning technique of the specimens may induce artificial enamel cracks. However, this possibility was excluded in this study because enamel microfractures were not observed with the Cerec inlay restorations.

Malmström and others (2002) reported greater leakage at the cervical enamel margins than at the occlusal enamel margins of Class II direct composite restorations. They attributed this finding to differences in the amount of enamel remaining at the cervical margin, which may increase the risk of fracture. Belli and others (2001) reported similar magnitudes of enamel cracks in occlusal and cervical enamel walls of Class II direct composite restorations. In this study, however, occlusal enamel showed greater gaps than cervical enamel. If the deeper layer of direct resin composite (Groups A and C) does not polymerize completely, it will exhibit lower shrinkage (Hassan & others, 1987) and exert lower stress on the bonded surface, leading to a reduction in possible enamel microfracture. In this study, since the vertical dimensions of the proximal box preparation and the filling technique were similar to those of the previous study (Belli & others, 2001), incomplete polymerization may not have been the reason for the lower number of microfractures in cervical enamel. Another possibility is the debonding of resin from the dentin walls. As separation from the dentin walls was much greater in this study than the previous one (Belli & others, 2001), some polymerization shrinkage might be compensated for by debonding the restoration from the dentin surface, thus, leading to lower stress on the cervical enamel walls.

Table 2: *Percentages of Gaps or Cracks at the Adhesive Interfaces: Groups A and B*

Without Liner			
	Direct Restoration Group A	**VS**	**Inlay Group B**
Enamel (Occlusal)	**65.8**(38.6-88.8)	> *p*=0.0002	**0**
Enamel (Cervical)	**43.9**(25.7-59.2)	> *p*=0.0002	**0**
Dentin	**37.1**(30.0-44.6)	= NS	**39.2**(33.8-44.2)

Median *(min-max)*
N*=10 (number of cavities tested)*
NS*=No significant difference*

Table 3: *Percentages of Gaps or Cracks at the Adhesive Interfaces: Groups C and D*

With Liner			
	Direct Restoration Group C	**VS**	**Inlay Group D**
Enamel (Occlusal)	**65.6**(56.0-95.8)	> *p*=0.0002	**0**(0-20.3)*
Enamel (Cervical)	**43.8**(37.4-63.9)	> *p*=0.0002	**0**(0-13.5)*
Dentin	**27.1**(19.2-36.2)	= NS	**24.7**(22.5-28.1)

Median *(min-max)*
N*=10 (number of cavities tested)*
NS*=No significant difference*
**7 out of 10 cases have no gap or crack*

Table 4: *Percentages of Gaps or Cracks at the Adhesive Interfaces: Groups A and C*

Direct Restoration			
	Without Liner Group A	**VS**	**With Liner Group C**
Enamel (Occlusal)	**65.8**(38.6-88.8)	= NS	**65.6**(56.0-95.8)
Enamel (Cervical)	**43.9**(25.7-59.2)	= NS	**43.8**(37.4-63.9)
Dentin	**37.1**(30.0-44.6)	> *p*=0.0065	**27.1**(19.2-36.2)

Median *(min-max)*
N*=10 (number of cavities tested)*
NS*=No significant difference*

Adhesively-luted Class II Cerec inlay restorations showed no microfractures of the occlusal and cervical enamel walls in this study. Manhart and others (2001) reported satisfactory sealing of the cervical enamel margins in Class II Cerec inlay restorations. Using resin composites as luting cements for porcelain or resin inlay restorations has claimed to reduce the adverse effects of bulk polymerization contraction of posterior resin composite restorations because the volume of resin used in bonding the inlay is reduced (Shortall & others, 1989; Crispin & others, 1994). Regarding the Cerec inlay restorations placed in this study, the layer of adhesive resin cement was thinner compared to a full volume of setting resin composite in the direct composite restorations (Inokoshi & others, 1992; Iida, Inokoshi & Kurosaki, 1999) and the influence of the polymerization shrinkage of the cement did not affect marginal integrity.

Table 5: *Percentages of Gaps or Cracks at the Adhesive Interfaces: Groups B and D*

Inlay	Without Liner Group B	VS	With Liner Group D
Enamel (Occlusal)	**0**	= NS	**0**(0-20.3)*
Enamel (Cervical)	**0**	= NS	**0**(0-13.5)*
Dentin	**39.2**(33.8-44.2)	> *p*=0.0065	**24.7**(22.5-28.1)

Median *(min-max)*
N*=10 (number of cavities tested)*
NS*=No significant difference*
**7 out of 10 cases have no gap or crack*

Table 6: *Percentages of Gaps or Cracks at the Adhesive Interfaces: Occlusal and Cervical Enamel*

	Enamel (Occlusal)	VS	Enamel (Cervical)
Group A	**65.8**(38.6-88.8)	> *p*=0.0233	**43.9**(25-7-59.2)
Group B	**0**	= NS	**0**
Group C	**65.6**(56.0-95.8)	> *p*=0.0009	**43.8**(37.4-63.9)
Group D	**0**(0-20.3)*	= NS	**0**(0-13.5)*

Median *(min-max)*
N*=10 (number of cavities tested)*
NS*=No significant difference*
**7 out of 10 cases have no gap or crack*

Although gaps at the enamel interface have decreased significantly with inlay restorations, no difference was found in the separation of resin from dentin walls between direct and inlay restorations. The percentages were 37% to 39% without a liner (Groups A and B) and 27 to 28% with a liner (Groups C and D), respectively. Clearfil Photo Bond, used as a bonding resin for Groups A and B, is a primer-free dual cure bonding system marketed in 1986. The reason for using this older type of adhesive resin is that the resin composite cement used in Group B, Clapearl DC, requires dual-cure type adhesive resin. When planning this experiment, the authors thought that an inlay restoration produced better adaptation to dentin walls compared to a direct restoration due to less volume of polymerizing resin, but they could not find any difference between them. This result was unexpected.

Liner Bond 2 used in Groups C and D is a self-etching primer two-step system marketed in 1992. Since this system has better adhesion properties than Clearfil Photo Bond (Harada & others, 1998), it explains better adaptation of the dentin walls in Groups C and D compared to Groups A and B. In this study, as the adhesion of resin to dentin wall was not perfect, even with Liner Bond 2, further improvement in the dentin bonding system is necessary.

Lining with an elastic, flowable resin has been reported to reduce the influence of polymerization shrinkage of resin composite and adhesive resin cements (Van Meerbeek & others, 1992; Labella & others, 1999). The liner layer acted as a buffer layer for polymerization shrinkage and compensated for the space created by polymerization shrinkage (Alhadainy & Abdalla, 1996; Kemp-Scholte & Davidson, 1990). However, no reduction in enamel cracks was found in direct resin restorations when a microfilled flowable composite (Protect Liner F) with a low filler weight and low elastic modulus (approximately 40 w/w% and 3.9 GPa, according to the manufacturer) was used in this study. This finding supports previous studies (Belli & others, 2001), where the rate of separation and cracks was 42.8% in the occlusal enamel area and 61.5% in the cervical enamel area in Class II direct composite restorations in which lining by Liner Bond 2 and Protect Liner F was used. Chuang and others (2001) reported that a flowable composite lining in Class II restorations could not improve the sealing of gingival enamel margins. If the lining had acted as a buffer layer for polymerization shrinkage of resin as reported by Alhadainy and Abdalla (1996), the rate of crack and separation would have decreased in both the enamel and dentin walls.

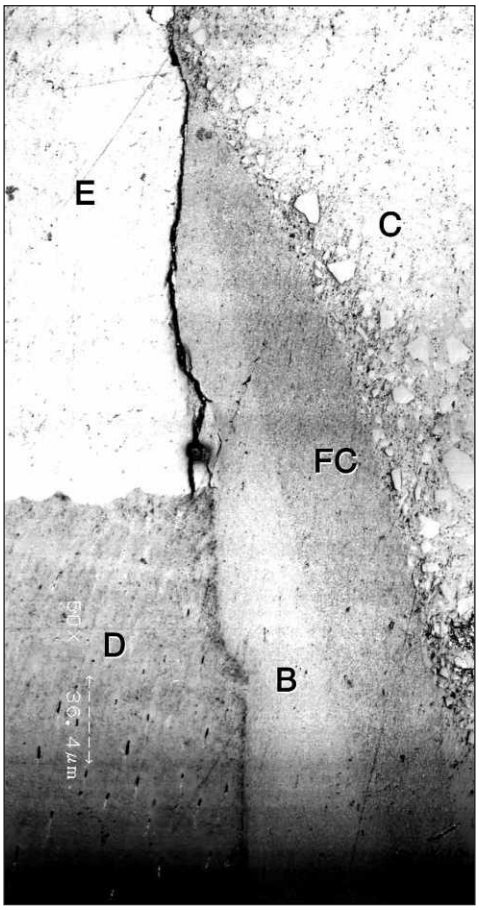


Figure 3. *Scanning laser microscope image of a polished cross section of a tooth bonded with Clearfil Liner Bond 2, Protect Liner F and AP-X resin composite. Note the presence of a gap in the enamel-resin interface but not in the dentin-resin interface. Preparation design: Mesio-distal section view, proximal view and occlusal view.*

Therefore, the thin layer of flowable composite was insufficient to act as an elastic buffer to compensate for the polymerization shrinkage of resin composite in preventing enamel microcracks.

The thickness of the flowable resin composite liner varied from 10 to 500 μm in this study. It was impossible to place a uniform thickness of the flowable liner even in this laboratory study. Malmström and others (2002) reported a pronounced reduction in leakage in Class II restorations with gingival enamel margins when the thickness of the flowable composite liner was 2.0 mm compared to 0.5 mm. Therefore, thickness of the flowable composite liner may have been insufficient to serve as an elastic buffer in this study.

The gingival margin in this study was prepared within enamel above the CEJ, and the Cerec inlay restorations showed no enamel microfractures at cervical margins. Gingival margins located below the CEJ are potentially a greater source of marginal opening not only in Class II direct composite restorations, but also in indirect inlay restorations (Manhart & others, 2001). As the adhesion of resin to the dentin wall was not perfect with Liner Bond 2, the authors cannot expect perfect sealing at the gingival margin below the CEJ.

When comparing resin composite restorations with ceramic inlay restorations, although similar gap formation was observed in the dentin walls in both types of restoration, no separation or cracks were observed in the enamel walls with Cerec inlay restorations, unlike direct resin composite restorations. Although inlay restorations require the cavity walls to be prepared diverging occlusally, which results in additional tooth loss, this has the advantage of minimizing enamel microfractures. Within the limitations of this study, the authors suggest that an adhesive inlay restoration is preferable for a large Class II cavity to prevent marginal enamel microfracture.

CONCLUSIONS

Adhesively-luted Cerec inlay restorations showed no interfacial gaps at the enamel walls, whereas, direct composite restorations showed a significantly higher incidence in gap formation at the interface or cracks in interfacial enamel. A flowable resin composite liner reduced interfacial gaps at dentinal walls but could not prevent enamel microfracture with direct resin composite restorations.

Acknowledgements

The authors thank to Dr Richard M Foxton, Tokyo Medical and Dental University, for his help in preparing this manuscript.

(Received 2 August 2002)

References

Aguiar FH, Ajudarte KF & Lovadino JR (2002) Effect of light curing modes and filling techniques on microleakage of posterior resin composite restorations *Operative Dentistry* **27(6)** 557-562.

Alhadainy HA & Abdalla AI (1996) 2-year clinical evaluation of dentin bonding systems *American Journal of Dentistry* **9(2)** 77-79.

Banks RG (1990) Conservative posterior ceramic restorations: A literature review *Journal of Prosthetic Dentistry* **63(6)** 619-626.

Belli S, Inokoshi S, Ozer F, Pereira PN, Ogata M & Tagami J (2001) The effect of additional enamel etching and a flowable composite to the interfacial integrity of Class II adhesive composite restorations *Operative Dentistry* **26(1)** 70-75.

Beznos C (2001) Microleakage at the cervical margin of composite Class II cavities with different restorative techniques *Operative Dentistry* **26(1)** 60-69.

Chuang S-F, Liu J-K & Jin Y-T (2001) Microleakage and internal voids in Class II composite restorations with flowable composite linings *Operative Dentistry* **26(2)** 193-200.

Crim GA (1991) Microleakage of three resin placement techniques *American Journal of Dentistry* **4(2)** 69-72.

Crispin BJ, Hewlett ER, Jo YH, Hobo S & Hornbrook DS (1994) *Contemporary Esthetic Dentistry: Practice Fundamentals* Chicago Quintessence Publishing Co, Ltd p 205-240.

Davidson CL (1997) Factor influencing the quality of composite restorations; theory and practice Bologna International Symposium Nov 22-23, Ariesdue Srl-Carimate-Italy 87-93 1997.

Dietschi D & Spreafico R (1997) *Adhesive Metal Free Restorations: Current Concepts for the Esthetic Treatment of Posterior Teeth* Chicago Quintessence Publishing Co, Ltd p 60-77.

Dietschi D, Bindi G, Krejci I & Davidson C (2002) Marginal and internal adaptation of stratified compomer-composite Class II restorations *Operative Dentistry* **27(5)** 500-509.

Feilzer AJ, de Gee AJ & Davidson CL (1988) Curing contraction of composites and glass ionomer cements *Journal of Prosthetic Dentistry* **59(3)** 297-300.

Han L, Okamoto A & Iwaku M (1992) The effect of various clinical factors on marginal enamel microcracks produced around composite restoration *Dental Materials Journal* **11(1)** 26-37.

Harada N, Yamada T, Inokoshi S & Tagami J (1998) Tensile bond strengths and adhesive interfaces of ten dentin bonding systems *Journal of Medical and Dental Science* **45(2)** 85-96.

Hassan K, Mante F, List G & Dhuru V (1987) A modified incremental filling technique for Class II composite restorations *Journal of Prosthetic Dentistry* **58(2)** 153-156.

Iida K, Inokoshi S & Kurosaki N (1999) Marginal and inside adaptation of MOD ceramic inlay made by Cerec 2 system *Japanese Journal of Conservative Dentistry* **42(6)** 1060-1065.

