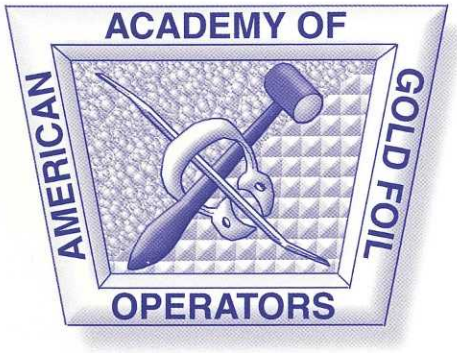
OPERATIVE DENTISTRY



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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.

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Guest Editorial

OPERATIVE DENTISTRY: The Vanishing Discipline

When someone asks my occupation, I generally reply that I am a dentist and a member of a dental school faculty. If additional interest is expressed, I may say that I teach operative dentistry, knowing that further explanation will be necessary since laymen often associate the term with dental surgery. This is an interesting phenomenon since "operative dentistry" is so clearly defined in my own mind and is, in fact, the very essence of my professional life. Unfortunately, this lack of understanding of the nature and importance of operative dentistry seems to be pervasive at all levels of dental education, organized dentistry and the practicing profession. I believe the reasons for this are many and merit close scrutinization, consideration and, hopefully, correction.

Those responsible for teaching the knowledge and skills necessary for the various disciplines within dentistry should have training and education beyond the basic dental degree. This usually requires that these educators focus their activities in a specified area to allow them to present information at a highly informed and current level. The dental specialties are very conscious of this and know that it has a direct influence on the capability of their residents. It would be rare for a school of dentistry to assign general practitioners the responsibility for the primary training of dental students in endodontics, periodontics or oral surgery, but many voices suggest this as the ideal for operative dentistry training.

As a discipline, operative dentistry has suffered over the years because we have not been as militant in stressing its importance in dental practice. This has resulted in limited availability of advanced training in operative dentistry, and the growth of an attitude that such training is not really necessary and that all dental school graduates and general practitioners are perfectly capable of teaching all restorative clinical procedures. This is akin to suggesting that a recreational snow skier, such as myself, can offer the same level of instructional expertise as a professionally trained ski instructor. In my opinion, the traditional values that accompany advanced education in all disciplines certainly apply to training and education in operative dentistry. If there are logistical problems for faculty in satisfying the need for advanced education, an excellent alternative in securing credentials beyond the minimum is the American Board of Operative Dentistry certification program. Preparation for the three-part examination (didactic, clinical performance and case presentations) encourages focused study on the part of the candidate and, upon completion, offers tangible evidence of advanced knowledge and skills.

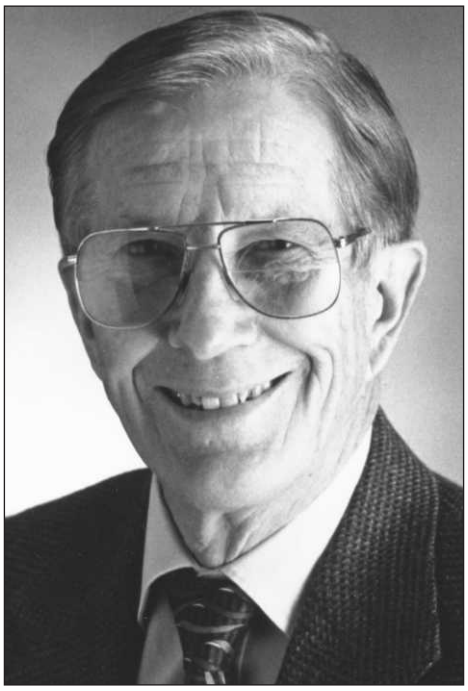
Another area experiencing decline within our discipline is the interaction of operative dentistry teachers within and between educational institutions and other dental specialties. Historically, the importance of such interaction received strong emphasis with the formation of ACORDE (A Consortium on Restorative Dentistry Education) in the early 1970s. This project was funded by the US Department of Health, Education and Welfare and allowed all dental schools in the country to work together on a common project—developing expanded function auxiliaries. ACORDE influenced the scope of operative procedures and instrumentation for the duration of its existence. As the project concluded, the value of its communication format gained recognition and resulted in the formation of the Conference of Operative Dentistry Educators (CODE). Initially, the combination of sectional meetings and the distribution of a composite report to all North American dental schools was extremely positive in promoting discussions and consensus on the myriad aspects of teaching operative dentistry. Unfortunately, busy schedules and the pressures of academic life resulted in a decline in active participation by all schools and faculty over the years. Even the activity of the Operative Dentistry Section at the annual meeting of the American Dental Education Association has been cut from more than one-half day

to two hours or less. This certainly makes it more challenging for operative faculty to justify the expense and time associated with attending this meeting.

What are some of the results of the problems discussed above? Anecdotal reports from dental faculty, various post-graduate programs, the Dental Services sections of the branches of our Armed Forces and some Dental Board Examiners would suggest that the overall clinical ability of recent graduates is not at the levels we enjoyed during the '60s, '70s and early '80s. This is significantly problematic since the intelligence, educational experience and capability of today's dental student is certainly equal to if not better than that of earlier graduates. One must wonder whether the current trends in dental education are not contributing factors, including:

- the lack of emphasis on advanced training when hiring operative faculty.
- the fostered perception that since dental school is preparing general practitioners that generalists should supervise the bulk of the students' clinical training.
- the de-emphasis on the importance of operative dentistry as a discipline mirrored in the creation of large restorative dentistry or general dentistry departments with, at most, an operative section or division.
- the overall reduction in both laboratory and clinical curricular time for operative dentistry with, perhaps, an overemphasis on cosmetic procedures and tooth-colored materials to the exclusion of more traditional and proven techniques.
- more and more reliance on continuing education programs that are industry driven to not only supplement dental education but also to provide needed funding for the dental school.

It seems obvious that in order to maintain a state of health, dental schools should encourage the maximum educational output of all the disciplines in dentistry.



Melvin R Lund

Unfortunately, this does not seem to be the direction operative dentistry is heading. Operative dentistry as a discipline appears to be an endangered species and may disappear if steps are not taken now. Certainly, the Academy of Operative Dentistry should be a prime mover in this task, along with CODE and the Operative Dentistry Section of the ADEA. It is time for these groups to stop worrying about being perceived as "interfering" with dental education and take aggressive, positive action to promote operative dentistry and return it to the position of influence it deserves.

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Clinical Research

The Effect of 10% Carbamide Peroxide Bleaching Material on Microhardness of Sound and Demineralized Enamel and Dentin *In Situ*

RT Basting • AL Rodrigues Jr • MC Serra

Clinical Relevance

In clinical situations, treatment with 10% carbamide peroxide agent can alter the microhardness of sound and demineralized enamel although it does not affect the microhardness of sound and demineralized dentin.

SUMMARY

This in situ **study evaluated the microhardness of sound and demineralized enamel and dentin submitted to treatment with 10% carbamide peroxide for three weeks. A 10% carbamide peroxide bleaching agent—Opalescence/Ultradent (OPA)—was evaluated against a placebo agent (PLA). Two hundred and forty dental fragments—60 sound enamel fragments (SE), 60 demineralized enamel fragments (DE), 60 sound dentin fragments (SD) and 60 demineralized dentin fragments (DD)—were randomly fixed on the vestibular surface of the first superior molars and second superior premolars of 30 volunteers. The volunteers were divided into two groups that received bleaching or the placebo agent at different sequences and periods at a double blind 2 x 2 crossover study with a wash-out period of two weeks. Microhardness tests were performed on the enamel and dentin surface. The SE and DE submitted to treatment with OPA showed lower microhardness values than the SE and DE submitted to treatment with PLA. There were no statistical differences in microhardness values for SD and DD submitted to the treatment with OPA and PLA. The results suggest that treatment with 10% carbamide peroxide bleaching material for three weeks alters the enamel microhardness, although it does not seem to alter the dentin microhardness.**

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INTRODUCTION

The demand for conservative aesthetic dentistry has dramatically grown. So has the rapid development of new non-restorative treatments for discolored teeth. Frequently, vital teeth present changes in color that substantially compromise the smile. As nightguard vital bleaching has gained popularity with patients and dentists as a conservative technique to lighten natural teeth (Haywood & Heymann, 1989), modifications, improvements and variations of the technique were introduced, including using a soft custom-fitted tray (from 0.2 to 0.4 mm thick), different concentrations of carbamide peroxide agents (10 to 22%) with carbopol and using these agents for one or more intervals during the day. Different products and systems have appeared on the market for in-office use, such as 35% hydrogen peroxide or as over-the-counter products (Haywood, 1994). However, 10% carbamide peroxide is still the most used at-home bleaching technique, with several reports in literature about its safety and effectiveness. The decomposition products of carbamide peroxide—water, oxygen, ammonia and carbon dioxide—are easily found in the normal processes of the human body. Therefore, the Food and Drug Administration (Haywood, 1993) classifies carbamide peroxide as safe and effective for human use as an oral antiseptic in 10% concentration and has ADA approval (Haywood & Robinson, 1997).

Ten percent carbamide peroxide dissociates into 3% hydrogen peroxide and 7% urea. These products are obtained after the dissociation of the bleaching product with the contact of saliva and oral fluids. Urea is degraded into ammonia and carbon dioxide. The hydrogen peroxide, because of its instability and ease of decomposition into water and oxygen, penetrates through the pores of enamel and dentin to provide the lightening of the teeth. Due to the low amount of hydrogen peroxide in the home bleaching products (only 3%), there is a need for a prolonged contact of the agent with the dental structure to initiate the oxidation process (Goldstein & Kiremidjian-Schumacher, 1993; Goldstein & Garber, 1995). Bleaching agents affect the lightening of tooth structure through decomposition of peroxides into free radicals. The free radicals break down large pigmented molecules in enamel and dentin into smaller, less pigmented molecules. This is the point (the saturation point) at which whitening should be terminated (Goldstein & Garber, 1995). If a prolonged period is spent—that is, when patients want their teeth "over" whitened—protein matrix breaks can occur in enamel and dentin (Goldstein & Garber, 1995). Therefore, one possible side-effect of bleaching products is that the enamel and dentin may be weakened by oxidation of the organic or inorganic elements.

As bleaching of vital teeth involves direct contact of the whitening agent on the outer enamel surface for an extensive period of time, many studies have evaluated the potential adverse effects of these carbamide peroxide agents. When using SEM evaluations, changes in enamel (Ben-Amar & others, 1995; Bitter, 1998; Bitter & Sanders, 1993; Ernst, Marroquin & Willershausen-Zonnchen, 1996; Flaitz & Hicks, 1996; Josey & others, 1996; McGuckin, Babin & Meyer, 1992; Nam, Kugel & Habib, 1999; Shannon & others, 1993; Smidt & others, 1998; Zalkind & others, 1996) and dentin surface morphology (Zalkind & others, 1996) were reported.

However, not only the micromorphology of the dental tissues can be affected by bleaching agents, but changes in the mineral content of the enamel and dentin should be evaluated as well, due to the acidic property of the bleaching agents (Ben-Amar & others, 1995; Ernst & others, 1996; Leonard & others, 1994; Murchison, Charlton & Moore, 1992; Smidt & others, 1998; Zalkind & others, 1996) that could affect tooth structure. Loss of mineral content or demineralization alters enamel and dentin microhardness (Featherstone & others, 1983; Rotstein & others, 1996), even though saliva, fluorides or other remineralizing solutions can maintain the balance between the phenomena of demineralization and remineralization.

In vitro studies have reported some alterations in enamel microhardness and loss of calcium after exposure to 10% carbamide peroxide (McCracken & Haywood, 1996; Rotstein & others, 1996; Attin & others, 1997; Smidt & others, 1998; Rodrigues & others, 2001). No changes in enamel microhardness were reported by Murchison & others (1992), Seghi & Denry (1992), Nathoo, Chmielewski & Kirkup (1994) and McCracken & Haywood (1995). In dentin, Nathoo, Chmielewski & Kirkup (1994) showed no changes in dentin microhardness, although Pécora & others (1994) and Rotstein & others (1996) showed significant alterations in the mineral content when using 10% carbamide peroxide agents.

In a combined *in vitro-in vivo study*—the bleaching treatment was performed in *in vitro* conditions and the remineralization period was performed inside the mouth—Shannon & others (1993) showed a decrease in the initial microhardness of enamel due to the bleaching agent, followed by an increase in enamel microhardness resulting from a possible remineralization phenomenon of saliva. However, interactions of the bleaching agent with the oral environment were not evaluated. Therefore, *in situ* studies should be conducted to evaluate the direct interaction among product, saliva, soft tissue and sound or demineralized teeth.

It is also possible that bleaching agents have been applied on active carious lesions in enamel and dentin

because there is a frequent absence of procedures to arrest incipient lesions before the aesthetic/restorative procedures. Since the bleaching agent penetrates through demineralized dental tissues, there is a need for additional research on the effects of the nightguard vital bleaching agent on these tissues in clinical situations.

This paper evaluated *in situ* the microhardness of sound and demineralized enamel and dentin when submitted to treatment with 10% carbamide peroxide bleaching material and a placebo agent for three weeks.

METHODS AND MATERIALS

A) Experimental Design

Thirty volunteers took part in this double-blind experiment performed in two periods with a two-week washout period. The volunteers were randomly divided into two groups of 15. Each group received the bleaching or the placebo agent for three weeks in different sequences, in two distinct periods (bleaching agent—placebo agent; placebo agent—bleaching agent) in a crossover 2 x 2 study (Montgomery, 1991).

The factors under study were:

Treatment Agents: (two levels) experimental—Opalescence/ Ultradent; control –placebo agent;

Quality of Dental Tissue Fragments: (two levels) sound and demineralized.

The experimental units consisted of 240 dental fragments: 60 sound enamel fragments; 60 demineralized enamel fragments; 60 sound dentin fragments and 60 demineralized dentin fragments. One fragment of each dental tissue was randomly distributed in complete blocks among 30 volunteers. Each volunteer was considered a block. All the volunteers underwent treatment with the bleaching agent for three weeks, then for another three weeks with the placebo agent.

B) Selection of Volunteers

The volunteers were 30 adults (23 females and 7 males) from 19 to 25 years of age. Each volunteer was informed of the objectives, benefits and possible risks involved in this experiment and participated only after providing written formal consent. This study had the approval of the FOP/UNICAMP Ethical Committee Guidelines in agreement with the National Health Council (Brazil, 1996).

The volunteer candidates for bleaching treatment were undergraduate students from the Dental School of Piracicaba, São Paulo, Brazil. Each volunteer's need for a bleaching treatment was evaluated by assessing tooth color using a Vita shade guide (Vita Zahnbabrik & H Rauter GmbH & Co KG, Bad Säckingen, Germany, D-79704). Exclusion criteria for participating in this study was whether volunteers wore fixed or removable dentures or orthodontic appliances, were pregnant or nursing women, smokers or had dentin sensitivity.

C) Tray (Nightguard) Preparation

Superior and inferior dental arch impressions were taken with alginate (Jeltrate/Dentsply, Mildford, DE 19963, USA) and stone cast molds were made (Vigodent, Rio de Janeiro, RJ, Brazil, 21041-150). The maxillary casts were horseshoe-shaped without a palate to avoid interference with the efficiency of the vacuum pull on the hot thermoplastic sheet.

In the molds, vestibular reservoirs with three coatings of nail varnish (Colorama/CEIL Com Exp Ind Ltda, São Paulo, SP, Brazil, 05113-900) were prepared on all teeth except for the last teeth of the arches.

On the superior first molars and superior second premolars (or, when the latter were missing, the superior first premolars), larger reservoirs of 5 mm x 5 mm x 4 mm were prepared with composite resin (Charisma/Heraus Kulzer, Wehrheim, TS, Germany, D-61273) corresponding to the dental fragments that would be fixed in the volunteers.

Two scalloped trays were manufactured for each volunteer in a vacuum-forming machine (P7/Bio-Art Equip Odontológicos Ltda, São Carlos, SP, Brazil, 13568-000) using a flexible ethyl vinyl acetate (EVA) polymer (Bio-Art Equip Odontológicos Ltda, São Carlos, SP, Brazil, 13568-000) that was 0.4 mm thick.

D) Specification of the Materials

A 10% carbamide peroxide bleaching agent (Opalescence/Ultradent, South Jordan, Utah 84095, USA)—recognized by the American Dental Association (ADA)—was evaluated. The pH level of the bleaching

Table 1: *Composition, pH, Presentation Form and Manufacturer of Each Treatment Agent*

Treatment Agents	Composition	pH	Packaging	Manufacturer
Opalescence	10% carbamide peroxide; carbopol; glycerin; flavoring*	6.68	Dispensable syringe	Ultradent Products Inc, South Jordan, Utah 84095 USA
Placebo	5% glycerin; 1.2% carbopol 940	7.0	Dispensable syringe with package identical to Opalescence	Mixed formula, Proderma–Phamacy, Piracicaba, 13414-000, Brasil

* The manufacturer does not indicate the percentage of each component.

agent was measured using a pH meter (Procyon, SA 720, São Paulo, SP, Brazil, 04530-970). The control group consisted of a placebo agent prepared with carboximethilcellulose and glycerin. The color, taste, flavor, consistency and packaging of the placebo agent was similar to the bleaching agent, but the placebo was pH neutral and had no active component (carbamide peroxide). Table 1 presents the basic composition, pH level, packaging and the manufacturer of each treatment agent.

E) Preparation of the Dental Fragments

Forty non-erupted third molars were used in this study. Immediately after extraction, the teeth were kept in 10% formaldehyde at pH 7.0. They were sectioned with double-faced diamond discs (KG Sorensen, Barueri, SP, Brazil, 06454-920) at a low motor speed (Kavo do Brasil, Joinville, SC, Brazil, 89221-040), dividing the root from the coronary portion. In the root portion, the apical third was discarded and only the cervical region was used.

Two hundred and forty dental fragments 4 mm x 4 mm x 2 mm (120 enamel fragments and 120 dentin fragments) were obtained. The fragments presenting stains or cracks after observation on stereomicroscope at 30x (Meiji Techno EMZ Series, Saitama, Japan, 356) were not used.

The dental fragments were embedded individually in a self-curing polyester resin with the external surface of the enamel or dentin exposed. The external surfaces of the dental fragments were leveled by a water-cooling mechanical grinder (Maxgrind/Solotest, São Paulo, SP, Brazil, 01328-000). For the enamel fragments, aluminum oxide discs of 400, 600 and 1000 grit were used sequentially (Carborundum/3M do Brasil Ltda, Sumaré, SP, Brazil, 13001-970) with water coolant. Polishing was performed with polishing cloths (Top, Gold and Ram, Arotec Ind e Com Ltda, Cotia, SP, Brazil, 06709-150) and diamond pastes of 6, 3, 1 and 0.25 µm (Arotec Ind e Com Ltda) with mineral oil coolant (Lubrificante azul modelo LA, Arotec Ind. e Com Ltda). For the dentin fragments, only aluminum oxide discs were used in a sequential granulation of 600, 1000 and 1200 grit (Carborundum/3M do Brasil Ltda) with water coolant.

The fragments were removed from the polystyrene resin with a probe. All dental fragments were immersed in containers with distillated and deionized water and steam sterilized (Tuttnauer 2340MK, Ronkonkoma, NY 11779, USA) for 20 minutes at 121ºC. Steam sterilization is the most effective method to avoid bacterial contamination (Pantera & Schuster, 1990; Amaechi, Higham & Edgar, 1998; Dewald, 1997) and does not change the mineral content of the teeth (Amaechi & others, 1998, Oliveira, Sperandio & Souza, 1999).

F) Induction of Artificial Caries Lesion

To obtain 60 demineralized enamel fragments and 60 demineralized dentin fragments, caries-like lesions were generated by a dynamic model of demineralization and remineralization cycles similar to the model proposed by Featherstone & others (1986) and modified by Serra & Cury (1992).

The enamel fragments were submitted to seven cycles of de-remineralization (Serra & Cury, 1992), while the dentin fragments were submitted to three cycles of de-remineralization (Hara & others, 2000). The 60 enamel dental fragments and 60 dentin fragments that made up the sound group of each dental tissue were not submitted to de-remineralization cycles but kept immersed in distillated and deionized water.

G) Preparing the Volunteers for the Experimental Phase

The initial color of the teeth was determined by Vita scale and photographs were taken to compare the initial to the final color after the experimental phase.

Two weeks before initiation of the experiment, volunteers received toothbrushes (Oral B 35/Gillette do Brasil Ltda, Manaus, AM, Brazil, 69075-900) and fluoride toothpastes (Colgate MFP/ Kolynos do Brasil Ltda, Osasco, SP, Brazil, 06020-170). They received instructions on how to perform the Bass dental hygiene technique to standardize the toothbrushing method and the fluoride levels in the mouth. This phase was called the run-in period and lasted for two weeks.

The 30 volunteers were randomly divided into two groups of 15. Group 1 received the bleaching treatment while Group 2 received the placebo. In a second phase, Group 1 received the placebo treatment while Group 2 received the bleaching treatment.

H) Experimental Phases

Four dental fragments, one of sound enamel, one of demineralized enamel, one of sound dentin and one of

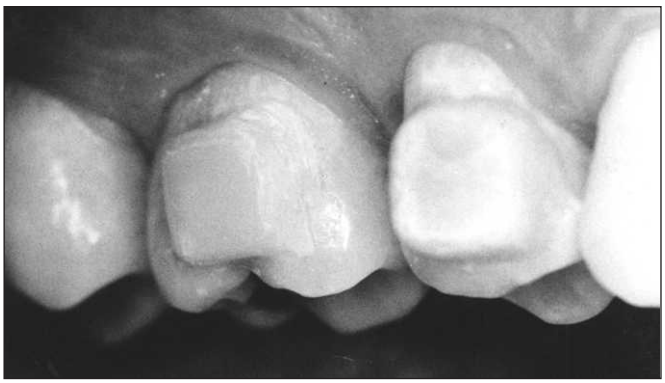

Figure 1. *Dental fragments fixed to the vestibular surfaces of the superior first molar and second premolars.*

Table 2: *Experimental Phases and Periods of the Study According to the Volunteer Groups*

Phase	Period	Group 1 (15 volunteers)	Group 2 (15 volunteers)
Run-In	2 weeks	Standardized brushing with toothbrushes and toothpastes provided; tray manufacturing	Standardized brushing with toothbrushes and toothpastes provided; tray manufacturing
Experimental Phase I	3 weeks	Bleaching agent	Placebo agent
Wash-Out	2 weeks	Standardized brushing with new toothbrushes and toothpastes provided	Standardized brushing with new toothbrushes and toothpaste provided
Experimental Phase II	3 weeks	Placebo agent	Bleaching agent
Post-Experimental Phase	Time required for the volunteer	Evaluation; continuation of the bleaching treatment on both arches; follow-up	Evaluation; continuation of the of the bleaching treatment on both arches; follow-up

demineralized dentin, were randomly fixed to the vestibular surfaces of the superior first molars and superior second premolars (or, when the latter were missing, the superior first premolars) of each volunteer (Figure 1). The fragments were fixed using an adhesive system (Scotchbond Multi-Purpose/3M Dental Products, St Paul, MN 55144-1000 USA) and a composite resin (Charisma/Heraus Kulzer). The bleaching or placebo treatment was applied in the superior dental arch of each volunteer, where the experimental units were attached (vestibular surfaces of the teeth).

Fifteen volunteers applied the bleaching agent (Group 1), while 15 volunteers applied the placebo agent (Group 2) in the tray and wore it during the night for about eight hours. They were instructed to clean the tray after removing it from the mouth and keep it in a container provided.

After three weeks of the treatment with the bleaching or placebo agent (experimental Phase 1), the fragments were removed with appropriate pliers. The composite resin that adhered to the volunteer's tooth was removed with resin polishing carbide burs (KG Sorensen) and aluminum oxide discs (Sof-Lex/3M).

Volunteers were then submitted to a washout period of two weeks to eliminate the residual effects of the treatment previously applied. Volunteers received new toothbrushes and toothpastes and the toothbrushing technique was reinforced. New trays were used to eliminate any possible residues left by the previously applied agent, eliminating the possibility of it interfering with the effects of the second agent used.

Four other dental fragments—sound and demineralized enamel and sound and demineralized dentin—were fixed in the same way as that used for experimental Phase 1. This time the volunteers used the treatment agent (placebo or bleaching agent) not received at experimental Phase 1 (experimental Phase 2) for another three weeks.

The fragments were again removed with appropriate pliers. During experimental Phases 1 and 2, volunteers received a weekly syringe of bleaching or placebo agent. Table 2 shows the experimental phases and periods of the study.

I) Microhardness Tests

An acrylic device allowed the fragments to be held, keeping the long axis of the indentor perpendicular to the dental surface. Three microhardness indentations were performed on the leveled surface of each enamel and dentin fragment with a microhardness tester (Future Tech-FM-1e, Tokyo, Japan, 140) and a Knoop indentator. A load of 25 gr was used for the enamel fragments and a load of 10 gr was used for the dentin for five seconds.

J) Statistical Analysis

For statistical analysis, the average of the three Knoop Hardness Numbers was taken. Before the Analysis of Variance, the *carry-over* effect was determined by the *t*-Student test, in each volunteer. The Analysis of Variance for Greco-Latin Squares 2x2 design was employed to compare the treatment agents, using a three-dimensional block composed of "different sequences," "periods" and "quality of the dental fragment" (Montgomery, 1991). The statistical analysis was made by Statgraphics plus software (Manugistics, Inc, Rockville, Maryland 20852, USA).

RESULTS

The *t*-Student test showed no presence of the carry-over effect for enamel (p-value=0.0269) or for dentin (p-value=0.0356). These results permitted a comparison between treatment agents and quality of the dental fragments without the *carry-over* effect at the 5% level of significance.

Table 3 shows the mean of the Knoop microhardness values for enamel and dentin according to the quality of the dental fragments, treatment agents and periods. Mean Knoop microhardness values for each group, period and quality of the dental fragments are illustrated in Figures 2 and 3.

The Analysis of Variance of the experimental design considering the Greco-Latin Squares 2x2 was employed. For enamel, there were significant differences between bleaching and placebo agents (p-

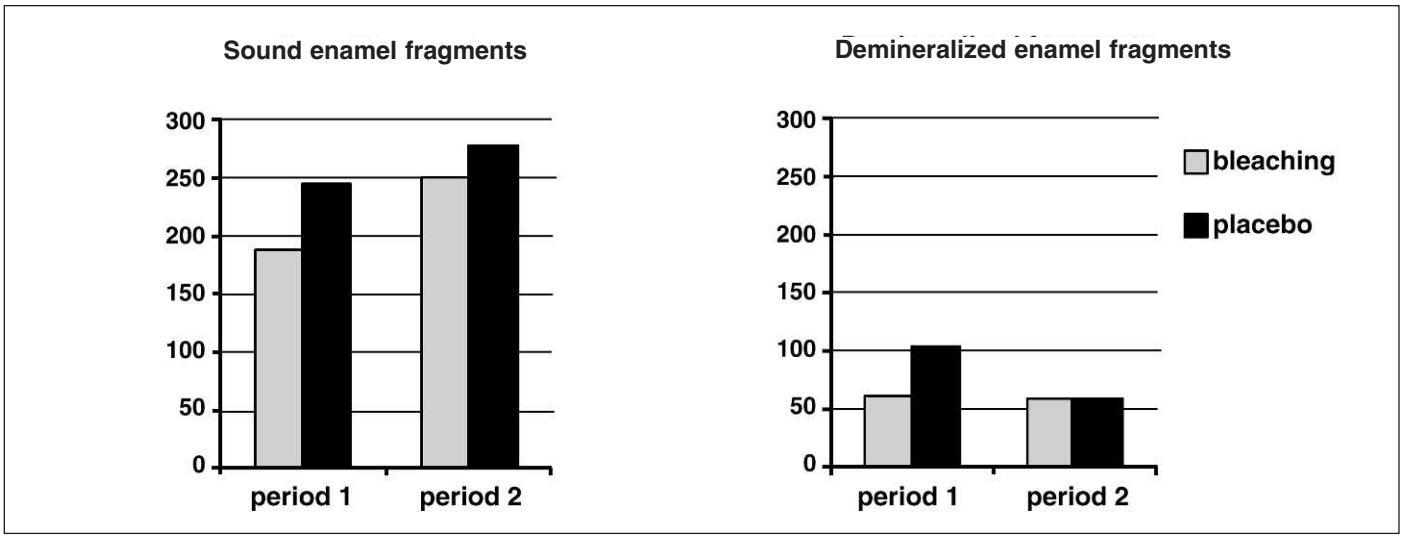

Figure 2. *Bar diagram of the mean Knoop microhardness illustrating the effects of the treatment agents, quality of the dental fragments and periods for the enamel fragments.*

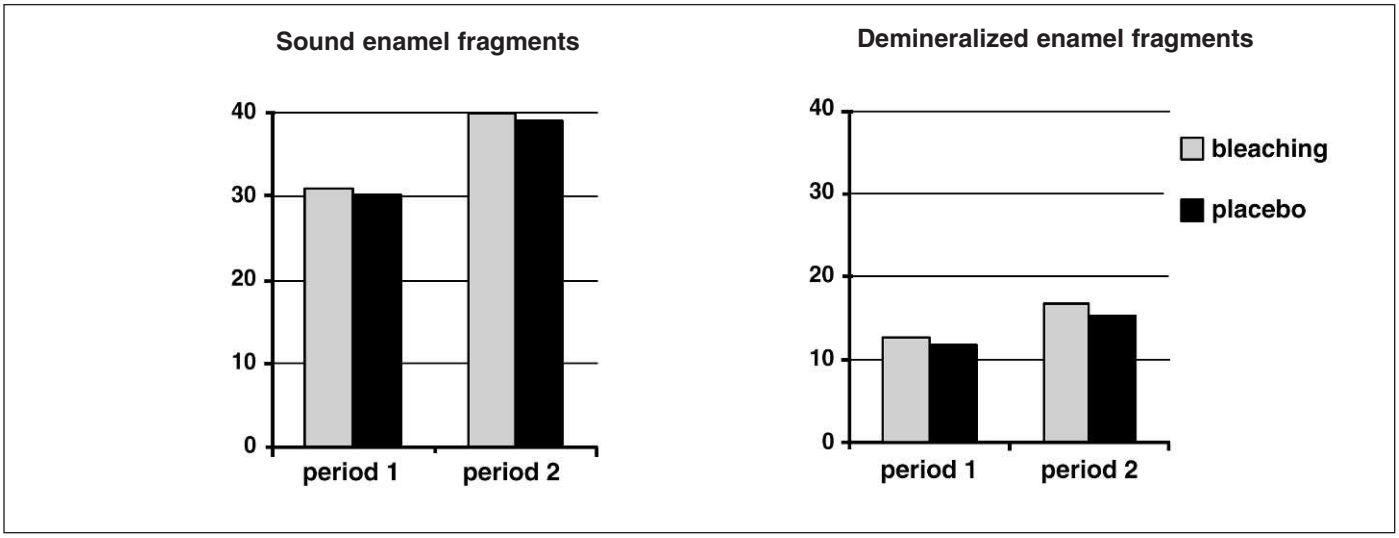

Figure 3. *Bars diagram of the mean Knoop microhardness illustrating the effects of the treatment agents, quality of the dental fragments and periods for the dentin fragments.*

value=0.0045) and between sound and demineralized dental fragments (p-value<0.0001). The sound and demineralized enamel submitted to 10% carbamide peroxide bleaching agent for three weeks showed significant lower values of microhardness than those submitted to a placebo agent. The Knoop microhardness values for sound enamel fragments were significantly higher than the Knoop microhardness values for demineralized enamel fragments in both treatments.

For dentin, there were significant differences between sound and demineralized dental fragments (p-value<0.0001). There were no significant differences between sound and demineralized dentin treated with bleaching or placebo agents, but the sound and demineralized dentin submitted to 10% carbamide peroxide bleaching agent for three weeks showed slightly higher values of microhardness than those submitted to a placebo agent. The Knoop microhardness values for sound dentin fragments were significantly higher than the Knoop microhardness values for demineralized dentin fragments for bleaching and placebo agents.

DISCUSSION

Since its introduction by Haywood & Heymann (1989), nightguard vital bleaching is a procedure that has dramatically grown in dental offices due to its efficiency and simplicity in removing intrinsic or extrinsic stains from teeth (Haywood, 1992; Haywood, 1994). Many bleaching products contain 10% carbamide peroxide (Goldstein & Kiremidjian-Schumacher, 1993;

Table 3: *Exploratory Estimates* for Enamel and Dentin Responses on Knoop Microhardness According to the Quality of Dental Fragments, Treatment Agents and Periods*

Quality of the Dental Fragments	Treatment Agents	Enamel		Dentin	
		Period 1	Period 2	Period 1	Period 2
Demineralized	OPA	59.6 (7.0)	57.4 (14.4)	12.7 (1.5)	16.7 (1.0)
	PLA	102.6 (14.7)	57.7 (13.9)	11.8 (1.2)	15.3 (0.9)
Sound	OPA	187.4 (21.6)	250.2 (13.8)	30.8 (4.2)	39.8 (2.7)
	PLA	244.2 (14.9)	275.5 (13.4)	29.8 (2.5)	38.8 (3.2)

* Mean (standard error) - m (se) - confidence interval at 95% is taken by m ± 1.96.se

Haywood, 1992) with carboxypolymethylene polymer as a thickening agent to improve tissue adherence and allow for a time or sustained release of the whitening agent (Haywood, 1994).

Although the bleaching agent is applied on the enamel surface, the oxidation processes of carbamide peroxide take place within the teeth by an interaction with their structural components. The effects and the mechanism of the bleaching agents should be evaluated to understand possible damages in detriment to the benefits of a more aesthetic smile offered by this technique.

In vitro studies using SEM analysis have demonstrated that applying 10% carbamide peroxide on enamel surface causes morphological changes with an increase in porosity and erosions (Ben-Amar & others, 1995; Bitter, 1998; Bitter & Sanders, 1993; Ernst & others, 1996; Flaitz & Hicks, 1996; Josey & others, 1996; McGuckin & others, 1992; Nam & others, 1999; Shannon & others 1993; Smidt & others, 1998; Zalkind & others, 1996). In dentin, an increase in the superficial roughness was verified (Zalkind & others, 1996). Although only one paper reported no alterations in enamel (Haywood & others, 1990), the acidic properties of bleaching agents, the prolonged contact time between the lightening product and dental surface, and the presence of greater amounts of carbopol have been claimed as possible factors that can cause these superficial changes. Clinically, increased porosity allows the bleaching agent to easily penetrate through enamel and dentin and could explain the transitory dental sensitivity during its use.

Regarding inorganic and organic components in dental structure, studies should take into account the structure's mechanical properties. Changes in the inorganic and organic components ratio could be deleterious to teeth (Featherstone & others, 1983; Featherstone & others, 1986). Free radicals of the decomposition of carbamide peroxide may react with organic components of the dental structure and the low pH of bleaching systems may cause demineralization. Loss of mineral can be related to the acidic properties of bleaching agents, even though Leonard & others (1994) observed an increase in the pH levels of 10% carbamide peroxide after its dissociation in the mouth. A loss of calcium after the exposure of enamel to 10% carbamide peroxide was observed in some *in vitro* studies (McCracken & Haywood, 1996; Rotstein & others, 1996), though this reduction of mineral content was not clinically significant.

Differences in organic and inorganic content can also be verified by microhardness tests (Featherstone & others, 1983). Although some *in vitro* studies have shown no microhardness changes on sound enamel and dentin submitted to the treatment with 10% carbamide peroxide (Murchison & others, 1992; Seghi & Denry, 1992; Nathoo & others, 1994; McCracken & Haywood, 1995), significant differences were found in other experiments (Pécora & others, 1994; Attin & others, 1997; Rodrigues & others, 2001; Shannon & others, 1993; Smidt & others, 1998).

A dynamic model of inducing artificial caries lesions through pH cycles of demineralization and remineralization solutions (Featherstone & others, 1986; Serra & Cury, 1992; Hara & others, 2000) was used in this study. This model presents a correlation with the initiation and progression of carious lesions in patients at high-risk for caries (Featherstone & others, 1986), leading to changes in the mineral and organic content of teeth. The applicability of the model for inducing caries can be verified by the significant differences between the quality of enamel and dentin fragments (p-value<0.0001; p-value<0.0001). It shows that the demineralization-remineralization model was effective in producing artificial caries lesions. The Knoop microhardness values for sound enamel fragments were significantly higher than those for demineralized enamel fragments in both treatments. The same occurred for sound and demineralized dentin fragments.

For enamel fragments, it was verified that sound and demineralized fragments submitted to effects of the bleaching agent presented lower microhardness values than sound and demineralized fragments submitted to the placebo agent. These results were obtained by fixing the dental fragments in a vestibular location—close to the parotid salivary gland duct exit where there is a low-risk for caries. Because the fragments were maintained in the mouth during all experimental phases and at the same location (with constant flow of saliva, temperature and pH changes, fluorides, toothbrush abrasiveness and effects of liquids and foods in

the oral environment), the authors consider the results reliable. The bleaching agent caused a mineral loss in human dental fragments, even though saliva, plaque control and fluorides were present in the oral environment. These factors could be responsible for maintaining the balance between the demineralization and remineralization phenomena. This mineral loss can not only be related to the pH level of the bleaching agent. Leonard & others (1994) showed that a 10% carbamide peroxide solution with a moderately low-pH presented an increase of its pH level after five minutes of degradation, reaching a neutral pH. However, the pH level of OPA used in this experiment was not as acidic, but an increase of OPA pH level can also be expected. Thus, prolonged contact between the product and dental structure can be responsible for the decrease in microhardness values. Due to a high level of mineral content of enamel, the bleaching agent seems to cause a demineralization effect in the enamel structure, even though a slight decrease in organic content in the enamel could take place (the percentage of organic content and water in enamel is around 4% in volume (ten Cate, 1988).

For dentin fragments, there was no significant difference between the bleaching and placebo agent. The percentage of mineral content in dentin (70%) is lower than in enamel (ten Cate, 1988). Therefore, the authors' results also suggest that, although some alterations occur in the dentin, these effects do not damage the inorganic/organic content in dentin structure, but significantly affect the mineral content of the enamel fragments.

This experiment can also elucidate the importance of not applying bleaching agents on early carious lesions due to their damaging effects. On sound dental structures, bleaching agents can be used as an aesthetic treatment, but one should be aware of the lower microhardness values obtained in this study. There is a possibility that human enamel could be damaged, although the effects of higher concentrations of fluorides and a post-bleaching time were not evaluated. Perhaps a prolonged time contact between the dental structure with saliva and fluorides could help reverse the ratio between organic and inorganic mineral content and to return to the initial conditions which could increase the enamel microhardness values. This emphasizes that at-home bleaching agents require future research, even though professional supervision can ensure correct selection of the proper case, correct application and steps to prevent adverse reactions.

CONCLUSIONS

The results suggest that:

a) treatment with 10% carbamide peroxide bleaching material for three weeks alters the microhardness of sound and demineralized enamel;

b) treatment with 10% carbamide peroxide bleaching material for three weeks does not alter the microhardness of sound and demineralized dentin.

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A Prospective Clinical Study of a Multipurpose Adhesive Used for the Cementation of Resin-Bonded Bridges

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Clinical Relevance

The clinical retention rate of anterior resin-bonded bridges cemented with Panavia 21 Opaque adhesive appears similar to that of a multipurpose adhesive; greyness of the abutments, however, was a major disadvantage in using the multipurpose adhesive.

SUMMARY

A clinical trial was conducted to assess comparatively the clinical performance of anterior resin-bonded bridges (RBBs) cemented with Panavia 21 Opaque (PO) or Scotchbond Multi-Purpose Plus (SBMP) used in conjunction with Scotchbond Resin Cement (SRC). Thirty-three bridges were cemented with PO and 31 bridges with SRC. The bridges were assessed six months after placement via a postal questionnaire. Two bridges (one in each group) failed at that stage. Clinical assessments at one year revealed that one PO bridge and five SRC bridges had failed. Chi-squared analysis, however, showed no significant difference (p>0.05) in the retention rate of the two groups. At the end of two years, eight bridges had failed (three PO and five SRC). Chi-squared analysis again revealed no significant difference in the retention rate provided by the two adhesives. Greyness of the abutment teeth of bridges cemented with SRC was a drawback in using this material. Many operators who participated in the trial felt the use of SRC/SBMP was time-consuming and rather complicated.

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INTRODUCTION

The advantages of replacing missing teeth with an RBB include conservation of tooth substance, lack of pulp irritation, minimal periodontal involvement, reversibility and reduced cost.

Since Howe & Denehy (1977) reported on the clinical use of bridges with perforated retainers (Rochette bridges), research has been directed towards improving the resin-to-metal bond. The research has attempted to overcome the disadvantages of the perforated retainer (Simonsen, Thompson & Barrak, 1983), so that with expanding the application and improving the performance of the restoration, the results have been the introduction of several mechanical mechanisms for bonding resins to metals (including electrolytic etching) and the development of conventional resin cements, especially those formulated for use with RBBs.

While many clinicians worked on their first electrolytically-etched (Maryland) bridges, resin cements with the ability to form strong bonds to sandblasted metal surfaces were being developed (Tanaka & others, 1981; Omura & others, 1984; Rheinburger & Beham, 1985).

Use of these resin cements, commonly referred to as adhesive resins, served to simplify laboratory procedures, reduce expense and eliminate disadvantages of the electrolytic etching technique (Hill, Zidan & Gomez-Martin, 1986). In the mid-1990s, SBMP adhesive was introduced by the 3M Dental Products, St Paul, MN 55144, USA. SBMP adhesive claimed to be useful for a variety of adhesive procedures, including bonding of direct resin composite restorations and indirect procedures involving cementation of metal or porcelain restorations.

Because of the versatility of such a system, a laboratory study (Aboush, 1995) comparatively assessed the tensile strength of metal-resin-enamel joints formed with adhesives for resin-bonded bridge applications. This work showed that joints formed with the use of SBMP were as strong as those formed with C & B Metabond (Parkell, Farmingdale, NY 22735, USA) but weaker than those formed with Panavia EX (Kuraray, Osaka, Japan). Although laboratory testing of a material may give an indication of its performance, it cannot accurately indicate how the material will perform in everyday clinical practice. Therefore, this research was designed as a single blind clinical trial to assess the clinical performance of SBMP/SBC when used for cementing RBBs. The control adhesive was PO (Kuraray).

METHODS AND MATERIALS

The trial was conducted at Bristol Dental Hospital, University of Bristol. The protocol was approved by the Healthcare Authorities.

Patients, aged 15 years or older without contraindications in their medical history, were included. Those exhibiting parafunctional habits were excluded. Patient selection was limited to individuals with only one or two missing anterior teeth in the maxillary arch, with sound or minimally restored abutment teeth. Before starting treatment, the procedure was explained to the patients and written details provided. They were not given information on which cement would be used for placing their prosthesis.

Materials

The metal framework for all the bridges was cast from a beryllium-free nickel-chromium alloy (Wiron 88, Bego, Germany). The aesthetic pontics were made of dental porcelain. SBMP resin cements used in conjunction with SRC (3M Dental Products) and PO (Kuraray).

Clinical Procedures

Staff and students in the last year of their dental studies placed the bridges. The students were closely supervised by academic staff, and the author provided guidelines for the cementation procedures. In the majority of cases, tooth preparation was minimal, involving only removing bulbous areas of enamel to create a non-undercut path of insertion. In a few cases, occlusal rests or proximal grooves were also prepared. Bridges were designed to have maximal coverage of the palatal surfaces. The metal periphery of the retainer extended to within 1 mm of the free gingival margin.

After adding porcelain to the pontic(s), the prosthesis was tried in the mouth to check marginal adaptation of the retainer wings. In addition, the shape and shade of the pontics were assessed and the occlusion checked. Occlusal interferences were adjusted and the path of insertion of the restoration carefully checked. The bonding procedure was performed only after all adjustments had been made.

Before cementation, the bridges were returned to the laboratory for glazing of the porcelain pontics and final sandblasting and cleaning of the fitting surfaces of the retainers. Sandblasting took place with 50 µm aluminum oxide at 2 bar pressure. The bonding surfaces were sandblasted until a matt finish was achieved. The bridge was then steam cleaned and placed in a plastic container.

The laboratory stages of bridge fabrication were undertaken by qualified technicians at the laboratory of the Bristol Dental Hospital. The clinical stages of bridge cementation using either of the two cements were those recommended by the manufacturers.

Once the cementation procedure was completed, excess cement was removed with flame-shaped burs in an air-turbine handpiece used with water coolant.

Clinical data were recorded on a Case Report Form. This included level of operator, presence of overbite or overjet, condition of occlusal wear (normal, slight, severe), teeth replaced, abutment teeth and design of the bridge (fixed-fixed, cantilever).

Data related to the abutment teeth were recorded before the cementation stage. These included the preparation (underprepared, overprepared and satisfactory). Satisfactory preparation of the abutment tooth meant that, for an anterior tooth, axial walls, a cingulum pit and/or fine finishing line were prepared. For a premolar, a rest seat plus wrap-around were provided. Other recorded data included the presence of restorations and exposed dentin, mobility, percentage of enamel covered and fit rating of the bridge before cementation (poor, acceptable, excellent).

Sixty-four resin bonded bridges were placed in 57 patients. They were evaluated after six, 12 and 24

Table 1: *Distribution of the Resin-Bonded Bridges According to the Type of Bridge*

		PO	SRC	Total
Cantilever	1A, 1P	18	19	37
Cantilever	2A, 1P	2	2	4
Cantilever	2A, 2P	2	0	2
Fixed/fixed	2A, 1P	8	8	16
Fixed/fixed	2A, 2P	3	2	5
Total		33	31	64

A = Abutment, P = Pontic

Table 2: *Factors Evaluated Immediately After Bridge Cementation*

	PO	SRC
Underprepared abutments	34	27
Satisfactorily prepared abutments	12	14
Overprepared abutments	2	2
Use of rubber dam	10	10
Non-use of rubber dam	23	21
Dentine exposure	1	0
Restoration present	8	9
Contacts in centric occlusion A	25	24
Contacts in excursions A	23	24
Contacts in centric occlusion P	8	5
Contacts in excursions P	11	7
Greyness of abutments*	1	19

A = Abutment, P = Pontic
* Statistical analysis $c^2 = 21.97$, DF = 1, $p < 0.05$

Table 3: *Number of Successes and Failures of the Resin-bonded Bridges Placed Using the Two Cements One-Year After Placement*

	Lost to Review	Successes	Failures	Total
PO	3	29	1	33
SRC	2	24	5	31
Total	5	53	6	64

Statistical analysis (with regard to number of failures): $c^2 = 2.67$, DF = 1, $p>0.05$.

Table 4: *Number of Successes and Failures of the Resin-bonded Bridges Placed Using the Two Cements Two-Years After Placement*

	Lost to review	Successes	Failures	Total
PO	4	26	3	33
SRC	3	23	5	31
Total	7	49	8	64

Statistical analysis (with regard to number of failures): $c^2 = 0.50$, DF = 1, $p>0.05$

months. Thirty-three bridges were cemented with PO and 31 with SRC. Patients ranged in age from 15 to 73 years, with a mean of 35.2 years.

Thirty-two bridges were placed by University academic members, 30 by undergraduate students and two by postgraduate students.

After bridge cementation, further parameters were evaluated, including the level of patient satisfaction, presence of gingivitis adjacent to the bridge, periodontal index (CPITN), presence of gaps at margins of retainers, whether the cement line was visible, and greyness of the abutments.

Six-Month Assessment

Six months after placement, patients were mailed a questionnaire. Those failing to respond were sent a letter and an attempt was made to contact them by telephone. The questionnaires inquired whether the bridge was still in place, was loose, and if it had caused any pain or discomfort.

Clinical Examination After One and Two Years

During the one- and two-year recalls, the following information was recorded:

- evidence of debonding.
- presence of caries in abutment teeth.
- presence of gingival inflammation around the abutment teeth and the CPITN score.
- mobility of abutment teeth.
- presence of greyness, gaps around the margins of the metal wings and visible cement line.
- fracture of pontic or metal framework.

RESULTS

Data Collected From the Registration Form

Table 1 shows the number of bridges placed according to the design and resin luting material. Twenty-nine bridges cemented with SRC had one pontic and two bridges had two pontics. Twenty-eight bridges placed with PO had one pontic and five bridges had two pontics. One bridge cemented with PO was used to correct the position of the midline diastema and not to replace a tooth. The most common teeth replaced in the study were the upper right central incisor and the upper right lateral incisor.

Thirty-four of the abutments cemented with PO and 27 with SRC were underprepared. Satisfactory preparation was recorded for 12 abutments cemented with PO and 14 cemented with SRC. Only two abutments in each group were overprepared. A rubber dam was used in only 10 cases using each of the two materials. Dentin was exposed in only one patient during abutment

Table 5: *Clinical Data Recorded for Failed Bridges*

	PO	SRC	SRC	SRC	SRC	SRC	PO	PO
Time of failure (months)	3	5	6	8	8	9	14	23
Bridge type	Cant	Cant	Cant	F/F	Cant	F/F	Cant	Cant
Number of pontics	1	1	1	1	1	2	1	1
Number of abutments	1	2	1	2	1	2	1	2
Operator grade	U/g	U/g	U/g	U/g	U/g	Ac staff	Ac staff	Ac staff
Preparation	U	S	U	S	U	S	S	S
Restoration present	No	No	No	Yes	Yes	No	No	No
Mobility	0	0	0	0	0	0	0	0
CPITN	0	0	0	0	0	0	0	0
Use of rubber dam	Yes	No	No	No	No	No	Yes	Yes

Ac Staff = Academic staff
U/g = Undergraduate student

preparation. This bridge was placed using PO. Palatal composite resin restorations were present in eight of the abutments cemented with PO and nine abutments cemented with SRC.

Immediately after the restoration was cemented, greyness was noticed in only one case cemented with PO, but in 19 bridges cemented with SRC. These results are shown in Table 2. Chi-squared analysis of the results concerning the greyness of the abutments indicated a significant difference between the two materials ($p<0.05$).

Six Month Assessments

After six months, a questionnaire was sent to the patients; this allowed evaluation of 61 bridges. Successful contact by postal questionnaire was 98%. Two bridges were reported by the patients as having fallen out. One bridge cemented with PO fell out three months after placement and the other, placed using SRC, lost retention after five months.

In the questionnaire, three patients mentioned dissatisfaction concerning the discoloration of the abutment teeth. SRC was the cement used in these three cases.

One-Year Clinical Assessments

At the one-year clinical assessments, 30 bridges cemented with PO (a recall rate of 91%) and 29 bridges cemented with SRC (a recall rate of 94%) were assessed; three bridges cemented with PO and two bridges cemented with SRC were lost to clinical review.

Six bridges (one PO and five SRC) failed during the one-year evaluation (Table 3). Therefore, the failure rate was 9%. The bridge cemented with PO debonded after three months. The five bridges cemented with SRC debonded after five, six, eight, eight and nine months, respectively.

No significant difference ($c^2=2.67$, DF = 1, $p>0.05$) could be shown in bridge retention between the two resin luting materials at one year. During the clinical examination, no caries lesions were noted on the abutment teeth and no failures of porcelain pontics or cast metal retainers were recorded. The gingival status, mobility of abutment teeth and marginal gaps were unchanged one year following bridge placement. Eleven patients fitted with SRC bridges had some greyness of the abutment teeth; two were dissatisfied with their bridges because of this.

Two-Year Clinical Assessments

At two years, 29 bridges cemented with PO and 28 bridges cemented with SRC were assessed; four bridges cemented with PO and three bridges cemented with SRC were lost to clinical review.

Eight bridges (three PO and five SRC) failed during the two-year evaluation (Table 4). The failure rate was 12.5%. The PO bridges failed after three, 14 and 23 months, respectively, and the SRC bridges failed after five, six, eight, eight and nine months, respectively. In other words, all the SRC failures occurred during the first year after the placement. Table 5 shows the clinical data concerning the failed bridges.

Chi-squared analysis showed no significant difference ($c^2=0.50$, DF=1, $p>0.05$) in the retention rates provided by the two luting cements (Table 4).

One patient with a PO bridge presented with a loose retainer at the two-year recall. The bridge replaced the left upper lateral incisor and used the two upper central incisors as abutments. The wing on the right upper central incisor became loose and the bridge had to be removed and recemented. The bridge was considered one of the failures.

No carious lesions were diagnosed on any of the abutment teeth. None of the bridges caused mobility of an

abutment tooth nor did they have an adverse effect on the surrounding gingival tissues. Only one porcelain pontic presented with a small fracture at the two-year recall. It had to be adjusted and smoothed.

Twelve 28 SRC bridges showed greyness of the abutment teeth. None of the PO bridges presented with such a drawback. Three SRC patients expressed dissatisfaction with the aesthetics of their bridges because of this.

DISCUSSION

The study's clinicians were informed of its aims and advised on the proper use of the luting cements. However, they were not advised to apply a particular design or follow certain tooth preparation procedures. It was thought that most bridges would be the fixed-fixed type, but as it turned out, the vast majority were of the cantilever type. This was due to recent articles that highlighted the acceptable performance of cantilevered bridges and their advantages (Hussey & Linden, 1996).

Hussey, Pagni & Linden (1991) reported that cantilevered resin-bonded bridges appear to perform as well as those with a fixed-fixed design and it was suggested that the former design might be more resistant to debonding as a result of occlusal forces.

Many, but not all bridges in this study were done with no or little preparation of abutment teeth. It has been suggested, however, that preparation of abutments provides good resistance to displacement and more favorable bonding thickness for the cement, which improves its rigidity (Sadd & others, 1995; de Kanter & others, 1998).

The Panavia resin cement has been in clinical use in the UK since 1985, and since then, has been the only resin cement used in the Bristol Dental School for the cementation of resin-bonded bridges. However, staff members offered no resistance to trying a new product. After using the SRC for the first time, almost all operators were disappointed and declared that they would not consider using this material again for cementing resin-bonded bridges. This was mainly due to the multiple steps required in using this material and the change in the translucency (greyness) of the abutment teeth.

After noticing the greyness effect in the first few cases, the manufacturing company (3M) was asked whether it could provide an opaque version of the cement. However, it appeared that this was not available.

The failure rate encountered in this study was in line with that of similar studies. Most failures encountered (especially with SRC) occurred during the first few months of function. This was expected, as several authors reported resin-bonded bridges being at greatest risk for debonding in the first few months of function (Williams & others, 1989; Hussey & others, 1991; Dune & Millar, 1993). It has also been suggested that shrinkage stress during the setting of resin cement might be responsible for the occurrence of early failures (Hoppenbrouwers, Verzdijden & Creugers, 1995). Other factors that can contribute to early debonding include inadequate mechanical retention, poor retainer design and/or bonding to contaminated metal or tooth surface (Creugers & van't Hof, 1991; Barrack, 1993; Boening, 1996).

No incidence of caries of abutment teeth was noticed in this study. Also, the gingival response was favorable. These findings are in line with those of Thayer & others (1993). Romberg & others (1995) and Wood & others (1996) also stated that the overall long-term periodontal response to resin-bonded bridges was favorable, despite gingival recession and higher plaque rating.

CONCLUSIONS

A clinical trial was undertaken to compare the clinical performance of anterior resin-bonded bridges cemented with SRC/SBMP or PO. The restorations were assessed at six months, and one and two years post-operatively. At the end of two years, five SRC/SBMP bridges and three PO bridges had failed. Chi squared analysis revealed no significant difference in the retention rate provided by the two cements.

Among the bridges cemented with SRC/SBMP, 42% showed greyness of abutment teeth. Many clinical operators felt using SRC/SBMP was time-consuming and rather complicated.

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Treatment of Dentin with Polyacrylic Acid—A Retrospective Observational Study of the Effect Upon the Durability of Glass Ionomer Restorations

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Clinical Relevance

Although pre-treatment of cavities restored with conventional glass ionomer cements with polyacrylic acid (Tooth Cleanser) offers no statistical improvement to the long-term durability, it improves the chances of obtaining a dependable restoration.

SUMMARY

This paper reports on the results of a material specific, retrospective observational study. It sought to determine the consequence of pre-treatment of cavity margins with the conventional glass polyalkenoate Chemfil II prior to restoration with Tooth Cleanser on restoration durability. All restorations were placed and varnished by senior dental students under the supervision of a staff member. Manufacturer recommendations were followed. Data on each restoration was collected and entered into a relational computer database. Data included details of cavity morphology and the use or non-use of Tooth Cleanser. Six years following the study start a manual search of all records was carried out to determine, for those patients continuing to attend the Dental Hospital, the number of failed and surviving restorations. This yielded a dataset of 149 restorations, of which 41 had been placed with the aid of Tooth Cleanser and 108 without. The number of failed restorations was 20 and 62, respectively. Survival analysis by the Kaplan-Meier method revealed median survival times of 2,094 days, when Tooth Cleanser was used and 1,748 days when not. Although 80% survival times of 1111.0 (SE=6.3) (With Tooth Cleanser) and 285.0 (SE=3.8) (Without Tooth Cleanser) days were

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observed, together with a Hazard Ratio of 1.49 (95% Confidence Intervals 0.92 & 2.31), a Logrank test revealed no statistically significant difference between the survival curves (p=0.12). It was concluded that although there was a trend for the application of Tooth Cleanser to improve the chances of obtaining a dependable restoration, this effect was not statistically significant.

INTRODUCTION

With the advent of clinical governance in the United Kingdom, currently, there is much interest in the efficacy and durability of healthcare treatments. In dentistry, the durability of simple plastic restorations is an area that has received much attention. Indeed, a recent systematic review (Downer & others, 1999) revealed 124 studies, the majority of which focused upon dental amalgam. The papers cited were mostly retrospective in nature but assessed the durability of restorations across the whole spectrum of operating environments. For both dental amalgam and resin composite, the majority of studies reported mean survival times (MSTs—the lifetime which any restoration has a 50% chance of exceeding) in the range of six to 10 years. A paucity of information on the durability of glass polyalkenoate restorations was highlighted. This was because the majority of studies involved an observation period of less than five years.

Since their development glass (ionomer) polyalkenoate restorations have found widespread application in the restoration of surfaces not subjected to occlusal force. It has been demonstrated previously that removal of the dentinal smear layer prior to placing a glass polyalkenoate restoration improves both the shear bond strength of the material to dentin (Powis & others, 1982; Hewlett, Caputo & Wrobel, 1991) and the clinical durability of such restorations (Ngo, Earl & Mount, 1986). Polyacrylic acid is effective in removing the smear layer (Hewlett & others, 1991) and is commercially available in products such as Chemfil Tooth Cleanser (Dentsply Ltd, Weybridge, Surrey, UK) that contains 25% polyacrylic acid and is marketed as a cavity conditioner for glass polyalkenoate restorative materials. The manufacturer's claim that this agent cleanses the cavity margins to facilitate bonding and promotes micromechanical retention by opening up dentinal tubules as a result of smear layer removal.

This paper reports on the results of a material specific retrospective observational study that sought to determine the consequences upon restoration durability of pre-treatment of the cavity margins prior to restoration with the conventional glass polyalkenoate Chemfil II (Dentsply Ltd, Weybridge, Surrey, UK), with Tooth Cleanser.

METHODS AND MATERIALS

This study was conducted in the Integrated Treatment Unit of Dundee Dental Hospital and School (Scotland). All restorations were placed by senior dental students under the supervision of a staff member. For each Chemfil II (Dentsply Ltd, Weybridge, Surrey, UK) restoration placed between January 1992 and February 1998, students completed a data collection form in consultation with a staff member. This recorded the patient's hospital identification number, cavity site, cavity morphology and the use of both lining material and any application of Tooth Cleanser (Dentsply Ltd, Weybridge, Surrey, UK) to the cavity prior to restoration. All completed restorations were varnished following placement, as recommended by the manufacturer, to protect them from moisture contamination for the first 24 hours. The use of lining material or application of Tooth Cleanser was left to the clinical judgement of the supervising staff member. No restorations were placed under rubber dam.

All responses were coded and entered into a relational computer database (Paradox 3.5, Borland International, CA 95067-0001, USA) that had been customized by the investigators. To conform to the Data Protection Act (1984), all computer records associated with this study were registered with the Data Protection Registrar through the University of Dundee and the Tayside Health Board.

In October 1998, hospital records of all patients included in the relational database were retrieved and, for each study restoration, manually checked for accuracy and any patient follow-up. Where patients continued to attend for routine check ups, the records were scrutinized closely for evidence of restoration failure or replacement. If these had occurred, the date when this was first noted was taken to be the date of failure and it was entered into the database. Thereafter, the database was interrogated to reveal the:

- number of restorations of Chemfil II placed.
- anatomical site of each restoration.
- proportion of Chemfil II restorations placed following the treatment of the prepared cavity with Tooth Cleanser.
- number of restorations that failed during the follow-up period.

In addition, the failure times for Chemfil II restorations placed with and without the application of Tooth Cleanser were calculated. Survival curves were generated by the Kaplan-Meier method and Prism (Version 3.0, GraphPad Software, Inc, 5775 Oberlin Drive #110,

Table 1: *The Median Survival Time (Days), 80% and 60% Survival Times (Days) for Restorations of Chemfil II Placed With and Without Tooth Cleanser*

Parameter	Chemfil II With Tooth Cleanser	Chemfil II Without Tooth Cleanser
Median Survival Time	2,094	1,748
80% Survival Time (SE)	1,111.0 (6.3)	285.0 (3.8)
60% Survival Time (SE)	1,818.0 (8.2)	1,020.0 (4.8)

San Diego, CA 92121, USA). Only data drawn from followed-up restorations was used. Restorations that had survived were treated as censored data, whereas failures were classified as uncensored.

RESULTS

Data for a total of 149 followed-up Chemfil II restorations were available for scrutiny. Of these, 41 had been placed with the aid of Tooth Cleanser, and during the period of study, 20 had failed. Of the 108 restorations placed without Tooth Cleanser, 62 restorations had failed.

Table 1 summarizes the median survival time for each material combination, together with the predicted 80% and 60% survival times.

Figure 1 shows the survival curves for each method of restoration. A comparison of the two survival curves by the Log Rank test revealed no statistically significant difference between them (p=0.12) and a hazard ratio of 1.49, with 95% confidence intervals of 0.91 and 2.31.

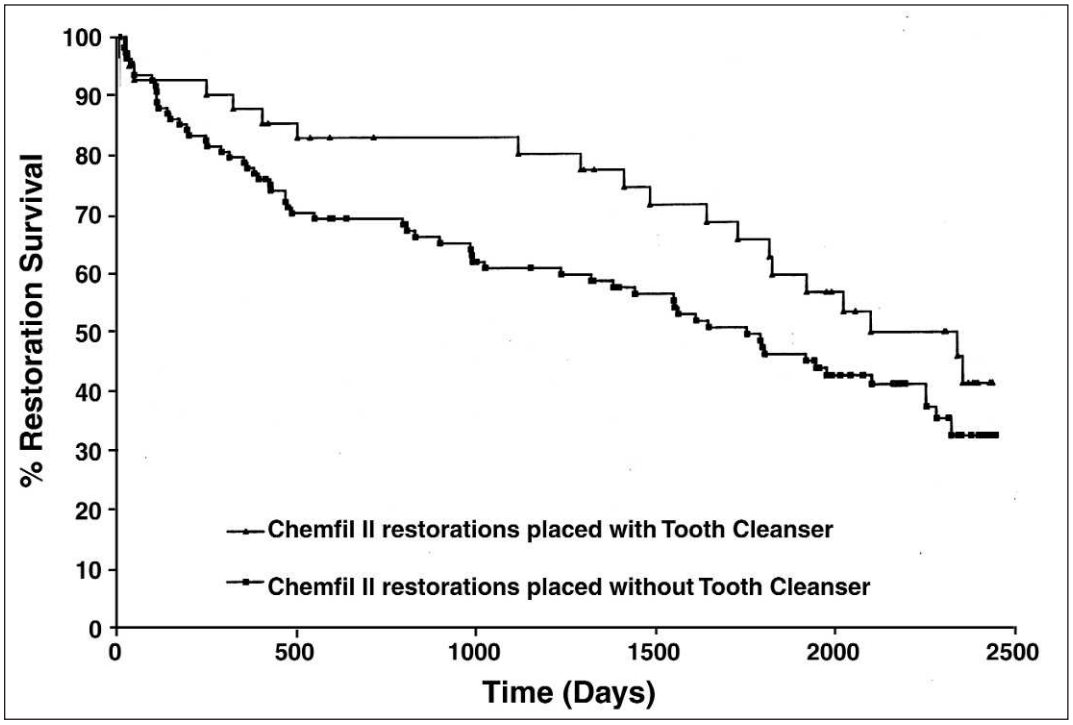

Figure 1. *A graph of the percent of restoration survival versus time to failure (days) for restorations of Chemfil II placed with and without the application of Tooth Cleanser.*

DISCUSSION

This study is one of a few that has examined the longevity of glass polyalkenoate restorations over a period greater than five years. It only utilized survival data from patients who had continued to attend the Dental Hospital for routine examination over the follow-up period. As a consequence, the assessment of durability was based on clinical examinations—important criteria concerning the validity and quality of such a study (Downer & others, 1999). In order to make survival predictions concerning the longevity of the restorations, the data was subdivided according to whether or not Tooth Cleanser was applied to the cavity prior to restoration placement but not cavity design. Such a mixture model has previously been applied successfully to assess the survival characteristics of named dental restorative materials (Smales, Webster & Leppard, 1991). All restorations were placed in non-stress bearing situations.

The median survival times observed (Table 1) of 1,748 (without Tooth Cleanser) and 2,094 days (with Tooth Cleanser) are comparable to, if not slightly better than the four years reported by Mjör, Dahl & Moorhead (2000) for conventional glass ionomer restorations placed in permanent teeth in general dental practice. This difference may perhaps be attributed to the different operating environments in which each study was conducted (Elderton, 1983).

Although the application of polyacrylic acid to dentin prior to placement of the restoration has previously been shown to improve the clinical retention rate of glass ionomer restorations (Ngo & others, 1986) over a one year period, the longer term effects of this procedure have not been reported. This work followed up restorations for a six-year period and demonstrated no statistically significant effect of this procedure on restoration survival rate.

Paradoxically, however, greater chances of restoration survival were observed if Tooth Cleanser were used (Table 1) and, though not statistically significant, this may encourage some to routinely use this agent to maximize the chances of success of glass polyalkenoate restorations.

CONCLUSIONS

1. The pre-treatment of cavities restored with Chemfil II with tooth cleanser offered no statistical improvement to the durability of such restorations compared to those where none was applied.
2. There was a trend, although not statistically significant, that pre-treatment of cavities to be restored with Chemfil II improved the chances of retaining the restoration.

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Laboratory Research

Influence of Time and Thermocycling on Marginal Sealing of Several Dentin Adhesive Systems

C Lucena-Martín • MP González-Rodríguez • CM Ferrer-Luque
V Robles-Gijón • JM Navajas

Clinical Relevance

The levels of leakage in enamel were minimal regardless of the experimental conditions applied or the adhesive used. Optibond Solo showed the best outcomes in dentin/cementum under the three experimental conditions.