Inokoshi S, Fujitani M, Otsuki M, Sonoda H, Kitasako Y, Shimada Y & Tagami J (1998) Monkey pulpal responses to conventional and adhesive luting cements *Operative Dentistry* **23(1)** 21-29.

Inokoshi S, Shimada Y, Fujitani M, Otsuki M, Shono T, Onoe N, Morigami M & Takatsu T (1995) Monkey pulpal response to adhesively luted indirect resin composite inlays *Operative Dentistry* **20(3)** 111-118.

Inokoshi S, Van Meerbeek B, Willems G, Lambrechts P, Braem M & Vanherle G (1992) Marginal accuracy of CAD/CAM inlays made with the original and the updated software *Journal of Dentistry* **20(3)** 171-177.

Jorgensen KD, Asmussen E & Shimokobe H (1975) Enamel damages caused by contracting restorative resins *Scandinavian Journal of Dental Research* **83(2)** 120-122.

Kemp-Scholte CM & Davidson CL (1990) Marginal integrity related to bond strength and strain capacity of composite resin restorative systems *Journal of Prosthetic Dentistry* **64(6)** 658-664.

Labella R, Lambrechts P, Van Meerbeek B & Vanherle G (1999) Polymerization shrinkage and elasticity of flowable composites and filled adhesives *Dental Materials* **15(2)** 128-137.

Malmström HS, Schlueter M, Roach T & Moss ME (2002) Effect of thickness of flowable resins on marginal leakage in Class II composite restorations *Operative Dentistry* **27(4)** 373-380.

Manhart J, Schmidt M, Chen HY, Kunzelmann KH & Hickel R (2001) Marginal quality of tooth-colored restorations in Class II cavities after artificial aging *Operative Dentistry* **26(4)** 357-366.

Otsuki M, Yamada T, Inokoshi S, Takatsu T & Hosoda H (1993) Establishment of a composite resin inlay technique. Part 7 Use of low viscous resin *Japanese Journal of Conservative Dentistry* **36(5)** 1324-1330 (in Japanese).

Peutzfeldt A & Asmussen E (2002) Composite restorations: Influence of flowable and self-curing resin composite linings on microleakage *in vitro Operative Dentistry* **27(6)** 569-575.

Prati C, Simpson M, Mitchem J, Tao L & Pashley DH (1992) Relationship between bond strength and microleakage measured in the same Class I restorations *Dental Materials* **8(1)** 37-41.

Shortall AC, Baylis RL, Baylis MA & Grundy JR (1989) Marginal seal comparisons between resin-bonded Class II porcelain inlays, posterior composite restorations, and direct composite resin inlays *International Journal of Prosthodontics* **2(3)** 217-223.

Van Meerbeek B, Lambrechts P, Inokoshi S, Braem M & Vanherle G (1992) Factors affecting adhesion to mineralized tissues *Operative Dentistry* **17(Supplement 5)** 111-124.

©Operative Dentistry, 2003, **28-4,** 453-460

Three Different Methods to Evaluate Microleakage of Packable Composites in Class II Restorations

JB Almeida • JA Platt • Y Oshida
BK Moore • MA Cochran • GJ Eckert

Clinical Relevance

The amount of microleakage seen *in vitro* with packable resin composites depends on the type of dye used to visualize it.

SUMMARY

This *in vitro* study compared three different methods to evaluate detectable levels of microleakage in Class II restorations placed with five commercially available packable resin composites: Alert, Glacier, Pyramid, Solitaire 2 and SureFil. A flowable resin composite, Flow-It, was used to line all packable composites. The hybrid resin composite Z100 was also included. The adhesive system used with all groups was Scotchbond MultiPurpose Plus. Standard Class II cavities were prepared on the mesial (enamel margins) and distal (dentin margins) sides of the teeth with no communication between them.

Based on a power analysis, 180 permanent human molars were randomly assign to each of six groups with 30 specimens per group. All restorative materials were placed following manufacturers' recommendations. Following restoration and thermocycling, the specimens were stored at room temperature in solutions of ^{45}Ca, methylene blue and rhodamine B, sequentially. Microleakage was ordinal scored as 1 (no penetration), 2 (penetration up to one-third of the cervical floor), 3 (penetration beyond one-third of the cervical floor to the axial wall) and 4 (penetration along the axial wall) by two independent evaluators. Analysis of the occlusal surfaces was also accomplished following the same scheme.

In this study, tracers/dyes were evaluated for differences in penetration using generalized estimating equation methodology applied to cumulative logistic regression models. The results showed that Rhodamine B detected more microleakage than ^{45}Ca or methylene blue, and ^{45}Ca generally detected more microleakage than methylene blue.

Janaina Bertoncelo de Almeida, DDS, MSD, PhD student, Department of Restorative Dentistry, Faculdade de Odontologia de Piracicaba–UNICAMP, Piracicaba, Brazil

*Jeffrey A Platt, DDS, MS, assistant professor, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN

Yoshiki Oshida, PhD, professor, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN

B Keith Moore, PhD, professor, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN

Michael A Cochran, DDS, MSD, professor, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN

George J Eckert, MAS, staff biostatistician, Indiana University School of Medicine, Indianapolis, IN

*Reprint request: 1121 West Michigan Street, Indianapolis, IN 46202; e-mail: jplatt2@iupui.edu

INTRODUCTION

The ability of a material to prevent microleakage has been regarded as a primary concern of modern dentistry. Many commercially available products have attempted to minimize the interfacial gap between the tooth and restoration, the main pathway of microleakage (Sano & others, 1995).

Traditional posterior composites have never been ideal amalgam alternatives because they: 1) require more handling time; 2) exhibit significant polymerization shrinkage; 3) present more microleakage with age; 4) provide questionable durability and 5) cannot be condensed or packed into the preparations, which makes establishing adequate proximal contact difficult. However, the evolution of composites has recently introduced packable composites as a possible alternative to amalgam restorations. These materials have been referred to as high density, condensable or packable composites (Pearson & Bouschlicher, 2001; Unterbrink & Liebenberg, 1999).

Manufacturers have claimed packable composites to be condensable like amalgam and fulfill the requirements for use in posterior primary and permanent teeth in stress-bearing and non-stress bearing areas. Some are claimed to make rapid bulk placement possible without concerns of poor polymerization. Higher strengths and lower shrinkage would make composites more competitive alternatives to amalgam and result in enhanced clinical performance.

A common property used by dental researchers for primary evaluation of new adhesive materials is microleakage. However, the wide variety of dye materials and different techniques used in *in vitro* tests lead to confusion because they lack common parameters for comparison. Examples are dye concentrations, immersion periods, temperature, chemical nature of tracers and even methods to evaluate or score microleakage tests (Kersten & Moorer, 1989; Pashley, 1990). Several authors (Christen & Mitchell, 1966; Delivanis & Chapman, 1982; Wu & others, 1983; Tangsgoolwatana & others, 1997) have compared different dye techniques and the results vary between studies. Most of these studies used different teeth for each of the experimental groups. Utilizing the same teeth and restorations for comparing techniques decreases the number of variables involved in the comparison and could improve results.

This *in vitro* study evaluated the extent of microleakage in Class II restorations using five commercially available packable resin composites lined with a flowable resin composite when they are submitted to different dye systems and a radioactive tracer.

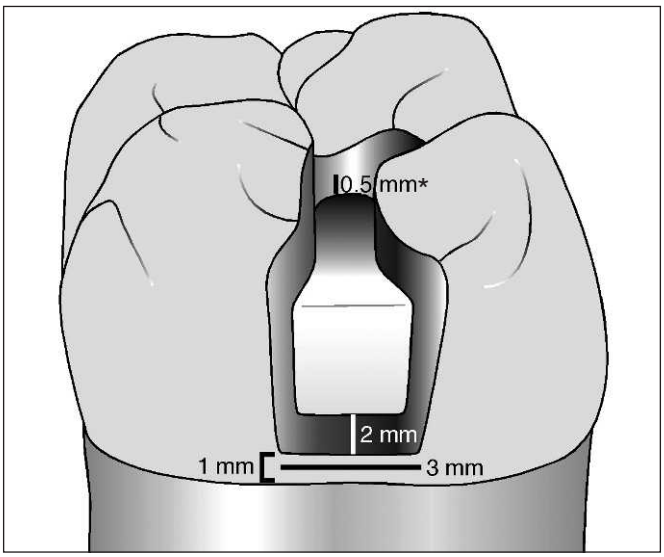


Figure 1. *Mesial view of the cavity design. *0.5 mm below dentin-enamel junction.*

Table 1: *Resin Composition by Manufacturers and Lot Numbers*

Trade Mark and Company	Lot #	Matrix	Filler Content*	Weight %	Volume % Type
Z100 3M Dental Products	20000314	Bis-GMA/TEGDMA	Zirconia Silica	84.5	66
FLOW-IT! Pentron Corp	28844	Ethoxylated BIS-GMA/TEGDMA	Barium-boron Silicate	68	53
ALERT Pentron Corp	28935	EBPADMA/PCDMA	Barium-boron Silicate	82	67
GLACIER Southern Dental Industries, Inc	991027	Urethane Dimethacrylate	Glass Powder Strontium Based	77	62
PYRAMID BISCO Inc	0000001123	Ethoxylated BIS-GMA/TEGDMA	Barium Glass Strontium Glass	80	**
SOLITAIRE 2 Heraeus Kulzer, Inc	010225	Bis-GMA/UDMA	***	75	90
SureFil DENTSPLY Caulk	990820	Urethane modified BIS-GMA dimethacrylate	Barium Glass	82	65

**Values by manufacturers.*
*** Manufacturer does not have the data.*
**** Proprietary.*

The study's hypothesis was that there is no significant difference in microleakage between groups when differentiated by the dyes or tracer used.

METHODS AND MATERIALS

Table 1 lists the five commercially available packable resin composites used in this study. A total of 180 sound, extracted permanent human molars were used. They were free of visual caries, previous restorations, hard or soft tissues and visible structural defects. Teeth with white lesions, visible fractures or cracks, abnormal shape or other coronal defects were excluded. When collected, the molars were treated with a 10% formalin solution and stored in water at 3°C. After initiation of the cavity preparations and restorations, the teeth were stored in 37°C deionized water. They were randomly divided into groups of 30. Data from Gonzalez (1992) was used to estimate that the study had 80% power at a 0.05 significance level to find a difference between a group with a microleakage distribution of 1:80%, 2:20% and a second with a microleakage distribution of 1:35%, 2:40%, 3:25%. Two Class II preparations were made in each tooth, with no connection between them. The mesial cavity had its cervical margins located 1 mm occlusal to the cemento-enamel junction, while the distal cavity had its cervical margins located 1 mm apical to the cemento-enamel junction. A modified adhesive design that included no bevels was used. Both mesial and distal cavities were prepared in a standard fashion; a minimum of 1 mm of tooth tissue remained occlusally between the two cavities. Occlusally, the depth of the preparation was at least 0.5 mm pulpal to the dentin-enamel junction. The bucco-lingual width of the cavity was 3 mm on the occlusal and the gingival side. The gingival floor was 2-mm deep to the axial wall. Internal angles were rounded (Figure 1).

All preparations were accomplished with a #245 tungsten carbide bur in a high-speed handpiece with copious water spray. The bur was replaced after every six preparations. The same operator performed all the procedures involved in this study.

The groups were restored and labeled as follows:

Group 1: Z100 (3M Dental Products, St Paul, MN)

Group 2: Alert (Pentron Corp, Wallingford, CT)

Group 3: Glacier (SDI Inc, Bensenville, IL)

Group 4: Pyramid (BISCO Inc, Schaumburg, IL)

Group 5: Solitaire 2 (Heraeus Kulzer Inc, South Bend, IN)

Group 6: SureFil (Dentsply Caulk, Milford, DE)

After preparation, 15-second acid conditioning of enamel and dentin was performed according to manufacturers' recommendations using the 35% phosphoric acid provided with the adhesive system (Scotchbond MultiPurpose Plus, 3M Dental Products, St Paul, MN, USA). After conditioning, the tooth was rinsed under running water and gently air dried for two seconds. A moist surface was left, with no visible pools. Scotchbond Multi-Purpose Prime was applied to enamel and dentin and gently air dried for five seconds. Scotchbond Multi-Purpose Adhesive was applied to enamel and dentin and immediately light cured for 10 seconds. The same dentin adhesive system was used with all groups. The output of the light-curing unit (Optilux, Demetron Research Corp, Danbury, CT, USA) was checked prior to initiation of curing procedures using a radiometer (Model 100 Curing Radiometer, Demetron Research Corp) and was 490 mW/cm^2 for all groups.

Following placement of the adhesive, a metal matrix band was secured and positioned around the specimen, simulating clinical procedures for a Class II restoration. The hybrid resin, Z100, was placed following the manufacturer's recommendations (Table 2).

For the groups where packable composite was used, the flowable resin composite Flow-It was placed after the adhesive in a thickness of 0.5 mm to 1 mm to cover all prepared surfaces of the cavities on the mesial and the distal sides. It was light cured for 40 seconds. The packable composites were placed following manufacturers' recommendations as described in Table 2. To reduce variation in the extent of polymerization caused by differences in color of the resin composites, shade A2 was used to restore all groups.

Table 2: *Manufacturer Recommendations for Placement Technique and Time of Cure*

Z100	Incremental placement. First increment, of 1.5 mm in thickness, on the floor of the proximal box. Light cure for 40 seconds. Following increments of 2.5 mm in thickness. Each increment cured separately.
Alert	Bulk placement up to 5-mm deep. Light cure from the occlusal for 40 seconds. If preparation exceeds a depth of 5 mm, fill the cavity halfway and light cure for 20 seconds. Cure final increment 40 seconds.
Glacier	Incremental placement. Each increment of a maximum of 2 mm in thickness. Light cure each increment for 20 seconds.
Pyramid	Incremental placement. Initial increment covering the floor of proximal box to a depth of 2 mm. Light cure for 10 seconds. Following increments of 2 mm in thickness and each one light cured for 10 seconds.
Solitaire 2	Incremental placement of maximum 2 mm in thickness. Light cure each increment for 40 seconds.
SureFil	Bulk placement up to 5 mm of the proximal box, level with the pulpal floor. Light cure for 40 seconds. Additional increments up to 5 mm and light cured for 40 seconds.