SUMMARY

This study evaluated the in vitro **microleakage of six dentin adhesive systems. Triangle-shaped Class V cavities with coronal margin in enamel and gingival margin in cementum or root dentin were cut in the buccal surfaces of 90 non-carious single-root human teeth. These teeth were randomly assigned into six groups (n=15) for the evaluation of six different dentin adhesive systems: One Step, Prime & Bond 2.0, Syntac Single, Single Bond, Optibond Solo and Syntac Sprint. The preparations were restored with Degufill Ultra composite and polished using the Enhance system. Each group was randomly divided into three subgroups (n=5): samples of the first subgroup were immersed in 2% methylene blue solution for seven days; those of the second subgroup remained in a similar solution for 31 days; those of the third subgroup were thermocycled 500x at 5-55°C and immersed in 2% methylene blue for seven days. All 90 teeth were then embedded in methacrylate and bucco-lingually sectioned; the dye penetration was evaluated using an 0-4 ordinal scale. All of the dentin adhesive groups showed minimal leakage at the enamel margins with increased leakage at the gingival margins. Optibond Solo showed the best outcomes among the dentin adhesives tested.**

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INTRODUCTION

Dentin adhesives have continuously evolved over the past 30 years. Since replacement of selective enamel etch by combined etching of enamel and dentin, most dentin adhesives on the market have required three basic steps: demineralization, priming and application of adhesive resin: this procedure results in the formation of the zone described by Nakabayashi as the interdiffusion or hybrid layer zone (Nakabayashi, 1985).

In the last few years, research in this field has focused on developing simpler systems that are less sensitive to the application technique. Bonding systems that combine the primer and resin in one bottle have been mar-

Table 1: *Application Method of the Adhesives Used in the Study*

Material	Application Method			
	Etching	Time	Nº layers	Photo-curing
One Step	H_3PO_4 at 32%	15 seconds	5 (2+2+1)	10 seconds
Prime & Bond 2.0	H_3PO_4 at 37%	20 seconds	2 (1+1)	20 seconds (10+10)
Syntac Single	H_3PO_4 at 37%	30 seconds	2 (1+1)	20 seconds (10+10)
Single Bond	H_3PO_4 at 35%	15 seconds	2 (2)	10 seconds
Optibond Solo	H_3PO_4 at 37.5%	15 seconds	1 (brush-stroke)	20 seconds
Syntac Sprint	H_3PO_4 at 37%	15 seconds	1 (brush-stroke)	No

H_3PO_4: Orthophosphoric acid. Nº: number of layers applied.

keted, thus reducing the clinical procedure from three steps to two. Additionally, autoetch primers that combine the demineralization and priming steps have been developed to simultaneously treat the enamel and dentin. Acceptance of these new products requires their prior study in controlled clinical trials, but by the time the results of these trials are published, new products have already come on the market. For this reason, *in vitro* studies are of immense value. While bond strength is the most widely studied parameter, microleakage testing is a useful and simple method to evaluate a property of the material that is fundamental to its clinical success: the failure of an adhesive to provide an adequate marginal seal to the restoration are strictly connected to the possibility of penetration of bacteria or oral fluids in the dentin, which consequently leads to the development of postoperative sensitivity, marginal discoloration and secondary caries (Cox, 1987).

Long-term durability of the bonding between the composite and dentin is essential to ensure the longevity of the restorations. Previous *in vitro* studies (Chan, Reinhardt & Boyer, 1985; Burrow, Satoh & Tagami, 1996; Lucena & others, 1999) demonstrated a major reduction in bond strength after long periods of storage in water, which may be related to the slow absorption of water and/or the hydrolysis of the adhesive resins (Burrow & others, 1996; Gwinnett & Yu, 1995).

This study aimed:

- to evaluate the sealing ability of Class V restorations bonded with six different bonding systems.
- to study the influence of thermocycling and time on marginal leakage.

METHODS AND MATERIALS

Ninety caries-free single-root human teeth extracted for different reasons were cleaned and preserved in an aqueous solution of 5% chlorhexidine until their study. A #59 (Maillefer, Swiss) diamond bur was used at high speed with abundant water coolant to make triangle-shape Class V cavities of approximately the same dimensions (4 mm wide x 4 mm deep x 5 mm high) in the buccal surface of all the teeth. The coronal margin was located on enamel and the gingival margin on cementum or root dentin. The bur was replaced after every five preparations. The teeth were randomly assigned to one of six experimental groups (n=15), and a different dentin bonding system was used in each group: One Step (BISCO, Schaumburg, IL 60193 USA), Prime & Bond 2.0 (Dentsply/DeTrey, Konstanz, Germany), Syntac Single (Vivadent, Schaan, Liechtenstein), Single Bond (3M Dental Products, St Paul, MN 55144, USA), Optibond Solo (Kerr, Orange, CA 92867, USA) and Syntac Sprint (Vivadent, Schaan, Liechtenstein). All bonding systems were applied according to the manufacturer's instructions (Table 1).

The composite used to restore the preparations was Degufill Ultra (Degussa AG, Hanau, Germany), applied in two increments. Each increment was light cured for 40 seconds. The restorations were then carefully polished using Enhance system disks (Dentsply/DeTrey, Konstanz, Germany).

The teeth were preserved in water at 37°C for two days. To prevent apical leakage, retentive cavities were made in the root apex and sealed with zinc oxide-eugenol cement. The dental surface was coated with two layers of nail varnish except for 1 mm around the restoration margin. The groups were then randomly divided into three subgroups of five teeth each for the following different treatments:

Group 1: The teeth were immersed in an aqueous solution of 2% methylene blue at 37°C for one week.

Group 2: The teeth were immersed in an aqueous solution of 2% methylene blue at 37°C for 31 days.

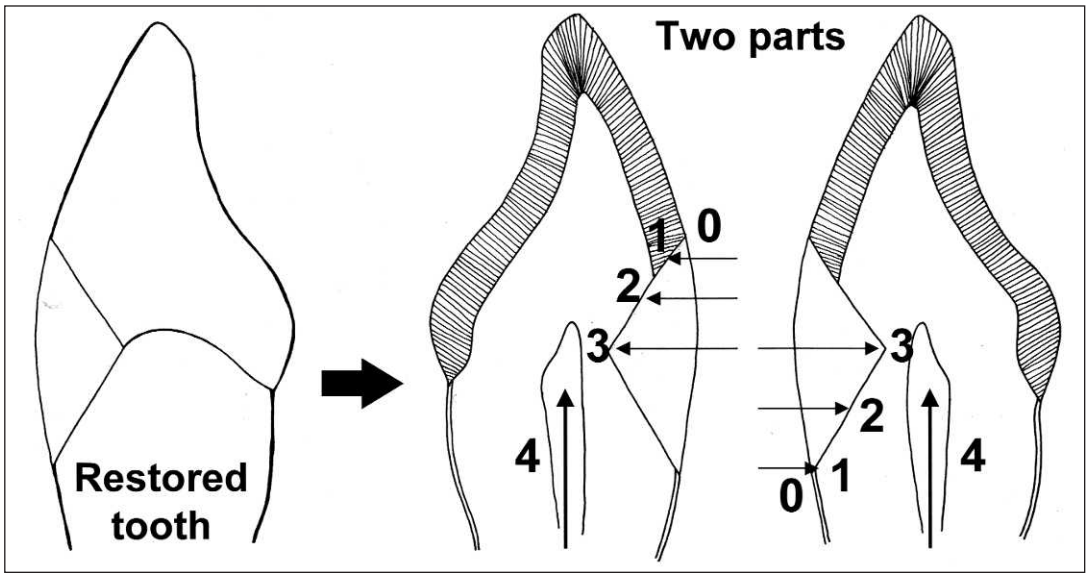

Figure 1. *Numerical scoring scale for microleakage.*

Group 3: The teeth were thermocycled 500 cycles in water baths at 5°C and 55°C, for 30 seconds at each temperature, then immersed in an aqueous solution of 2% methylene blue at 37°C for one week.

After being removed from the solution and drying, the teeth were embedded in methacrylate blocks and a longitudinal bucco-lingual cut was made through the center of the restoration using a hard tissue microtome (Accutom-50, Struers, Copenhagen, Denmark). Thus, two halves of each specimen were obtained, and microleakage was assessed in each half, both at the coronal and the gingival margins (Figure 1), using a 10x optic microscope (Olympus SZ-PT, Tokyo, Japan). The teeth were assessed according to the following scale:

Grade 0: No leakage.

Grade 1: The dye only penetrates the enamel (at the coronal margin) or the cementum (at the gingival margin).

Grade 2:The dye penetrates the dentin.

Grade 3:The dye penetrates to the depth of the cavity.

Grade 4: The dye penetrates through the apex.

The highest score of the halves of each specimen was taken as the leakage score. The leakage in enamel and dentin of the different dentin adhesives was compared using the Wilcoxon test for paired samples. The comparisons by groups used the Kruskall-Wallis test and comparisons by pairs of groups was done with the Mann Whitney's U test.

RESULTS

The scores for each adhesive are listed in Table 2. There was no microleakage at the coronal margin (enamel) for most of the adhesives, regardless of the duration or conditions of dye immersion. Microleakage at the gingival margin (cementum) varied widely between the adhesives: there was no marginal leakage in specimens in which Optibond Solo was used, except for four samples with Grade 1 leakage (two in the seven-days group and two in the 31-day group), while the greatest leakage occurred with Syntac Sprint, particularly in specimens submitted to thermocycling plus seven days of dye immersion.

The results, according to the time categories, are listed in Tables 3 and 4:

Table 2: *Scores Obtained by the Bonding Agents*

	Leakage 7 Days		Leakage 31 Days		Leakage 7 Days Thermocycling	
	Enamel	Cementum	Enamel	Cementum	Enamel	Cementum
	0 1 2 3 4	0 1 2 3 4	0 1 2 3 4	0 1 2 3 4	0 1 2 3 4	0 1 2 3 4
Adhesive						
One Step	5 0 0 0 0	3 1 0 1 0	3 1 1 0 0	2 2 0 1 0	4 0 0 0 1	1 0 1 1 2
Prime & Bond 2.0	4 1 0 0 0	3 1 0 1 0	2 1 0 1 1	2 0 2 0 1	4 0 0 0 1	0 0 0 4 1
Syntac Single	5 0 0 0 0	2 1 2 0 0	4 0 0 0 1	1 0 2 1 1	3 0 0 0 2	0 0 0 3 2
Single Bond	5 0 0 0 0	2 2 0 1 0	4 0 0 0 1	0 0 1 3 1	5 0 0 0 0	0 1 2 2 0
Optibond Solo	5 0 0 0 0	2 2 0 0 1	5 0 0 0 0	3 2 0 0 0	5 0 0 0 0	5 0 0 0 0
Syntac Sprint	4 0 0 0 1	0 0 2 1 2	5 0 0 0 0	0 0 1 3 1	5 0 0 0 0	0 0 0 5 0

0: No leakage.
1: The dye only penetrates the enamel (at the coronal margin) or the cementum (at the gingival margin).
2: The dye penetrates the dentin.
3: The dye penetrates to the depth of the cavity.
4: The dye penetrates through the apex.

Seven days: For all adhesives tested, the leakage at the coronal margin did not significantly differ from that at the gingival margin. When the behavior of the adhesives by pairs was compared, there were no significant differences at the coronal margin (p=0.082), where the dye penetration was always minimal. At the gingival margin, Syntac Sprint showed significantly worse results compared to the other adhesives.

Thirty-one days: There was a significantly greater dye penetration at the gingival compared to the coronal margin in the Syntac Sprint group ($p< 0.05$), while there were no differences between the two locations in the other groups. In the paired comparisons, there were no significant differences in leakage at coronal margin, and Optibond Solo and One Step had better results than the Single Bond and Syntac Sprint groups at the gingival margin.

Thermocycling plus seven days: There was significantly greater leakage at the gingival compared to the coronal margin in the Prime & Bond 2.0, Single Bond and Syntac Sprint groups. In the paired comparisons, there were no significant differences in leakage at the coronal margin (p=1.0). Optibond Solo had the best results at the gingival margin.

Table 3: *Comparison of Leakage at the Margins According to the Adhesive Used and the Time of Immersion in the Dye*

	Enamel vs Cementum[1]		
	Leakage 7 Days	**Leakage 31 Days**	**Leakage 7 Days Thermocycling**
One Step	NS[2]	NS	NS
Prime & Bond 2.0	NS	NS	$p<0.05$
Syntac Single	NS	NS	NS
Single Bond	NS	NS	$p< 0.05$
Optibond Solo	NS	NS	NS
Syntac Sprint	NS	$p<0.05$	$p<0.05$

[1] Wilcoxon test for comparison of paired samples.

[2] NS: Not significant.

The results listed in Table 5 show that the adhesion to the enamel was not significantly affected either by the passage of time or by the thermocycling, regardless of the adhesive used. Leakage at the gingival margin was significantly increased by thermocycling in the Prime & Bond and

Table 4: *Paired Comparison of the Adhesives at Both Cavity Margins According to the Time of Immersion in the Dye*

Leakage 7 Days		**Leakage 31 Days**		**Leakage 7 Days Thermocycling**	
Enamel	**Cementum**	**Enamel**	**Cementum**	**Enamel**	**Cementum**
One Step	One Step	One Step	Optibond Solo	One Step	Optibond Solo
Prime&Bond 2.0	Prime&Bond 2.0	Syntac Single	One Step	Prime&Bond 2.0	One Step
Syntac Single	Syntac Single	Single Bond	Prime&Bond 2.0	Syntac Single	Single Bond
Single Bond	Single Bond	Optibond Solo	Syntac Single	Single Bond	Prime&Bond 2.0
Optibond Solo	Optibond Solo	Syntac Sprint	Single Bond	Optibond Solo	Syntac Single
Syntac Sprint	Syntac Sprint	Prime&Bond 2.0	Syntac Sprint	Syntac Sprint	Syntac Sprint

The adhesives joined by lines showed no statistical differences between them.

Table 5: *Comparison of the Results of Each Adhesive According to the Time of Immersion in the Dye*

	Enamel 7 Days vs Enamel 7 Days Thermocycling[1]	**Cementum 7 Days vs Cementum 7 Days Thermocycling**	**Enamel 7 Days vs Enamel 31 Days**	**Cementum 7 Days vs Cementum 31 Days**
One Step	NS[2]	NS	NS	NS
Prime & Bond 2.0	NS	$p<0.05$	NS	NS
Syntac Single	NS	$p<0.01$	NS	NS
Single Bond	NS	NS	NS	$p<0.05$
Optibond Solo	NS	NS	NS	NS
Syntac Sprint	NS	NS	NS	NS

[1] Mann Whitney U test for paired comparisons of groups.

[2] NS: Not significant.

Syntac Single groups. There was also significantly increased leakage at the gingival margin with Single Bond after 31 days of immersion.

DISCUSSION

The bonding of composite to enamel and dentin is mainly based on micromechanical retention. The hybrid layer, resin tags and adhesive lateral branch formation have been suggested as the essential mechanisms of adhesion between a dentin bonding system and the conditioned dentin (Walshaw & McComb, 1996).

Although the efficacy of conventional acid etching of the enamel is considered to be adequately demonstrated in the clinical setting (Crim & Shay, 1987), currently, there is controversy regarding the strength and durability of bonding to enamel that can be obtained with weak acids or with phosphoric acid applied for shorter periods. In fact, attempts to replace phosphoric acid with citric acid, maleic acid, nitric acid or oxalic acid have demonstrated worse outcomes, both *in vitro* (Van Meerbeek & others, 1994; Van Meerbeek & others, 1996a) and *in vivo* (Ziemiecki, Dennison & Charbeneau, 1987; Swift & Cloe, 1993).

All of the adhesives tested in this study use phosphoric acid as etching agent, although at different concentrations (range, 32-37.5%) and for different lengths of time (range, 15-30 seconds). The levels of leakage in enamel were minimal, regardless of the bonding system used or the experimental conditions applied. The behavior of all of the adhesives was similar, except in the samples prepared with Prime & Bond 2.0, which unexpectedly leaked significantly more in enamel after 31 days in the dye than did the other samples studied. Thus, the application of phosphoric acid for 20 or 30 seconds (Prime & Bond and Syntac Single, respectively) does not appear to produce better marginal sealing on enamel than does its application for 15 seconds (all of the other adhesives studied).

Leakage at the gingival margin was found with all the adhesives and the worst results occurred with the use of Syntac Single and Syntac Sprint. Single Bond and Prime & Bond 2.0 showed significantly worse leakage at the gingival margin of samples submitted to thermocycling. Some authors reported that single-component adhesives achieved an incomplete infiltration of the hybrid layer (Van Meerbeek & others, 1996b; Powell, Johnson & Gordon, 1995). This drawback relates to an excessive demineralization of the dentin, leading to the exposure of too much collagen. This problem may be resolved by using shorter etching times. It has been suggested that adequate enamel etching without excessive demineralization can be achieved by placing phosphoric acid at >30% concentration first on the enamel surfaces, then extending it to the dentin for an additional 15 seconds (Triolo & others, 1993).

An efficacious hybridization also requires that the collagen network exposed after the acid application retain its sponginess. In the early 1990s, Kanca & Gwinnett, (Kanca, 1992a; Kanca, 1992b; Gwinnett, 1992) introduced their technique of adhesion to wet dentin, and all of the adhesives in this study employ this method and contain hydrophilic monomers as primers that either dissolve in water (Syntac Single) or in volatile solvents, such as acetone (Prime & Bond 2.0, One Step and Syntac Sprint) or ethanol (Single Bond and Optibond Solo). The use of wet dentin involves a risk of overwetting (Perdigão & others, 1996) that can to lead to separation between the primer and hydrophobic resin layers and to the formation of aqueous vesicles at the dentin-resin interface, thus weakening the bonding at the tubular and intertubular level and producing an incomplete sealing (Perdigão & others, 1996; Tay, Gwinnett & Wei, 1996). This study design prevented the authors from testing the characteristics of the interdiffusion zone obtained with the different adhesives. However, they demonstrated the difficulty of achieving the precise degree of wetness that provides an adequate hybridization. Some investigations showed that re-wetting the dried dentin surface with an aqueous HEMA solution maintained or improved bond strengths to levels comparable to those obtained by application of the dentin adhesive on moist dentin (Perdigão & others, 1998).

Comparing the six materials tested, Optibond Solo and One Step showed the best results under the three experimental conditions, both in enamel and cementum. Optibond Solo contains a resin with a filling of low elasticity that favors a stronger, more long-lasting bonding by acting as a relatively flexible layer that absorbs stress due to the contraction of the first layer of composite. This absorption reduces overall stress at the tooth-restorative material interface (Kemp-Scholte & Davidson, 1990a; Kemp-Scholte & Davidson, 1990b; Unterbrink & Liebenberg, 1999) and helps to preserve the marginal integrity. Another advantage of filled adhesives is that the film is thick enough to eliminate the problem of inhibition by oxygen (Unterbrink & Liebenberg, 1999). However, one drawback is that they are minimally radio-opaque, and since the thickness of the layer they produce is around 5-10 µm, this could induce errors in the diagnosis of caries at subsequent check-ups. The manufacturer of One Step recommends applying five layers of adhesive for Class V cavities. According to published reports (Walshaw & McComb, 1994), the presence of an adequate thickness of adhesive resin is an important factor in developing the secure bond. The application of one-bottle adhesives in multiple layers substantially improves the bonding to dentin, since it ensures displacement of the moisture

and the complete impregnation of the collagen network. However, this procedure unquestionably increases the treatment time.

While some of the requisites of the ideal bonding agent have been met, in that the authors can now etch the enamel and condition the dentin with one single application of acid and prime and bond without a washing step, there is a need for improvements in the bonding at the gingival margin.

CONCLUSIONS

1. All the adhesive systems tested have a very similar performance in enamel.
2. The adhesion to enamel was not significantly affected either by the passage of time or by thermocycling, regardless of the adhesive used.
3. Optibond Solo showed the best outcomes at the gingival margin under the three experimental conditions.

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Influence of Eugenol-Containing Temporary Restorations on Bond Strength of Composite to Dentin

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Clinical Relevance

Using temporary restorations prior to composite placement should be avoided, if possible. Eugenol-containing temporary restorations mixed at a ratio of 10g powder: 2g liquid significantly decreased bond strength of composite to dentin and should not be employed clinically.

SUMMARY

This study investigated the influence of eugenol-containing temporary restorations on bond strength of composite to dentin. Thirty-two freshly extracted human molars were embedded and horizontally sectioned at a level 2 mm from the central fossa to obtain a flat dentin surface. The teeth were randomly divided into four groups of eight teeth. Specimens in Group 1 (control) received no pretreatment with any temporary restorations. Group 2 and 3 specimens were covered with IRM (eugenol-containing) mixed at powder: liquid (P:L) ratio of 10g: 1g and 10g: 2g, respectively. Specimens in Group 4 were covered with polycarboxylate cement (eugenol-free) mixed at a P:L ratio of 2.85g: 1g. The temporary restorations were mechanically removed with an ultrasonic scaler after one-week storage in distilled water at 37ºC. The dentin surfaces were cleaned with pumice-water slurry and treated with Scotchbond Multi-Purpose Plus bonding system according to manufacturer's instructions. Composite (Z100) columns (3 mm diameter, 2 mm high) were applied and shear bond testing was carried out after 24 hours storage in distilled water at 37ºC using an Instron Universal testing machine with a cross-head speed of 0.5 mm/minute. The mode of failure was examined using a stereomicroscope at X40 magnification. Results were analyzed using one-way ANOVA/Scheffes's post-hoc test at significance level 0.05. Ranking of bond strengths was as follows: Group 1 (22.58 MPa) > Group 2 (21.14 MPa) > Group 4 (15.35 MPa) > Group 3 (13.02 MPa). Group 3 had significantly lower bond strength than Groups 1 and 2. No significant difference in dentin bond strength

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was observed between the Group 1 (control) and Groups 2 and 4. Although the predominant mode of failure for Groups 1, 2 and 4 was cohesive in dentin, all specimens in Group 3 exhibited adhesive failure. Pre-treatment with polycarboxylate cement or IRM mixed at P:L ratio of 10g: 1g did not affect shear bond strength of composite to dentin. IRM mixed at a lower P:L ratio of 10g: 2g significantly decreased bond strength.

INTRODUCTION

The clinical usage of composite restoratives has increased substantially over the last few years due to improvements in formulation, simplification of the bonding procedures, increased aesthetic demands by patients and the decline in amalgam usage due to fear of mercury toxicity and changes in government regulations. They can be used to restore all cavity classes in both anterior and posterior teeth except for large stress-bearing Class II restorations (Bayne, Heymann & Swift, 1994). Placing composite restorations requires a dry field and is very time consuming and technique sensitive. The lack of clinical time, intermediate restoration of multiple carious teeth and use of indirect pulp capping procedures may warrant using temporary restorations. Zinc oxide-eugenol (ZOE) cements are widely used as temporary restorations for cavities restored with amalgam and endodontically-treated teeth. In addition to being inexpensive and easily removable, they also have a sedative effect on sensitive teeth.

Mechanical removal of temporary cements is not 100% effective, and cement remnants were observed microscopically on surfaces that appeared macroscopically clean (Terata, 1993). Eugenol released from ZOE mixtures can also penetrate dentin (Hume, 1984; Kielbassa, Attin & Hellwig, 1997). Like other phenolic compounds, eugenol is a radical scavenger that inhibits the polymerization of resin materials (Tiara & others, 1992). The inhibited polymerization results in increased surface roughness, reduced microhardness and decreased color stability of composites cured in contact with ZOE cement (Grajower, Hirschfeld & Zalkind, 1974; Lingard, Davies & Von Fraunhofer, 1981; Marshall, Marshall & Hardcourt, 1982). Residual cement and eugenol penetration may also impair polymerization of resin adhesives and reduce wettability/reactivity of dentin (Baier, 1992), leading to a decrease in bond strength and marginal sealing ability. For these reasons, it has been considered state-of-the-art not to use eugenol-containing temporary restorations in cavities filled with composite resins. Hansen & Asmussen (1987) substantiated this view. They found markedly increased contraction gaps in dentin pre-treated with ZOE temporary restorations.

Although eugenol does not affect the bond strength of composites to enamel (Schwartz, Davis & Mayhew, 1990; Jung, Ganss & Senger, 1998), contradictory findings exist regarding bond strength to dentin, while earlier research has found pre-treatment with eugenol-containing temporary cements lowers bond strength of composites to dentin (Xie, Powers & McGuckin, 1993; Terata & others, 1994), later research have found otherwise (Ganss & Jung, 1998; Kelsey, Latta & Blankenau, 1998). It was hypothesized that current dentin-bonding systems effectively remove residual cement and eugenol-contaminated dentin and consequently are insensitive to pre-treatment with eugenol-containing temporary restorations.

This study assessed the influence of eugenol-containing temporary restorations of different powder: liquid ratios on the bond strength of composite to dentin. A eugenol-free cement was included to determine whether any negative effects result from the presence of residual cement or eugenol.

METHODS AND MATERIALS

Table 1 lists the restorative materials used and their manufacturers. Thirty-two freshly extracted, non-carious human third molars were selected for the study. The teeth were cleaned, disinfected in 10% formalin-saline solution for 10 minutes and stored in distilled water at 4ºC until required for use. The roots were serrated and the teeth embedded in auto-curing acrylic resin (Tray Resin II; Shofu Inc, Kyoto, Japan) within customized plastic holders. The mounted teeth were then sectioned horizontally at a level 2 mm from the central fossa with a 64 μm grit diamond blade in a sectioning apparatus (CP300; Exakt, Norderstedt, Germany) to obtain a flat dentin surface. The sectioned teeth were randomly divided into four groups of eight teeth. Specimens in Group 1 (control) received no pre-treatment with any temporary restorations. Group 2 specimens were covered with IRM (eugenol-containing) mixed at manufacturer's recommended powder: liquid (P:L) ratio of 10g: 1g (1 scoop powder: 1 drop liquid). Specimens in Group 3 were also covered with IRM but mixed at a lower P:L

Table 1: Materials Used and Their Manufacturers

Material	Manufacturer	Batch #
Intermediate Restorative Material (IRM)	Dentsply-Caulk, Milford, DE 19963	000614
Hy-Bond Polycarboxylate Cement	Shofu Inc, Kyoto, Japan	9903
Scotchbond Multi-Purpose Plus	3M Dental Products St Paul, MN 55144	19980121
Z100 (A2 shade)	3M Dental Products St Paul, MN 55144	19980106

ratio of 10g: 2g (1 scoop powder: 2 drops liquid). Group 4 specimens were covered with polycarboxylate cement (eugenol-free) mixed at a P:L ratio of 2.85g: 1g (1 scoop powder: 2 drops liquid). Customized square molds (5 mm long/wide and 1.5 mm deep) were used for placement of temporary restorations. The molds were first slightly overfilled with temporary restorative materials. Excess material was extruded by applying pressure through a glass slide. All specimens were then stored in distilled water at 37ºC for one week.

After the one-week incubation, the temporary materials were mechanically removed with an ultrasonic scaler until the dentin surfaces were macroscopically free of material. The specimens were then cleaned with pumice-water slurry, rinsed and treated with Scotchbond Multi-Purpose Plus system (SBMP) according to manufacturer's instructions. The dentin surfaces were first etched with SBMP etchant (37% phosphoric acid) for 15 seconds, rinsed and blotted dry. SBMP primer was then placed and gently dried for five seconds. A thin layer of SBMP adhesive was placed and light-cured for 10 seconds using a PolyLux II light-curing unit (Kavo Dental, Warthausen, Germany). A split Delrin mold with a cylindrical hole (3 mm diameter and 2 mm high) was clamped to the tooth assembly and the mold was filled with Z100. The composite was light-cured for 40 seconds using the PolyLux II light-curing unit with a light-intensity of 540 mW/cm^2. The mold was removed immediately after light-curing and the samples were stored in distilled water at 37ºC for 24 hours. The shear bond strengths were determined using an Instron Universal testing machine (Instron Corp, Canton, MA 02021, USA) with a cross-head speed of 0.5 mm/minute. A 0.5 mm spacer was placed so that a constant distance was maintained between the bonded interface and the point of force application through the force rod. The mean bond strength value and standard deviation for each experimental group was computed. Results were analyzed using one-way analyses of variance (ANOVA) and Scheffe's post-hoc test at significance level 0.05.

To determine the mode of failure, samples were examined with a VM stereomicroscope (Olympus, Tokyo, Japan) at X40 magnification. Some specimens showed multiple modes of failure, however, they were classified according to the dominant mode of failure as advocated by Yap, Sau & Lye (1999). Three categories were defined. Type I, adhesive failure at the dentin-composite interface; Type II, cohesive failure within the composite and Type III, cohesive failure within dentin.

RESULTS

Table 2 shows the mean shear bond strength values for the different experimental groups. The failure modes by percentage are reflected in Table 3.

The highest bond strength to dentin was obtained with the control group (Group 1). Ranking of mean bond strength was as follows: Group 1 (22.58 MPa) > Group 2 (21.14 MPa) > Group 4 (15.35 MPa) > Group 3 (13.02 MPa). The bond strength for Group 3 was significantly lower than that for Groups 1 and 2. Thus, pre-treatment with IRM mixed at a P:L ratio of 10g: 2g significantly reduced bond strength of composite to dentin. No significant difference was observed between the control group (Group 1) and Groups 2 and 4.

The predominant mode of failure for Groups 1, 2 and 4 was cohesive in dentin (Type III). Adhesive failure (Type I) was observed in about a third to quarter of the specimens in these groups. Type I failure was, however, observed with all specimens in Group 3.

DISCUSSION

Eugenol is the principal constituent of clove oil and has the structure 4-ally 2 methoxy phenol. A chelating reaction occurs when zinc oxide is mixed with eugenol in the presence of water due to hydrolysis. The set

Table 2: Mean Bond Strength Values (MPa) and Standard Deviations for the Different Experimental Groups

Group	Description	Mean Bond Strength (MPa)	Standard Deviation
1	Control – No pre-treatment	22.58	7.61
2	Covered with IRM mixed at 10g powder: 1g liquid	21.14	3.50
3	Covered with IRM mixed at 10g powder: 2g liquid	13.02	1.63
4	Covered with polycarboxylate cement mixed at 2.85g powder: 1 g liquid	15.35	4.83

Table 3: Failure Modes by Percentage

Group	Failure Mode Type I Adhesive	Type II Cohesive in Composite	Type III Cohesive in Dentin
1	37.5%	0%	62.5%
2	25.0%	0%	75.0%
3	100%	0%	0%
4	25%	0%	75.0%

cement consists of grains of zinc oxide embedded in a zinc eugenolate matrix (Markowitz & others, 1992). ZOE cement is one of the least irritating of all dental materials and provides an excellent seal against leakage (Anusavice, 1991). Various formulations and uses are reflected in ADA specification No 30 for ZOE restorative materials, which lists four types. Type I cement is used for temporary cementation. Type II cement is intended for permanent cementation of restoration or appliances fabricated outside the mouth. Type III cement is used for temporary restorations and thermal-insulating bases, where as Type IV is used as a cavity liner. ZOE cements are not recommended as liners and bases under composites restorations.

Studies on the influence of Type III ZOE cements on bond strength of composite restorative materials to dentin are limited. Most research had focused on Type I ZOE cements and their effects on resin luting cements or composite resin cores (Ganss & Jung, 1998; Kelsey & others, 1998; Al-Wazzan, al-Harbi & Hammad, 1997; Paul & Scharer, 1997; Millstein & Nathanson, 1992). Peutzfeldt & Asmussen (1999) studied the effect of a Type III ZOE cement (IRM) on two dentin-bonding systems (Gluma CPS and Scotchbond Multi-Purpose Plus) and a composite resin. The tooth surfaces were exposed to IRM or a eugenol-free cement (Cavit) for one week before application of the dentin-bonding systems and shear bond strength testing was conducted on one-day old specimens as with this study. They concluded that ZOE temporary restorations did not influence the efficacy of the two dentin-bonding systems. For SBMP, mean dentin bond strengths were 20 ± 6 MPa for the freshly cut tooth, 20 ± 7 MPa for specimens pre-treated with IRM and 22 ± 5 MPa for specimens treated with Cavit. No significant difference in dentin bond strengths was observed among the three groups. The P:L ratio of IRM was not mentioned in their study and can be assumed to be 1 scoop powder: 1 drop liquid as recommended by the manufacturer. The bond strengths obtained are similar to those observed in this study. The recommended ratio of 1 scoop powder: 1 drop liquid (10g: 1g) results in a rather dry mix that is difficult to manipulate. Hence, a lower P:L ratio of 1 scoop powder: 2 drops liquids (10g: 2g), which is more commonly employed by clinicians, was also evaluated in this study.

The highest bond strength was observed with the control group and a reduction in bond strength was observed after pre-treatment with both eugenol-containing and eugenol-free cements. Using temporary restorations prior to composite placement is, therefore, best avoided, if possible. This was also observed in other studies (Xie & others, 1993; Terata & others, 1994). Woody & Davis (1992) suggested that the negative effect of temporary cements on bond strength was not caused by eugenol, but by the presence of residual cement. The latter may explain the lower bond strength observed with specimens pre-treated with polycarboxylate cement (Group 4) as compared to IRM mixed at the P:L ratio of 10g: 1g (Group 2). Polycarboxylate cements chemically bond to tooth via an ion-exchange mechanism. This bonding mechanism is identical to that of glass ionomer cements. The complexities are not yet fully understood but, in essence, the union of the cement with tooth structure arises following the displacement of phosphate ions from the surface of the tooth by the carboxyl group of polyacrylic acid. Each phosphate ion takes with it a calcium ion to maintain electrolytic balance. A new material is developed as an intermediary between the tooth and cement, and this is firmly attached to both (Mount, 1998). This intermediate or ion-exchange layer may be harder to remove and more acid resistant, resulting in decreased efficacy of the dentin-bonding system and lower bond strength observed. IRM does not bond to tooth, and the "total-etch" technique employed by SBMP appears to effectively remove any residual cement or eugenol contaminated dentin when IRM is mixed in the P:L ratio of 10g: 1g. Alternatives to polycarboxylate and ZOE cements for the temporary restoration of teeth include silicophosphate and glass ionomer cements.

Although no significant difference in bond strength between the control group and the groups pre-treated with polycarboxylate cement or IRM mixed at a P:L ratio of 10g: 1g, pre-treatment with IRM mixed at a ratio of 10g powder: 2g liquid lowered bond strength significantly. Bond strength values obtained for the latter (Group 3) were significantly lower than the control group and Group 2, where IRM was mixed in the P:L ratio of 10g: 1g. The eugenol content was doubled in Group 3, compared to Group 2. Wetter ZOE mixtures have been shown to have significantly higher diffusion rates (Kielbassa & others, 1997) and may be more susceptible to hydrolysis. Because water cannot penetrate the set bulk materials, only dentinal tubule fluid has an effect on the rate of release of eugenol towards the pulp. The latter may explain why eugenol does not affect the bond strength of composites to enamel (Schwartz & others, 1990; Jung & others, 1998). Dentin bond strength *in vivo* may be even lower due to the presence of intra-pulpal pressure and the exudation of tubule fluid from freshly cut dentin that leads to increased hydrolysis and the presence of free eugenol. The inhibition of polymerization of the dentin-bonding system and composite by eugenol results in the decreased bond strength and 100% adhesive failure observed with all specimens in Group 3. Hence, it is not accurate to state that ZOE cements do not influence the efficacy of new dentin-bonding systems, as results depend highly on the concentration of eugenol present in the cement.

The predominant mode of failure for Groups 1, 2 and 4 was cohesive in dentin. This testifies to the effectiveness of the dentin-bonding system and the "total-etch" technique. Phosphoric acid etching successfully removes residual cement, smear layer, eugenol-containing dentin and demineralizes dentin. Its effectiveness in removing the ion-exchange layer associated with the use of polycarboxylate cements needs to be verified. The hydrophilic monomers contained in the primer and adhesive infiltrates the demineralized dentin to form a hybrid layer of polymerized resin intermingled with collagen bundles (Nakabayashi, Kojima & Masuhara, 1982). The bond strength values observed may better reflect the strength of the hybrid layer-dentin interface rather than the composite-dentin interface. The moderate standard deviations observed could be attributed to inter-individual differences in dentin quality including density, length and diameter of the dentin tubules.

CONCLUSIONS

Based on the results of this *in vitro* study, using temporary restorations prior to composite placement should be avoided, if possible. If temporary restorations are required, both polycarboxylate (2.85g powder: 1g liquid) and ZOE cement mixed in the ratio of 10g powder: 1g liquid is permissible. ZOE cements mixed at a lower ratio of 10g powder: 2g liquid significantly decreased bond strength of composite to dentin and should not be employed clinically.

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3-D Surface Profile Analysis: Different Finishing Methods for Resin Composites

L Marigo • M Rizzi
G La Torre • G Rumi

Clinical Relevance

High levels of smoothness are produced by certain procedures on resin composite surfaces.

SUMMARY

A microfilled and hybrid resin composites used for esthetic restoration were finished and polished using four methods: Enhance system, Sof-Lex system, Multi-step system and Identoflex points.

The tested materials were condensed into cylindrical molds, covered with a Mylar matrix at the surface to be tested and incrementally cured according to manufacturers' instructions. Samples were randomized into four groups of three for each material and were finished/polished using the different methods. The samples were then analyzed by a 3-D surface profiler to obtain roughness average (Ra), root mean square value (rms), greatest distance peak-valley (PV), measure of profile about the center line (Rsk) and measure of steepness of the amplitude density curve of the roughness profile (Rku) directly from the tested area. This method offers the advantage of being error-free. All parameters were determined for each sample and the mean of each parameter was determined for each group. ANOVA and Sheffé's test were employed to determine whether significant differences existed. The Enhance and Multi-step systems gave the best finish and polish for both materials.

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INTRODUCTION

The longevity and esthetic appearance of a composite restoration greatly depends on the quality of finishing and polishing techniques employed. Moreover, as reported by other authors (Barsotti & others, 1989), gingival health is subject to surface texture of the restoration. A smooth surface has always been the prime objective of composite restorations due to secondary caries and discoloration that results from plaque accumulation. Whereas surface roughness seems to affect the initial adhesion of cells, it appears to be independent of bacterial accumulation once initial adhesion has occurred. Weitman & Eames (1975) reported that plaque accumulates on composite samples with a surface roughness of 0.7-1.44 µm. A well-finished and polished surface is difficult to obtain because the resin matrix and the inorganic fillers differ in hardness, preventing homogeneous abrasion. Finishing and polishing can also affect the color and gloss of a composite restoration (Stanford & others, 1985).

Many researchers agree that the smoothest surface is obtained when composites set against a cellulose strip and any subsequent finishing/polishing techniques can be detrimental to this surface (Heath & Wilson, 1976; Bauer & Caputo, 1983).

Quiroz & Lentz (1985) found that even the finest Brassler ET diamond finishing bur caused extensive damage to the surface areas of enamel and that composites finished with diamond burs were rough. Using Scanning Electron Microscopy, Grundy (1985) observed that diamond burs had a tendency to tear filler particles and leave irregularities on the composite surface. Diamond burs gave rougher surfaces overall with superficial irregularities that were more easily polished than surfaces treated with carbide burs (Goldstein & Waknine, 1989). Several studies stated that the large particles embedded in Sof-Lex disks tend to rip through the surface of the composites and Sof-Lex disks used with certain hybrid composites tend to cut and abrade filler particles and resin matrix equally, resulting in a smoother surface (Van Noort & Davis, 1984; Van Dijken & Ruyter, 1987). These authors also found that frictional heat generated by Sof-Lex disks cause microcracks in the polymer matrix. Tate & Powers (1996) found that fluted finishing bur left a roughened irregular surface on hybrid ionomers and composites, requiring further finishing and polishing. Overall, Sof-Lex disks (aluminum-oxide) produced the smoothest finished surface for both the materials.

To date, research on finishing/polishing techniques has employed either qualitative SEM methods or quantitative mechanical surface profile analysis (Eide & Tveit, 1988; Stoddard & Johnson, 1991; Ashe & others, 1996). Berastegui & others (1992) concluded that microfilled resin composites provided a better finish when treated with aluminum-oxide disks. Results obtained by a profilometer were statistically significant when compared with arkansas, tungsten and diamond burs ($p<0.0001$). In another study, Chung (1994) demonstrated that three different polishing systems produced significantly less roughened surfaces ($p<0.01$) for microfilled composites than hybrid composites. In 1997, Hondrum & Fernandez showed that Enhance System, in association with finishing burs, silicone points and polishing pastes, produced the smoothest surface. On the contrary, other authors demonstrated that Enhance System provided a less smooth surface for hybrid and microfilled resin composites than other finishing/polishing systems (Kaplan & others, 1996; Setcos, Tarim & Suzuki, 1999).

Rapisarda & others (1999) compared four different finishing methods of composite and compomers. The authors found that silicone points did not remove much material but continued to smooth the outline of the restoration and the disks were not suitable for finishing the composites and compomers.

This study evaluated surface plots and roughness parameters of two composites subjected to different finishing and polishing techniques using an interferometric method.

METHODS AND MATERIALS

Table 1 lists the products, manufacturers, finishing and polishing materials and techniques used in this study. The composites employed were a microfil, Silux Plus (shade L) and a hybrid and Filtek Z250 (shade A2) (3M Dental Products, St Paul, MN 55144, USA).

Samples were made by placing each material into a cylindrical mold (8 mm diameter x 3 mm depth) shaped by a computer-aided bur (Ferrari turning-lathe, Milano, Italia 20080) in an aluminum alloy plate. The composites were pressed into the mold in two increments to form two layers, each polymerized with a light curing unit (Curing light XL 1500, 3M Dental Products) for 20 seconds.

Table 1: Finishing/Polishing Products with Sequence Passages and Manufacturers

Finishing/Polishing	Description/Procedures Systems	Manufacturers
Enhance System	Finisher disk aluminum oxide (40 µm),slow speed Polishing paste aluminum oxide (1 µm), prophy cup Extra fine paste aluminum oxide (0.3 µm), prophy cup	LD Caulk/Dentsply Milford, DE 19963
Sof-Lex System	Coarse disk aluminum oxide (55 µm), slow speed Medium disk aluminum oxide (40 µm), slow speed Fine disk aluminum oxide (24 µm), slow speed Superfine disk aluminum oxide (8 µm), slow speed	3M Dental Products St Paul, MN 55144
Identoflex Points	Finisher yellow silicone point, slow speed Polishing white silicone point, slow speed Pumice silicone polisher blue point, slow speed	Identoflex AG Buchs Switzerland CH 9471
Multi-step System	3117 diamond bur (25 µm), high speed 5017 diamond bur (15 µm), high speed Pumice silicone polisher blue point, slow speed Diamond paste Shiny A (3 µm), hair goat brush, slow speed Diamond paste Shiny B (1 µm), felt disk, slow speed	Intensiv, Switzerland Identoflex AG Buchs Switzerland CH 9471 Enamel plus Micerium srl, Rosbach, Germany 61191

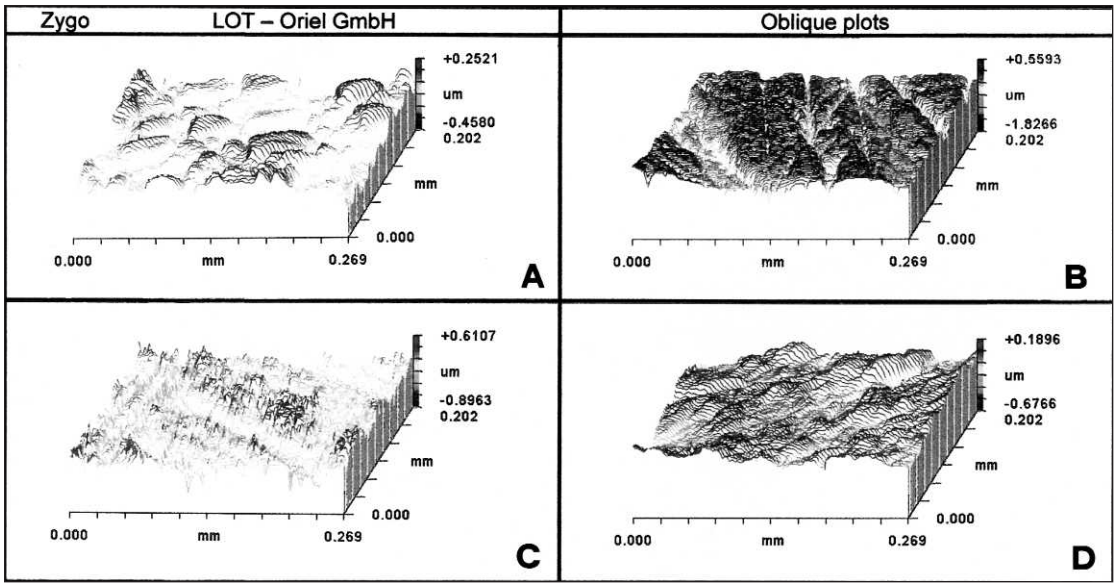

Figure 1. *3-D oblique plots by Zygo for representative samples of Filtek Z250 after treatments with Enhance System (A), Sof-Lex System (B), Identoflex Points (C), Multi-step System (D).*

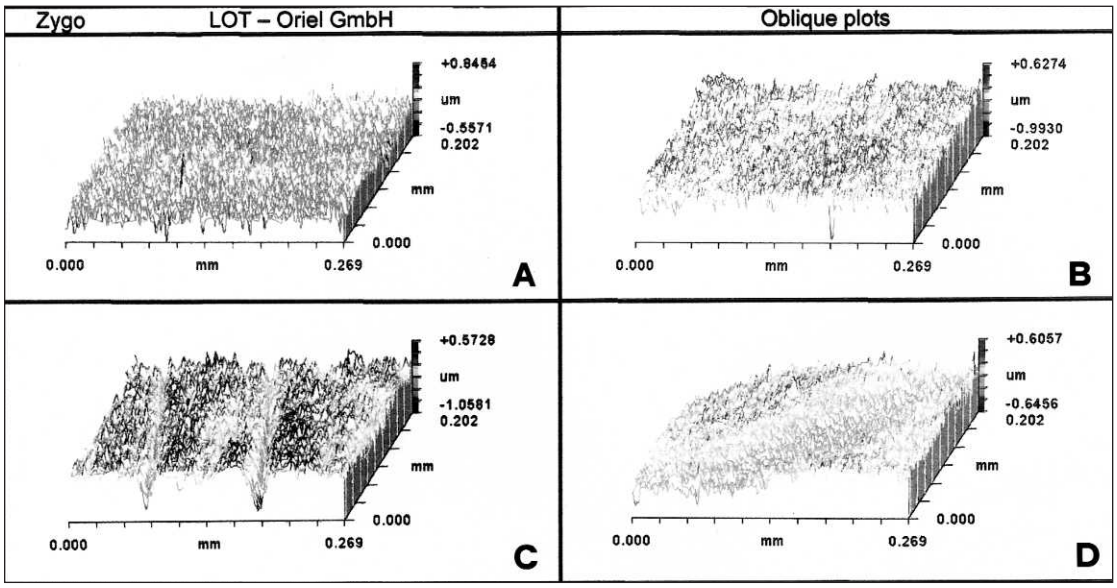

Figure 2. *3-D oblique plots by Zygo for representative samples of Silux Plus after treatments with Enhance System (A), Sof-Lex System (B), Identoflex points (C), Multi-step System (D).*

The second increment was placed to allow overflow of the plate surface. This surface was then covered with a Mylar matrix strip (SS White Co, Philadelphia, PA 19177, USA) and a glass slide using hand pressure for 10 seconds during light polymerization. The surface was then polymerized for an additional 10 seconds without hand pressure.

The samples were removed with a pointed instrument pushed through a hole in the bottom of the mold. All samples were finished and polished by one operator. The various techniques were applied according to manufacturers' instructions (Table 1). Diamond burs were used with a high-speed handpiece. All samples were tested after each polishing/finishing step. All disks, silicone points and polishing instruments were used only once. All polishing steps were performed for 30 seconds using minimal pressure and continual irrigation, except for the steps that used pastes.

Sample surfaces were analyzed using a Zygo 3-D surface profiler (Zygo New View 5000, Lot Oriel, Milano, Italia). This instrument, which is based on scanning white-light interferometry by splitting incoming light, produces a light and dark fringe pattern. Together, a vertical scanning transducer and camera generate a three-dimensional interferogram of the surface processed by the computer and transformed by frequency domain analysis to give a quantitative non-contact 3-D image (Figure 1).

For each finishing system, three samples were prepared and only the final step was analyzed. Three random readings covering an area of 269 µm x 202 µm totaling 163.014 µm^2 were executed. Since readings taken at the periphery may not truly represent the surface profile, the authors decided to exclude a circular surface of 2 mm from the edge; in fact, using Zygo 3-D is possible to defined the zone to be analyzed. The parameters obtained were: Ra, arithmetic mean of the sum of roughness profile values; rms, root mean square value obtained from the ordinate values of the roughness profile; PV, greatest distance peak-valley; Rsk, measure of the shape or sym-

Table 2: Mean Values by 3-D Surface Profile Analysis for Filtek Z250 (μm)

System Employed	Ra	rms	PV	Rsk	Rku
Enhance System	0.105	0.135	1.402	0.032	3.712
Sof-Lex System	0.134	0.182	3.143	-1.895	21.916
Identoflex Points	0.120	0.159	1.682	-0.813	5.326
Multi-Step System	0.095	0.119	1.181	-0.171	3.348

Table 3: Mean Values by 3-D Surface Profile Analysis for Silux Plus (μm)

System Employed	Ra	rms	PV	Rsk	Rku
Enhance System	0.058	0.073	0.749	0.266	3.582
Sof-Lex System	0.124	0.205	3.196	-3.307	28.512
Identoflex Points	0.127	0.176	2.705	-0.230	11.644
Multi-Step System	0.062	0.078	0.763	-0.135	5.075

metry of the amplitude density curve of the roughness profile; Rku, measure of the steepness of the amplitude density curve of the roughness. Ra, rms, Rsk and Rku data were first analyzed using a multifactorial analysis of variance. When differences were significant, one-way ANOVA and Sheffé's tests at the 99% confidence level determined whether significant differences existed between composite and roughness values. The authors considered a normal distribution of pro-

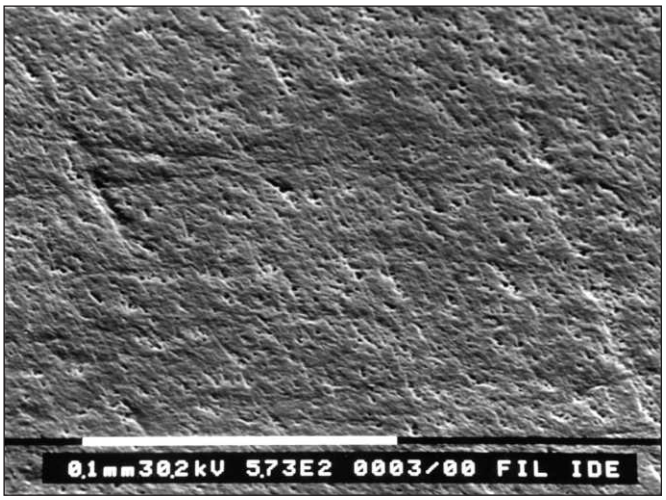

Figure 3. *Filtek Z250, Identoflex points (573x).*

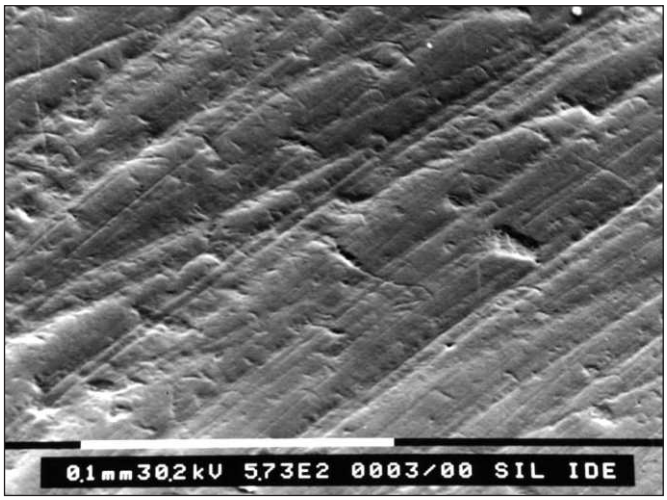

Figure 4. *Silux Plus, Identoflex points (573x).*

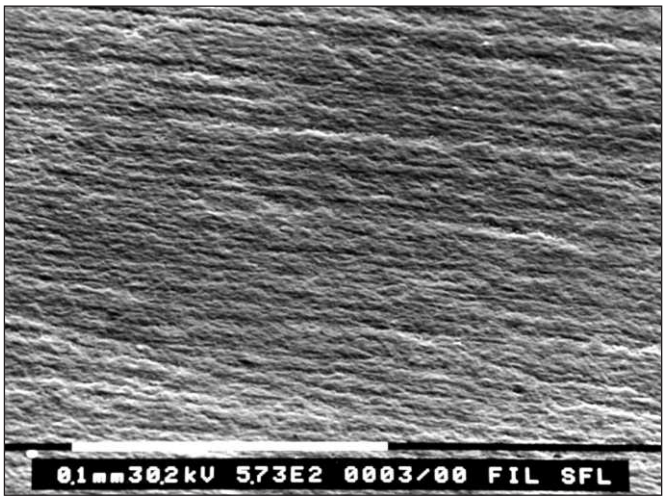

Figure 5. *Filtek Z250, Sof-Lex System (573x).*

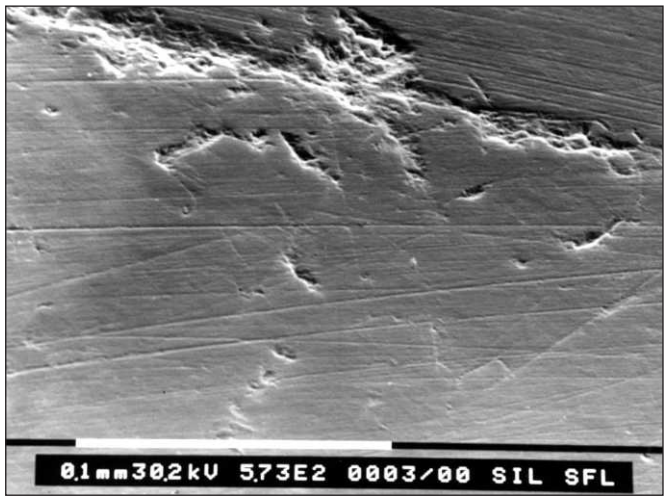

Figure 6. *Silux Plus, Sof-Lex System (573x).*

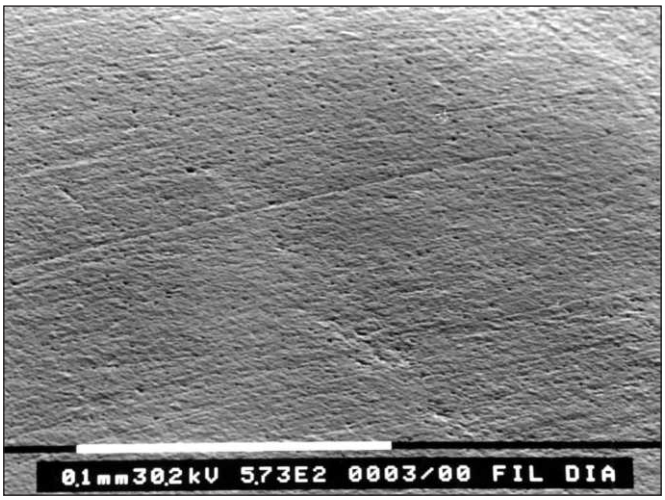

Figure 7. *Filtek Z250, Multi-step System (573x).*

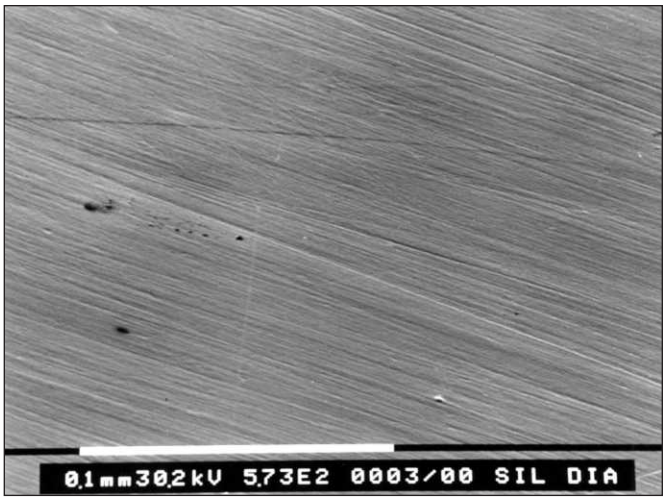

Figure 8. *Silux Plus, Multi-step System (573x).*

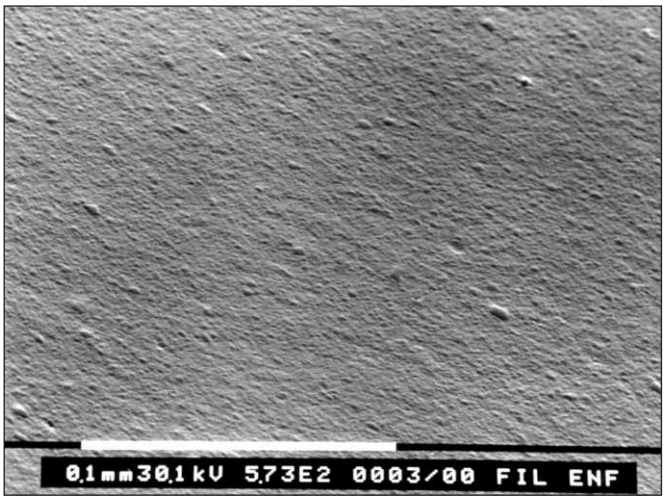

Figure 9. *Filtek Z250, Enhance System (573x).*

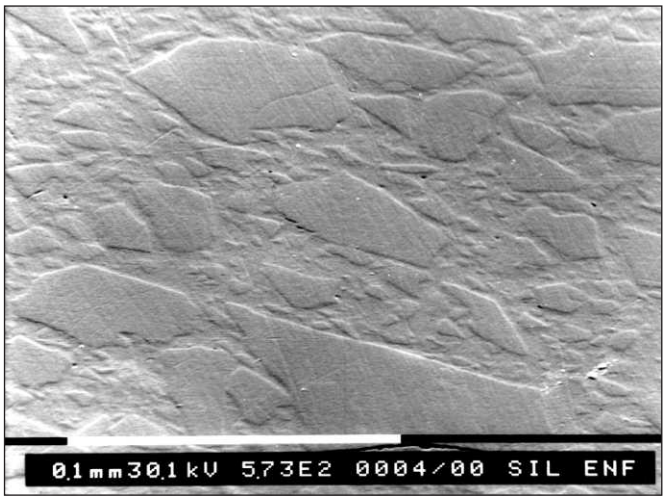

Figure 10. *Silux Plus, Enhance System (573x).*

file values results in zero skewness (Rsk=0) and the curtosis value of a roughness profile with a normal distribution of profile values results in three (Rku=3).

Representative samples of the different finishing/polishing techniques for each material were selected for Scanning Electron Microscopy (SEM Philips 500, Amsterdam, Netherland).

RESULTS

The 3-D surface profiler provides a filled and an oblique plot, both with a colorimetric evaluation to a range from –1.8266 µm to 0.5593 µm (Figures 1 and 2). The histogram recorded for each area of each sample tested is characterized by counts (on the Y-axis) associated with a different surface level. A narrow histogram indicates a homogeneous area.

Tables 2 and 3 report the results. Interferometry showed that for Silux Plus, the Enhance and the Multi-step systems gave similar results. Sof-Lex disks and Identoflex points showed similar values for Silux Plus and were rougher than those obtained using the other two systems.

The Filtek Z250 surface finished and polished with the Enhance and the Multi-step systems had less roughness than with Sof-Lex disks or Identoflex silicon points. The results obtained with the Enhance system and the Multi-step system differed from those obtained for Silux Plus microfilled composite.

One-way ANOVA test demonstrated significant differences for the finishing methods of microfilled composite ($p<0.01$) both for Ra and rsm values. The results of Sheffé's tests showed that Identoflex points had a roughness value higher than Enhance system ($p<0.05$).

According to Rsk parameter, the Sof-Lex system showed the worst performances for microfilled and hybrid resin composites ($p<0.005$). According to Rku,

the Sof-Lex and Identoflex systems showed significant values for the microfilled and hybrid resin composites ($p<0.05$).

Scanning Electron Microscopy qualitative analysis of the representative samples confirm the differences among the surfaces obtained using the four different finishing and polishing procedures. Microphotographs of hybrid composites finished with Identoflex points show a uniform surface dotted with pores produced by detached filling particles (Figure 3). The same thing occurred to microfilled composite surfaces, but resulted in less homogeneousness due to deep scratches in accordance with PV data (Figure 4).

Scratched surfaces were observed on the hybrid composites when finished with the Sof-Lex System and the organic fillers were not distinguished from the resin matrix (Figure 5). Silux Plus surfaces were smoother, even if variable ridges were produced by coarse and medium disks in the same areas (Figure 6).

Multi-step and Enhance System produced smooth, high quality surfaces for hybrid resin composites (Figures 7 and 9). After the last step, the filling particles were seen in the resin matrix and appeared rounded-off and well located. Shining surfaces without roughness were produced (Figures 8 and 10) when both finishing systems were used on Silux Plus.

DISCUSSION

Several previous studies (Chung, 1994; Tate & Powers, 1996; Setcos & others, 1999) use Ra parameter to examine the average surface roughness of some aesthetic materials, but Ra only provides very limited information and is insensitive to extreme profile peaks and valleys. Obtaining a reliable quantitative and qualitative evaluation of surface microstructure using Zygo 3-D surface profiler is possible. This is the first time to the authors' knowledge that interferometry has been employed for that purpose. Surface profile analysis showed that smoothness depends on use of a systematic series of instruments and polishing materials characterized by decreasing abrasive particle size.

Rsm, Rsk and Rku represent the standard deviation of profile values and measures of the amplitude density curve of the roughness profile.

Rsk and Rku parameters showed significant differences in this research that did not appear when Ra and Rms parameters were used. In fact, Rsk measures the shape or symmetry of the amplitude density curve of the roughness profile: skewness values are greatly influenced by the individual extreme profile peaks and valleys and a normal distribution of profile values results in zero skewness (Sof-Lex/Silux plus Rsk= -3.307). Rku measures steepness of the amplitude density curve of the roughness profile: a profile with flattened peaks and valleys has smaller Rku values. More pointed peaks and valleys in the roughness profile would result in Rku values of >3 (Sof-Lex/Z250 Rku=21.916).

Using finishing burs, alone, gave a markedly rougher surface than other finishing methods tested. This agrees with a previous study (Hoelscher & others, 1998).

The final glossy surface obtained by polishing depends on the flexibility of the backing materials in which the abrasive is embedded, the hardness of the abrasive, the geometry of the instruments and the instruments employed.

This study agrees with that of Hondrum & Fernandez (1997), which showed abrasive impregnated disks followed by polishing pastes (Prisma Gloss and Prisma Gloss Extra-fine aluminum—oxide pastes) for the Enhance system and finishing burs followed by silicon points followed by diamond polishing pastes for the Multi-step system, as providing a considerably smoother surface than the other systems. Moreover, it also showed that using polishing paste following carbide burs, abrasive impregnated finishing cups or aluminum oxide disks did not greatly improve the surface smoothness of composite and glass ionomer materials (Chen, Chan & Chan, 1988; Tate & Powers, 1996; Tate, Deschepper & Cody, 1992). Other studies have recommended using aluminum oxide disks following carbide finishing burs to obtain a clinically smooth restoration surface (St Germain & Meiers, 1996; Tate & Powers, 1996).

This study's data suggests that using diamond finishing burs, sometimes routinely employed in clinical practice, followed by secondary finishing passages and final polishing (with diamond polishing pastes), results in a surface with smoothness and gloss superior to that obtained with an abrasive impregnated disk or aluminum oxide disks, alone.

CONCLUSIONS

Filtek Z250 hybrid composite is rougher than microfilled Silux Plus after testing the finishing and polishing techniques used in this study. The multi-step finishing and polishing techniques tested for Silux Plus (Enhance and Multi-step) produced an excellent surface smoothness. The smoothest surfaces were achieved with the Enhance and Multi-step systems, which stress the importance of using aluminum oxide polishing and diamond polishing pastes. Diamond polishing burs, Sof-Lex disks and Indentoflex points should not be used alone to polish composite if the smoothest surface of esthetic restorations is to be obtained. According to roughness tests, statistic results indicated that Rms, Rku and Rsk parameters give more information than Ra parameter.

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Effect of Preliminary Treatment of the Dentin Surface on the Shear Bond Strength of Resin Composite to Dentin

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Clinical Relevance

After acid conditioning, treating dentin surfaces with aqueous HEMA solution before applying Prime & Bond 2.1 or 2% chlorhexidine solution prior to One Step is recommended to enhance resin-dentin shear bond strength.

SUMMARY

This study evaluated the effect of two dentin disinfectants (Consepsis, Tubulicid), one aqueous HEMA solution (Aqua Prep), a combination of Aqua Prep and Tubulicid and an air abrasion treatment (50 µm aluminum oxide) on the shear bond strength (SBS) of two acetone-based single bottle adhesives (One Step and Prime & Bond 2.1). The occlusal surfaces of 167 freshly extracted human third molars were ground flat to expose the dentin, then polished with a 600 grit-polishing disc. The teeth were randomly assigned to 12 test groups (two bonding agents, six pretreatment protocols). The exposed dentin was etched with 35% phosphoric acid for 20 seconds, rinsed and briefly (1-2 seconds) air dried. Six pretreatment protocols were then applied. The air abrasion groups were exceptional, as etching was carried out only after pretreatment. One Step, or Prime & Bond 2.1 was applied according to the manufacturer's instructions. Cylinders of Z-100 composite were bonded to the flat dentin surfaces by transparent gelatin capsules. Specimens were thermocycled in water baths between 5° and 55°C, then sheared in an Instron Testing Machine. One-way and two-way ANOVA and Tukey HSD post-hoc tests were used for statistical analysis. In the One Step group, Consepsis yielded a significantly higher SBS (17.8 MPa) than air abrasion (9.5 MPa), Control (11.8 MPa) and Aqua Prep + Tubilicid (11.9 MPa), and a comparable SBS with Tubilicid (12.5 MPa) and Aqua

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Prep (14.8 MPa). In the Prime & Bond 2.1 group, Aqua Prep (24.9 MPa) showed a significantly higher SBS than all other groups: air abrasion (9.3 MPa), Control (9.97 MPa), Tubilicid (12.2 MPa), Consepsis (13.0 MPa) and Tubilicid +Aqua Prep (13.3 MPa).

INTRODUCTION

Current materials for treating dentin after etching and before applying hydrophilic resins include cavity disinfectants, re-wetting agents and "coworker" agents. The objective of a disinfectant wash is to eliminate residual bacteria from the prepared cavity. Bacterial growth under the restoration could lead to secondary caries or injury to the pulp (Meirs & Shook, 1996). Commercially available disinfectants may contain chlorhexidine digluconate, benzylkonium chloride, disodium EDTA dihydrate or iodine. The etched dentin surface rinsed, dried and re-moistened with an aqueous HEMA solution, maintains a collagen framework and intertubular porosity patent for monomer infiltration. As excess water is partially evaporated by a gentle air stream, HEMA concentration increases and the water vapor pressure decreases, making it more difficult to remove the last residual water. Thus, the dentin remains moist, a prerequisite for optimal interfacial integrity, especially with the current generation of acetone-based single bottle adhesives (Perdigão & others, 1998, 1999; Pashley & others, 1998).

An aqueous solution of HEMA and 5% gluteraldehyde has been reported to be an effective "coworker." When applied after conditioning the dentin, the marginal contraction gap could be significantly reduced. This may result from the collagen fibers being crosslinked by the glutaraldehyde, which strengthens the organic part of the hybrid layer (Hansen & Asmussen, 1997).