Following placement of the restorations, any proximal excess was removed using a #11 surgical blade (Miltex Instrument Company, Inc, Bethpage, NY, USA). On the occlusal areas, finishing was accomplished using finishing burs in a low-speed handpiece with water spray and all visible flash outside the cavity margins was removed. The specimens were stored in 37°C deionized water.

Microleakage Test

After storage for one week, all specimens were subjected to 2,500 thermocycles between 8°C (± 2°C) and 48°C (± 2°C) water baths. The dwell time in each bath was 30 seconds and the transfer time was 10 seconds (Tangsgoolwatana & others, 1997).

Following thermocycling, open apices of the specimens were blocked with resin composite. The unprepared coronal and root surfaces were painted with one coat of fingernail polish placed as close as possible to the restoration margins to prevent dye penetration anywhere other than the interfaces. Special attention was given to the apex region, where a thicker coat of fingernail polish was used to ensure maximum sealing capability. No evidence of apical leakage was noted during evaluation.

The fingernail polish was allowed to air dry. Tin foil was wrapped over this layer of polish and burnished against the surface with a hand instrument, and a second coat of fingernail polish was applied to seal the edges of tin foil and also allowed to air dry. After completing the sealing, the specimens were returned to 37°C deionized water until they were submitted to the dye tracers.

Previous work has shown that no significant interaction occurs between ^{45}Ca and subsequent dye exposure (Gonzalez, 1992). To control variations within groups, all groups were sequentially subjected to three different leakage tracers to evaluate the degree of microleakage. The sequence was (1) ^{45}Ca, a radioactive tracer, (2) methylene blue dye and (3) rhodamine B fluorescent dye.

The total time that had elapsed between completing the restoration and immersion into the tracer and dyes was nine days for all groups. One group at a time was immersed into the radioactive tracer. The specimens were immersed for two hours at room temperature in an aqueous solution (pH 5.5) of $^{45}CaCl_2$ having a radioactivity of 0.1 mCi/mL. The specimens were then kept for one hour under running water, scrubbed with a toothbrush and neutral liquid soap, rinsed and blot dried to avoid any excess water that could alter the concentration of the dye solutions. Careful handling and scrubbing was done to avoid removing the fingernail polish or tinfoil.

Solutions of 0.5-percent were prepared for methylene blue and rhodamine B dye by dissolving 5 g of dye powder in 1000 ml of deionized water. After completing the process for ^{45}Ca, and on the same day, the specimens were immersed at room temperature into the aqueous solution of methylene blue for 10 hours. The specimens were then rinsed with water and blot dried. Following completion of the process for methylene blue, the specimens were immersed into the rhodamine B aqueous solution at room temperature for 10 hours, rinsed with water and blot dried.

Once the dye immersions were completed, the tinfoil and fingernail polish were removed. A longitudinal mesiodistal section was accomplished using a high-speed diamond saw (Gillings-Hamco, Hamco Machines, Inc, Rochester, NY, USA) under copious water irrigation. A wafering diamond blade 0.38-mm thick (Leco Corporation, St Joseph, MI, USA) was used to section the specimens. The resulting two sections from the same specimen contained both mesial and distal portions of each restoration. Remaining pulp debris was removed and the specimens were allowed to air dry prior to exposure to autoradiographs and obtaining images from the metallograph and confocal microscope.

Autoradiographs were made using Ultra-speed intraoral dental film (Eastman Kodak Company, Rochester, NY, USA). Specimens were individually wrapped with tinfoil and the exposure maintained for 17 hours. The exposed films were developed in an automatic film processor and evaluated against white paper under a light microscope with 40x magnification.

Each coded tooth section was further evaluated under a metallograph at 36x magnification for methylene blue dye microleakage. Digital images were obtained with a Polaroid Digital Camera Model DMC 1 (Polaroid Corp, Cambridge, MA, USA).

For the fluorescence technique with rhodamine B, images were obtained using a Confocal Laser Scanning Microscope (Odyssey, Noran Instruments, Inc, Middleton, WI, USA) with an argon laser (a 488 nm excitation wavelength), a 10 µm confocal slit, a 515 nm long pass barrier filter and 25x magnification.

The sites for evaluation were cervical and occlusal enamel on the mesial side (the latter being referred to as mesial occlusal) and cervical dentin and occlusal enamel on the distal side. The occlusal on the distal side was called distal occlusal as a matter of differentiation. Therefore, each sectioned tooth produced four sites for evaluation on the facial and lingual sections: cervical enamel, mesial occlusal, cervical dentin and distal occlusal, totaling eight sites of evaluation for each tooth.

In this study, two experienced evaluators familiar with *in vitro* techniques scored for microleakage and the data were recorded. Each site was evaluated once for ^{45}Ca, once for methylene blue and once for rhodamine B by each evaluator.

Microleakage was determined by observing the distance of uniaxial penetration of the dye or tracer into the specimen. The criteria for cervical sites were:

1 = no penetration.

2 = penetration up to one-third of the cervical floor.

3 = penetration beyond one-third of the cervical floor to the axial wall.

4 = penetration along the axial wall.

For the occlusal sites the criteria followed the same scheme, with differences related only to the cavity wall nomenclature. Leakage that was evident along the entire extent of the tooth-restoration interface was scored as four for both sites.

Agreement between the two evaluators by comparing corresponding sites was assessed using weighted kappa statistics (Landis & Koch, 1977). Weighting treats disagreements that were "closer" (2 vs 3) as having less disagreement than those that were not as close (2 vs 4).

For data reduction, the maximum score that represented the largest degree of tracer/dye penetration from the facial and lingual sections scored by both evaluators was determined for comparisons between the tracers/dyes. Comparisons among the three tracers/dyes for differences in penetration scores were performed using the generalized estimating equation (GEE) methodology (Lipsitz, Kim & Zhao, 1994) applied to cumulative logistic regression. A cumulative logistic regression model is appropriate when the observed outcomes are ordered categories and the model compares the distributions of the scores between groups. Standard cumulative logistic regression requires all observations to be independent. Data from the enamel and dentin sections of a specimen cannot be treated as independent, and the use of all three tracers/dyes on each specimen adds additional non-independence. The GEE methodology allows cumulative logistic regression to be extended to analysis of correlated data. In addition to the term for tracers/dyes, the model also included terms for material, an indicator for whether the observation was from enamel or dentin, and all two-way and three-way interactions. Fisher's Protected Least Significant Differences was used to control the significance level for multiple comparisons. Comparisons were considered to have a statistically significant difference when the *p*-values were less than 0.05.

RESULTS

Table 3 depicts the agreement between evaluators. The categories of agreement for weighted kappa statistical analysis are given in Table 4. Comparisons between the mesial and distal and occlusal and cervical sites are depicted in Tables 5 and 6, respectively.

The tracer/dye main effect and the interactions with the tracer/dye were significant ($p<0.05$). The significant interactions did not affect comparisons against rhodamine B. Results of the comparisons between tracer/dyes showed that for every resin and location combination, rhodamine B had significantly deeper dye penetration than ^{45}Ca ($p=0.0001$) and methylene blue ($p=0.0001$). These significant interactions affected comparisons between ^{45}Ca and methylene blue. With all groups and locations combined, ^{45}Ca had significantly

Table 3

	^{45}Ca	Methylene Blue	Rhodamine B
CE	Moderate	Substantial	Fair
CD	Substantial	Almost perfect	Fair
MO	Moderate	Substantial	Slight
DO	Moderate	Substantial	Slight

Agreement between evaluators (WK)*
Cervical in enamel (CE), cervical in dentin (CD)
Mesial occlusal (MO), and distal occlusal (DO)

Table 4: *Weighted Kappa (WK*) Categories of Agreement (Landis & Koch, 1977)*

Below 0.0	→	Poor
0.00 - 0.20	→	Slight
0.21 – 0.40	→	Fair
0.41 – 0.60	→	Moderate
0.61 – 0.80	→	Substantial
0.81 – 1.00	→	Almost perfect

Table 5: *Comparisons Between Mesial and Distal Sites*

^{45}Ca		Methylene Blue		Rhodamine B	
M x D	***ND***	M x D	***ND***	M x D*	***D***
*CD x CE	***D***	*CD x CE	***D***	*CD x CE	***D***
*MO x DO	***D***	MO x DO	***ND***	MO x DO	***ND***

M (mesial); D (distal); CD (cervical in dentin); CE (cervical in enamel); MO (mesial occlusal); DO (distal occlusal).
Higher microleakage;* **ND *= not different (*p*>0.05);* **D** *= different (*p*<0.05)*

Table 6: *Comparisons Between Occlusal and Cervical Sites*

^{45}Ca		Methylene Blue		Rhodamine B	
*C x O	***D***	C x O	***ND***	*C x O	***D***
*CD x DO	***D***	CD xDO	***ND***	*CD x DO	***D***
CE X MO	***D***	CE x MO*	***D***	CE x MO	***ND***

C (cervical); O (occlusal); CD (cervical in dentin); CE (cervical in enamel); MO (mesial occlusal); DO (distal occlusal).
Higher microleakage;* **ND *= not different (*p*>0.05);* **D** *= different (*p*<0.05)*

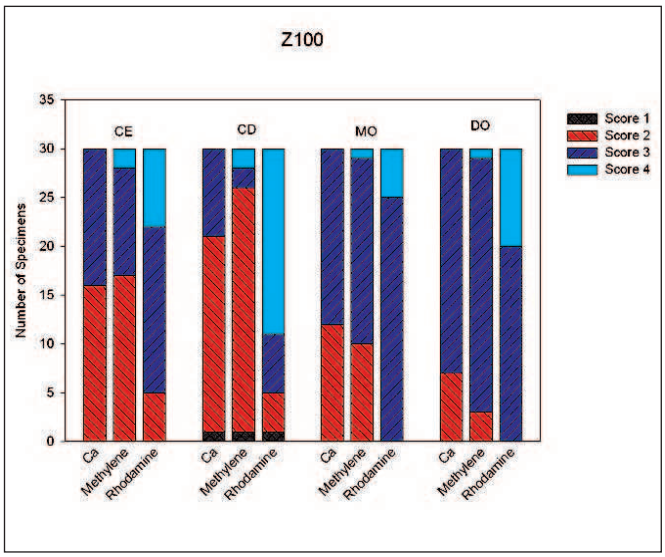


Figure 2. *Number of specimens, and scores for ^{45}Ca, methylene blue and rhodamine B for Z100 at the different sites: cervical in enamel (CE), cervical in dentin (CD), mesial occlusal (MO) and distal occlusal (DO).*

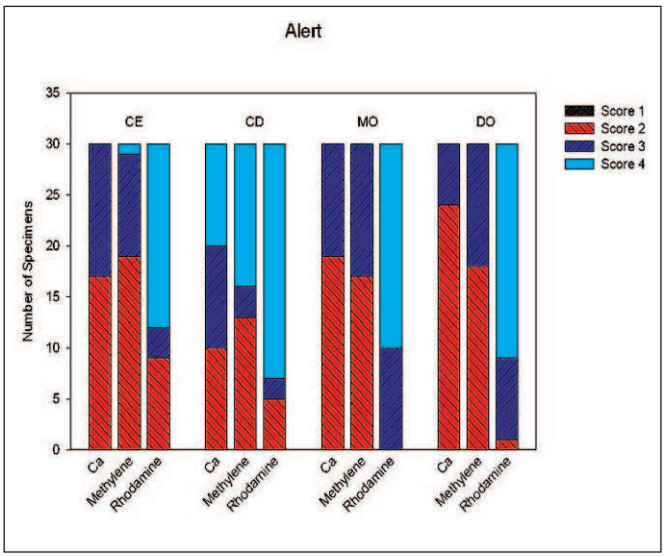


Figure 3. *Number of specimens, and scores for ^{45}Ca, blue, and rhodamine B for Alert at the different sites: cervical in enamel (CE), cervical in dentin (CD), occlusal (MO) and distal occlusal (DO).*

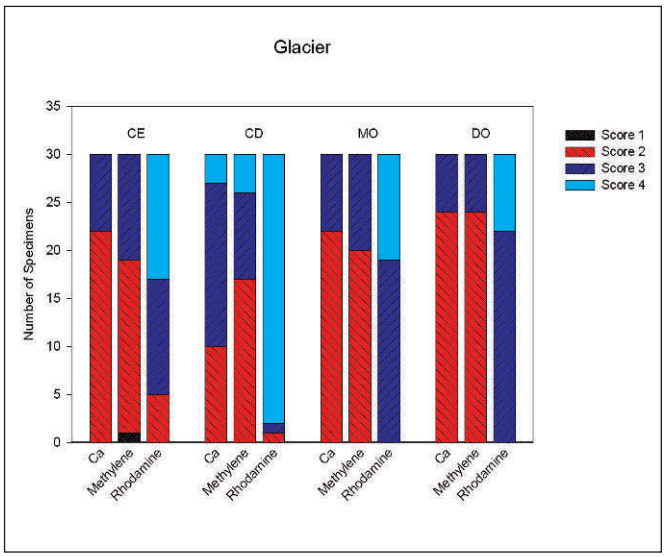


Figure 4. *Number of specimens, and scores for ^{45}Ca, methylene blue, and rhodamine B for Glacier at the different sites: cervical in enamel (CE), cervical in dentin (CD), mesial occlusal (MO) and distal occlusal (DO).*

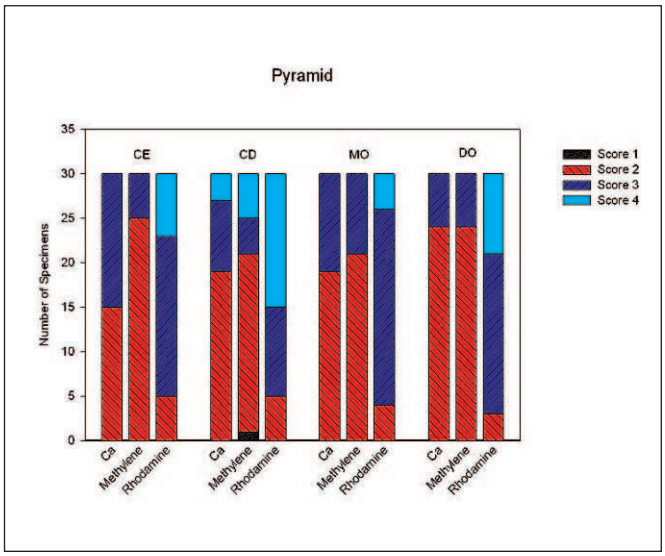


Figure 5. *Number of specimens, and scores for ^{45}Ca, blue, and rhodamine B for Pyramid at the different sites: cervical in enamel (CE), cervical in dentin (CD), mesial occlusal (MO) and distal occlusal (DO).*

deeper dye penetration than methylene blue (p=0.0470) (Figures 2 to 7). For the specific comparisons, ^{45}Ca had significantly deeper dye penetration than methylene blue for Pyramid (p=0.0006), while methylene blue had significantly deeper dye penetration than ^{45}Ca for SureFil in dentin (p=0.0076).