Chlorhexidine gluconate, disodium EDTA dihydrate and iodine/copper sulfate-based disinfectants have no significant effect on the SBS of composite to dentin when fourth generation dentin bonding agents are used, as these require complete smear layer removal (Gwinnett, 1992; Perdigão, Denehy & Swift, 1994; Meirs & Shook, 1996). Similarly, Tubilicid red label (Global Dental Products, North Bellmore, NY 11710, USA) containing disodium EDTA dihydrate, 1% benzylkonium chloride and 1% sodium fluoride did not reduce the tensile bond strength of composite to root dentin mediated by a system with only a partial removal of the smear layer (Surmont & others, 1989).

Applying Aqua Prep (BISCO Inc, Itasaca, IL 60143, USA), a commercial aqueous solution of HEMA, to dried dentin surfaces restored or increased the bond strength of acetone-based water-free dentin adhesives to the same level as bonding to moist substrates (Perdigão & others, 1998). The same phenomenon has been reported for ethanol-based, and ethanol- and water-based one-bottle systems (Perdigão & others, 1999).

A different approach to dentin pretreatment procedures is the mechanical alteration of dentin with air abrasive techniques prior to bonding procedures. Air abrasion, alone, significantly lowers the SBS of fourth and fifth generation dentin bonding agents. However, air abrasion with etching results in SBS values similar to the etched specimens (Los & Barkmeier, 1994; Rinaudo, Cochran & Moore, 1997).

This study evaluated the effect of two dentin disinfectants, an aqueous HEMA solution, a combination of dentin disinfectant and HEMA solution and an air abrasion treatment on the shear bond strengths of two acetone-based single bottle adhesives.

METHODS AND MATERIALS

For this study, 167 freshly extracted, caries-free human molar teeth were cleaned and stored in refrigerated tap water. Each tooth was mounted in a teflon cylinder filled with self-curing acrylic. The occlusal surfaces were ground flat to expose the dentin surface using a water-cooled 240 grit grinding disc followed by a 600 grit polishing disc (Ecomet 3, Buheler, Lake Bluff, IL 60044, USA).

The teeth were randomly assigned to two main groups according to the bonding agent used, either One Step (BISCO) (Group A) or Prime & Bond 2.1 (Dentsply/Caulk, Milford, DE 19963, USA) (Group B). The teeth were then radiographed and redivided into six sub-groups with similar distributions of remaining dentin thickness. Each group was assigned to a different treatment protocol.

The exposed dentin was etched with 35% phosphoric acid (Ultra-Etch 35%, Ultradent Products Inc, South Jordan, UT 84065, USA) for 20 seconds, washed with an air-water spray for 10 seconds, then briefly dried for 1-2 seconds with oil-free compressed air keeping the syringe tip 2 cm from the dentin surface. Pretreatment protocols were then applied as follows:

Subgroup 1: (Control) no further treatment.

Subgroup 2: 2% Chlorhexidine digluconate (Consepsis, Ultradent Products Inc) was gently rubbed on the dentin surface with a cotton wool pledget for 30 seconds, washed with a water jet for 10 seconds and gently air dried.

Subgroup 3: Tubulicid (Tubulicid Blue Label, Global Dental Products, North Bellmore, NY 11710, USA) containing 2% EDTA and 1% benzylkonium chloride was gently rubbed on the dentin with a cotton wool pledget for 10 seconds, gently air dried but not washed off (in accordance with the manufacturer's instructions).

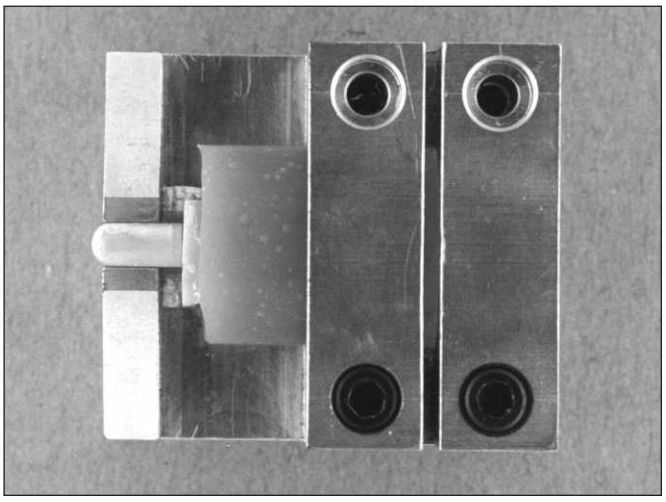

Figure 1. *Device to adhere the composite cylinder perpendicular to the dentin surface.*

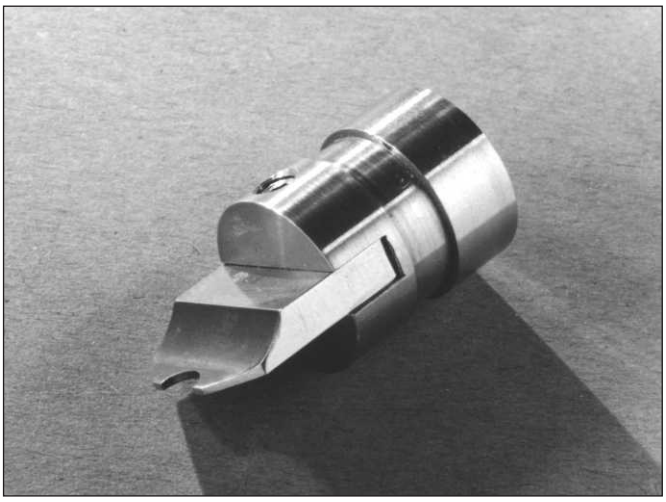

Figure 2. *Semicircular knife shearing device.*

Table 1: *Mean Shear Bond Strengths and SD of Bonding Agents Following Various Pretreatments of Dentin*

Pretreatment	Bonding Agent	Number of Specimens	Mean Shear Bond Strength (MPa)	SD
All	A+B	167	13.488	6.729
All	A	82	13.131	5.332
All	B	85	13.832	7.863
Control	A+B	28	10.832	5.865
Consepsis	A+B	28	15.444	6.194
Tubilicid	A+B	27	12.355	4.925
Aqua Prep	A+B	29	19.748	6.662
Tubilicid +Aqua Prep	A+B	26	12.628	6.149
Air abrasion	A+B	29	9.737	5.441
Control	A	13	11.806	4.339
Consepsis	A	14	17.819	3.276
Tubilicid	A	13	12.505	4.844
Aqua Prep	A	15	14.855	4.547
Tubilicid +Aqua Prep	A	13	11.930	6.256
Air abrasion	A	14	9.542	4.927
Control	B	15	9.970	6.966
Consepsis	B	14	13.069	7.537
Tubilicid	B	14	12.216	5.177
Aqua Prep	B	14	24.990	4.015
Tubilicid + Aqua Prep	B	13	13.327	6.211
Air abrasion	B	15	9.937	6.048

A = One Step; B = Prime & Bond 2.1

Subgroup 4: Aqua Prep (35% HEMA solution) (Aqua Prep, BISCO) was applied to the dentin with a disposable brush, left undisturbed for 20 seconds, then dispersed with a gentle air stream keeping the air-spray nozzle 2 cm from the dentin surface.

Subgroup 5: A mixture of Aqua Prep and Tubilicid (ratio 1:1) was applied for 20 seconds and gently dried with an air-spray at a distance of 2 cm.

Subgroup 6: The exposed dentin surface was air abraded with 50 µm aluminum oxide particles at an air pressure of 85 psi (Micro Etcher, Model ERL, Danville Engineering Inc, San Ramon, CA 94583, USA) for three seconds. The prepared surfaces were then rinsed with an air-water spray and dried with compressed air. Phosphoric acid (35%) was applied for 20 seconds, washed for 10 seconds and briefly air-dried. This group

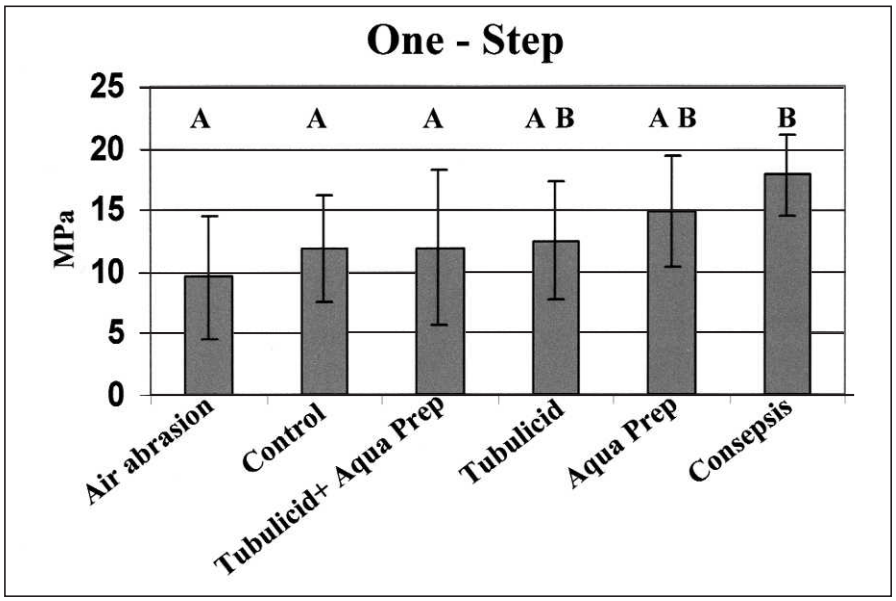

Figure 3. *Mean shear bond strength (MPa) of composite resin to dentin for One Step subgroups (letters refer to Tukey's post hoc ranking at* p<*0.05).*

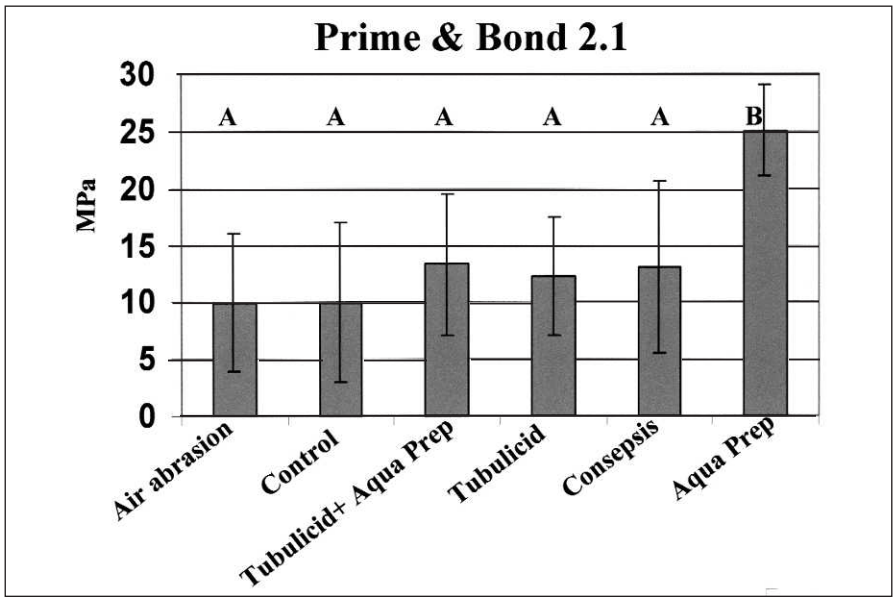

Figure 4. *Mean shear bond strength (MPa) of composite resin to dentin for Prime & Bond 2.1 subgroups (letters refer to Tukey's post hoc ranking at* p<*0.05).*

was exceptional, as etching was carried out only after air abrasion.

Following the experimental protocol, either One Step or Prime & Bond 2.1 was applied to the specimens according to the manufacturer's instructions.

In Group A, two layers of One Step were applied consecutively, gently dried with an air-spray for five seconds and light-cured for 10 seconds at a distance of approximately 2 mm with an Optilux 401 curing light (Demetron Research Corp, Danbury, CT 06810, USA). The curing light output was monitored with a Curing Radiometer (Demetron model #100) and found to be in excess of 400 mW/cm^2. An extra layer of One Step was placed as a wetting agent without curing.

In Group B, Prime & Bond 2.1 was applied for 20 seconds, dried with an air stream and light-cured for 10 seconds. Another layer was applied, dried and light cured.

A hybrid composite resin (Z-100, 3M Dental Products, St Paul, MN 55144, USA) was condensed into a #5 transparent gelatin capsule (Lock Ring Capsules, Torpac Inc, Fairfield, NJ 07004, USA), approximately two-thirds full and light-cured for 60 seconds. The remaining third of the capsule was filled, applied to the treated bonded dentin and seated securely perpendicular against the flattened tooth surface with a special device (Figure 1). Excess composite was removed from the capsule surface and the composite was light cured from each side for a further 40 seconds (total 160 seconds).

Specimens were thermocycled 1,000 times in a water bath for 10 seconds at 55°C and 5°C, then stored in tap water at 37°C for six weeks.

To measure the SBS of composite to dentin, specimens were mounted at right angles to a semi-circular knife-edge shearing device (Figure 2) on an Instron Universal Testing Machine (Model 4502, High Wycombe, Buckinghamshire, HP12 3SY, UK). A loading speed of 0.5 mm/minute was used until failure and the SBS were recorded in MPa units.

Data were analyzed using a one- and two-way ANOVA. The independent variables were surface treatments (n=6) and bonding agents (n=2). A Tukey HSD post-hoc test was used to identify significant differences between pairs of means at a confidence level of 95%.

RESULTS

The mean SBS for One Step ranged from 9.5 ± 4.9 MPa, (air abrasion) to 17.8 ± 3.2 (Consepsis). For Prime & Bond 2.1, SBS ranged from 9.9 ± 6.0 (air abrasion) to 24.9 ± 4.0 (Aqua Prep). All data are presented in Table 1 and Figures 3 and 4. Two-way ANOVA showed no significant difference for the variable bonding system (p=0.322), but there were significant differences for the

variable pretreatment protocol ($p<0.001$) as well as the interaction, bonding agent combined with pretreatment protocol ($p<0.001$). One-way ANOVA revealed significant differences ($p<0.01$) between the various pretreatment protocols for both One Step and Prime & Bond 2.1. The post-hoc Tukey HSD Test ranked the means at $p<0.05$ for One Step (Figure 3) and Prime & Bond 2.1 (Figure 4).

In Group A (One Step, Figure 3) Consepsis showed a significantly higher SBS than the subgroups Control ($p=0.02$), air abrasion ($p=0.001$) and a combination of Aqua Prep and Tubilicid ($p=0.02$), but a non-significant higher SBS than Aqua Prep alone ($p=0.55$) and Tubilicid alone ($p=0.053$). Other pretreatments did not differ significantly from the Control subgroup.

In Group B (Prime & Bond 2.1, Figure 4), dentin pretreated with Aqua Prep showed a significantly higher SBS ($p<0.001$) than all other subgroups.

DISCUSSION

After etching the dentin, maintaining a moist surface is advised to prevent collapse of the unsupported collagen and promote subsequent wetting and infiltration of the resin. This is mandatory with acetone-based bonding systems. Bonding agents dissolved in acetone have a higher SBS to moist dentin than to a dry dentin (Kanca, 1992; Jacobsen & Soderholm, 1998). Finger & Uno (1996) report similar findings. They suggest that moist dentin keeps a porous collagen framework from collapsing, thus allowing it to be more readily penetrated by the hydrophilic primer, particularly when dissolved in acetone. The acetone displaces the water, and upon its evaporation, monomer occupies the interfibrillar spaces where it polymerizes to create a greater interfacial bond strength.

A certain degree of gentle drying after washing the etchant, either by blotting dry or by a short (1-2 seconds) burst of air is necessary to avoid the "overwet" phenomenon (Tay, Gwinnett & Wei, 1996). Excess water within the dentinal tubules and at their openings on the dentin surface severely hinder the infiltration of intratubular resin. Blister-like spaces on the dentin surface and globules around the tubular orifices cause deterioration in continuity of the resin layer (Tay & others, 1996). However, it is difficult to control wetness of the dentin surface in complex cavities. Therefore, there is a risk that large variations in surface moisture exist clinically which may result in variations in bond strength at different locations. Use of a re-wetting agent may render the procedure less technique-sensitive (Jacobsen & Soderholm, 1998).

When dentin is dried after etching and washing, re-wetting the dentin with water partially restores the plasticity and permeability of the demineralized collagen fibrillar network. Such a recovery, although not complete, appears adequate for the interfibrillar spaces to reopen for resin infiltration (Tay, Gwinnett & Wei, 1997). Carvalho & others (1996) have shown that air-dried dentin specimens shrink to a residual volume of 33.7% of their original demineralized volume. When immersed in water, they completely revert to their original dimensions.

An effective re-wetting agent is 35% HEMA in water. Together with the plasticizing effect of water on the collapsed collagen fibers, HEMA may act as a stiffening agent to prevent any subsequent shrinkage (Perdigão & others, 1999). HEMA may slow down water evaporation from the dentin surface by bonding to water molecules through hydrogen bonds (Tay & others, 1997). A mechanical interlocking of HEMA into the demineralized dentin web by hygroscopic expansion after polymerization has also been described (Schumacher, Eichmiller & Antonucci, 1992). In this study, Aqua Prep (35% HEMA) groups yielded the highest SBS values (mean 19.75 MPa). However, this effect was more pronounced with Prime & Bond 2.1 (25.0 MPa) than with One Step (14.85 MPa). A possible explanation is that One Step contains HEMA and, therefore, benefited slightly (14.8 vs 11.8 MPa in the Control) from the re-wetting step, whereas Prime & Bond 2.1 contains no HEMA, and thus benefited greatly (25.0 vs. 9.9 MPa in the Control). Another explanation is that the bonding mechanism of Prime & Bond 2.1 may primarily rely on its penetration into the intertubular dentin, making it very sensitive to the degree of air drying of the etched dentin. Air drying etched dentin decreased the bond strength of Prime & Bond 2.1 to half of that obtained to moist dentin (Barkmeier, Hammesfahr & Latta, 1999; Perdigão & others, 1999). Although air dried for 1-2 seconds, the Control group of Prime & Bond 2.1 in this study was probably too dry and thus yielded low SBS values (9.9 MPa). These results are in accordance with Perdigão & others (1998), who report that SBS of Prime & Bond 2.1 bonded to dentin re-wetted with Aqua Prep improved nearly 100% as compared to moist or dried (one second) dentin.

The Consepsis (2% chlorhexidine) groups also benefited from the re-wetting procedure (mean 15.4 MPa). In previous studies, another agent, Cavity Cleanser (BISCO) containing 2% chlorhexidine, was applied either before (Meiers & Kresin, 1996) or after (Perdigão & others, 1994) the etching process. Perdigão & others (1994) used the All-Bond 2 adhesive system (BISCO) and found that pretreatment with chlorhexidine had no significant effect on SBS of composite to dentin. However, as the chlorhexidine was not washed off the dentin, debris remaining on the dentin surface and in the tubules may account for this result. Meiers & Kresin (1996) found that cavity washing with 2% chlorhexidine did not affect SBS or microleak-

age of composite resins. Since chlorhexidine was applied before etching, its effect on bond strength values could have been neutralized by the etching process. In this study, the approach was to gently rub chlorhexidine on dentin after the etching process, then wash it away. Thus, the benefit of chlorhexidine was gained without interference of the washed-away debris. A second explanation involves the difference in composition of the materials. The manufacturer declined to reveal the exact composition of Consepsis except that in addition to 2% chlorhexidine, it contains a surfactant to reduce surface tension and allow better wetting. Chlorhexidine has a strong affinity for tooth surfaces (Nordbo, 1972). It is possible that the surfactant caused more chlorhexidine molecules to bind to the tooth structure. A water jet might only partially drive away these chlorhexidine molecules and, therefore, the bound molecules might serve as a co-surfactant on the wet acid-conditioned dentin before resin is applied. This would allow the bifunctional monomer HEMA to more fully saturate the collagen network. One Step, which contains HEMA, benefited more (17.8 MPa) than Prime & Bond 2.1 (13.0 MPa).

Pretreatment with Tubilicid showed only slightly higher SBS values than the Control group. Values for One Step (12.50 MPa) and Prime & Bond 2.1 (12.21 MPa) were almost identical but not statistically significantly different from the Control group. Tubilicid partially removes the smear layer, leaving the dentinal plugs undisturbed (Surmont & others, 1989). Since removing the smear layer in its entirety is accomplished by etching before applying the disinfecting agent, it has no effect on bond strength values (Surmont & others, 1989; Gwinnett, 1992). Benzylkonium chloride is known to cross-link to collagen but does not impair hybridization (Gwinnett, 1992). The primary positive effect of Tubulicid is its bactericidal properties, and it could also conserve moisture on the dentin surface since the manufacturer advises that it should not be washed off the dentin. A combined use of Tubilicid and Consepsis yielded bond strength values very similar to Tubilicid. Benzylkonium chloride apparently masked the positive effect of HEMA observed when Consepsis, alone, was applied.

The air abrasion group was exceptional, as the air abrasion was carried out before etching. It showed the lowest average SBS of all pretreatment groups and was similar to the Control group. This is in accordance with Rinaudo & others (1997), who reported that air abrasion, alone, significantly lowered the SBS of dentin bonding agents. However, air abrasion plus etching results in SBS similar to the etched specimens. Los & Barkmeier (1994) and Roeder & others (1995) have demonstrated that air abrasion does not enhance the SBS of fourth generation dentin bonding agents. It appears that with the availability of the current bonding agents, using air abrasion on dentin is superfluous.

CONCLUSIONS

With the exception of air abrasion, pretreatment of the etched dentin with either a disinfectant or re-wetting agents may have a positive effect on the shear bond strength of resin to dentin, but this effect is bonding-agent specific. An aqueous HEMA solution was the most effective material and could render the bonding procedure less technique-sensitive.

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Surface Roughness Assessment of Resin-Based Materials During Brushing Preceded by pH-Cycling Simulations

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Clinical Relevance

Depending on the restorative material, brushing under a pH-cycling condition provided a remarkable initial increase in surface roughness which, thereafter remained unaltered or showed further increase.

SUMMARY

This study evaluated the surface roughness pattern of resin-based restorative materials during brushing preceded by a regimen that simulated a dynamic pH-cycling. Restoratives included two resin composites (Renamel Microfill and Charisma), two polyacid-modified composite resins (Compoglass-F and Dyract AP) and one resin-modified glass ionomer cement (Fuji II LC Improved). Twenty standardized cylindrical specimens of each material were prepared according to a randomized complete block design. After finishing and polishing, the average surface roughness (Ra) and profile-length ratio (LR) of the specimens were determined. The experimental units were subjected to a pH-cycling regimen, and then to 10,000 brushing strokes. New readings of both the Ra and LR parameters were obtained. The same protocol of pH-cycling, brushing simulation and surface roughness measurements were repeated 10 times. Data was analyzed using ANOVA according to split-plot design and Tukey's test. Results showed the pH-cycling followed by 10,000 brushing strokes provided a remarkable increase in Ra for all restorative materials except for Renamel. Throughout the brushing simulation, Renamel, Charisma, Compoglass-F and Dyract AP showed steady textures, whereas Fuji II LC Improved exhibited a progressive increase in surface roughness. Among the materials tested, Renamel presented the smoothest surface, followed by Charisma and Compoglass-F, which did not differ from each other except at the baseline. Dyract AP was different from both these materials except at

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the baseline. Fuji II LC Improved had the roughest surface texture.

INTRODUCTION

Long-term clinical performance of restorative materials may be impaired by a number of factors, including degradation in the oral environment (Øilo, 1992). Wear processes, including diverse phenomena such as sliding, abrasion, chemical degradation and fatigue (Söderholm & Richards, 1998), contribute to this breakdown. The outcome of these phenomena may be submargination (Gladys & others, 1997; Browning, Brackett & Gilpatrick, 2000) and changes in surface roughness (Sulong & Aziz, 1990; Sidhu, Sherriff & Watson, 1997). As a consequence, restorative materials may be a source of increased plaque retention and gingival irritation (Dunkin & Chambers, 1983). Moreover, these restorations may become susceptible to staining (Hachiya & others, 1984).

Among the wear processes, toothbrushing abrasion constitutes an important consideration in non-stress-bearing areas, such as cervical locations (Asmussen, 1985). Hence, considering that resin-based restoratives are often the materials of choice for restorations of carious and noncarious lesions located in these surfaces, studies to predict their clinical performance (Heath & Wilson, 1977; Goldstein & Lerner, 1991; Frazier, Rueggeberg & Mettenburg, 1998) have been undertaken. Various material-related features, such as their inter-particle filler spacing, filler particle size and the degree of cure of the resin, have been reported to influence resistance to abrasion (Draughn & Harrison, 1978; Bayne, Taylor & Heymann, 1992; Söderholm & Richards, 1998). Furthermore, direction and magnitude of acting forces and time may also be strongly related to the abrasion process (Milleding & others, 1998). On the other hand, certain physical characteristics of abrasive particles, including their size, hardness and shape, have been shown to have a pronounced effect on their ability to wear surfaces (Mair & others, 1996).

In addition to abrasive processes, numerous factors in the oral cavity, such as low pH due to cariogenic micro-organisms or acidic food, ionic composition and ionic strength of the saliva, are important parameters that may play a role in the physical and mechanical characteristics of a restorative material (Geurtsen, Leyhausen & García-Godoy, 1999). The chemical environment is, therefore, one aspect of the oral cavity that can have an appreciable influence on the *in vivo* wear process of resin-based restoratives (Roulet & Wälti, 1984; van Groeningen, Jongebloed & Arends, 1986). Only very limited information, however, about the abrasion resistance of these materials under conditions presumably encountered in the mouth is available (Attin & others, 1998).

Considering these facts, it seems important to not only compare the performance of different restorative materials, but also to estimate their long-term behavior. Hence, this study assessed the surface roughness pattern of resin-based materials throughout brushing under a condition that simulated a dynamic pH-cycling.

Table 1: *Resin-Based Materials Under Investigation and Their Technical Profiles*

Material	Manufacturer	Type	Basic Composition*	Filler size (μm)*	Filler Content (% by volume)*	Batch #
Renamel	Cosmedent Inc Chicago, IL 60640 USA	microfilled resin composite	Pyrogenic silic acid; highly molecular multi-functional methacrylate ester	0.02-0.04	80	993460 G
Charisma	Heraus Kulzer GmbH, Wehrheim, Germany D-6 1723	hybrid resin composite	Barium aluminum fluoride glass; highly dispersive siliciumdioxide; bisGMA	0.02-2.0	64	74
Compoglass-F	Vivadent Ets, Liechtenstein, Germany FL-9494	polyacid-modified resin composite	YF3, Ba-Al-fluorosilicate glass; bisGMA, UDMA; TEGDMA; cyclo-aliphatic dicarboxylic acid dimethacrylate	0.2-3.0	55	B0069
Dyract AP	Dentsply De Trey GmbH, Konstanz, Germany 78467	polyacid-modified resin composite	Strontium-AL-Na-fluoro-P-silicate-glass; strontium fluoride; UDMA; TCB resin; highly cross-linking methacrylate-monomer	0.8	47	9904001505
Fuji II LC	GC America Inc Tokyo, Japan 174	resin-modified glass-ionomer cement	Al, fluorosilicate glass; HEMA; tataric acid; poly-acrylic acid, water	0.1-25	60	Powder: 120191 Liquid: 060191

*As disclosed by the manufacturers
BisGMA = Bisphenol-A-glycidyl methacrylate; UDMA = Urethane dimethacrylate; TEGDMA = Triethylene glycol dimethacrylate; HEMA = Hydroxyethyl methacrylate

METHODS AND MATERIALS

Experimental Design

This study observed surface roughness as a response variable in relation to two factors: (1) restorative materials and (2) pH-cycling followed by brushing strokes. The average roughness (Ra) and the profile-length ratio (LR) values were taken from each experimental unit and evaluated separately. The restorative materials factor was taken into five levels (Renamel; Charisma; Compoglass-F; Dyract AP; and Fuji II LC Improved) and pH-cycling followed by brushing strokes into 11 levels (baseline; 10,000; 20,000; 30,000; 40,000; 50,000; 60,000; 70,000; 80,000; 90,000 and 100,000). This study used 20 experimental units for each restorative material made in 10 blocks with two replicates each. The randomized, complete block design was used to reduce the experimental error arising from known and controlled nuisance sources of variability (Montgomery, 1991). The split-plot design was employed, supported by repeated measurements taken from the same experimental unit at different pH-cycling followed by brushing stroke levels.

Specimen Preparation

Table 1 lists the restorative materials used in this study, together with other information on their basic composition, particle size and filler content. Materials were handled according to manufacturers' instructions and inserted into stainless-steel matrixes of internal dimensions of 4 mm diameter by 2 mm thickness. A Centrix syringe (Centrix Inc, Shelton, CT, 06484, USA) was used to insert Fuji II LC Improved, whereas syringes supplied by the manufacturers were employed for Dyract AP and for Compoglass-F and a metal spatula was used for the other materials. The surface of the restorative materials was covered with a polyester strip (Probem Ltda, Catanduva, SP, Brazil, 15800-000) that was pressed using a glass slide with a load of 500 g for 30 seconds to remove the excess material. The restoratives were then polymerized for the recommended exposure times through the polyester strip with a light unit (Optilux 401, Demetron/Kerr Corp, Danbury, CT 06810, USA). The output from the curing light was periodically monitored using a light meter (Curing Radiometer, Model 100, Demetron/Kerr Corp) and ranged from 400 to 520 mW/cm^2.

After polymerization, specimens were individually stored for 24 hours at 37(±1)°C at 100% relative humidity. Thereafter, samples were finished and polished using medium, fine and superfine aluminum oxide abrasive disks (Sof-Lex Pop On, 3M Dental Products, St Paul, MN 55144, USA). Each instrument was applied in a single direction for 15 seconds. Following each finishing and polishing step, specimens were flushed with air-water spray. Samples were then ultrasonically cleaned (Model T1440D, Odontobrás Ltda, Ribeirão Preto, SP, Brazil) in distilled-deionized water for 10 minutes to remove polishing debris and stored at 100% relative humidity.

Baseline Surface Roughness Measurements

Each sample was gently dabbed dry with absorbent paper and the surface roughness analyses conducted using the Surfcorder SE1700 surface roughness measuring instrument equipped with a diamond needle of 2 µm radius (Kosaka Corp, Tokyo, Japan). To record roughness measurements, the needle moved at a constant speed of 0.05 mm/second with a force of 0.7 mN. The cut-off value was set at 0.08 mm to maximize filtration of surface waviness. The surface roughness was characterized by the average roughness (Ra) and by the profile-length ratio (LR). Ra is the arithmetical average value of all absolute distances of the roughness profile from the centerline within the measuring length. LR is defined as the ratio between the true profile length, that is, the length of the profile being drawn out into a straight line, and the measuring distance. An ideal, smooth surface has an LR value of 1; the rougher the surface becomes, the greater the LR-value will be. Three traces were recorded on each specimen at three different locations in each direction—parallel, perpendicular and oblique to the finishing and polishing scratch directions—amounting to nine tracings per sample. The average of these nine mean surface roughness measurements was used as the score for each sample.

pH-cycling Protocol

The specimens were subjected to a pH-cycling regimen, as proposed by Featherstone & others (1986) and modified by Serra & Cury (1992). The samples were immersed in 5 ml of demineralizing solution for six hours at 37(±1)°C, followed by rinsing with distilled-deionized water and storage in 5ml of remineralizing solution (artificial saliva) for 18 hours at 37(±1)°C. The artificial saliva consisted of 1.5 mM of calcium, 0.9 mM of phosphate and 150 mM of potassium chloride in a buffer solution of 20 mM of Tris (hydroxymethyl-aminomethane) at pH 7.0. The acid solution contained 2.0 mM of calcium and 2.0 mM of phosphate in a buffer solution of 74.0 mM of acetate at pH 4.3. After this protocol, the specimens were rinsed with distilled-deionized water and stored at 37(±1)°C in 100% relative humidity.

Brushing Abrasion Protocol

Brushing abrasion of the specimens was performed with an automatic toothbrushing abrasion testing machine (Marcelo Nucci, São Carlos, SP, Brazil) with a motor that produced a reciprocating motion on 10 soft nylon bristle toothbrush heads (Oral-B Indicator 40, Gillette do Brasil Ltda, Manaus, AM, Brazil) in a thermostatically-controlled environment at 37(±0.5)°C. The experimental units were aligned so that the brushing heads moved parallel to their surfaces. Each toothbrushing head was loaded with a 300g weight and trav-

Table 2: *Mean and Standard Deviations for Ra (Average Roughness), Showing the Results From the Tukey's Test*

Condition	Renamel	Charisma	Compoglass-F	Dyract AP	Fuji II LC Improved
Baseline	0.0270 (0.0036)[a]	0.0517 (0.0046)[b]	0.0723 (0.0053)[c]	0.0650 (0.0084)[bc]	0.1747 (0.0100)[d]
10,000 brushing strokes	0.0390 (0.0036)[a]	0.0914 (0.0062)[b]	0.0952 (0.0062)[b]	0.1433 (0.0078)[c]	0.3216 (0.0228)[d]
20,000 brushing strokes	0.0407 (0.0024)[a]	0.0944 (0.0049)[b]	0.0937 (0.0087)[b]	0.1459 (0.0072)[c]	0.3774 (0.0252)[d]
30,000 brushing strokes	0.0429 (0.0036)[a]	0.0959 (0.0050)[b]	0.0945 (0.0062)[b]	0.1499 (0.0061)[c]	0.3968 (0.0324)[d]
40,000 brushing strokes	0.0449 (0.0108)[a]	0.0923 (0.0043)[b]	0.0930 (0.0037)[b]	0.1494 (0.0116)[c]	0.3922 (0.0855)[d]
50,000 brushing strokes	0.0428 (0.0024)[a]	0.0927 (0.0039)[b]	0.0895 (0.0055)[b]	0.1473 (0.0072)[c]	0.4310 (0.0379)[d]
60,000 brushing strokes	0.0447 (0.0027)[a]	0.0911 (0.0042)[b]	0.0913 (0.0063)[b]	0.1527 (0.0049)[c]	0.4367 (0.0255)[d]
70,000 brushing strokes	0.0485 (0.0036)[a]	0.0900 (0.0031)[b]	0.0916 (0.0048)[b]	0.1543 (0.0059)[c]	0.4624 (0.0281)[d]
80,000 brushing strokes	0.0528 (0.0045)[a]	0.0924 (0.0048)[b]	0.0961 (0.0080)[b]	0.1575 (0.0057)[c]	0.4750 (0.0178)[d]
90,000 brushing strokes	0.0524 (0.0043)[a]	0.0943 (0.0048)[b]	0.0948 (0.0061)[b]	0.1555 (0.0067)[c]	0.4768 (0.0270)[d]
100,000 brushing strokes	0.0523 (0.0030)[a]	0.0922 (0.0061)[b]	0.0950 (0.0073)[b]	0.1583 (0.0059)[c]	0.4700 (0.0226)[d]

Standard deviations are given between parentheses

Means connected by vertical brackets did not differ from each other. Values with the same superscript letter were not statistically different (a=0.05) by row

eled horizontally for 20 mm at a speed of 4.5 strokes per second. Specimens were brushed with 10,000 strokes. An abrasive slurry was prepared by mixing dentifrice (Colgate MFP, Colgate Palmolive—Division of Kolynos do Brasil Ltda, Osasco, SP, Brazil) and distilled-deionized water at a ratio of 1:3 by weight, respectively, which was independently injected beside each brush at a frequency of 0.4 ml at two-minute intervals. By means of this intermittent regimen of injection, it was possible to reduce sedimentation of the abrasive and avoid a decrease in the amount of slurry.

After testing, specimens were removed, rinsed with tap water and ultrasonically cleaned in distilled-deionized water for 10 minutes. Samples were again stored at 100% relative humidity and data was collected using the surface roughness instrument as previously described.

Repeated Measurements Throughout the pH-cycling Followed by Brushing Strokes

The same protocols of pH-cycling and brushing simulation were subsequently repeated 10 times. After every pH-cycling followed by 10,000 strokes, the specimens were again subjected to surface roughness analysis. Data were also obtained after 20,000; 30,000; 40,000; 50,000; 60,000; 70,000; 80,000; 90,000 and 100,000 brushing strokes. The brush heads were replaced after simulating 50,000 brushing strokes.

Statistical Analysis

The statistical evaluation of the data was made by Analysis of Variance, according to split-plot design, followed by Tukey's test to perform pairwise comparisons between restorative materials and pH-cycling followed by brushing strokes at the level of 5% of significance. The regression method was used to fit a mathematical function of surface roughness (dependent variable) by pH-cycling followed by brushing strokes (independent variable) using a quartic-order additive model. Statistical analysis were performed by Statgraphics Plus (Manugistics, Rockville, MD 20852, USA).

RESULTS

The Analysis of Variance revealed significant effect for restorative materials, pH-cycling followed by

Table 3: *Mean and Sandard Deviations for LR (Profile-Length Ratio), Showing the Results From the Tukey's Test*

Condition	Renamel	Charisma	Compoglass-F	Dyract AP	Fuji II LC Improved
Baseline	1.0031 (0.0009)[a]	1.0040 (0.0012)[ab]	1.0051 (0.0023)[b]	1.0047 (0.0010)[ab]	1.0078 (0.0021)[c]
10,000 brushing strokes	1.0028 (0.0007)[a]	1.0070 (0.0013)[b]	1.0067 (0.0016)[b]	1.0092 (0.0015)[c]	1.0218 (0.0028)[d]
20,000 brushing strokes	1.0031 (0.0010)[a]	1.0072 (0.0015)[b]	1.0058 (0.0014)[b]	1.0106 (0.0018)[c]	1.0259 (0.0029)[d]
30,000 brushing strokes	1.0029 (0.0011)[a]	1.0077 (0.0011)[b]	1.0062 (0.0017)[b]	1.0103 (0.0017)[c]	1.0279 (0.0021)[d]
40,000 brushing strokes	1.0027 (0.0011)[a]	1.0074 (0.0020)[b]	1.0057 (0.0012)[b]	1.0104 (0.0018)[c]	1.0265 (0.0067)[d]
50,000 brushing strokes	1.0029 (0.0009)[a]	1.0070 (0.0012)[b]	1.0058 (0.0011)[b]	1.0103 (0.0021)[c]	1.0289 (0.0028)[d]
60,000 brushing strokes	1.0023 (0.0006)[a]	1.0062 (0.0006)[b]	1.0054 (0.0015)[b]	1.0103 (0.0013)[c]	1.0297 (0.0040)[d]
70,000 brushing strokes	1.0023 (0.0007)[a]	1.0062 (0.0006)[b]	1.0049 (0.0009)[b]	1.0101 (0.0014)[c]	1.0303 (0.0021)[d]
80,000 brushing strokes	1.0030 (0.0017)[a]	1.0075 (0.0023)[b]	1.0063 (0.0022)[b]	1.0110 (0.0024)[c]	1.0301 (0.0032)[d]
90,000 brushing strokes	1.0029 (0.0008)[a]	1.0062 (0.0006)[b]	1.0049 (0.0009)[b]	1.0099 (0.0010)[c]	1.0308 (0.0021)[d]
100,000 brushing strokes	1.0029 (0.0008)[a]	1.0075 (0.0023)[b]	1.0063 (0.0022)[b]	1.0108 (0.0014)[c]	1.0297 (0.0037)[d]

Standard deviations are given between parentheses

Means connected by vertical brackets did not differ from each other. Values with the same superscript letter were not statistically different (a=0.05) by row

brushing strokes and for the interaction between these factors for both Ra and LR response variables. Since the interaction was significant for each response variable, comparison of the different materials at baseline and within each 10,000 brushing strokes could be made. Tables 2 and 3 show the mean and standard deviation for Ra and for LR, respectively.

Figures 1 and 2, respectively, show the surface roughness pattern for both Ra and LR as a function of pH-cycling followed by brushing strokes. This was done by fitting the data according to a quartic-order mathematical equation. In regards to Ra, an increase in surface roughness for every restorative material except Renamel was shown after pH-cycling followed by 10,000 brushing strokes (Table 2 and Figure 1). Throughout the brushing simulation, that is, from 10,000 to 100,000 strokes, Renamel, Charisma, Compoglass-F and Dyract AP showed steady textures, whereas Fuji II LC Improved exhibited a progressive increase in surface roughness interspersed with periods of stabilization. Comparing the restorative materials within each level of the pH-cycling followed by brushing strokes factor (comparisons by rows in Table 2), Renamel presented the smoothest surface, followed by Charisma and Compoglass-F, which did not differ from each other except at the baseline. Dyract AP was different from both materials, except at the baseline. Fuji II LC Improved statistically had the roughest surface texture.

Regarding the LR response in each restorative material, a remarkable increase in surface roughness for Charisma, Dyract AP and Fuji II LC Improved was observed, whereas Renamel and Compoglass-F showed a steady state throughout the entire experimental period in relation to the baseline (Figure 2). Considering the levels of the pH-cycling followed by brushing strokes factor shown in Table 3, there were no statistical differences among Renamel, Charisma, Dyract AP and Compoglass-F, except when Compoglass-F was compared to Renamel.

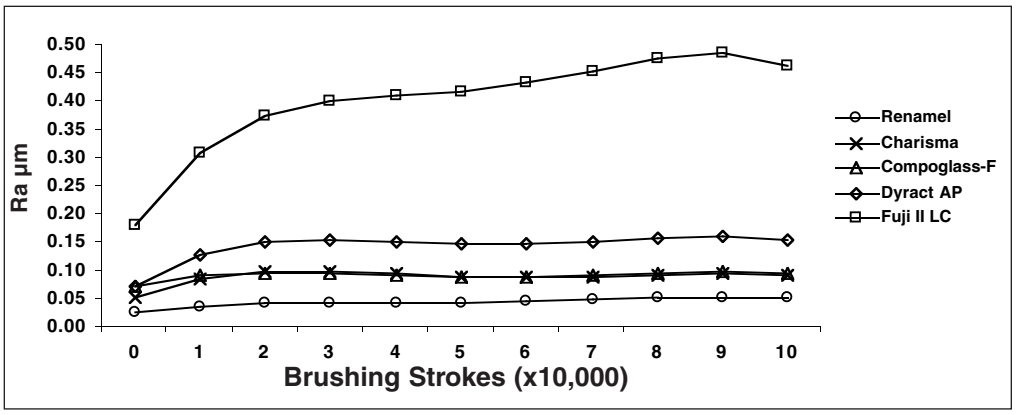

Figure 1. *Ra (average roughness) response of the different materials as a function of pH-cycling followed by brushing strokes. The experimental data were fitted according to a quartic-order mathematical equation.*

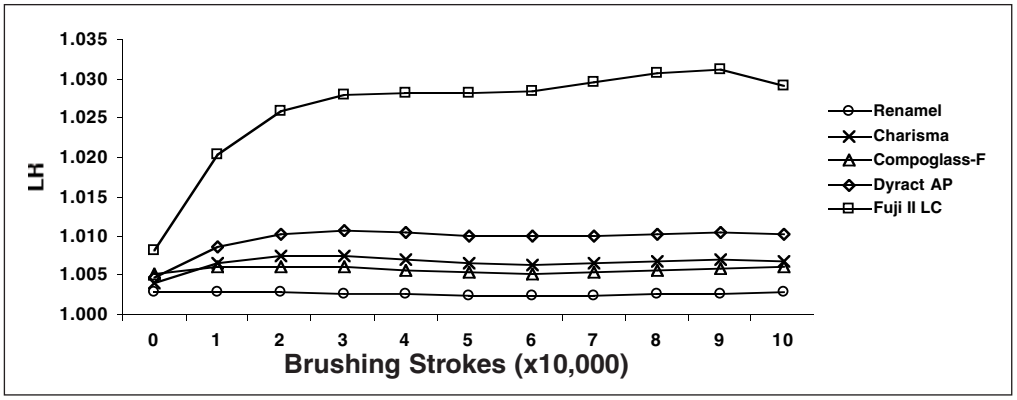

Figure 2. *LR (profile-length ratio) response of the different materials as a function of pH-cycling followed by brushing strokes. The experimental data were fitted according to a quartic-order mathematical equation.*

From the first pH-cycling followed by 10,000 brushing strokes to the last, Renamel statistically showed the smoothest surface; Charisma and Compoglass-F did not differ from each other; Dyract AP was rougher than both Charisma and Compoglass-F and Fuji II LC Improved showed the greatest roughness compared to any material, since the baseline.

DISCUSSION

Based on studies that examined various behavioral aspects of adult toothbrushing (Heath & Wilson, 1974; van der Weijden & others, 1996), attempts to develop *in vitro* abrasion tests intended to predict clinical abrasion reliability were made. In this study, factors previously described, such as brushing load, stroke rate (Heath & Wilson, 1974), temperature and schedule in which the test may simulate more closely oral conditions (Heath & Wilson, 1977), were incorporated into the abrasion procedure.

In previous laboratory studies, the load applied to the toothbrushes during brushing amounted to 100-576 g (Montes-G & Draughn, 1986; Hotta & Hirukawa, 1994; Wülknitz, 1997; Attin & others, 1998; Kawai, Iwami & Ebisu, 1998; Tanoue, Matsumura & Atsuta, 2000b). Considering this wide variation, the load chosen in this investigation—300 g—simulated a medium brushing force. Moreover, there was controversy over the number of strokes used to simulate one year's brushing. Numbers ranged from 4,320 (Kanter, Koski & Martin, 1982) to 16,000 (Aker, 1982). In this study, assigning 10,000 brushing strokes, the equivalent to one year of brushing in a clinical situation, was based on Goldstein & Lerner (1991).

The abrasion resistance of resin-based restorative materials to brushing has been evaluated by various methods, such as surface roughness (Kanter & others, 1982; de Gee, ten Harkel-Hagenaar & Davidson, 1985; Goldstein & Lerner, 1991; Whitehead & others, 1996; Gladys & others, 1997; Momoi & others, 1997; Tanoue & others, 2000b), profilometrical tracings (Heath & Wilson, 1976; de Gee & others, 1985; Goldstein & Lerner, 1991; Momoi & others, 1997, Attin & others, 1998; Tanoue & others, 2000b), weight loss (Aker, 1982; Kanter & others, 1982; Kaway & others, 1998; Frazier & others, 1998) and photomicrographs (Draughn & Harrison, 1978; Ehrnford, 1983). In this study, surface roughness assessment was chosen because it has been documented that surface texture can play a role in bacterial colonization of restorative materials (Dunkin & Chambers, 1983). Although contradictory results have been reported in the literature regarding the effect of surface properties on these phenomena (Quirynen & Bollen, 1995), adherence and metabolic activities of micro-organisms in the mouth are well known to be the primary causes of a variety of conditions including dental caries and inflammatory diseases of gingival and periodontal tissues (Bollen, Lambrechts & Quirynen, 1997).

Although the arithmetical mean of absolute values of profile departures within the evaluation length (Ra) is the most common roughness parameter used to describe surface texture (Sidhu & others, 1997), roughness height has been advocated to be merely one esti-

mator of surface quality (Nowicki, 1985). As stated by Jung (1997), the horizontal aspect of roughness remains largely unconsidered. In order to deal with this limitation, the same author has proposed the measure of the profile-length ratio (LR), which takes into account the vertical and horizontal dimensions of roughness at the same time. For this reason, Ra and LR parameters were recorded.

In addition to brushing simulation, the specimens in this investigation were subjected to pH cycles comprising alternating storage in de- and remineralizing solutions. Although this model has been introduced to simulate the caries process in cariology research, it incorporates a severe acid challenge at a pH value that has been reported to occur *in vivo* (Featherstone & others, 1986). For this reason, it was used with the intent to mimic chemical processes or dissolution already reported to occur in the mouth (Roulet & Wälti, 1984; van Groeningen & others, 1986).

Regarding the performance of restorative materials, the microfilled composite, Renamel, showed the lowest surface roughness, followed by Charisma. These findings might be ascribed to the filler particles size of these materials, which is lower for Renamel, as shown in Table 1. The presence of small fillers has been suspected to result in decreased interparticle space and reduced wear (Söderholm & Richards, 1998). Throughout the brushing strokes preceded by pH-cycling, the hybrid composite and polyacid-modified composite resin Compoglass-F were not different from each other. It was also hypothesized that although Compoglass-F was less filled, it was as smooth as Charisma, probably due to the presence of a wide range of small particles. These findings might be explained by the presence of smaller filler particles in both composites, as showed in Table 1, which would result in decreased interparticle space and reduced wear (Söderholm & Richards, 1998). Although Compoglass-F and Dyract AP are both polyacid-modified composite resins, they differ significantly during brushing. This is probably due to dissimilarities in their microstructure. Fuji II LC Improved was rougher than the other materials under evaluation. Unlike polyacid-modified resin composites, the coherence between the cross-linked polyacrylate network and the polymer chain of resin-modified glass ionomers seems insufficient (Kanchanavasita, Anstice & Pearson, 1998). Moreover, in an aqueous environment, this material may take up great amounts of water, swell, become plastic and mechanically less resistant than other resin-based materials (Meyer, Cattani-Lorente & Dupuis, 1998; Cattani-Lorente & others, 1999).

Although dissimilarity in surface roughness of materials may mainly be attributable to the differences in their size and content of filler particles, these restoratives differ in many other ways, for example, type of fillers, degree of conversion of the polymer matrix and silane coupler, which may also influence their abrasion resistance (Jaarda, Wang & Lang, 1997; Kawas & others, 1998; Tanoue, Matsumura & Atsuta, 2000a).

Showing the restorative materials as a function of pH-cycling followed by brushing strokes was thought to best demonstrate the process. For response variables Ra and LR, the best approximations of how these restoratives work were obtained when a quartic-order mathematical equation was used. With Ra, the surface roughness of all restorative materials increased after the first 10,000 brushing simulations except for Renamel. Throughout the pH-cycling followed by brushing strokes, Renamel, Charisma, Compoglass-F and Dyract AP exhibited steady textures. These findings may be attributed to the difference between the abrasion of the matrix and filler particles. This discrepancy is large enough to provide an anisotropic degradation, which gradually slows down as the filler becomes exposed to the surface (de Gee & others, 1985). Fuji II LC Improved exhibited a progressive increase in surface roughness, interspersed with stable periods. One possible explanation is the deficient coherence between the matrix and the fillers of this material, which may cause exfoliation of some particles as the matrix is worn away (Aker; 1982; Condon & Ferracane, 1996).

When evaluating LR throughout the experimental period, both Renamel and Compoglass-F showed steady surface textures. Since the profile-length ratio takes into account the vertical and horizontal dimensions of roughness simultaneously, the overall aspect of the microstructure of these materials has likely remained unconsidered when recording Ra values. However, under the conditions adopted in this study, it may be inferred that the surface roughness description may be determined on the basis of Ra. This is based on the consideration that after 10,000 brushing strokes preceded by pH-cycling, the Ra and LR results were very similar and a lack of equipment suitable for its direct measurement exists (Nowicki, 1985).

In laboratory-based experiments, the inherent complexity of the oral environment is disregarded so as to highlight the main factor in analysis. To evaluate the surface roughness pattern of resin-based materials during brushing in this investigation, an approach that only simulated an abrasive wear preceded by a dynamic pH-cycling was considered. Not included were other aspects, such as thermal stress (Montes-G & Draughn, 1986; Sulong & Aziz, 1990) and cuspal flexure resulting from occlusal loading (Rees & Jacobsen, 1998), which may alter the process of restoration wear located in cervical areas. Therefore, *in vitro* models may not necessarily provide a full, realistic indication of what occurs in the mouth. However, considering the high turnover of new restorative materials, this and

other laboratory studies for predicting the behavior of dental restorations, are important.

CONCLUSIONS

Under this study's conditions, it may be concluded that:

1. pH-cycling followed by brushing provided a remarkable increase in Ra for all restorative materials strokes except for Renamel after 10,000 brushing.
2. Throughout the brushing strokes preceded by pH-cycling, Renamel, Charisma, Compoglass-F and Dyract AP showed steady textures, whereas Fuji II LC Improved exhibited a progressive increase in surface roughness.
3. Among the materials tested, Renamel presented the smoothest surface, followed by Charisma and Compoglass-F, which did not differ from each other, except at baseline. Dyract AP was different from both these materials except at the baseline. Fuji II LC Improved had the roughest surface texture.

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Depth of Cure and Marginal Adaptation to Dentin of Xenon Lamp Polymerized Resin Composites

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Clinical Relevance

The high-powered xenon lamp unit is clinically effective in the polymerization of visible light-cured resin composites.

SUMMARY

Marginal adaptation of four resin composites (Clearfil APX, Estelite, Silux Plus and Z-100) cured with two xenon lamp units (Plasma Arc Curing System or Apollo 95E) or a halogen lamp unit (Witelite) were evaluated by measuring the wall-to-wall contraction gap width. A cylindrical dentin cavity (ø3 mm x 1.5 mm) prepared in an extracted human molar was treated with the Megabond system or an experimental bonding system consisting of 0.5M EDTA, 35% GM and Clearfil Photo Bond prior to composite filling and was irradiated for three seconds (xenon lamp) or 40 seconds (halogen lamp). The contraction gap was measured with a light microscope. In addition, the curing capability of these three light sources was evaluated by measuring the curing depth of the composites filled in a split Teflon mold (ø4 mm x 8 mm). There was no marginal gap formation for Clearfil APX, Estelite and Silux Plus treated with the experimental bonding system regardless of the type of light sources. The curing depth of the xenon lamp was significantly higher than the halogen lamp, while marginal adaptation did not suffer any significant deterioration.

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INTRODUCTION

To reduce the irradiation time required for polymerization of a visible light-cured resin composite, the high-power xenon lamp unit was introduced. The relationship between polymerization velocity and cavity adaptation of a resin composite has been discussed, though no consistent conclusions have been reported (Kato, 1987; Uno & Asmussen, 1991). It is essential to establish marginal integrity of the resin composite restoration to compensate for the quantity of polymerization contraction from the free surface into the cavity by the flow of resin composite during polymerization (Asmussen & Jørgensen, 1972; Davidson & de Gee, 1984a). The contraction gap width of a light-cured resin composite in a cylindrical dentin cavity has been reported to increase significantly compared to a chemical-cured resin composite (Itoh, Yanagawa & Wakumoto, 1986). Such a difference in marginal integrity between chemical and light-cured resin composite restorations might result from the difference in early polymerization stress (Davidson, de Gee & Feilzer, 1984b). The initial contraction stress of a light-activated resin composite is considered to be larger than that of the chemical-cured resin

composite (Feilzer, de Gee & Davidson, 1993). Higher polymerization velocity is thought to cause lower marginal adaptation of the resin composite because the contraction stress is accelerated and resin composite paste separates from the cavity wall. To improve cavity adaptation of a light-cured resin composite, soft-start curing (Mehl, Hickel & Kunzelmann, 1997) was introduced, by which the resin composite was initially irradiated with a low-power halogen lamp followed by high-power irradiation. Thus, initial contraction stress was reduced and cavity adaptation was reported to be improved (Yoshikawa, 1997). In addition, it has been claimed that the distance between the resin composite and the tip window of the lamp source should be increased in order to decrease the light intensity on the resin composite restoration and reduce polymerization contraction stress (Rueggeberg & Jordan, 1993). On the contrary, the higher power irradiation of the xenon lamp may damage the marginal adaptation of resin composite restoration due to rapid polymerization. This study investigated the effect of the xenon lamp and the halogen lamp on curing depth and dentin cavity adaptation of four commercially available resin composites.

Table 1: *Lamp Units*

Lamp Unit	Light Source	Manufacturer
Apollo 95E	Xenon lamp	Dental Medical Diagnostic Systems, Inc, Westlake Village, CA 91362, USA
Plasma Arc Curing System	Xenon lamp	American Dental Technologies, Corpus Christi, TX 78405, USA
Witelite	Halogen lamp	Takarabelmont Co, Osaka, 5420083 Japan

Table 2: *Commercially Available Resin Composites*

Resin Composite	Base Resin Matrix/Filler	Manufacturer	Shade/ Batch
Clearfil APX	Bis-GMA & TEGDMA/ irregular-shaped filler	Kuraray, Osaka 5300021 Japan	A-3 /0430
Estelite	Bis-GMA & TEGDMA/ spherical shaped filler	Tokuyama, Tokyo 1500002 Japan	A-3 (U) /1471
Silux Plus	Bis-GMA & TEGDMA/ irregular shaped micro-filler and smashed pre-polymerized filler	3M Dental Products St Paul, MN 55144 USA	U /8AH
Z-100	Bis-GMA & TEGDMA/ spherical shaped filler	3M Dental Products St Paul, MN 55144 USA	A-3 /8EK

Table 3: *Bonding Systems*

Bonding System	Manufacturer	Batch
MEGA BOND		
Primer	Kuraray, Osaka, 5300021 Japan	00009A
Bond	Kuraray, Osaka, 5300021 Japan	00017A
Experimental System		
0.5M EDTA	Experimental	
35% GM solution	Experimental	
Clearfil Photo Bond		
Universal	Kuraray, Osaka, 5300021 Japan	#389
Catalyst	Kuraray, Osaka, 5300021 Japan	#287

METHODS AND MATERIALS

Two commercial xenon lamp units (Apollo 95E, Dental Medical Diagnostic Systems Inc, Westlake Village, CA 91362, USA; Plasma Arc Curing System, American Dental Technologies, Corpus Chrispi, TX 78405, USA) and a conventional halogen lamp unit (Witelite, Takarabelmont Co, Osaka, Japan) were employed as the light sources (Table 1). Four commercial resin composites (Clearfil APX, Kuraray; Estelite, Tokuyama, Tokyo, Japan; Silux Plus and Z-100, 3M Dental Products, St Paul, MN 55144, USA) and two bonding systems (Megabond, Kuraray; and an experimental bonding system) were employed in this study (Tables 2 and 3). The experimental bonding system, consisting of 0.5M EDTA, 35% glyceryl mono-methacrylate solution and Clearfil Photo Bond (Kuraray) has been reported to eliminate contraction gap formation in a cylindrical dentin cavity (Chigira & others, 1994).

Analysis of the Spectro-Photo Distribution

The light intensities of these lamp sources were analyzed at regular wavelength intervals of one nanometer between a 200-nm and 800-nm wavelength using a multi-channel spectro-photo detector (MCPD-1000, Otsuka Electronics, Tokyo, Japan). The relative light intensity was calculated as the proportion to the light intensity at a wavelength of 480-nm for the commercial halogen lamp source. The spectro photo distribution of the light source was plotted by relative light intensity and a wavelength between 200-nm and 800-nm.

In addition, the light intensity of the three lamp sources mentioned above were also measured by a commercially available light meter, Apollo Meter (Hilux P/N:950-700 #9020844; Dental Medical Diagnostic System Inc) according to the manufacturer's instructions.

Curing Depth of a Resin Composite

An experimental split Teflon mold with an inner diameter of 4 mm and a height of 8 mm was fixed on a glass plate (Figure 1). In a preliminary examination, 8 mm, full length of the hardened resin composite cylinders were recognized on all specimens of the four composites irradiated for 10 seconds with a xenon lamp unit or for 60 seconds with a halogen lamp unit. Each commercial resin composite paste was slightly over-filled in the cavity of the Teflon mold, and the top surface of the unpoly-

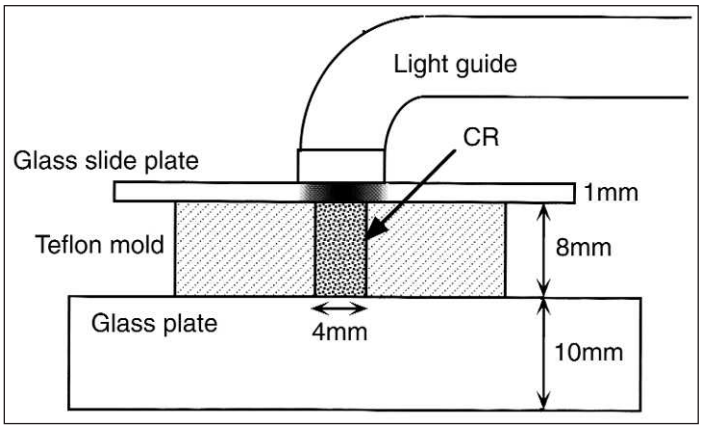

Figure 1. *Set up for determination of curing depth.*

merized resin composite paste was gently pressed with another glass plate, 1 mm in thickness. Subsequently, the composite was irradiated with a commercial xenon lamp source for a maximum of 10 seconds or with a halogen lamp for a maximum of 60 seconds from the top opening of the mold mediated with a glass plate, 1 mm in thickness. Immediately after irradiation, the resin composite cylinder was removed from the Teflon mold and the unpolymerized resin composite paste was eliminated by ethanol. The curing depth of the resin composite was determined by measuring the longitudinal length of the hardened resin composite cylinder with a slide caliper. Five specimens were prepared for each composite. The data was analyzed by ANOVA and Tukey's multiple comparison test (Kleinbaum, Kupper & Muller, 1988) for each of the four composites at the same irradiation condition (the light source and the irradiation time).

Contraction Gap Width Measurement

Marginal integrity of the commercial light activated resin composite irradiated with the xenon lamp was evaluated by measuring the contraction gap width in the cylindrical dentin cavity. The proximal enamel of the extracted human molar was eliminated on a wet carborundum paper grit number of 220, and a cylindrical cavity approximately 3.0 mm in diameter and 1.5 mm in depth was prepared in the exposed dentin. Figure 2 presents the experimental design for the resin composite restoration samples. For the commercial dentin bonding specimen, the cavity wall was treated with a commercial dentin bonding system (Megabond, Lot 00009A & 00017A, Kuraray) according to the manufacturer's instructions, and the cavity was slightly

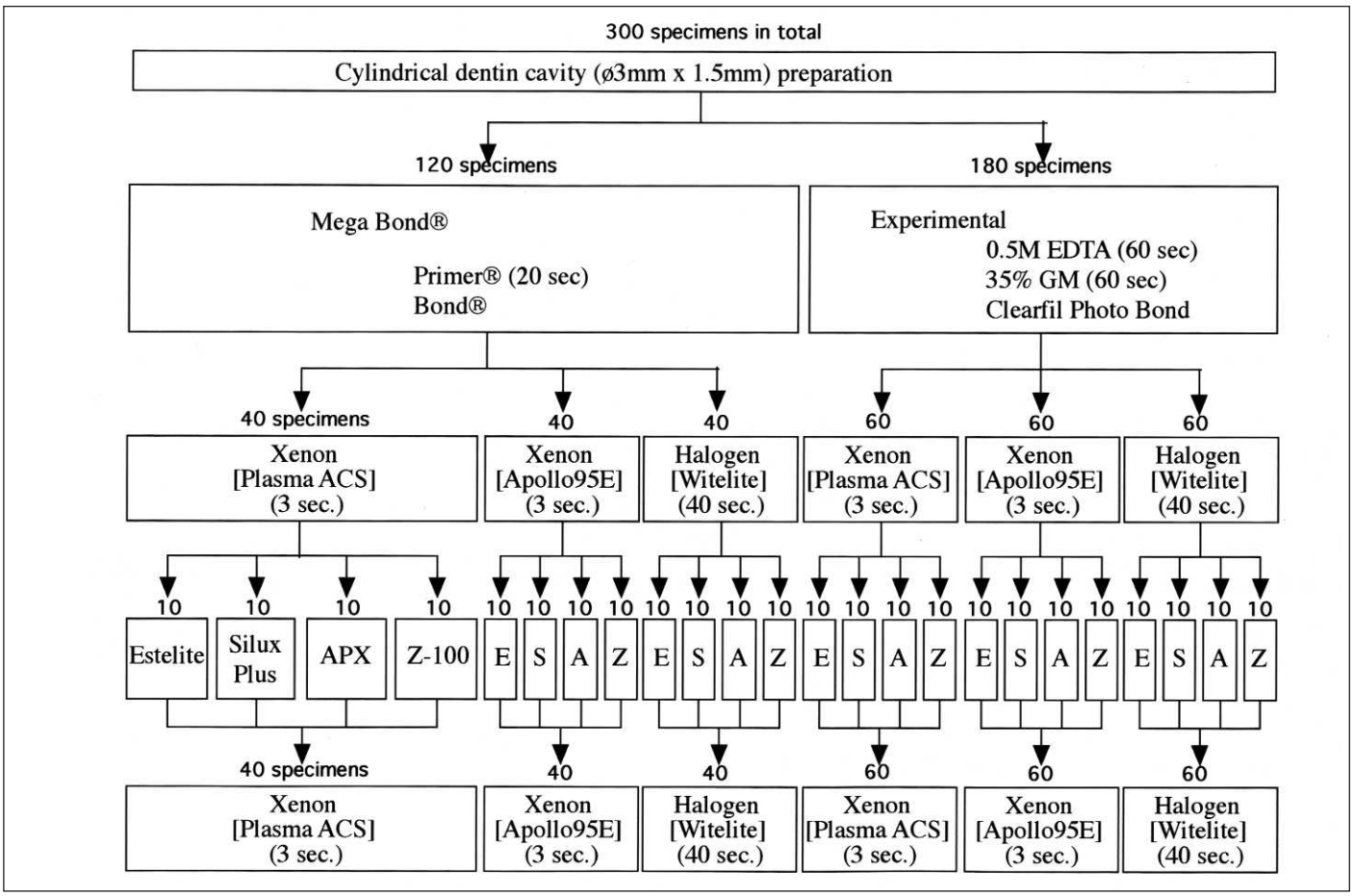

Figure 2. *Experimental design for determination of wall-to-wall contraction gap.*

over-filled with one of the commercial resin composites. The composite surface was momentarily flattened on a glass plate mediated with a plastic matrix (Frasaco Polyester Strip, GC, Tokyo, Japan) and irradiated with one of the lamp units. After final exposure, the specimen was stored in tap water at a room temperature of 24 ± 1°C for 10 minutes. The over-filled resin composite on the cavity margin was eliminated on wet carborundum paper grit number 1000, and the resin composite surface, including the surrounding dentin surface, was polished on a linen cloth mediated with a 0.03 μm-grain-sized alumina slurry. The marginal integrity of the restoration was inspected under a light microscope (Orthoplan, Leitz, Germany), and the width of the possible contraction gap was measured every 45 degrees along the cavity margin. The contraction gap value was presented by the sum of the diametrically opposing gap widths in percent to the cavity diameter, and the maximum of the four values was given as the contraction gap of the specimen. For the control group, the dentin cavity wall was conditioned with 0.5 mol/L EDTA which was neutralized to pH 7.4 for 60 seconds, followed by rinsing and drying. The cavity was then primed with a 35 vol % glyceryl mono-methacrylate (Blemmer GLM, NOF Co, Tokyo, Japan) solution for 60 seconds followed by air blast. After applying the commercial dentin bonding agent (Clearfil Photo Bond, Lot 389 & 287, Kuraray) and irradiation, the resin composite was filled and polymerized as mentioned above. Observations of the marginal adaptation and gap width measurement were performed in the same method as the Megabond group. Ten specimens for the Estelite

Table 4: *Light Intensity of the Light Sources and the Total Energy Density*

Lamp Unit	Light Intensity (mW/cm^2)	Irradiation Time (Sec)	Total Energy Density (mJ/cm^2)
Apollo 95E	1040	3	3120
Plasma Arc Curing System	1310	3	3930
Witelite	300	40	12000

Table 5: *Curing Depths of Resin Composites*

Plasma Arc Curing System	Irradiation Time 1	2	3	5	10 (sec)
APX	2.68 ± 0.08 *	4.28 ± 0.28 *	5.06 ± 0.22 *	6.62 ± 0.22 *	>8
Silux Plus	2.70 ± 0.16 *	4.04 ± 0.35 *	4.84 ± 0.11 *	6.94 ± 0.18 *	>8
Estelite	3.54 ± 0.17	4.78 ± 0.23	5.58 ± 0.30	7.08 ± 0.26 *	> 8
Z-100	5.30 ± 0.37	6.70 ± 0.19	7.60 ± 0.10	>8	--- (mm)
Apollo 95E	Irradiation Time 1	2	3	5	10 (sec)
APX	2.50 ± 0.20	4.24 ± 0.21 *	4.68 ± 0.13 *	6.10 ± 0.32 *	>8
Silux Plus	2.00 ± 0.12	3.48 ± 0.19	4.72 ± 0.16 *	5.16 ± 0.23	>8
Estelite	3.26 ± 0.17	4.48 ± 0.11 *	5.16 ± 0.24	6.22 ± 0.19 *	>8
Z-100	4.42 ± 0.18	6.30 ± 0.12	6.78 ± 0.15	7.48 ± 0.04	>8 (mm

Witelite	Irradiation Time 1	3	5	10	20	40	60 (sec)
APX	0	1.52 ± 0.22	2.60 ± 0.16 *	3.94 ± 0.24 *	5.22 ± 0.18	6.32 ± 0.28	>8
Silux Plus	0	2.22 ± 0.08 *	3.38 ± 0.13	5.20 ± 0.10	6.58 ± 0.19	7.70 ± 0.10 *	>8
Estelite	0	2.02 ± 0.18 *	2.58 ± 0.15 *	3.94 ± 0.19 *	5.76 ± 0.21	7.52 ± 0.28 *	>8
Z-100	1.40 ± 0.12	4.12 ± 0.28	5.68 ± 0.61	7.38 ± 0.29	7.76 ± 0.15	>8	-- (mm)

N=5.