DISCUSSION

The microleakage process is a phenomenon of diffusion of substances, organic or inorganic, into the tooth through the interface between the restorative material and the tooth structure.

In this study, the volume concentration for methylene blue and rhodamine B dyes was 0.5 percent. The molar concentration was 13.37 mM/L for methylene blue (pH=3.6), 10.44 mM/L for rhodamine B (pH=2.5) and 0.157 mM/L for ^{45}Ca. This was selected after maximum solubility for rhodamine B in water at room temperature was determined to be approximately 0.5 percent and 6.0 percent for methylene blue.

Rhodamine B has been described as a relatively large molecule with an area of 1.75 nm^2 (Müller, Mächtle & Helm, 1994). Using bond lengths, the area of methylene blue was calculated to be approximately 0.52 nm^2. From

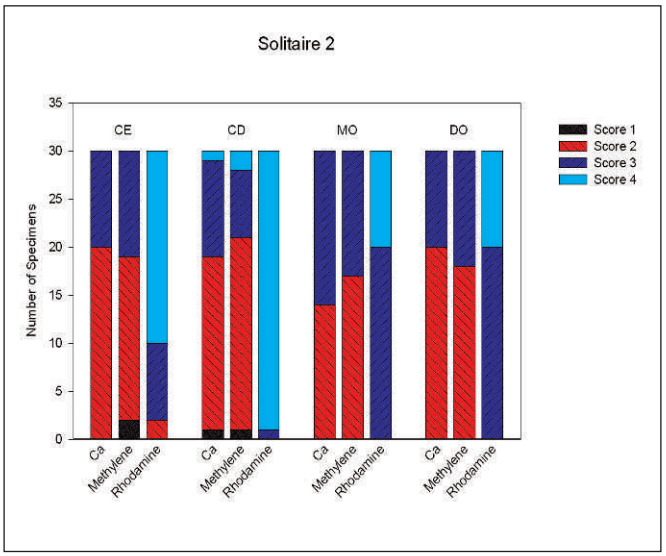


Figure 6. *Number of specimens, and scores for ^{45}Ca, methylene blue, and rhodamine B for Solitaire 2 at the different sites: cervical in enamel (CE), cervical in dentin (CD), mesial occlusal (MO) and distal occlusal (DO).*

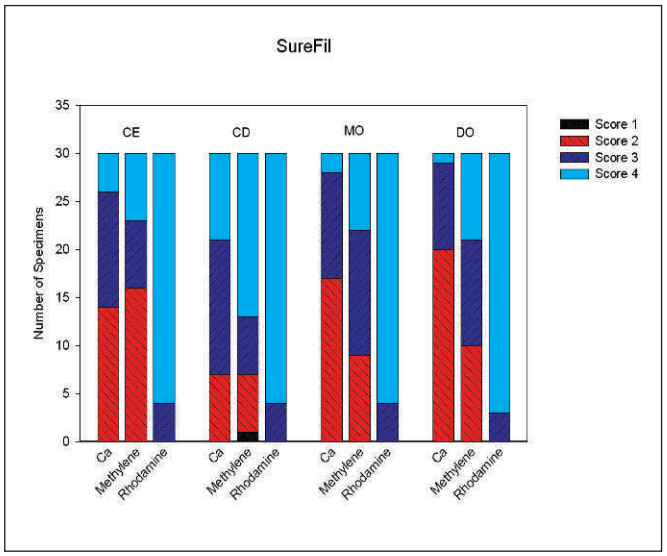


Figure 7. *Number of specimens, and scores for ^{45}Ca, methylene blue, and rhodamine B for SureFil at the different sites: cervical in enamel (CE), cervical in dentin (CD), mesial occlusal (MO) and distal occlusal (DO).*

the *CRC Handbook of Chemistry and Physics*, ^{45}Ca has an approximate area of 0.031 nm^2. If the size of the tracer molecule were of primary importance in microleakage evaluation, one would expect to see ^{45}Ca exhibit the largest amounts of microleakage. The results of this study showed that, individually, rhodamine B always presented more microleakage than ^{45}Ca and methylene blue (Figures 2-7). These results agree with the findings by Chan and Jones (1992) that suggested the size of the tracer is not the only important factor influencing microleakage. The results also support findings by Wu and others (1983), where ^{45}Ca was not considered to be the most aggressive material for microleakage tests in spite of its small ionic size. A possible explanation for this observed phenomenon is that ion exchange can occur between calcium ions in dental tissues and those in the isotope solution (Going, Myers & Prussin, 1968).

Trowbridge (1987) stated that an affinity possibly exists between Ca isotope and the structure of the tooth and restorative materials. Youngson and others (1998), studying several dyes used in *in vitro* microleakage tests, including methylene blue, concluded that all dyes undergo chemical reaction that may alter distribution of the dye depending on the biochemical constituents of dentin. The results showed that dyes also penetrated dentin to a similar degree regardless of their molecular size, pH or the sectioning technique employed.

Interaction with the tooth structure of one tracer could interfere with the performance of a subsequent tracer. The tracer's ability to demonstrate microleakage is likely dependent upon its penetration coefficient, a property that is tied to the contact angle of the tracer on the underlying structure. Internal data has indicated no discernable impact on the contact angle with tooth structure that was treated following the sequence outlined in this study. Previous work by Gonzalez (1992) evaluated the impact of sequential exposure to dyes after exposure to ^{45}Ca in restored teeth. No statistical changes in microleakage were discernable when basic fuchsin, fluorescent or reactive orange 14 dyes were used after ^{45}Ca when compared to their use alone. This provided justification for the study design reported here.

The results of this study showed that, when utilizing methylene blue, the agreement between the two evaluators was considered substantial to almost perfect. This may be partially credited to easy visualization of the prepared cavity in the digital images, providing the evaluators with a clear reference point from which to score. The dye also provides an excellent contrast with the surrounding environment.

Results of tracer material ^{45}Ca showed substantial agreement between evaluators for cervical dentin. The agreement was only moderate for cervical enamel and occlusal microleakage. For five of the six materials, cervical dentin sites tended to have more leakage than other sites. Agreement appears to increase as the amount of microleakage increases. Therefore, this method may be less reliable for distinguishing small differences.

Agreement between evaluators for rhodamine B varied from fair to slight. It cannot be ruled out that using rhodamine B was a relatively new technique for both evaluators, which could have impaired the outcome of the results. A major drawback to the technique appeared to be that although the fluorescent dye enables easy observation of the areas where it is present, it also appears to interact with enamel. In several

specimens, it was possible to observe the presence of rhodamine B in the dentin-enamel junction associated with the cervical enamel. This may have significantly affected the ability to differentiate microleakage in the restoration-tooth interface from dye penetration through the adjacent tooth structure. Further investigation is necessary to assess standardization of the parameters involved with this methodology.

All restorations were bonded with the same dental adhesive. The flowable resin composite used with packable resins was the same for all groups. All manufacturers involved in this study were consulted and stated that their packable resin composites were chemically compatible with any flowable resin available on the market. When questioned specifically about Flow-It, the answer was the same. However, the placement techniques used (for example, incremental vs bulk placement) were based on manufacturers' recommendation and were not uniform between groups. Therefore, any apparent differences between materials should be viewed as limited by the conditions of the study.

CONCLUSIONS

1. Under the conditions of this study, the null hypothesis is rejected.
   a. Rhodamine B had significantly deeper dye penetration than ^{45}Ca and methylene blue.
   b. ^{45}Ca generally had significantly deeper dye penetration than methylene blue.
2. The results of microleakage studies that used different tracer/dyes materials should not be compared.

(Received 5 August 2002)

References

Chan MF & Jones JC (1992) A comparison of four *in vitro* marginal leakage tests applied to root surface restorations *Journal of Dentistry* **20(5)** 287-293.

Christen AG & Mitchell DF (1966) A fluorescent dye method for demonstrating leakage around dental restorations *Journal of Dental Research* **45(5)** 1485-1492.

CRC Handbook of Chemistry and Physics 61st ed Weast RC & Astle MJ eds (1980) Boca Raton CRC Press p F216.

Delivanis PD & Chapman KA (1982) Comparison and reliability of techniques for measuring leakage and marginal penetration *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics* **53(4)** 410-416.

Going RE, Myers HM & Prussin SG (1968) Quantitative method for studying microleakage *in vivo* and *in vitro Journal of Dental Research* **47(6)** 1128-1132.

Gonzalez, MAG (1992) Comparison of four microleakage tests [Thesis] Indianapolis: Indiana University School of Dentistry.

Kersten HW & Moorer WR (1989) Particles and molecules in endodontic leakage *International Endodontic Journal* **22(3)** 118-124.

Landis J R & Koch GG (1977) The measurement of observer agreement for categorical data *Biometrics* **33(1)** 159-174.

Lipsitz SR, Kim K & Zhao L (1994) Analysis of repeated categorical data using generalized estimating equations *Statistics in Medicine* **13(11)** 1149-1163.

Müller C, Mächtle P & Helm CA (1994) Enhanced absorption within a cavity. A study of thin dye layers with the surface forces apparatus *Journal of Physical Chemistry* **98(43)** 11119-11125.

Pashley DH (1990) Clinical considerations of microleakage *Journal of Endodontics* **16(2)** 70-77.

Pearson J & Bouschlicher MR (2001) Polymerization contraction force of packable composites *General Dentistry* **49(6)** 643-647.

Sano H, Takatsu T, Ciucchi B, Horner JA, Matthews WG & Pashley DH (1995) Nanoleakage: Leakage within the hybrid layer *Operative Dentistry* **20(1)** 18-25.

Tangsgoolwatana J, Cochran MA, Moore BK & Li Y (1997) Microleakage evaluation of bonded amalgam restorations: Confocal microscopy versus radioisotope *Quintessence International* **28(7)** 467-477.

Trowbridge HO (1987) Model systems for determining biologic effects of microleakage *Operative Dentistry* **12(4)** 164-172.

Unterbrink GL & Liebenberg WH (1999) Flowable resin composites as "filled adhesives": Literature review and clinical recommendations *Quintessence International* **30(4)** 249-257.

Youngson CC, Glyn Jones JC, Manogue M & Smith IS (1998) *In vitro* dentinal penetration by tracers used in microleakage studies *International Endodontic Journal* **31(2)** 90-99.

Wu W, Cobb E, Dermann K & Rupp NW (1983) Detecting margin leakage of dental composite restorations *Journal of Biomedical Materials Research* **17(1)** 37-43.

©Operative Dentistry, 2003, **28-4,** 461-464

Invited Paper

Putting It All Together: My Journey in Dentistry

BW Small

Editor's Note

This information was presented on February 28, 2003 as the final didactic session of the annual meeting of the Academy of Operative Dentistry. It was so well received and generated so much positive discussion that I asked Dr Small if he would be willing to condense his verbal presentation into an Invited Paper. I am grateful for his willingness to do so and for his promptness, which enabled me to get this to our readership in a very timely manner.

INTRODUCTION

As a speaker at the February 2003 Academy of Operative Dentistry meeting, I was asked to wrap-up the meeting and try to "Put It All Together." After considerable thought, I decided to attempt this huge task by telling my story of 30 years in dentistry, particularly regarding posterior direct and indirect restorations. Through personal experience and the refereed literature, I related what I had seen and experienced in the clinical and corporate setting. The following is a condensed, written version of that lecture.

The Early Years

I attended dental school between 1970 and 1973 at the University of Medicine and Dentistry of New Jersey. During the last two years, other potential dentists and myself were doing amazing things in the operative clinic in Jersey City, NJ, USA. With the help of Dr Zia Shey, who studied with Dr Michael Buonocore in Rochester, NY, USA, we were actually attaching resin composite to enamel without any retention or resistance form! We were using Nuva-fil and Nuva-Light (LD Caulk, Milford, DE, USA), the first adhesive composite and visible light curing system.

During my early dental practice career I placed many of these adhesive restorations, some with, but many without any preparation, particularly when restoring Class V lesions.

Initial results of placing many of these restorations were excellent, and patients were excited about having their teeth restored in such a manner but, unfortunately, many of those restorations did not last very long.

The refereed dental literature was already warning of problems with posterior composites. Phillips (1985) stated, "Until we have a more accurate picture of exactly what place dental bonding agents will rightfully assume in dentistry, some slight retention should be placed in the dentin at the gingiva when restoring an eroded area with resin."

Fulfil (LD Caulk) was the first posterior composite approved in 1986 by the American Dental Association as an accepted and "clinically-proven material" but, unfortunately, I was already seeing posterior composites in patients on recall visits that were not ideal (Figures 1 and 2). Marginal degradation, loss of anatomic form and wear were all noticed readily. In a published study, Lui and others (1987) concluded "This study indicates that the marginal adaptation at the cervical aspect for conventional Class II composite restorations may present a problem." The literature was ignored by others and myself and poor, short-lived operative dentistry was seen (Figures 3-5). Even popular lecturers at that time were warning about the limitations of posterior composite: Jordan and Suzuki (1991) stated, "Composite resin restorations do not fare well in wide buccolingual preparations in the molar region. Amalgam or cast gold restorations sometimes offer the best solutions to a difficult problem."