* Couple values joined by vertical lines are not significantly different when analyzed by Tukey's multiple comparison test ($p>0.05$).

Table 6: *Wall-to-Wall Contraction Gap of Resin Composites*

Experimental System	Plasma ACS		Apollo 95E		Witelite	
Estelite	0	(10/10) * *	0	(10/10) * *	0	(10/10) * *
Silux Plus	0	(20/20) *	0	(20/20) *	0	(20/20) *
APX	0	(10/10) *	0	(10/10) *	0	(10/10) *
Z-100	0149 ± 0.094	(3/20)	0.112 ± 0.123	(6/20)	0.113 ± 0.124	(8/20)
Mega Bond System					%	
Estelite	0.003 ± 0.010	(9/10) *	0.001 ± 0.004	(0/20) *	0.009 ± 0.020	(8/10) * *
Silux Plus	0.023 ± 0.052	(8/10) *	0.020 ± 0.047	(7/20) *	0.024 ± 0.035	(6/10) *
APX	0.029 ± 0.037	(5/10) *	0.089 ± 0.090	(4/10) *	0.016 ± 0.025	(6/10) *
Z-100	0.211 ± 0.228	(3/10)	0.099 ± 0.099	(1/10) *	0.188 ± 0.111	(2/10)
					%	

N=10, Mean ± SD (number of gap-free specimens/total specimen number).

* Couple values joined by vertical lines are not significantly different when analyzed by Mann-Whitney test ($p>0.05$).

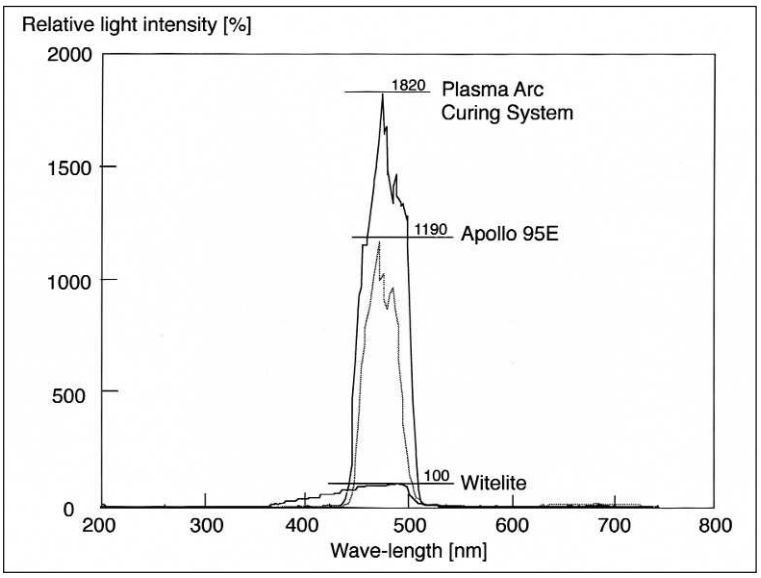

Figure 3. *Set up for determination of curing depth.*

and Clearfil APX groups, 20 specimens for the Silux Plus and Z-100 group, 180 specimens in total, were prepared with the experimental bonding system. Ten specimens for the four composite groups, 120 specimens in total, were prepared with the Megabond system The data were analyzed by the Kruskal-Wallis and the Mann-Whitney tests (Siegel & Castellan, 1988).

RESULTS

Figure 3 presents the spectroscopic distributions of the analyzed xenon and halogen lamp sources. The light intensity at a wavelength of 480 nm, considered effective to activate the camphoroquinone, was 1820% and 1190% for the Plasma ACS and Apollo 95E, respectively, compared to the commercial halogen lamp source (Witelite). The light intensities of Witelite, which was 100%. Apollo 95E and Plasma Arc Curing System measured with the Apollo Meter were 300, 1040 and 1310 mW/cm^2 (Table 4). The curing depths of the resin composite cylinder are given in Table 5. The length of the hardened Z-100 cylinder increased significantly compared to the other three resin composites regardless of the light source. In addition, the curing depths of the xenon lamp groups were greater than the halogen light source in all resin composites. The irradiation time required to harden the resin composite cylinder, 4 mm in diameter and 8 mm in height in the Teflon mold, was determined to be less than 60 seconds, and less than 10 seconds for the halogen and xenon lamp light sources, respectively. When the cavity was pretreated by the experimental dentin bonding system prior to resin composite filling, contraction gap formation of the three resin composites except Z-100 was completely prevented, regardless of the kind of light source. The gap value of Z-100 irradiated by the three light sources was not significantly different (p=0.3433) according to the Kruskal-Wallis test, though gaps were observed in more than 12 out of 20 specimens. The marginal integrity of the above mentioned three contraction gap-free resin composites combined with the experimental dentin bonding system deteriorated when the cavity was treated with the commercial dentin bonding system; gaps were observed in all tested groups. In the commercial dentin bonding system group, gap value of the composites irradiated by the three light sources was not significantly different (Estelite; p=0.6704, Silux Plus; p=0.7612, APX; p=0.1518, Z-100; p=0.2311) according to the Kruskal-Wallis test. The Mann-Whitney test revealed the gap value of Z-100 increased significantly when compared with the other three composites in the Plasma Arc Curing System group and Witelite group.

DISCUSSION

A light-cured resin composite is at a disadvantage because its polymerization stress is accelerated by rapid polymerization compared to that of a chemically-cured resin composite. To establish complete cavity adaptation of the resin composite, it is essential to compensate for the entire quantity of polymerization contraction of the resin composite with the flow of the composite into the cavity during polymerization. Not only the quantity but also the quality of polymerization contraction stress, "post gel contraction," is considered to play an important role in the marginal integrity of the resin composite (Sakaguchi & others, 1991). As demonstrated in this study, the xenon lamp has a remarkably high light intensity—more than 1000% of the conventional halogen lamp at a wavelength of 480 nm. Such a high light intensity accelerates the initial polymerization velocity of the composite. Despite rapid polymerization with xenon lamp irradiation, marginal contraction of the three resin composites did not occur with the experimental dentin bonding system. The efficacy of the experimental dentin bonding system employed in this study was characterized by EDTA conditioning and GM priming.

Chiba, Itoh & Wakumoto (1989) recommended using EDTA as a dentin conditioner because it possibly removes the smear layer without decalcifying sound dentin. Furthermore, Chigira & others (1998) reported that glyceryl mono-methacrylate solution prevents monomer penetration into the dentin, keeps the functional monomer in the bonding agent high concentration at the adhesive interface and establishes the chemical interaction by the calcium-monomer compound between the resin material and the dentin cavity wall. The contraction gap width increases when the dentin conditioner decalcifies the dentin cavity wall or when the adhesive monomer, such as 10-methacryloxydecyl dihydrogen phosphate (10-MDP) or 4-methacryloxyethyl trimellitate anhydride (4-META), is eliminated from the dentin bonding agent (Manabe & others, 1999). The contraction gap width of Z-100 increased significantly com-

pared to that of Silux Plus and Estelite among both the Megabond system group and the experimental system group. In addition, the curing depth of Z-100 was greater than Silux Plus, Estelite and APX. Thus, the poor marginal adaptation of Z-100 might be caused by the higher speed polymerization of Z-100. However, marginal adaptation of the composites irradiated by the three light sources was not significantly different in the Megabond system (Estelite; p=0.6704, Silux Plus; p=0.7612, APX; p=0.1518, Z-100; p=0.2311) and the experimental system (Z-100; p=0.3433). Polymerization velocity of the resin composite enhanced by xenon lamp may have less affect to the marginal adaptation of resin restoration than the curing depth measured. Marginal adaptation of the resin restoration is determined by interaction between the polymerization contraction stress and the efficacy of the bonding system. Versluis, Tantbirojn & Douglas (1998) reported the physical behavior of resin composite paste during its polymerization. Furthermore, Kinomoto & Torii, (1998) analyzed the polymerization contraction stress in the resin composite restoration. While polymerization velocity was considered to be influenced by the initiator content and translucency of the resin composite, marginal discrepancy of Z-100 might be improved by adjusting the concentration of the polymerization initiator. From a clinical viewpoint, applying the xenon light source for polymerization of the resin composite has the advantage of reducing irradiation time without damaging the marginal integrity if the dentin cavity is treated with an optimum dentin bonding system. Furthermore, when rubber dam isolation could not be applied, the chance of contaminating the cavity wall by saliva or moisture is possibly minimized by rapid polymerization using the xenon lamp.

CONCLUSIONS

Marginal gap formation of Clearfil APX, Estelite and Silux Plus treated with the experimental bonding system were completely prevented when the resin composites were polymerized with not only the conventional halogen lamp unit but also the xenon high power lamp unit. The curing depth enhanced by the xenon lamp was significantly greater than the halogen lamp, while marginal adaptation did not suffer any significant deterioration.

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Color Stability of a Resin-Modified Glass Ionomer Cement

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Clinical Relevance

Color stability of a resin-modified glass ionomer cement was determined to be shade dependent. As all shades darkened with time, selection of a shade lighter than the original tooth shade is recommended during placement.

SUMMARY

The color stability of a resin-modified glass ionomer cement (Fuji II LC) was investigated over six months using colorimetry. Five shades (A2, A3, A4, B3 and C4) were selected and 10 square specimens (7 mm wide and long, and 1.5 mm deep) were made for each shade using special grit molds. CIE L*, a*, b* color parameters of the specimens were taken at one day, one week, one month, three months and six months. Results were subjected to MANOVA and ANOVA/Scheffe's test at significance level 0.05. The effects of time on color parameters (L*, a*, b* values) were found to be shade dependent. All shades exhibited a decrease in L* values over time. With the exception of shade B3, significant differences in L* values were observed at six months. A general decrease in b* values was also observed but differences among the various time intervals were not significant except for shades A3 and C4. No apparent trends were observed for changes in a* values. For all shades, the largest color change (ΔE) was observed between one day and one week. The color stability of the resin-modified glass ionomer investigated was shade dependent. A general decrease in lightness and yellow chroma was observed.

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INTRODUCTION

Glass ionomer cements were first introduced to the dental profession in the early 1970s (Wilson & Kent, 1972). They are derived from aqueous polymeric acids and a glass component, which is usually a fluoroalumino silicate. Other non-fluoride glasses, such as aluminosilicates or aluminoborates, have also been used (Anstice & Nicholson, 1992). Glass ionomers possess

certain properties that make them useful as restorative materials. These include a low coefficient of thermal expansion similar to tooth structure, direct physico-chemical bonding to enamel and dentin and release of fluoride ions into adjacent tooth tissue (Naasan & Watson, 1998). Resin-modified glass ionomer cements were introduced to help overcome the problems of moisture sensitivity and low early mechanical strengths associated with conventional glass ionomer cements, and at the same time, maintain their clinical advantages (Sidhu & Watson, 1995). In resin-modified cements, a second, light-initiated curing process supplements the fundamental acid-base curing reaction. In their simplest form, they are glass ionomer cements with a small quantity of resin components such as hydroxyethyl methacrylate (HEMA) or BisGMA. More complex materials have been developed by modifying the polyacid with side chains that can polymerize by light-curing mechanisms.

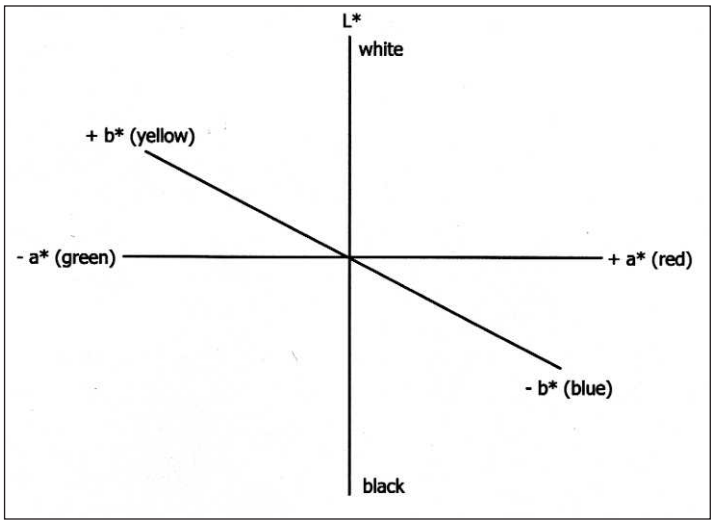

Figure 1. *Diagram of the CIELAB color space.*

Resin-modified glass ionomers undergo color changes during light polymerization (Yap, Sim & Loganathan, 1999). This color change may be primarily attributed to the photo-polymerization of the resin components as the acid-base reaction is retarded (Nicholson, 1998; Wan, Yap & Hastings, 1999). The delayed acid-base reaction, in addition to water sorption by the resin components (Yap, 1996), may result in post-polymerization color changes. Although the color stability of composite restoratives had been extensively studied (Tyas, 1992; Inokoshi & others, 1996; Uchida & others, 1998; Hosoya, 1999), the color stability of resin-modified glass ionomer cements has not been widely investigated. Inokoshi & others (1996) studied the opacity and color changes of five chemically cured composites, seven light-cured composites and three resin-modified glass ionomer cements. All tooth-colored restorative materials tested, including resin-modified glass ionomer cements, showed a similar tendency to become dark yellow or dark brown, the magnitudes of which depended on the product used. Only one shade of glass ionomer (A3) was evaluated and color changes were measured up to four weeks. This study determined the color stability of

Table 1: *Mean L*a*b* Values at the Various Time Intervals*

Shade	Time	L* Values	a* Values	b* Values
A2	**1 day**	72.98 (0.81)	1.94 (0.54)	23.85 (1.28)
	1 week	70.08 (1.14)	0.40 (0.82)	21.07 (1.60)
	1 month	69.25 (1.19)	-0.15 (0.75)	22.01 (1.90)
	3 months	69.04 (0.87)	-0.18 (0.80)	21.07 (1.04)
	6 months	69.72 (1.71)	-0.12 (0.70)	23.68 (1.54)
A3	**1 day**	70.64 (0.92)	3.97 (0.64)	32.20 (1.14)
	1 week	68.47 (1.09)	2.83 (0.51)	30.48 (0.91)
	1 month	67.56 (0.96)	2.05 (0.54)	30.25 (0.94)
	3 months	67.32 (1.17)	2.32 (0.75)	28.73 (1.31)
	6 months	67.25 (0.84)	2.27 (0.48)	29.25 (1.16)
A4	**1 day**	63.99 (0.74)	6.57 (0.54)	32.25 (1.76)
	1 week	62.78 (0.49)	5.53 (0.51)	31.42 (0.95)
	1 month	62.26 (0.48)	5.13 (0.55)	31.79 (0.91)
	3 months	62.08 (0.50)	5.62 (0.65)	30.23 (1.33)
	6 months	61.73 (0.42)	5.57 (0.53)	30.79 (1.09)
B3	**1 day**	69.13 (0.54)	0.61 (0.60)	26.04 (0.82)
	1 week	67.35 (1.16)	0.01 (0.20)	25.34 (0.72)
	1 month	66.68 (1.45)	-0.85 (0.47)	25.19 (1.21)
	3 months	66.74 (1.07)	-0.02 (3.16)	24.74 (0.79)
	6 months	66.13 (0.60)	-0.62 (0.27)	24.80 (0.74)
C4	**1 day**	60.45 (0.36)	0.94 (0.57)	20.11 (0.58)
	1 week	58.77 (0.38)	0.24 (0.36)	19.12 (0.66)
	1 month	58.11 (0.42)	-0.20 (0.36)	18.87 (0.58)
	3 months	58.53 (0.55)	0.32 (0.42)	18.54 (0.85)
	6 months	58.40 (0.54)	1.24 (0.53)	16.99 (1.48)

Standard deviations in parentheses.

Table 2: *Results of Statistical Analysis*

Shade	Color Parameters	Differences
A2	**L***	1 day > 1 week, 1 month, 3 months & 6 months
	a*	1 day > 1 week, 1 month, 3 months & 6 months
	b*	NS
A3	**L***	1 day > 1 week, 1 month, 3 months & 6 months
	a*	1 day > 1 month, 3 months & 6 months
	b*	1 day > 3 months & 6 months
A4	**L***	1 day > 6 months
	a*	NS
	b*	NS
B3	**L***	NS
	a*	1 day > 1 month & 6 months
	b*	NS
C4	**L***	1 day > 1 month, 3 months & 6 months
	a*	1 day > 1 month; 6 months > 1 week & 1 month
	b*	1 day, 1 week, 1 month > 6 months

> indicates statistically significant differences while NS denotes no significant difference in L*a*b* values between the different time intervals (results of one-way ANOVA/Scheffe's post-hoc test at significance level 0.05).

a resin-modified glass ionomer cement as a function of time and shade.

METHODS AND MATERIALS

Five shades of a resin-modified glass ionomer cement (Fuji II LC Capsule; GC, Tokyo, Japan) were selected for this study. They were Vita A2, A3, A4, B3 and C5. Special grid molds were used to prepare square specimens, which were 7-mm wide and long and 1.5-mm thick. The capsules were activated, mixed for 10 seconds and injected directly into the molds. The molds were slightly over-filled and sandwiched between two quartz glass plates to extrude excess material. Specimens were then light-polymerized for 20 seconds using a visible light curing unit (Spectrum; Dentsply Inc, Milford, DE 19963, USA) with a power output of approximately 420 mW/cm^2. No overlapping irradiation was necessary, as the diameter of the exit window of the light source (13 mm) was larger than the specimens. The glass slides were subsequently removed and the specimens stored in distilled water at 37°C. A small-area colorimeter (Dental Colorimeter; Minolta Camera Co Ltd, Tokyo, Japan) with a 3 mm diameter measuring area was used to determine the CIELAB coordinates of each specimen at the following time intervals: one day (baseline), one week, one month, three months and six months. The CIE color system (CIELAB) was determined in 1978 by the International Commission on Illumination. The three attributes of color in this system are L*, a* and b*, where L* is the lightness variable proportional to Value in the Munsell system and a* and b* are chromaticity co-ordinates (Figure 1). The a* and b* co-ordinates designate positions on a red/green and yellow/blue axis, respectively (+a = red, -a = green; +b = yellow, -b = blue).

Illumination corresponding to "average" daylight (CIE illuminant D65) from a pulsed xenon light source was utilized. The colorimeter was calibrated before each measurement session using the white calibration tile supplied by the manufacturer. Readings were taken at the centre of each specimen with a standardized white background below. The specimens were not allowed to dehydrate and were blotted dry just prior to measurement. Ten specimens were evaluated for each shade. The L* a* b* values at each time interval were averaged to obtain a single set of values for each shade using SPSS for Windows version 10.0 (SPSS Inc, Chicago, IL 60611, USA). All statistical analysis was conducted at a significance level of 0.05. The interaction between time, color parameters and shades was assessed using MANOVA. For the various shades, significant differences in L*, a* and b* values between the different time intervals were determined by one-way ANOVA and Scheffe's post-hoc test. The color change (ΔE) between time intervals was calculated using the equation:

$$\Delta E = \div \overline{\quad (\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

Where ΔL, Δa and Δb are the mathematical difference in mean L* a* b* values between time intervals.

RESULTS

Table 1 reflects the mean L*, a* b* values for the different shades at the various time intervals. Results of statistical analysis are showed in Table 2. Tables 3 and 4 reflect the color change (ΔE) between the various time intervals and between one day (baseline) and the different time intervals, respectively.

A significant interaction was observed between shade and time for L*, a* b* values. The effects of time on color parameters (L*, a*, b* values) were therefore shade dependent. All shades exhibited a decrease in L* values over time. At six months, a significant decrease in L* values as compared to baseline was observed for all shades except B3. For shades A2 and A3, the significant decrease in L* values was observed as early as

Table 3: Color Change (ΔE) Between the Various Time Intervals

Shade	1 Day and 1 Week	1 Week and 1 Month	1 Month and 3 Months	3 Months and 6 Months
A2	4.30*	1.37	0.96	2.70
A3	2.99	1.22	1.56	0.53
A4	1.80	0.75	1.65	0.66
B3	2.00	1.10	0.95	0.86
C4	2.07	0.83	0.75	1.81

*Change is perceptible to the human eye when the value of ΔE is ≥ 3.3

Table 4: Color Change (ΔE) Between One Day (baseline) and the Different Time Intervals

Shade	1 Day and 1 Week	1 Day and 1 Month	1 Day and 3 Months	1 Day and 6 Months
A2	4.30*	4.65*	5.27*	3.86*
A3	2.99	4.12*	5.08*	4.80*
A4	1.80	2.30	2.94	2.87
B3	2.00	2.98	2.79	3.47*
C4	2.07	2.88	2.56	3.75*

*Change is perceptible to the human eye when the value of ΔE is ≥ 3.3

one week. A general chromatic shift towards the blue region of the color space was also observed but differences in b* values between the various time intervals were not significant except for shades A3 and C4. For shade A3, b* values at one day were significantly greater than at three and six months. For shade C4, b* values at one day, one week and one month were significantly greater than at six months. No apparent trends were observed for changes in a* values, although significant changes were observed for all shades with the exception of A4.

For all shades, the largest color change was observed between one day and one week. This ranged from 4.30 for shade A2 to 1.80 for shade A4. Based on ΔE≥ 3.3 as the lower limit of perceptibility for color change by the human eye (Ruyter, Nilner & Moller, 1987), only shade A2 had perceivable color change between time intervals (Table 3). The largest color change between baseline, which can be used to represent the tooth shade, and the different time intervals, was also observed for shade A2 (Table 4). A ΔE of 5.27 was obtained at three months. For shade A2 and A3, perceptible color change with respect to the baseline was observed from one week and one month, respectively. Perceptible color changes were observed only after six months for shades B3 and C4.

DISCUSSION

A major esthetic failure of direct tooth-colored restorations is discoloration. It results from surface staining, wear-related changes in surface morphology, marginal staining due to microleakage and internal material deterioration. Although regular cleaning and proper use of adhesive systems minimize extrinsic surface and marginal staining, intrinsic discoloration is material-dependent and difficult to control by clinicians. Several *in vitro* accelerated tests for color stability had been developed to predict clinical performance of tooth colored restoratives (Asmussen, 1981; Burrow & Makinson, 1991; Uchida & others, 1998). These accelerated tests usually involve exposing materials to daylight, ultraviolet light or elevated temperatures and may not be pertinent *in vivo*. Hence, a more enduring and clinically relevant approach involving the use of water at body temperature (37°C) was adopted.

The color stability of the resin-modified glass ionomer cement evaluated was shade dependent. Color degradation of composite resins has also been found to be shade dependent (Uchida & others, 1998; Hosoya, 1999). As with composite resins, the lighter shades (A2 and A3) appeared to be more susceptible to color changes (Uchida & others, 1998). Large ΔE values between baseline (one day) and the various time intervals were observed for these shades (Table 4). Assuming that the baseline shade is matched to tooth, the color mismatch between tooth and restoration will be perceptible to the human eye as the latter can sense ΔE values of 3.3 or greater under clinical conditions (Ruyter & others, 1987). In controlled settings, color changes between ΔE 1 and 2 are perceived by the human eye (Seghi, Johnston & O'Brien, 1986; Seghi, Hewlett & Kim, 1989). For darker shades B3 and C4, the color mismatch can only be perceived at six months. Hence, tooth-restoration color mismatch will be clinically visible at six months for all shades evaluated, with the exception of A4 (Table 4), if baseline shades were matched to tooth.

A characteristic shift towards the black region of the color space was observed with all shades. The darkening of resin-modified glass ionomer cements was also reported by Inokoshi & others (1996). This occurred abruptly after one week storage in 60°C water and was associated with a decrease in opacity. For shades A2 and A3, the decrease in L* values was significant from the first week. A significant difference in L* values was observed only at six months and after one month for shades A4 and C4, respectively. Only shade B3 had no significant decrease in L* values. The selection of a shade lighter than the original tooth shade is therefore suggested for Fuji II LC. Inokoshi & others (1996) made the aforementioned recommendation for both

Fuji II LC and Vitremer (3M Dental Products, St Paul, MN 55144, USA).

A general shift towards the blue region of the color space was also observed. With the exception of shade A2, all shades showed a decrease in yellow chroma. The decrease was, however, only significant for shades A3 and C4. Compensatory measures may, therefore, not be warranted. No apparent trend was observed for changes in a* values. This may not be surprising considering the relatively small a* values observed. The latter is due to the fact that teeth, to which the shades were matched, have little red-green components.

Unlike conventional glass ionomers where the acid-base reaction is essentially complete after one day, Fuji II LC's acid-based reaction is retarded and levels off only after one week (Wan & others, 1999). This may account for the large ΔE values between one day and one week (Table 3). The acid-base reaction in resin-modified cements proceeds more slowly as some of the water in the cement is replaced by water-soluble monomers (McLean, 1992). If too little water is in the system, only polymerization reaction takes place and the material is, strictly speaking, not a glass ionomer cement (Wilson, 1990). The set cement has two interpenetrating matrices. These are the ionic matrix from the acid-base reaction and the polymerization or resin matrix from the free radical reaction. While some research has suggested that the resin network reduces the diffusion of water into the cement (Mathis & Ferracane, 1989), others have found that resin-modified cements have the potential to uptake water from the environment (Anstice & Nicholson, 1992; Yap, 1996). The degree of water sorption is product-dependent and appears to be influenced by the resin (HEMA) content (Yap, 1996). Copolymers of HEMA can take up large amounts of water, possibly up to 80% by mass (Pedley, Skelly & Tighe, 1980). Materials with higher resin (HEMA) content are therefore expected to absorb more water. Restorative resin-modified glass ionomers that have lower resin content than lining cements was observed to have lower water sorption (Yap, 1996). The uptake of water by the resin matrix may account for the late changes in color parameters.

CONCLUSIONS

This study investigated the color stability of a resin-modified glass ionomer cement as a function of time and shade. Within the limitations of this study, the following conclusions can be drawn.

1. Color stability of the resin-modified glass ionomer cement evaluated is shade dependent.
2. The largest color change occurred between one day and one week for all shades.
3. All shades, with the exception of B3, darkened significantly by six months.
4. A general decrease in yellow chroma was also observed but was not significant except for shades A3 and C4.
5. Selection of a shade lighter than the original tooth shade is recommended during placement.

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The Effect of Elapsed Time Following Bleaching on Enamel Bond Strength of Resin Composite

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Clinical Relevance

Bond strength of resin composite to enamel can be significantly reduced after bleaching. However, the effects of carbamide peroxide on bond strength may be reversible and short-lived.

SUMMARY

Recent studies have concluded that carbamide peroxide bleaching agents significantly affect the bond strength of composite to bleached enamel. This study evaluated the effects of bleaching regimen with different carbamide peroxide concentrations and post-treatment times on composite bond strength to enamel. Two hundred and four flat buccal and lingual enamel surfaces obtained from erupted sound third molars were randomly divided into 17 groups (n=12). Sixteen experimental groups comprised the evaluation of four carbamide peroxide home bleaching agents (Opalescence 10%—20 % and Whiteness 10%—16%) and four time intervals after bleaching (one day, one, two and three weeks). Specimens of control group were not submitted to bleaching and were stored in artificial saliva at 37°C for 10 days. The specimens of experimental groups were exposed to one daily application of carbamide peroxide for six hours for 10 consecutive days. After each daily treatment and post-bleaching, the specimens were stored in artificial saliva solution. Bonds were formed with Scothbond MP and Z-100 composite resin, and shear bond test was carried out 24 hours after adhesive-composite application. Two-way ANOVA showed that the bond strengths were significantly different ($p<0.05$). For the first two weeks post-bleaching, the bond strengths of resin to enamel were low. After a lapse of three weeks, the bond strength returned to that of the untreated control group. Increased concentration did not prolong the time needed prior to bonding.

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INTRODUCTION

Nightguard vital bleaching as an esthetic dentistry treatment was introduced by Haywood & Heymann (1989). Many dentists have indicated treating intrinsically discolored teeth with bleaching agents due to current interest in aesthetic dentistry (Chong, 1993; Lyons

Table 1: *Home Bleaching Agents Evaluated*

Product	Bleaching Agent	Concentration	Other Components	pH
Opalescence	Carbamide peroxide	10% and 20%	Carbopol, glycerin	6.5
Whiteness	Carbamide peroxide	10% and 16%	Carbopol, glycol, potassium ions, humectant, deionized water	6.5 – 7.2

Table 2: *Means and Standard Deviations of Shear Bond Strengths (MPa)*

Treatment	Post Treatment Time			
	1 day	1 week	2 weeks	3 weeks
Opalescence 10%	5.13 ± 1.76*	8.37 ± 2.53*	8.36 ± 2.54*	13.84 ± 4.13
Opalescence 20%	5.54 ± 2.08*	8.16 ± 1.48*	6.20 ± 1.77*	14.72 ± 4.03
Whiteness 10%	6.95 ± 2.15*	6.86 ± 1.80*	7.89 ± 2.26*	13.37 ± 4.71
Whiteness 16%	7.47 ± 2.66*	7.28 ± 2.26*	7.17 ± 2.06*	14.94 ± 3.81

Control Group: 16.62 ± 3.29
* Significant differences from the control group (unbleached enamel) by Dunnett's test ($p<0.05$).
Means joined by vertical line were not significantly different by Tukey test ($p>0.05$).

& Ng, 1998; Cibirka & others, 1999; Leonard & others, 1999). This original technique involved applying a carbamide peroxide gel to the teeth within the confines of a soft mouthguard for a specified time for two-to-five weeks. Daily exposure to carbamide peroxide, based on clinical experiences and research, is an apparently safe and effective procedure for whitening teeth (Haywood, 1992; Haywood & others, 1994; Haywood & Robinson, 1997; Kelleher & Roe, 1999).

Several reports concerned themselves with possible adverse effects of carbamide peroxide gel due to the large use of dentist-prescribed bleaching agents. These effects include changes in ultra-morphological resin-enamel interface (Perdigão & others, 1998); alterations in enamel surface morphology (McGuckin, Babin & Meyer, 1992a; Bitter, 1992; Shannon & others, 1993; Ben-Amar & others, 1995; Josey & others, 1996); pulpal (Cooper, Bokmeyer & Bowles, 1992) and gingival irritation (Marshall, Cancro & Fischman, 1995); changes in salivary pH (Leonard, Bentley & Haywood, 1994); adherence of Streptococcus mutans to bleached enamel (Gürgan, Bolay & Alaçam, 1997); alterations on composite resin (Bailey & Swift, 1992; Monaghan, Lim & Lautenschlager, 1992) and changes on fracture toughness, hardness and abrasion characteristics of human enamel (Seghi & Denry, 1992).

Some adverse effects of carbamide peroxide are clinically relevant when bonding composite and porcelain restorations or orthodontic brackets to bleached enamel surfaces. A decrease in bond strength up to one week following bleaching was noted (Cvitko & others, 1991; Stokes & others, 1992; García-Godoy & others, 1993; Ben-Amar & others, 1995). Use of the acid-etch technique on bleached enamel with normal bond strength values has been a challenge for esthetic dentistry (McGuckin & others, 1992b; Titley, Torneck & Ruse, 1992; Barghi & Godwin, 1994; Sung & others, 1999).

This *in vitro* study evaluated the effect of different carbamide peroxide gel concentrations, using the mouthguard technique, on the shear bond strength of resin composite to bleached enamel surface after various post-bleaching intervals (one day and one, two or three weeks).

METHODS AND MATERIALS

One-hundred and two freshly extracted, erupted sound human third molars were stored in 10% formalin. The roots were separated from their crowns using a diamond disc (KG Sorensen, Barueri, SP 06454-920, Brazil). The crowns were sectioned mesiodistally to obtain two similar enamel halves, lingual and buccal. Each half-crown was embedded in self-curing polyestyrene resin cylinders (2.0 cm diameter by 2.0 cm high), and lingual or buccal enamel surfaces were polished with wet 600-, 1000- and 1200-grit aluminum oxide abrasive paper on a polishing machine (APL-4; Arotec, Cotia, SP 06709-150, Brazil) to create a flat enamel surface. Specimens were randomly divided into 17 groups of 12 specimens each, one control group and 16 experimental groups. The control group was not bleached and after 10-day storage in artificial saliva at 37°C, it was tested in shear.

The artificial saliva, with an electrolyte composition similar to that of human saliva, was prepared from 1g sodium carboxymethylcellulose, 4.3 g xylitol, 0.1 g potassium chloride, 0.1 g sodium chloride, 0.02 mg sodium fluoride, 5 mg magnesium chloride, 5 mg calcium chloride, 40 mg potassium phosphate, 1 mg potassium thiocyanate and 100 g distilled deionized water.