The Middle Years

The National Institute of Dental Research (NIDR) sponsored a symposium on esthetic dental materials at the ADA building in 1991. Many clinicians and researchers

*Bruce W Small, DMD

*Reprint request: 133 Franklin Corner Rd, Lawrenceville, NJ 08648; e-mail: bsmall@aosi.com

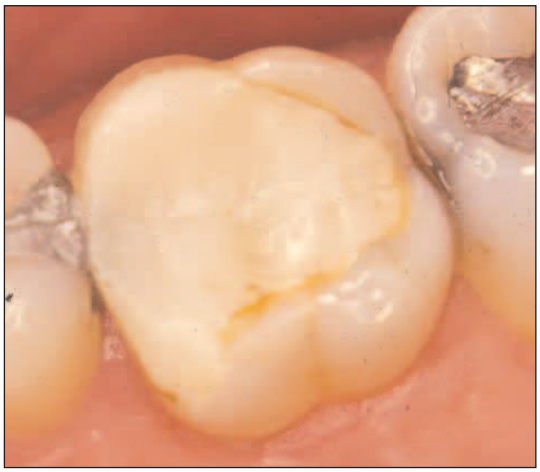


Figure 1. *Three-year post-op view of large posterior composite. Note marginal breakdown.*

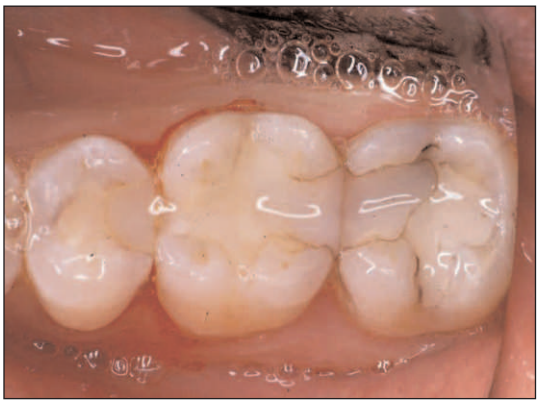


Figure 2. *Two-year post-op view of bicuspid and two molars restored with composite.*

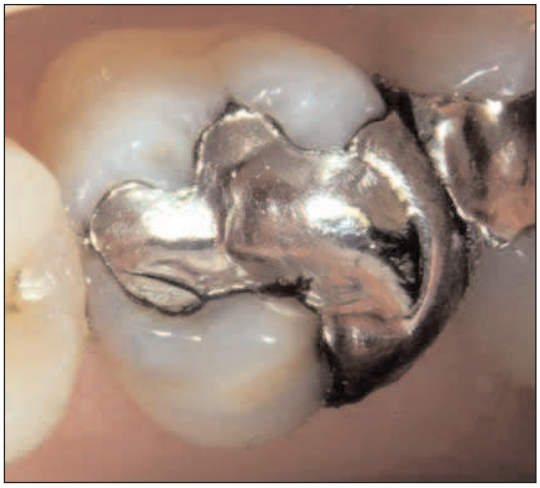


Figure 3. *Failing MODL amalgam.*

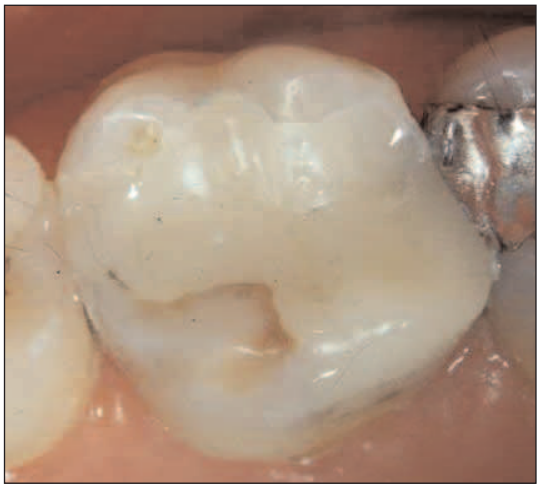


Figure 4. *Six-month post-op of MODL composite.*

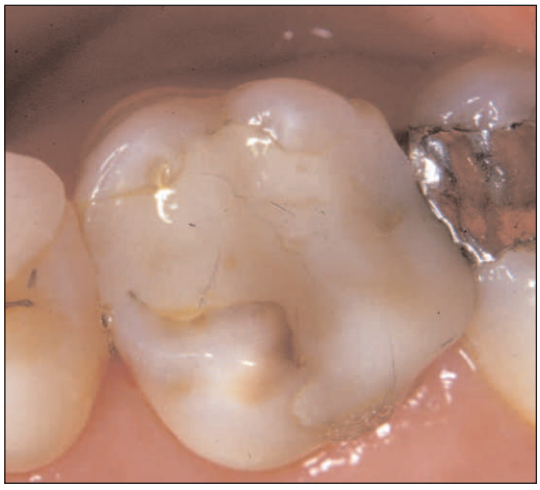


Figure 5. *One-year post-op of MODL composite.*

gave presentations on their experiences, and some warned of problems. Ferracane (1991) concluded, "Although many claims are made, we have no data to support the extended use of resin composites as amalgam replacements."

As a practicing dentist, I was concerned about restoration longevity and what I was seeing happening within my practice. How should I decide what material or technique to use? Should I believe the advertisements and marketing claims of certain dental manufacturers and lecturers who tout the benefits of "metal free" dentistry? Or should I believe people like Terry Donovan (2002) who said, "It is my considered belief that there is nothing inherently beneficial with metal-free dentistry and there is absolutely no evidence base to support such a concept. The current obsession with this concept is the result of marketing hype by several dental manufacturers and aggressive promotion by a handful of entrepreneurial, greedy, and in some cases, unethical practitioners." Christensen (2001) stated, "Has the esthetic

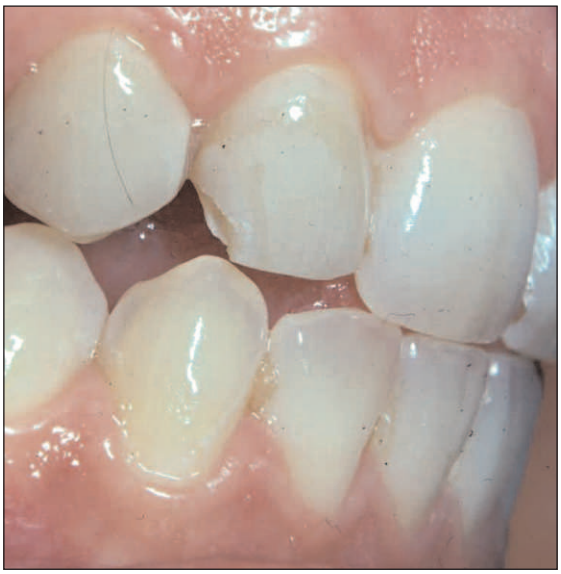


Figure 6. *Fractured porcelain laminate veneer.*

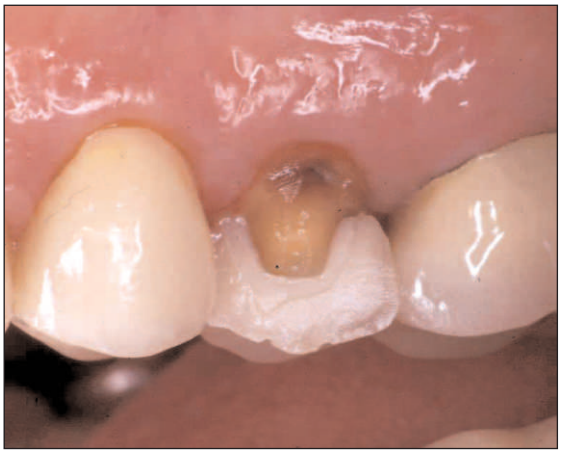


Figure 8. *Fractured pressed ceramic crown.*

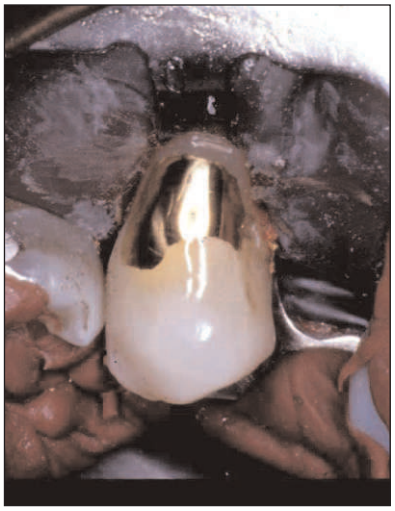


Figure 10. *Class V gold foil.*

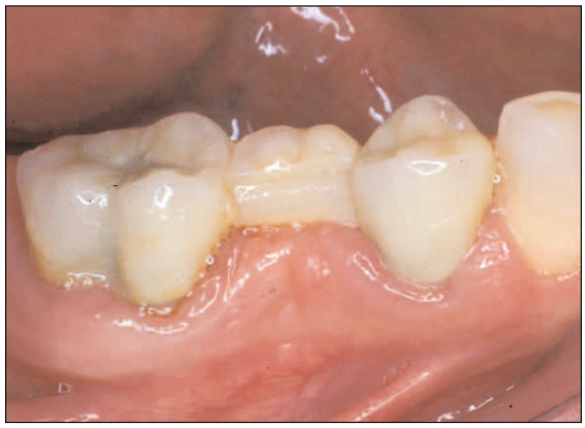


Figure 7. *Fractured composite bridge.*

Figure 9. *Cast gold inlay/onlay.*

pendulum swung too far? I propose a renewed observation of the desirability of restoration longevity interest rather than an obsession with esthetics."

During this time I noticed in the dental publications that all ceramic crowns were being used to restore teeth, complete arches and even full mouth reconstructions. I attempted to use these newer restorative materials since they were introduced in the mid-1980s and found that all was not as anticipated. I observed fractured ceramic veneers (Figure 6), inlays and onlays, all composite bridges (Figure 7) and even pressed ceramic crowns, both core and surface porcelain (Figure 8).

I questioned my choice of materials during these years and sought to improve my clinical skills. Thinking that my occlusal and operative skills were lacking, I began my journey through the continuums at the LD Pankey Institute for Advanced Dental Education and, then, soon afterward, joined the Academy of Operative Dentistry. This was followed by forming two operating study clubs; one for cast gold and one for gold foil. As a member of The Academy of Richard V Tucker Study Clubs and the American Academy of Gold Foil Operators, I was exposed to many of the finest operators in the world.

Through my association with these men and women, I saw the highest quality of clinical dentistry and aspired to learn a better way of restoring teeth that involved more use of gold.

The Recent Years

The recent years have been different for my clinical dental practice and for me. I have found that cast and direct gold are viable restorative materials in the 2000s. Mjör and Medina (1993) published a paper reviewing the age of gold restorations in selected dental practices. Of the 2,564 castings and gold foils examined, only 111 had failed. The average age of the failed restorations was 18.5 years (range 5 to 41), while the average age of those *in situ* was 17 years. I began placing many high quality intracoronal gold castings and compacted gold restorations (Figures 9-10). I noticed that by combining the three basic requirements of a stable occlusion (stable stops, anterior guidance and posterior disclusion) with gold restorations in the posterior of the mouth, my patients were having less fractures, less sensitivity and longer lasting restorations.

My evolution as a clinician was corroborated by the more current dental literature regarding posterior composites. Raskin and others (1999) published a 10-year multi-center study evaluating Class I and Class II posterior composites. They reported failure rates of 30% to 40%, which were defined as loss of approximal contact and loss of anatomic form. Kohler, Rasmussen and Odman (2000) reported a failure rate of 31% of Class II composite restorations after five years. Reasons for failure in this study were recurrent caries and marginal defects.

I also noticed that due to my improved operative skills and increased recognition of what a higher quality restoration should look like, the balance of my dental practice was also improved. I paid more attention to detail at every step of a restorative case and, particularly, the evaluation of laboratory work. I began using a microscope both chairside and in the lab where, together with my technician, we carefully fabricated our own cast gold inlays, onlays and crowns.

Except for the usual bumps in the road, dental practice is now a joy, eagerly anticipated and totally appreciated. To have patients thank you for your time, care, skill and judgment and knowing that you are delivering potentially lifetime restorations, is the greatest joy.

Mentorship

During the most recent part of my journey, I was guided by a mentor. A mentor is a dentist with superior skills who desires to pass on the knowledge and accomplishes this over an extended period of time, usually in an operating study club format. My study clubs are based on this concept from which I have benefited.

I strongly recommend this type of repetitive learning for those who desire to improve their operative skills and knowledge. Learning comes not only from your own work but also from seeing the dentistry of the others in your study club.

SUMMARY

I have grown and evolved as a clinical dentist primarily because I have been exposed to other dentists who have superior skills. It has been my choice to do this, and it has been very satisfying and professionally stimulating. Unfortunately, marketing hype and not understanding the total picture is influencing many of our younger, inexperienced dentists. The purpose of this lecture/paper was to help our younger colleagues realize some of the pitfalls in our profession and guide them in a direction where they may also see the benefits and rewards of our great profession.

Acknowledgements

The author thanks his mentor, Dr Warren Johnson, Seattle, WA, USA, for his continuing mentorship. Also, thanks go to Stony Brook Noble Gold for the lab work in this paper.

The opinions expressed in this paper are those of the author and do not necessarily reflect those of *Operative Dentistry*.

(Received 28 February 2003)

References

Christensen GJ (2001) Cast gold restorations. Has the pendulum swung too far? *Journal of the American Dental Association* **132(6)** 809-811.

Donovan T (2002) Metal free dentistry: Consumer generated or marketing hype? *Journal of Esthetic Dentistry* **14(2)** 71-73.

Ferracane J (1991) NIDR symposium on esthetic restorative materials Nov 11-13.

Jordan RE & Suzuki M (1991) Posterior composite restorations—where and how they work best *Journal of the American Dental Association* **122** 30-37.

Kohler B, Rasmussen CG & Odman P (2000) A five-year clinical evaluation of Class II composite resin restorations *Journal of Dentistry* **20(2)** 111-116.

Lui JL, Masutani S, Setcos JC, Lutz F, Schwartz ML & Phillips RW (1987) Margin quality and microleakage of Class II composite resin restorations *Journal of the American Dental Association* **114(1)** 49-54.

Mjör IA & Medina JE (1993) Reasons for placement, replacement and age of gold restorations in selected dental practices *Operative Dentistry* **18(3)** 82-87.

Phillips (1985) Visible light bonding. A review for the clinician *Journal of the American Dental Association* (interview) **(1165)** p 720-734.

Raskin A, Michotte-Theall B, Vreven J & Wilson NH (1999) Clinical evaluation of a posterior composite: Ten-year report *Journal of Dentistry* **27** 13-19.

©Operative Dentistry, 2003, **28-4,** 465-468

Clinical Technique/Case Report

Preserving Tooth Vitality

E Joffe

Clinical Relevance

Direct pulp capping has a long history of clinical application in dentistry. Since exposed pulp has the ability to heal, creating favorable conditions for pulpal healing is an important part of clinical and research dentistry. A variety of different materials have been used as pulp capping agents. Attempts have been made to seal the opening with well-known materials such as amalgam, glass ionomers, ZOE, IRM, SuperEBA, zinc phosphate cement, gutta-percha and more. Traditionally, covering mechanically-exposed vital, healthy pulp with calcium hydroxide $Ca(OH)_2$ has been used by many researchers with varying degree of success. The researchers were unable to demonstrate predictable results, especially with permanent teeth. New materials and new technology may help this challenging problem.

INTRODUCTION

The widespread use of $Ca(OH)_2$, alone, is a debatable topic. The existing references do not conclusively link dentin bridge formation under calcium hydroxide to the healing process. Bridge formation can occur under different materials and using $Ca(OH)_2$ is not necessarily a requirement for healing (Schroder, 1985; Cox, 1987). The disadvantage of calcium hydroxide is its tendency to dissolve under the restoration within one-to-two years (Cox & Suzuki, 1994). Also, since dentin bridges formed under this material contain tunnels in 50% of the cases, microleakage through these openings may lead to pulpal necrosis (Cox & others, 1985). One of the major factors leading to pulp healing is the suppression of bacterial activity (Matsuo & others, 1996) and hemorrhage control. In order to create and maintain the hermetic seal, the sealing material must be used in a bacterial-free environment, while pulpal bleeding must be completely stopped.

*Eugene Joffe, DDS, PhD, FAGD

*Reprint request: 79-10 34th Ave, Jackson Heights, NY 11372; e-mail: Amecom@earthlink.net

The emergence of a new adhesive technology has changed the conventional approach to "pulp protection" based on $Ca(OH)_2$. Numerous researchers (Cox & others, 1992; Cox & others, 1998) have been studying dentin adhesives and their ability to seal vital pulp. However, the technique sensitive nature of bonding makes pulp capping difficult. In addition, acidic monomers and etchants easily provoke pulpal bleeding.

Attempts to preserve pulp vitality on different stages of pulpitis are reflected in a partial or coronal pulpotomy (Mejare & Cvek, 1993), making the choice of pulp capping materials especially important due to a large, exposed area that need to be hermetically sealed.

Mineral Trioxide Aggregate may help in many clinical situations that require pulp capping. MTA was developed and first reported in 1993 by Lee, Monsef and Torabinejad (1993) and is essentially a modified Portland cement consisting of hydrophilic tricalcium silicate, tricalcium oxide and tricalcium aluminate with some other oxides. Calcium and phosphorus ions are the major components of this cement. With pH=12.5, MTA has low solubility, sets in the presence of water,

possess better sealing ability with less leakage then amalgam, IRM or SuperEBA (Torabinejad, Watson & Pitt-Ford, 1993; Torabinejad & others, 1995) and can be used after pulpotomy or accidental exposure (Myers & others, 1996). MTA is currently marketed by Dentsply under the name "Pro Root."

The following clinical cases demonstrate the use of this material in different clinical situations when pulp capping was used to maintain the vitality of the involved tooth.

Case 1.

Patient P, 32-years old, underwent routine replacement of an old, defective MODF amalgam filling in tooth #19. The tooth was asymptomatic and the existing filling on the periapical x-ray did not indicate any problem or a close proximity to pulp (Figure 1). After the old filling was removed, the secondary caries underneath was excavated using a #4 round bur in a low-speed headpiece under the control of caries detector (Caries Finder, Dunville Materials, USA). After completion of the cavity preparation, a small pulp exposure was detected. (Figure 2).

A cotton pellet with NaOCL was used to disinfect the cavity and stop the bleeding. The mixture of MTA was directly applied to the exposure (Figure 3) and lightly condensed with a moist cotton pellet. The cement was left to set for 15 minutes. The entire preparation was totally etched (Figure 4) using 32% phosphoric acid containing Benzalconium Chloride (BAC) (Uni-Etch, BISCO, Schaumberg, IL, USA). The etching gel was thoroughly rinsed and the cavity slightly dried. The rewetting agent (Aqua-PrepF, BISCO) was generously applied with a large brush on all cavity walls, left for 20 seconds, then slightly dried. Several layers of universal bonding agent (OneStep, BISCO) was applied to all cavity walls and polymerized for 20 seconds.

The cavity was then restored in the usual manner, using universal hybrid composite (Aelitfil, BISCO) (Figure 5).

Pulp testing six months later confirmed the vitality of the tooth. Periapical x-ray clearly shows the radiopacity associated with the cement used for pulp capping (Figure 6) with no periapical changes.

Case 2.

Patient B, a 55-year old. During preparation for porcelain onlay of tooth #15, the portion of carious dentin was left on the distal aspect of the tooth (Figure 7).

While preparing the tooth for bonding, the affected dentin was removed, leading to considerable pulp exposure (Figure 8). In addition, a partial pulpotomy was performed, then a cotton pellet with NaOCL was packed for a few minutes to stop hemorrhaging and disinfect the area. After the bleeding completely stopped, the

Case 1

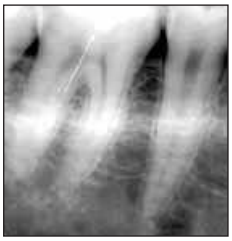


Figure 1. *Pre-operatve.*

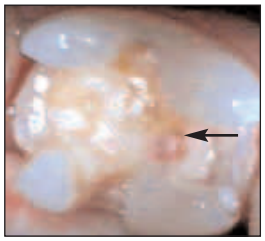


Figure 2. *Pulp exposure.*

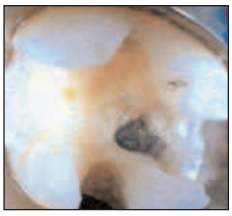


Figure 3. *MTA in place.*

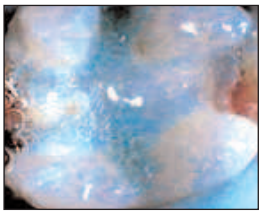


Figure 4. *Etching agent on tooth.*

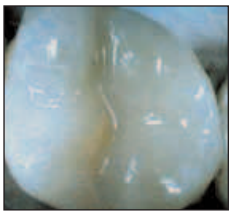


Figure 5. *Restored tooth.*

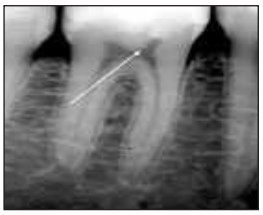


Figure 6. *Six months later.*

opening was filled with MTA (Figure 9) and the cement was left to set for 15 minutes. The corresponding area of internal onlay surface was relieved with a diamond bur, sandblasted and re-etched with (Porcelain etch, BISCO). The entire preparation was etched (UniEtch, BISCO), slightly dried and rewetted with (AquaPrepF, BISCO) for 20 seconds. Two layers of universal bonding agent (OneStep, BISCO) were applied to all tooth surfaces, dried by compressed air and polymerized. The porcelain primer was then applied to the internal surfaces of onlay and gently dried by air syringe for 20 seconds. The universal adhesive OneStep was also applied to all internal surfaces of onlay, dried and polymerized for 20 seconds.

To compensate for possible surface discrepancy between the internal surface of the onlay and the tooth, a chemically cured universal hybrid composite (Bisfill2B, BISCO) was chosen for final fixation of the onlay. This composite is strong, soft and very useful as a luting material in such situations. It fills the voids without compromising the strength of the restoration. Another consideration for using Bisfill2B is because it is chemically cured. This composite potentially produces less polymerization shrinkage stress that could affect the interface with a pulp capping cement. Using a

Case 2

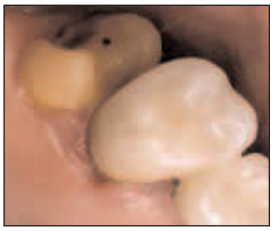


Figure 7. *Caries removal.*

Figure 8. *Pulpal exposure.*

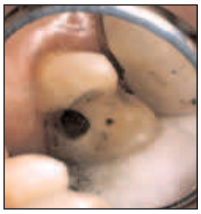


Figure 9. *MTA in place.*

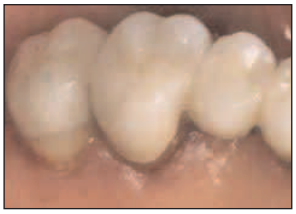


Figure 10. *Crown cemented.*

Centrix syringe (Centrix Inc, Shelton, CT, USA), Bisfill2B was injected into the preparation. As the restoration was placed, the excess composite was removed from around the margins and, as oxygen-blocking Vaseline had been generously applied all over the restoration, it was covered with DryFoil (Jelenko, USA) and left to set for about 15 minutes (Figure 10).

The tooth remained asymptomatic and pulp testing in six and 12 months confirmed the vitality of the restored tooth.

Case 3.

Patient M, a 21-year old presented complaining of pain to hot and cold in tooth #30 for the last two days. The tooth was previously restored with an amalgam filling (Figure 11). The patient was suffering from acute pulpitis. In its dynamic from the coronally-inflamed pulp tissue down to the radicular areas, which represented the transition from reversible to irreversible pulpitis, the degree of pulpal involvement was difficult to establish clinically. However, one may assume that some radicular areas may still be vital and not infected. In this case, considering the history of a recent onset, the authors hoped that the inflammation was still restricted to a coronal portion of the pulp. After removing the old filling and excavating all carious dentin, the exposure was widened and all coronal pulp tissue was completely removed with a sterile round bur in a low-speed handpiece (Figure 12). The cotton pellet with NaOCL was tightly packed inside the pulp chamber in order to stop the hemorrhage and disinfect the cavity and the wound (Figure 13). Then, MTA cement was condensed on top of the orifices and allowed to set for 15 minutes (Figure 14). After the cement hardened, the entire cavity was etched (Figure 15). Since the cement seal was already in place, the acidity

Case 3

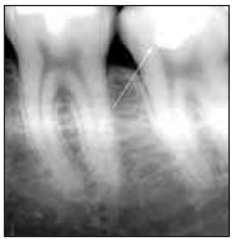


Figure 11. *Pre-operative.*

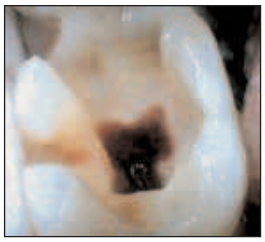


Figure 12. *Coronal pulp tissue removed.*

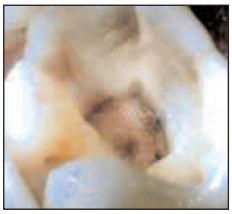


Figure 13. *NaOCL with cotton pellet.*

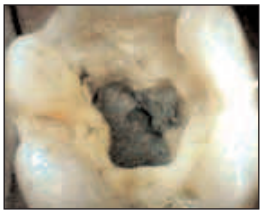


Figure 14. *MTA in place.*

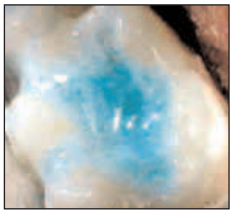


Figure 15. *Tooth etched.*

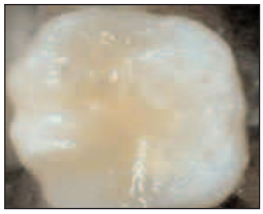


Figure 16. *Resin placed.*

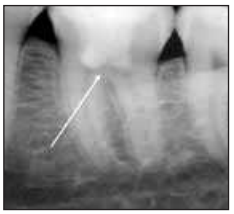


Figure 17. *Pulp cap in place.*

of the bonding components did not present any concern of possible contact bleeding. The tooth was conventionally restored with universal hybrid composite (Aelitfil, BISCO) (Figure 16). Post-operative periapical x-ray showed pulp capping covering the canal orifices (Figure 17).

A few days later, the tooth was asymptomatic and follow-up pulp testing in six and 12 months confirmed the vitality of the restored tooth.

Case 4.

Patient N, a 45-year old, complained of pain from heat and cold in tooth #30. The pain lasted for a few days and started as a mild discomfort. The tooth was previously restored with an amalgam filling (Figure 18). The patient was informed that there is a very high probability that she would need to undergo root canal therapy. The patient was not able to afford the proposed treatment and was ready to have this tooth extracted.

It was suggested that there was a limited chance to save the tooth by the technique described above. If it failed, the patient still had the same options opened to her: root canal therapy or extraction. The old filling was removed and all of the carious dentin excavated, exposing the pulp. The coronal pulp was amputated to the orifice level with a sterile large bur in a low-speed handpiece, making sure that the wound produced a substantial flow of clean, red blood. Hemorrhaging was stopped by tightly packing a cotton pellet with NaOCL, and the bottom of the pulp chamber was filled with MTA (Figure 19). The tooth was restored with hybrid composite (Figure 20), and no pain or tenderness was experienced during the follow-up appointment a few days subsequent.

Case 4

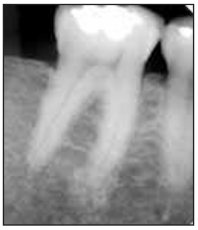


Figure 18. *Pre-operative.*

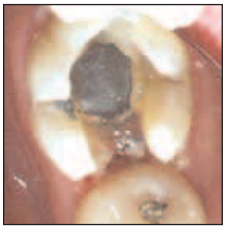


Figure 19. *MTA placed.*

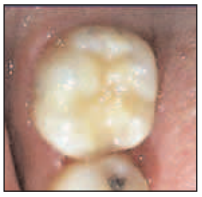


Figure 20. *Resin placed.*

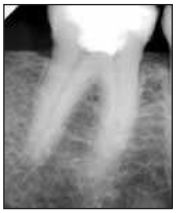


Figure 21. *Six-month post-operative.*

Follow-up appointments in three and six months indicated vitality of the tooth, with the pulp chamber completely obturated and the cement on the level of the canal orifices showing no periapical changes. (Figure 21).

Many times in everyday practice, practitioners face problems that require difficult decisions. One of these problems is pulp exposure, either accidental or intentional, as described in this article. Every time it occurs, we wish that we could avoid root canal therapy. The authors' decisions may have been based on different factors, including cost to the patient, who is not prepared for unexpected expenses related to the need for therapy itself, followed by core build-up and, often, a crown. With the patient's consent, the authors tried to maintain the vitality of the tooth, possibly eliminating root canal therapy, provided we were successful with pulp capping in selected cases. One of the promising materials for this technique seems to be MTA. It is extremely important in providing complete homeostasis and eliminating the chance of bacterial contamination during the procedure. It seems that NaOCL serves well for both of these tasks (Cox, 1999).

MTA is a substantial contribution to existing dental materials. However, difficulties in mixing, problems in delivery to the site and unpredictable setting time of the material present certain difficulties. As a result, these features need to be improved.

(Received 10 July 2002)

References

Cox CF (1987) Biocompatibility of dental materials in the absence of bacterial infection *Operative Dentistry* **12** 146-152.

Cox CF (1999) Lecture in St Petersburg, Russia.

Cox CF, Hafez AA, Akimoto N, Otsuki M, Suzuki S & Tarim B (1998) Biocompatibility of primer, adhesive and resin composite systems on non-exposed and exposed pulps of non-human primate teeth *American Journal of Dentistry* **11(Special Issue)** S55-S63.

Cox CF, Bergenholtz G, Heys DR, Syed SA, Fitzgerald M & Heys RJ (1985) Pulp capping of dental pulp mechanically exposed to oral microflora: A 1-2 year observation of wound healing in the monkey *Journal of Oral Pathology* **14** 156-168.

Cox CF & Suzuki S (1994) Re-evaluating pulp protection: Calcium hydroxide liners vs cohesive hybridization *Journal of the American Dental Association* **125** 823-831.

Cox CF, White KC, Ramus DL, Farmer JB & Snuggs HM (1992) Reparative dentin: Factors affecting its deposition *Quintessence International* **23** 257-270.

Lee SJ, Monsef M & Torabinejad M (1993) Sealing ability of mineral trioxide aggregate for lateral root perforation *Journal Endodontology* **19** 541-544.

Matsuo T, Nakanishi T, Shimizu H & Ebisu S (1996) A clinical study of direct pulp capping applied to carious-exposed teeth *Journal Endodontology* **22** 551-556.

Mejare I & Cvek M (1993) Partial pulpotomy in young permanent teeth with deep carious lesions *Endodontics and Dental Traumatology* **9** 238-242.

Myers K, Kaminski E, Lautenschlager E & Miller D (1996) The effects of mineral trioxide aggregate on the dog pulp *Journal Endodontology* **22(4)** Abstract #39 p 198.

Schroder U (1985) Effects of calcium hydroxide-containing pulp capping agents on pulp cell migration, proliferation and differentiation *Journal of Dental Research* **64(Spec No)** 541-548.

Torabinejad M, Rastegar AF, Kettering JD & Pitt-Ford TR (1995) Bacterial leakage of mineral trioxide aggregate as a root filling material *Journal Endodontology* **21(3)** 109-112.

Torabinejad M, Watson TF & Pitt-Ford TR (1993) Sealing ability of a mineral trioxide aggregate when used as a root end canal filling material *Journal Endodontology* **19(12)** 591-595.

©Operative Dentistry, 2003, **28,** 469-472

Modified Matrix Adaptation for Sub-Gingival Class II Amalgam Restorations

DCN Chan

Clinical Relevance

This technique for adapting two matrix bands in cases involving deep subgingival interproximal margins can result in improved cervical adaptation and proximal contour and contact.

INTRODUCTION

Occasionally, a clinician encounters a deep root caries lesion at the gingival cavosurface margin of a Class II cavity. This area is difficult to restore properly if the cavosurface is close to or right at the bone level. Often, even the gingival extension molar MOD matrix band is inadequate to seal off the subgingival cavosurface margin. Innovative methods are frequently proposed to tackle the challenges of concavity at the gingival cavosurface margin of a prepared cavity (Ireland, 1985; Khera & Swift, 1989; Woodmansey, 1998; Chan 2001). Other proposed techniques deal with severely broken down teeth (Knight 1996; Esquivel & Welsch, 1999). However, no method was proposed to deal with the deep root caries lesion at the gingival cavosurface margin of a Class II cavity.

This paper describes a technique for adaptation of the matrix band in cases where the gingival cavosurface margin extends subgingivally. An open sandwich restoration was prescribed. In such a case, the gingival area is first restored with a resin-modified glass ionomer material, then amalgam condensed on top of the cured glass ionomer layer.

*Daniel CN Chan, DMD, MS, DDS, associate professor and head, Division of Operative Dentistry, Department of Oral Rehabilitation, Medical College of Georgia, Augusta, GA

*Reprint request: Medical College of Georgia, Augusta, GA 30912-1260; e-mail: dchan@mail.mcg.edu

TECHNIQUE

Figures 1-9 illustrate the technique of using a combination of #1 Adult Universal band and #2 Adult MOD Wide stainless steel metal matrix band to seal the gingival cavosurface margin of a deep Class II preparation. Figures 1-3 show the pre-operative condition of tooth #2.

After cavity preparation has been completed, preselect a wooden wedge that will fit into the interdental space. Multiple wedges may be needed for the situation.

1. Place an extra thin (0.0015 in) #2 Adult MOD Wide matrix band (Waterpik Technologies, Inc, Ft Collins, CO, USA) on the tooth and adapt it in the usual manner; loosen the retainer one-half turn to make room for the additional #1 Adult Universal band (Waterpik Technologies, Inc).
2. Cut a 0.002-in #1 Adult Universal band in half (Figure 4). Place the additional #1 band inside the #2 matrix band. The thicker 0.002-in band is preferred because of its stiffness. Use a hemostat to insert the band firmly towards the gingival margin. Check visually to confirm that the mar-

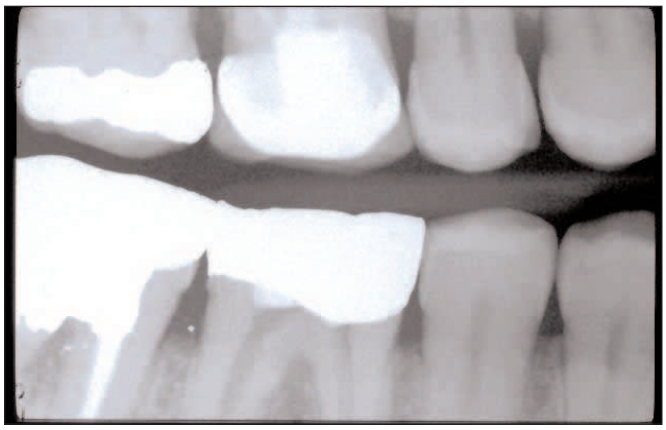


Figure 1. *Bitewing radiograph of an MO amalgam restoration on a maxillary molar #2 five years ago.*

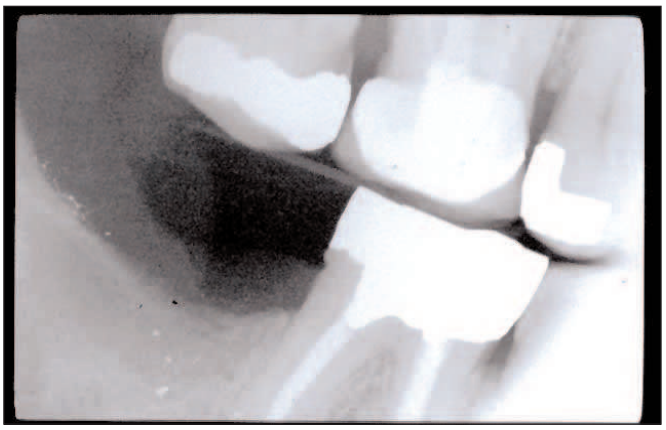


Figure 2. *Pre-operative bitewing radiograph of #2 as referred by a periodontist two months ago. Note extensive recurrent decay underneath MO restoration.*

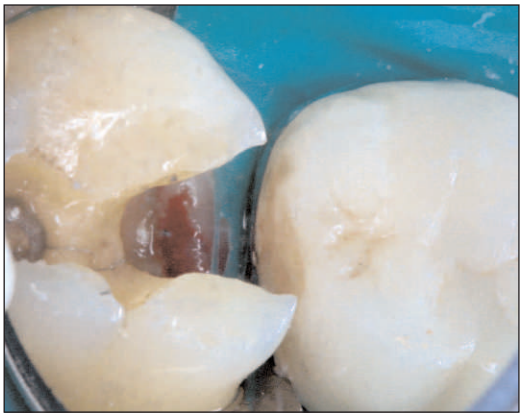


Figure 3. *The occlusal amalgam and decay removed. The mesial box of the molar extended deep gingivally. Even the #2 Adult MOD Wide matrix band is inadequate to seal off the subgingival cavosurface margin.*

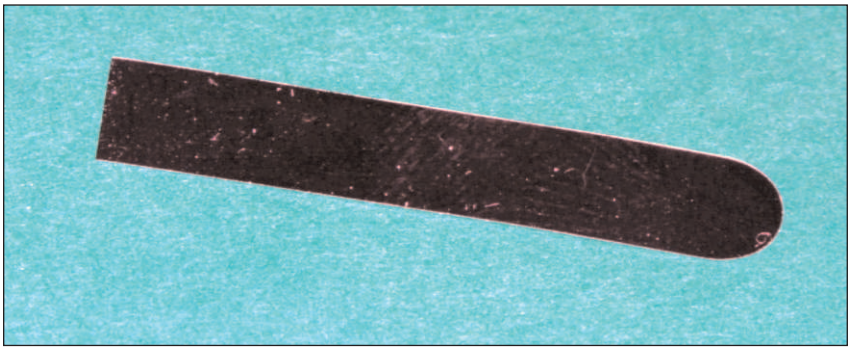


Figure 4A. *#1 Adult Universal band cut in half and ready for the gingival extension procedure.*

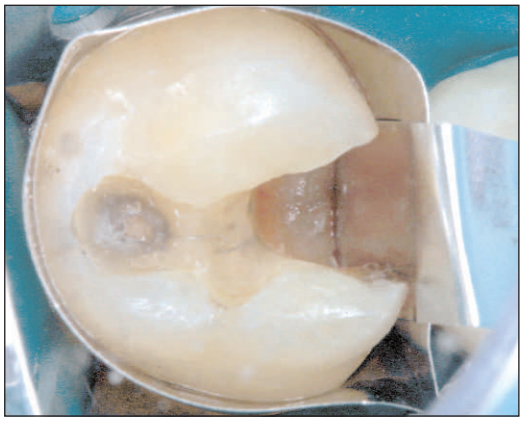


Figure 5. *Correct matrix adaptation has been accomplished with the additional #1 Adult Universal band placed inside the #2 Adult MOD Wide band and secured with a wedge.*

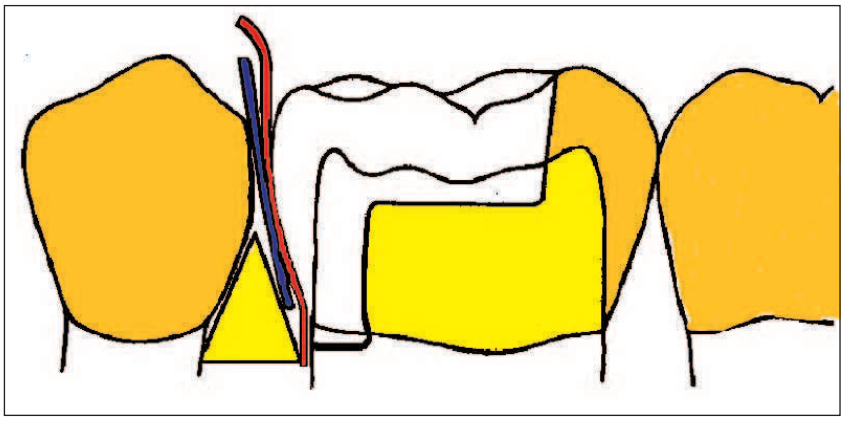


Figure 6. *Schematic diagram of #1/#2 matrix combination and wedge placement. Note the proximal box is sealed with no gap present between matrix band and tooth cavosurface.*

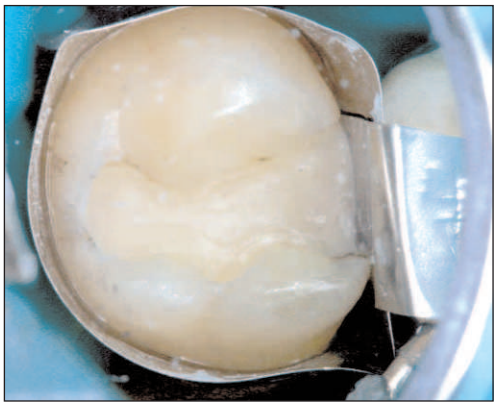


Figure 7. *Occlusal views of the first layer of restoration short of the contact area with a resin-modified glass ionomer. The resin-modified glass ionomer is prepared to receive an amalgam restoration.*

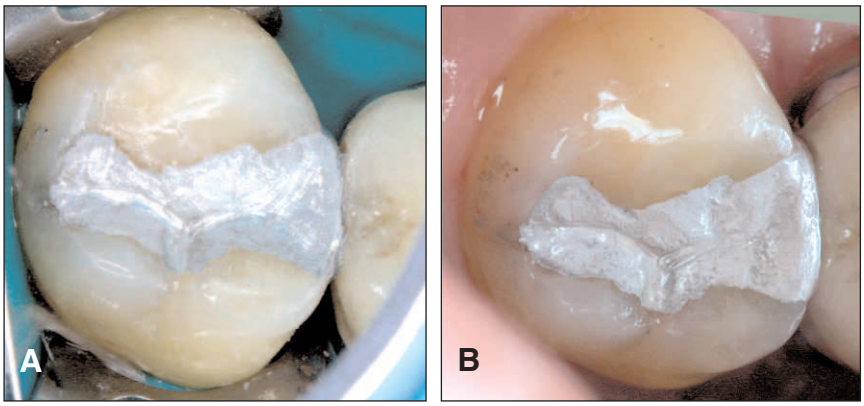


Figure 8A and B. *Occlusal views of the completed restoration with proper contour and contact.*

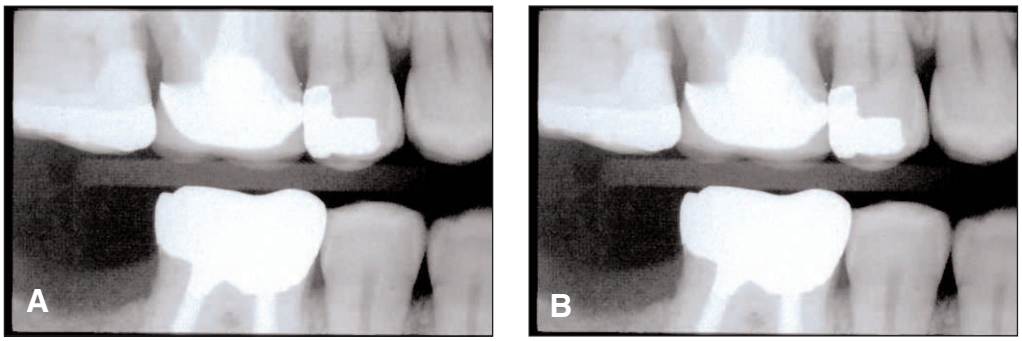


Figure 9A and B. *Post-operative bitewing radiograph of #2. Note proper seal of the gingival margin and proper interproximal contour.*

gins are sealed (Figure 5). Bend the #1 band towards the adjacent tooth for accessibility (Figure 6). One can also use LC block out resin (Ultradent Products Inc, South Jordan, UT, USA) to tag down the matrix band combination. Inject a smaller amount of LC resin over the bended portion of the #1 band and onto the occlusal portion of the adjacent teeth. After light curing, the blocking resin should provide additional retention and stability.

3. Apply the wedge(s) with adequate pressure. Make sure the matrix is tightly adapted against the gingival cavosurface margin and burnish it from the inside for desired interproximal contours and contacts.
4. Once the gingival area is sealed, burnish the proximal box towards the contact area, making the cervical part of the matrix more directly accessible.
5. The prepared cavity is ready to be restored. A resin-modified glass ionomer (Fuji II LC, GC America, Inc, Alsip, IL, USA) is prescribed for the gingival portion to take advantage of its fluoride-releasing property.
6. After restoring with resin-modified glass ionomer up to but shy of the contact area, prepare the occlusal portion to receive an amalgam restoration (Figure 7).
7. While condensing the amalgam restoration, progressively loosen the retainer a half to a full turn to provide for proper contact and contour.
8. Restore the rest of the restoration back to proper contour and anatomy (Figure 8A & 8B).
9. Post-operative radiographs confirm that the gingival margins are adequately sealed with proper contour and contact (Figure 9A & 9B).

SUMMARY

Proper placement of the matrix and wedge is critical for the success of Class II amalgam restorations. Improper placement of matrix and wedges can result in poor contours or contacts, overhangs or weakness resulting from poorly condensed restorative material. This paper presents a technique for adaptation of a combination of matrix bands in cases where the gingival cavosurface margin extends deep sub-gingivally.

(Received (21 March 2003)

References

Chan DCN (2001) Custom Matrix Adaptation with Elastic Cords *Operative Dentistry* **26(4)** 419-422.

Esquivel JF & Welsch BB (1999) Stable matrix for amalgam build-up *General Dentistry* **47(4)** 396-398.

Ireland EJ (1985) Evaluation of a new matrix band and wedge for amalgam preparations having lingual or facial extensions *General Dentistry* **33** 434-435.

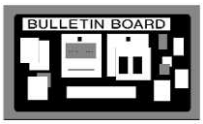
Khera SC & Swift EJ Jr (1989) Custom matrix adaptation for concavities along cavosurface margins *Compendium of Continuing Education in Dentistry* **10(3)** 171-176.

Knight JS (1996) A matrix solution for severely broken-down teeth *General Dentistry* **44(2)** 150-152.

Woodmansey KF (1998) Replacing compound with composite resin for quick and efficient matrices *Journal of the American Dental Association* **129(11)** 1601-1602.

Departments

Announcements



Annual Meeting

American Academy of Gold Foil Operators

Lincoln, NE
October 1-4, 2003

Three half-day essay sessions, plus clinical demonstrations and social activities. Emphasis will be on Direct Filling and Cast Golds.

For details and information, contact:

Dr Ronald K Harris, Secretary,
17922 Tallgrass Court, Noblesville, IN 46060

Phone: (317) 867-0414

Fax: (317) 867-3011

E-mail: piperon@earthlink.net

Funding for Students' Research in Operative Dentistry

Students wanting to carry out research related to Operative Dentistry may apply for a Ralph Phillips Research Award, sponsored by the Founder's Fund of the Academy of Operative Dentistry.

The application should consist of a protocol (and 15 copies) outlining the background, aim/hypothesis to be tested, the methodology to be employed, a time schedule and the expected outcome of the study. The protocol should not exceed three double-spaced type-written pages and a budget page (including where the funds should be sent provided the Award is granted). The budget may not exceed $6,000.

If an abstract, based on the research and acknowledging support from the Academy of Operative Dentistry, is accepted for presentation at the IADR/AADR meeting in 2004, additional travel funds not exceeding $1,000 will be made available to the recipient.

A Faculty Advisor should be named, and he/she should co-sign the application. The application must be submitted by December 11, 2003 to:

Academy of Operative Dentistry,
Research Committee
c/o Dr Ivar A Mjör, Chairman
UFCD, Box 100415
Gainesville, FL 32610

Applications may also be submitted by e-mail to: imjor@dental.ufl.edu followed by one signed original mailed to the above address. Award recipients will be announced during the Annual Meeting of the Academy of Operative Dentistry, February 18-20, 2004.

Operative Dentistry Home Page

We hope all our readers will take advantage of the information available by accessing our Internet home page. Our address is: **http://www.jopdent.org/**

The home page contains a search engine and buttons that, hopefully, will lead you to answers to any questions you may have related to ***Operative Dentistry***. These are:

Journal: Leads to information on the Editorial Staff and Editorial Board; a complete index of journal volumes; a compilation of direct gold references; highlights of the current, next, and future issues, as well as a more detailed look at published Editorials and Clinical Pearls.

Subscribe: Leads to complete information on subscription rates; purchasing back issues, reprints, and bound volumes; and subscription and change of address forms.

Affiliates: Provides links to the American Academy of Gold Foil Operators, the Academy of Operative Dentistry, the AADS-Operative Section, and our Corporate Sponsors. In addition, membership applications for the journal's parent academies are available for downloading.

News: Announcements of interest to our readers, including meeting information, advertised faculty positions, and upcoming CE courses.

Authors: Complete instructions for contributors to the journal.

Reviewers: Link for our Editorial Board to submit manuscript reviews electronically.

Corporate Sponsorship

Operative Dentistry invites dental manufacturers that share our commitment to timely publication of research and clinical articles relevant to the discipline of restorative dentistry to become Corporate Sponsors of the journal. ***Operative Dentistry*** is distributed in 54 countries with more than 1,800 subscribers. The cost of sponsorship is US $3,000.00 per year (January through December). Sponsors will be recognized by having their company logo displayed on a special page in each issue of the journal for the duration of their sponsorship.

A complimentary subscription to ***Operative Dentistry*** will be sent to the corporate contact person, and the sponsor's logo will appear in the Corporate Sponsors section of our web page, where it will act as a link to their company's website. Interested parties should contact the Editor at ***Operative Dentistry,*** 1121 West Michigan Street, Indianapolis, IN 46202-5186; phone: (317) 278-4800; fax: (317) 278-4900; e-mail: editor@jopdent.org; URL: http://www.jopdent.org/

Instructions to Contributors

Correspondence

Send manuscripts and correspondence regarding manuscripts to Dr Michael A Cochran, Editor, *Operative Dentistry*, Indiana University School of Dentistry, Room S411, 1121 W Michigan St, Indpls, IN 46202-5186; phone (317) 278-4800; fax (317) 278-4900; e-mail: editor@jopdent.org; URL: http://www.jopdent.org/.

Exclusive Publication

All material submitted for publication must be submitted exclusively to *Operative Dentistry*. Manuscripts not following the form outlined below may be returned for correction and resubmission.

Manuscripts

❒ Submit an original typed manuscript and three copies. The manuscript should include a short title for running headlines. Any identifying information (author's names, etc) should be on a separate page and not a part of the manuscript. Authors with English as a second language should consider having their manuscript reviewed for grammar, syntax and punctuation prior to submission.

❒ Submit a computer disk and identify the operating system (Macintosh or IBM-compatible) and the word processing program used.

❒ Identify the corresponding author and provide a complete address, fax number and e-mail address.

❒ Supply complete names, degrees, titles and affiliations for all authors (include addresses that are different from the corresponding author's).

❒ Proprietary names of equipment, instruments and materials should be followed in parenthesis by the name, company, city and state or country of the source or manufacturer.

❒ Research (clinical and laboratory) papers MUST include a one sentence Clinical Relevance statement, as well as a Summary, Introduction, Methods and Materials, Results, Discussion and Conclusions section. Funding other than material supply must be stated.

❒ Clinical Technique/Case Report papers should contain at least the following: Purpose, Description of Technique or Solution, along with materials and potential problems and a Summary outlining advantages and disadvantages.

❒ Type double-spaced, including references, and leave margins of at least 3 cm (1 inch). Spelling should conform to the *American Heritage Dictionary of the English Language*. SI (Système International) units are preferred for scientific measurement, but traditional units are acceptable.

❒ The editor reserves the right to make literary corrections.

Illustrations

Please do NOT submit any illustrations or graphs in Microsoft Power Point or Word format. They will not be accepted.

❒ Submit four copies of each illustration.

❒ Line drawings should be in India ink or its equivalent on heavy white paper, card or tracing velum. All lettering must be of professional quality, legible against its background and remain proportionally legible if reduced. Type legends on separate sheets.

❒ Photographs should be on glossy paper with a maximum size of 15x20 cm (6x8 inches). For best reproduction, a print should be one-third larger than its reproduced size.

❒ On the back of each illustration indicate lightly in pencil the top and the number of the figure ONLY (no names). Where relevant, state staining technique(s) and the magnification of the prints. Obtain written consent from holders of copyright to republish any illustrations published elsewhere.

❒ Illustrations may also be supplied on floppy disk, Zip disk or CD as TIFF files with a minimum resolution of 300 dpi (dots per inch) for grayscale and 1200 dpi for color.

❒ Photographs become the property of *Operative Dentistry*.

Tables and Graphs

❒ Submit tables and graphs on sheets separate from the text.

❒ Graphs are to be submitted with any lettering proportional to their size, with their horizontal and vertical axes values displayed.

❒ Data for constructing graphs MUST be provided with the manuscript in a spreadsheet (Excel) or word processing format on computer disk.

❒ Graphs may be supplied on floppy disk, Zip disk or CD as TIFF files with a minimum resolution of 300 dpi. or as Microsoft Excel files.

References

❒ References must be arranged in alphabetical order by authors' names at the end of the article, with the year of publication placed in parentheses immediately after the author's name. This is followed by the full journal title (no abbreviations and in italics), the full subject title, volume and issue number and first and last pages.

❒ In the text, cite references by giving the author and, in parentheses, the date: Smith (1975) found…; or, by placing both name and date in parentheses: It was found…(Smith & Brown, 1975; Jones, 1974).

❒ When an article being cited has three authors, include the names of all of the authors the first time the article is cited; subsequently, use the form (Brown & others, 1975). Four or more authors should always be cited in the text as (Jones & others, 1975). In the References section, always list all the authors.

❒ If reference is made to more than one article by the same author and is published in the same year, the articles should be identified by a letter (a, b) following the date, both in the text and in the list of references.

❒ Book titles should be followed by the publication address and the name of the publisher.

Reprints

Reprints of any article, report or letter can be ordered through the editorial office.

OPERATIVE DENTISTRY
CORPORATE SPONSORS

These Dental Manufacturers have joined Operative Dentistry in our commitment to publish quality dental literature in a timely manner. We thank them for their support.

SDI
SOUTHERN DENTAL INDUSTRIES

Den-Mat®

DENTSPLY
CAULK

BISCO
Specialist In Adhesive and Composite Technology
Bringing Science to the Art of Dentistry™

ULTRADENT

3M ESPE

sds Kerr
SYBRON DENTAL SPECIALTIES

ivoclar vivadent®

Please view the Corporate Sponsors Page at our website (http://www.jopdent.org) for direct links to these companies.

JULY/AUGUST 2003 • VOLUME 28 • NUMBER 4 • 325-476

10-9385
Operative Dentistry
Indiana University School of Dentistry, Rm S411
1121 West Michigan Street
Indianapolis, IN 46202-5186 USA

Periodicals