A heat and vacuum tray-forming machine (Plastivac P7, Bioart, São Carlos, SP 13568-000, Brazil) was used to fabricate 192 individual trays from 0.02-inch soft plastic. A reservoir in each tray helped to keep the bleaching gel and artificial saliva in contact with the teeth. Two commercially available bleaching agents,

Opalescence (Ultradent Products, Inc, Salt Lake City, UT 84095, USA) and Whiteness (FGM Produtos Odontológicos, Joinville, SC 89219-310, Brazil), with two different concentrations of carbamide peroxide for each bleaching agent, were used in this study: Opalescence 10%, Opalescence 20%, Whiteness 10% and Whiteness 16% (Table 1).

Experimental groups were exposed to one daily application of carbamide peroxide for six hours during 10 consecutive days. In each specimen, approximately 0.1 mL of bleaching agent and 0.05 mL of artificial saliva was applied on the enamel surface and covered with an individual tray. During bleaching, the specimens were placed in 100% relative humidity at 37°C, and after daily bleaching, the specimens were thoroughly rinsed with an air/water spray for 10 seconds and stored in artificial saliva at 37°C. The effect of carbamide peroxide on shear bond strength to enamel was tested on the first day, one week, two and three weeks post-bleaching treatments and storage in artificial saliva at 37°C.

Bleached and unbleached (control group) flat enamel surfaces were washed in tap water and dried with oil free compressed air for the bond strength test. A circular hole, 2 mm in diameter was punched in adhesive tape positioned on the enamel surfaces. The demarcated area was etched with 35% phosphoric acid for 15 seconds, rinsed with an air/water syringe for 15 seconds and dried with compressed air for five seconds. Scotchbond MultiPurpose Adhesive (3M Dental Products, St Paul, MN 55144, USA) was applied on the demarcated enamel bonding area in a thin layer and light cured for 10 seconds. The embedded half-crowns were mounted in an assembly apparatus as described by Kamel and others (1990). A split Teflon mold with a circular hole 2 mm in diameter and 4 mm high was locked in the device. Two increments of composite resin (Z-100; 3M Dental Products) were placed into the opening of the split mold and each one was light cured for 40 seconds. After curing, the split mold was removed and the specimens stored in 100% relative humidity at 37°C for 24 hours prior to testing.

Each specimen was locked in a special device that was seated on the compression load cell of a universal testing machine (DL 500; Emic, São José dos Pinhais, PR 83020-250, Brazil). A shear load was applied to the base of the composite cylinder with a knife-edge rod with 0.5 mm width at a crosshead speed of 0.5 mm/min^{-1}. The shear bond strengths were calculated and expressed in MPa. Results were statistically analyzed by two-way analysis of variance (split plot—ANOVA) and additional treatment (control group), Dunnett's test and Tukey test at the 5% level of significance. Means of bond strength were correlated with the respective post-bleaching time and analyzed by linear regression at $\alpha = 0.05$.

RESULTS

Table 2 displays the mean shear bond strengths and standard deviations for bleached groups and the control group. ANOVA revealed a statistically significant difference among groups ($p<0.05$), therefore, data were further analyzed by Dunnett's and Tukey test. Dunnett's test indicated that the shear bond strength of all bleached groups, except for the groups tested three weeks post-bleaching, were lower than control group ($p<0.05$). Analysis of the data by Tukey test showed no significant differences in shear bond strength among treatments for the same post bleaching time ($p>0.05$). Linear regression showed a direct relationship between bond strength and post-bleaching time for the four treatments ($p<0.05$) (Figures 1, 2, 3 and 4). Increased concentration did not prolong the time needed prior to bonding.

DISCUSSION

Previous studies have shown changes in enamel surfaces (Titley, Torneck & Smith, 1988a) and reduction on bond strength of composite resin to enamel exposed to high-concentration of hydrogen peroxide (Torneck & others, 1991; Stokes & others, 1992). Alterations in bleached enamel surface morphology with the use of 35% hydrogen peroxide could have resulted from exposure to acid solutions and peroxide, which also has a low pH, or from both agents (Titley & others, 1988b; McGuckin & others, 1992a). The loss in resin adhesiveness to enamel was related to possible presence of residual peroxide, which interfered with resin attachment and inhibited resin polymerization (Torneck & others, 1990; Titley & others, 1993; Dishman, Covey & Baughan, 1994), or to changes in the penetration and structure of resin tags induced by hydrogen peroxide treatment (Titley & others, 1991). Using 25-35% hydro-

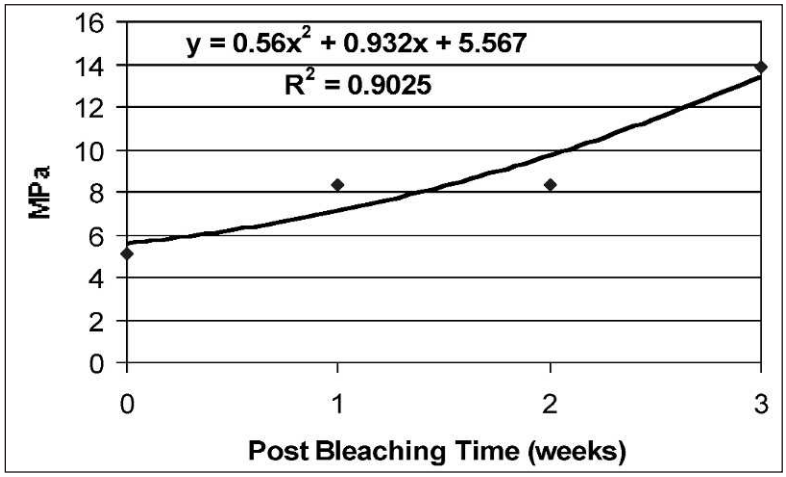

Figure 1. *Enamel shear bond strengths after bleaching with Opalescence 10%. The solid diamonds represent the mean bond strength at each time interval after the bleaching treatment.*

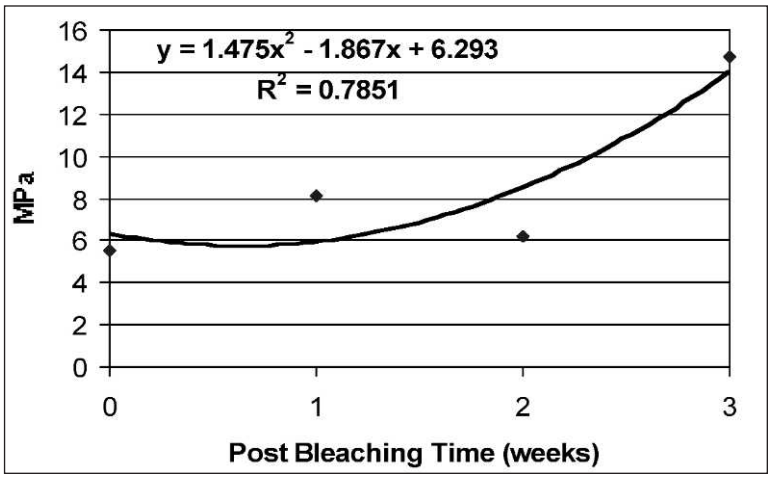

Figure 2. *Enamel shear bond strengths after bleaching with Opalescence 20%. The solid diamonds represent the mean bond strength at each time interval after the bleaching treatment.*

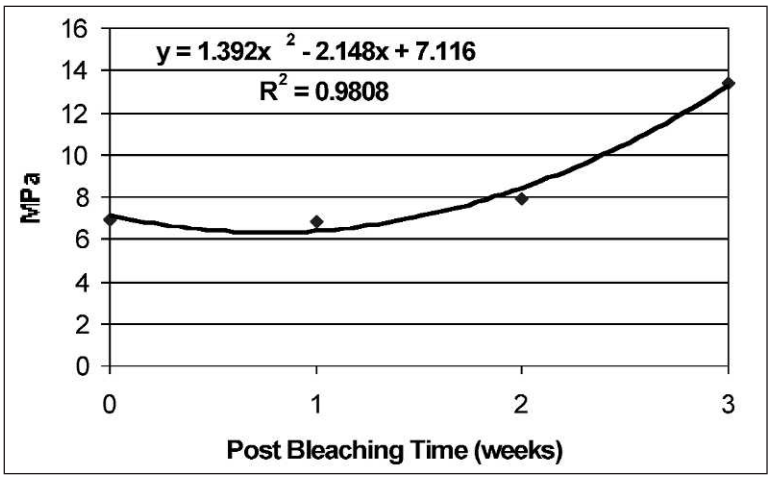

Figure 3. *Enamel shear bond strengths after bleaching with Whiteness 10%. The solid diamonds represent the mean bond strength at each time interval after the bleaching treatment.*

gen peroxide in the office as a vital bleaching procedure was indicated prior to nightguard home bleaching.

Products currently available on the dental market for the nightguard vital bleaching technique use 10% and higher concentrations of carbamide peroxide with a pH close to neutral. This solution is unstable and disassociates into 3% hydrogen peroxide and 7% urea on contact with tissue or saliva. The hydrogen peroxide further degrades into oxygen and water, while the urea degrades into ammonia and carbon dioxide, which elevate the pH. The oxidizers remove some unattached organic matter from the tooth without dissolving the enamel matrix, and these pigments are removed by diffusion, leading to bleaching. In this technique, after carbamide peroxide breakdown, the concentration of peroxide is lower than previous in-office bleaching techniques. However, home bleaching comprises daily applications of five-to-eight hours for two-to-five weeks (Haywood, 1992, Haywood & Robinson, 1997).

Hegedüs & others (1999) found that 30% hydrogen peroxide solution caused more severe changes in enamel surfaces than did carbamide peroxide gel. It was presumed that the difference in groove depth after treatment was caused by differences in hydrogen peroxide concentration. Morphologic alterations of the external enamel structure after carbamide peroxide bleaching have been reported (McGuckin & others, 1992a; Bitter, 1992; Shannon & others, 1993; Ben-Amar & others, 1995; Josey & others, 1996); however, other studies have demonstrated minimal topographic alteration in bleached enamel (Haywood & others, 1990; Haywood, Houch & Heymann, 1991; Wandera & others, 1994; Ernst, Marroquin & Willershausen-Zönnchen, 1996; Zalkind & others, 1996).

It has been hypothesized that the carbamide peroxide-containing bleaching agents affect the mineral content and organic phase of the superficial layer and the inner structure of enamel (Hegedüs & others, 1999; Perdigão & others, 1998). Carbamide peroxide is a denaturing agent for proteins (Yip, Beeley & Stevenson, 1995). Urea is capable of attacking protein structures and penetrating into the enamel affecting the surface and interprismatic regions (Arends & others, 1984; Goldberg & others, 1983). The mineral loss can be noted by decreased enamel micro-hardness after bleaching (Seghi & Denry, 1992). Thus, these changes in mechanical properties may be responsible for alterations in the superficial bleached enamel crystallites and ultra-structure of resin-enamel interfaces (Perdigão & others, 1998).

Reduction in bond strength due to bleaching with carbamide peroxide has been evaluated *in vitro* (Stokes & others, 1992; García-Godoy & others, 1993; Ben-Amar & others, 1995). Authors have advised of delays in bonding one week after bleaching due to the reduction of composite bond strength to freshly bleached enamel has been transient (McGuckin & others, 1992b; Titley & others, 1992; Miles & others, 1994). Similarly, removal of the superficial layer (Cvitko & others, 1991), pretreatment of bleached enamel with alcohol (Barghi & Godwin, 1994) and use of adhesives containing organic solvents (Sung & others, 1999) can result in complete reversal of the reduced enamel bonds.

Statistical evaluation of the data obtained by shear testing indicated a reduction in bond strength until two weeks post-bleaching and the bleaching agent concentration did not seem to affect the bond strength for the same post-treatment time. It took three weeks for the

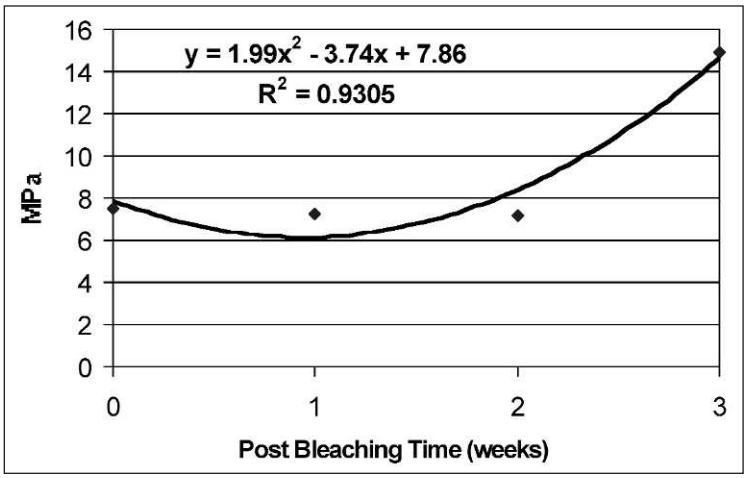

Figure 4. *Enamel shear bond strengths after bleaching with Whiteness 16%. The solid diamonds represent the mean bond strength at each time interval after the bleaching treatment.*

enamel to return to conditions that lead to normal bond strengths. Over an extended period of time, exposure of bleached enamel to artificial saliva may leach out peroxide absorbed by enamel during bleaching and reestablish the superficial morphology.

For bleached enamel surfaces, linear regression showed a tendency towards a reduction of the shear bond strength of the composite resin to enamel, which varied according to the post-bleach storage period. Although Opalescence and Whiteness bleaching agents weakened the resin bond strength, the buffering and remineralization potential of artificial saliva probably minimized the bleaching effects without interfering with the resin bonding if it was carried out three weeks post-bleaching.

CONCLUSIONS

The results of this study suggested that bonding of composite to enamel bleached with 10%, 16% and 20% carbamide peroxide gels result in a significant decrease in bond strength. Bond strength returns to values close to those of non-bleached enamel within three weeks following the procedure.

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Effect of Eccentric Load Cycling on Microleakage of Class V Flowable and Packable Composite Resin Restorations

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Clinical Relevance

The stresses induced on non-carious cervical lesions may increase microleakage of Class V composite restorations regardless of the elasticity of the restorative materials used.

SUMMARY

Class V composite restorations are subject to the stresses that induce non-carious cervical lesions. This study evaluated the effect of eccentric oblique load on microleakage of restorations. Class V cavities were randomly prepared on the buccal surfaces of 40 recently extracted premolars and restored with composite resin according to manufacturers' directions. Teeth were randomly assigned to one of four treatment groups with 10 teeth per group: (1) flowable resin (Revolution) without load cycling; (2) packable resin (Prodigy Condensable) without load cycling; (3) flowable resin with load cycling (200,000 cycles) and (4) packable resin with load cycling (200,000 cycles). All teeth were then immersed in 2% methylene blue solution for 24 hours after thermocycling (500 cycles). Dye penetration was measured (scores 0-3). The results indicated that adding load cycling significantly increased microleakage ($p<0.05$). No significant differences in microleakage were observed for flowable resin vs packable resin. Gingival margins had significantly more microleakage ($p<0.05$) than occlusal margins.

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INTRODUCTION

The marginal seal or adaptation of restorative materials to the cavity walls is a major factor for the long-term performance of any restoration.

Microleakage, or the movement of bacteria, fluids, molecules or ions, and even air between the prepared cavity walls and the subsequently applied restorative materials (Kidd, 1976), is a primary reason for restoration replacement, and approximately 30% or more of such replacements are attributed to microleakage (Hakimeh & others, 2000). Failure to prevent microleakage may produce post-operative pain, recurrent caries, marginal staining and possible pulpal pathology (Berry & Tjan, 1994, Castelnuovo, Tjan & Liu, 1996).

Bonded composites have commonly been the choice for esthetic restoration of Class V lesions. However, one disadvantage of composites is polymerization shrinkage, which can result in marginal discrepancies causing microleakage (Kaplan & others, 1992). In the mouth, additional stresses are applied to the restorative material due to temperature changes and mastication. Many new bonding agents and composite restorative materials have been introduced to bond to the dentin and cementum margins of cervical lesions, but microleakage at the dentin or cementum aspects of restorations remains a problem of clinical significance (Kidd, 1976; Kaplan & others, 1992; Gordon, Plasschaert & Stark, 1986). Longevity of a Class V restoration can be affected by stresses in the cervical area caused by mechanical, thermal and chemical factors (Kaplan & others, 1992; Davidson, 1986; Feilzer, de Gee & Davidson, 1990; Gordon & others, 1986).

Many studies dealing with microleakage; in particular, thermocycling effects have received considerable attention for many years (Kidd, 1976; Cooley & Barkmeier, 1991; Crim, 1993). Although load cycling on microleakage has not been extensively examined, some studies have reported important and interesting results showing some conflicting findings about the role of load cycling on microleakage (Abdalla & Davidson, 1996; Davidson & Abdalla, 1994; Rigsby & others, 1992, Darbyshire, Messer & Douglas, 1988; Munksgaard, Itoh & Jorgensen, 1985; Yap, Stokes & Pearson, 1996). Moreover, the effects of oblique load cycling on microleakage of Class V restorations are not well known. However, bending or flexing stresses are known to occur if the tooth is eccentrically loaded, generating tensile stress at the margins of Class V restorations (Lee & Eakle, 1984; Van Meerbeek & others, 1993). Also, Van Meerbeek & others (1993) reported the correlation of improved clinical results with lower moduli of elasticity, and Hasegawa & others (1999) have reported the tensile bond strength of the restoration correlated with mechanical properties of composites.

Recently, two new types of resin composites, known as "flowable composites" and "packable composites," have been introduced for restorative purposes. These new types of composites have shown inferior mechanical properties when compared to traditional hybrid composites (Manhart & others, 2000; Bayne & others, 1998).

Flowable composites have less filler load (Bayne & others, 1998). This formula results in a material that offers a lower Young's modulus that represents an average of three times less than hybrid composites, which can provide some elasticity in Class V situations (Labella & others, 1999). This property, unique to flowable composites, should help alleviate marginal stress during the life of a restoration (Van Meerbeek & others, 1993; Bayne & others, 1998; Unterbrink & Liebenberg, 1999).

On the other hand, the composition and physical properties of the packable composites revealed that none of the materials represent a remarkable improvement over more traditional universal composites (Leinfelder, Bayne & Swift, 1999; Jackson & Morgan, 2000; Perry, Kugel & Leinfelder, 1999). But generally, Young's modulus of these materials is higher than that of flowable composites (Leinfelder & others, 1999).

This study determined the effects of eccentric oblique load cycling on microleakage in Class V composite resin restorations and the correlation of microleakage between oblique load cycling and the elasticity of restorative materials. Flowable and packable composite resins were used for material comparison under load cycling.

METHODS AND MATERIALS

Forty non-carious human premolars stored in 0.1% thymol solution for up to four months at room temperature were used. After surface debridement with a hand-scaling instrument and cleaning with a rubber cup slurry of pumice, wedge-shaped Class V cavity preparations were made on the buccal surface at the cementoenamel junction so that half of the cavity preparation was above the cementoenamel junction and half below. Preparations were made with a #329 carbide bur in a

Table 1: Investigated Materials

	Product	Batch #	Composition	Manufacturer
Adhesive	OptiBond Solo Plus	911608	HEMA,BisGMA,GPDM,SiO2, barium glass, fluoride, acetone	Kerr Orange, CA 92867, USA
Flowable Resin	Revolution Formula 2	1473	Information not available	
Packable Resin	Prodigy Condensible	2943	Information not available	

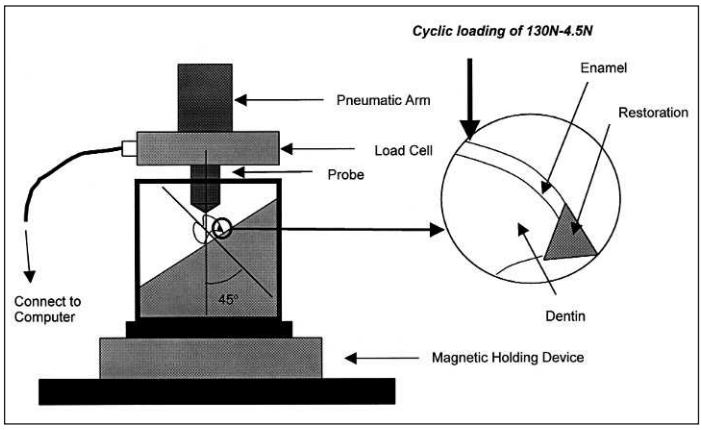

Figure 1. *Experimental setup for cyclic loading.*

high-speed handpiece to a uniform outline. Prepared cavities measured 5 mm long, 3 mm wide and 2.5 mm deep with diverging margins, with the occlusal one in enamel and the gingival one in dentin/cementum. Subsequently, teeth were randomly assigned to four groups of 10 teeth per group. Table 1 shows the tested materials.

Group 1: flowable resin without mechanical loading

Group 2: packable resin without mechanical loading

Group 3: flowable resin with mechanical loading

Group 4: packable resin with mechanical loading

In all cases, manufacturers' instructions for dentin conditioning, handling and placing were strictly followed. Restorations were cured with a visible light source (Optilux 400, Demetron Research Corp, Danbury, CT 06810, USA) in accordance with manufacturers' recommended time. The light source was calibrated for light output (>600mW/cm^2) before each use with a Demetron radiometer (Model 100, Demetron Research Corp). After curing the restorations, the teeth were finished to contour and cavosurface margins with a #7901 carbide finishing bur (SS White Burs Inc, Lakewood, NJ 08701, USA) with air and water spray in a high speed handpiece (Kavo, Biberach, Germany).

The samples were then thermocycled for 500 cycles in distilled water at 6°C and 60°C with a dwell time of 30 seconds. After thermocycling, the roots of the teeth in Groups 3 and 4 were mounted in a receptacle at a 45°angle to the long axis with clear orthodontic resin. This receptacle was mounted on a servo-pneumatic materials testing machine with a magnetic holding device (Figure 1). The entire tooth was submerged in a water bath with a pH of 7. This type of mounting apparatus was used to line-up the specimen directly under the probe on the test frame. The probe was attached to a load cell, which in turn, was attached to the upper member of the test frame. A cyclic load was applied to the tooth at a 45° angle to the long axis of tooth, slightly apical to the cusp tip. The probe was programmed to never leave the surface of the tooth. The loading cycle oscillated from 4.5N to 130N at 1Hz. Each specimen experienced 200,000 load cycles.

Teeth were prepared for microleakage evaluation by coating the entire tooth with nail varnish, except for 1 mm around the restoration margins. To avoid dye penetration through the apices, the root apices were sealed with acrylic resin and only the tooth portion that included the restoration margins was placed in a solution of 0.5% methylene blue for 24 hours at room temperature.

After removing the specimens from the dye solution, the superficial dye was removed with a pumice slurry and rubber cup. Teeth were then mounted in a cup filled with dye stone (Miles Inc, a Division of Heraeus Kulzer, South Bend, IN 46628, USA) to facilitate handling during sectioning. Teeth were sectioned longitudinally with a hard tissue microtome in the center of the cavity to evaluate dye penetration. The sectioned teeth were then separated and polished with wet 240-, 320-, 400- and 600-grit silicon carbide paper. The polished surfaces were examined at the occlusal and gingival margins with a stereomicroscope (Olympus Co, Tokyo, Japan).

Two evaluators independently examined dye penetration along the restoration interface according to the following criteria:

0= no dye penetration.

1= dye penetration up to one-third of the cavity depth.

2= dye penetration from one-third to two thirds of the cavity depth.

3= dye penetration greater than two-thirds of the cavity depth.

If the evaluators disagreed, a consensus was obtained after both investigators reexamined the specimen. Occlusal, gingival and overall scores for each group of restorations were compared with the Kruskal-Wallis one-way analysis of variance (ANOVA) non-parametric statistical test to identify any statistically significant differences among the four groups. If overall differences were confirmed to be significant, individual pairwise group comparisons were then performed using Bonferroni-adjusted Student's *t*-tests applied to the rank scores.

RESULTS

Tables 2 and 3 summarize the frequency of microleakage scores at the enamel and dentin margins.

The Kruskal-Wallis test revealed significant differences in microleakage for the four composite resins for the occlusal alone ($p<0.02$), gingival alone ($p<0.005$) and the total leakage ($p<0.001$). Adding eccentric oblique load cycling significantly increased microleakage. There were significant differences in Group 1 vs Group 3 and Group 2 vs Group 4 ($p<0.05$). No significant differences

Table 2: Frequency of Microleakage Scores at the Enamel Margins

Groups	Microleakage Score				
	0	1	2	3	
Group 1	8	2	0	0	*a
Group 2	8	2	0	0	*a
Group 3	3	7	0	0	*b
Group 4	3	5	2	0	*b

*Groups joined same line or same letter are not significantly different from the Kruskal-Wallis tests.

Table 3: Frequency of Microleakage Scores at the Cementum Margins

Groups	Microleakage Score				
	0	1	2	3	
Group 1	4	6	0	0	*a
Group 2	3	7	0	0	*a
Group 3	0	6	2	2	*b
Group 4	0	8	1	1	*b

*Groups joined same line or same letter are not significantly different from the Kruskal-Wallis tests.

were observed for Group 1 vs Group 2 and Group 3 vs Group 4.

In general, the gingival margins had significantly more leakage than the corresponding occlusal margins ($p<0.05$). The difference (gingival margin-occlusal margin) was not significantly different among the four groups by Kruskal-Wallis test.

DISCUSSION

This study showed no significant differences between flowable composites and packable composites whether or not load cycling was used. This is in contrast to a study of Class V restorations that reported the correlation of improved clinical results using a resin of a lower moduli of elasticity (Van Meerbeek & others, 1993) and one reporting the use of flowable composites decreasing microleakage (Payne, 1999; Ferdianakis, 1998). The materials used in this study were Revolution (flowable composite) and Prodigy Condensable (packable composite). Young's modulus of Revolution is 4.6 GPa (formula 2; Kerr technical manual and R&D group) and it was reported that the Young's modulus of Prodigy Condensable is 11.9 GPa (Leinfelder & others, 1999). Low Young' modulus of flowable composites can provide some elasticity in Class V situations (Labella & others, 1999) and can help alleviate marginal stress during the life of a restoration (Van Meerbeek & others, 1993; Bayne & others, 1998; Unterbrink & Liebenberg, 1999). However, Miranda & others (1999) reported that using packable composite resins with or without a flowable composite liner does not prevent marginal microleakage and the flowable composite had no influence on marginal sealing. Their results are similar to the results of this study in that they can be explained by using a dentin-bonding agent (OptiBond Solo Plus) that offers a layer of intermediate elasticity between the restorations and tooth structure (Van Meerbeek & others, 1992). This layer tends to increase the strain capacity of the restorations and contributes to relaxation of polymerization contraction stress (Kemp-Scholte & Davidson, 1990). Another possible factor is that higher filler levels of packable composite may reduce polymerization shrinkage and minimize marginal leakage (Leinfelder & others, 1999).

Teeth are subjected to heavy occlusal stresses during normal function and parafunction, and when occlusal forces are exerted on a tooth, stresses will be distributed throughout its structure (McCoy, 1982). McCoy (1982) was the first to notice the clinical significance of this stress and pointed out that bending or flexing stresses will occur if a tooth is eccentrically loaded. Lee & Eakle (1984) hypothesized that the primary etiology of non-carious cervical lesions is the tensile stress induced by mastication and malocclusion. Of course, when cervical lesions are restored, the corresponding Class V composite restorations are subjected to the same stresses. Such stresses have been reported to progressively dislodge the Class V restoration at the cavosurface margin (Van Meerbeek & others, 1993). To illustrate this problem, Jørgensen (1970) introduced the term "mechanical percolation" to indicate mechanical factors in the oral environment, which might produce asymmetric pressure on the restoration and the tooth. He showed that load cycling of teeth with filled cavities resulted in permanent or transitory gaps (Jørgensen, Matono & Shimokobe, 1976).

Load cycling in this study had a significant effect on microleakage. These results also agree with other studies (Abdalla & Davidson, 1996; Davidson & Abdalla, 1994; Rigsby & others, 1992; Miranda & others, 1999). In a study by Rigsby & others (1992), microleakage of restorations was found to be significantly greater at the cementum aspects of restorations subjected to thermocycling and occlusal load cycling when compared to restorations only subjected to thermocycling or load cycling. However, Hakimeh & others (2000) noticed that thermocycling and cavity preparation had a significant effect on microleakage, but load cycling did not. Moreover, others have also reported on the lack of effect of load cycling on microleakage (Darbyshire & others, 1988; Munksgaard & others, 1985; Yap & others, 1996). These differences in the results of load cycling on microleakage effects need careful evaluation and interpretation due to differences in experimental designs. For example, Darbyshire & others (1988) used Class II cavities in their experiments and Hakimeh & others (2000) used compomer as a restorative material.

It is important to note that most studies used axial compressive loading to stress on margins of restorations (Abdalla & Davidson, 1996; Davidson & Abdalla, 1994; Rigsby & others, 1992; Darbyshire & others, 1988; Munksgaard & others, 1985). In this study, load was applied obliquely on the buccal side to ensure the same type of stresses that induce non-carious lesions at the cervical area. The magnitude of these forces (4.5N-130N) is within the range expected *in vivo* (DeLong & Douglas, 1983). Total load cycling was 200,000 cycles, with a rate of 1 Hz. This load cycling corresponds approximately with at least four years of clinical mastication (Hakimeh & others, 2000).

SEM studies by Van Meerbeek & others (1993) revealed composite resin remnants in the dentinal surface of cavities of lost fillings. This finding clearly indicates stress-induced failure of the adhesive joint and confirms the presence of powerful forces at the cervical region. Palamara & others (2000) have described that a load applied at 45° on the buccal incline of the buccal cusp generates tensile strains on the buccal surface and compressive strains on the lingual surface. These studies agree with the results of this study in that they have shown that eccentric oblique load cycling could increase microleakage of Class V restorations.

When comparing microleakage at the gingival margins with that of the occlusal margins, the gingival margins had more leakage than the corresponding occlusal margins. This agrees with previous studies (Rigsby & others, 1992; Linden & Swift, 1994). Moreover, Rigsby & others (1992) found that the microstrain of restorations was significantly greater at the cementum aspects of the restorations than at the enamel aspects when Class V resin restorations were subjected to axial load cycling.

CONCLUSIONS

Adding eccentric oblique load cycling significantly increased microleakage. No significant difference in microleakage between flowable resin and packable resin was observed. In general, gingival margins had more leakage than their corresponding occlusal margins.

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The Effect of Long-Term Storage on Nanoleakage

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Clinical Relevance

Durability of bonds achieved with the dentin bonding systems used in this study may be affected by nanoleakage leading to hydrolytic degradation over time. Clinically nanoleakage may be important, especially for modern dentin bonding systems which can achieve gap-free margins.

SUMMARY

To evaluate the durability of dentin bonding over time, the nanoleakage of four dentin bonding systems (Single Bond, Stae, Clearfil SE Bond and PermaQuik) over 24 hours, three months, six months and 12 months, was investigated. Flat occlusal dentin surfaces from extracted human molars were finished with wet 600-grit silicon carbide paper and bonded with one of the dentin bonding systems following manufacturers' instructions. The bonded surface was covered with <1 mm thick layer of Silux Plus resin composite and light cured for 40 seconds. The specimens in each dentin-bonding group were randomly assigned to four sub-groups and kept in phosphate buffered saline solution (pH 7.4) containing 0.01% sodium azide at 37°C for 24 hours, three, six or 12 months. The margins of all specimens were finished and polished with Sof-Lex disks after initial 24-hour storage. At the end of each storage time, the surrounding tooth surfaces except for 1 mm adjacent to the restoration were coated with nail varnish. The samples were immersed in a 50% w/v solution of silver nitrate for 24 hours, placed in photodeveloping solution and exposed to fluorescent light for eight hours. The samples were cut longitudinally and buccolingually, polished, mounted on stubs, carbon coated and observed in a Field Emission-SEM using backscattered electron mode.

The results showed that systems using phosphoric acid as the etchant had a line of silver deposition at the base of the hybrid layer. Silver deposition increased in all systems over 12-months storage, with PermaQuik changing the least. Nanoleakage of the dentin bonding systems increased slightly during the 12-month storage period, indicating that they may be subject to hydrolytic attack over time.

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INTRODUCTION

With the rapid development of dentin bonding agents, continual evaluation of their performance is necessary. While clinical studies can provide the most reliable evi-

dence of dentin bonding agent efficacy, they are time-consuming, expensive and not always easy to perform. In addition, the rapid replacement of old versions of dentin bonding agents with new versions has reduced the usefulness of such studies. Laboratory tests, such as microleakage evaluation and bond strength measurement, are valuable as controlled pre-clinical screening tests of adhesive agents to attempt to predict their clinical performance (Van Meerbeek & others, 1994).

Van Meerbeek & others (1999) stated that a restoration may have leaked extensively and undermined the restoration's integrity before it is lost. A failed seal of the margin can result in post-placement sensitivity, marginal staining and recurrent caries, which are the most common reasons associated with clinical failure of adhesive restorations (Van Meerbeek & others, 1999).

The evaluation of dentin bonding systems is usually carried out in the laboratory 24 hours after specimen preparation. This time interval is adequate to test the adhesive ability of a material, but fails to provide any other information regarding the change in performance over time for a bonding system; thus, laboratory studies for long-term durability of bonds are important (Burrow, Tagami & Hosoda, 1993, Burrow, Satoh & Tagami, 1996).

Sano & others (1995a,b) found that nanoleakage occurred within the hybrid layer due to incomplete penetration of adhesive resin into the demineralized dentin, leaving porosity within the hybrid layer and exposure of collagen fibers. It is possible that the exposed collagen fibers are susceptible to hydrolytic degradation over time, thus weakening the bond and compromising its durability.

This study evaluated the possible changes within the hybrid layer over 24 hours, three months, six months and 12 months, as assessed by nanoleakage. The hypothesis advanced is that over time, the leakage pattern may change and more silver penetration would be expected.

METHODS AND MATERIALS

Eighty freshly extracted human molars stored in physiological saline containing 0.1% thymol were used. Occlusal enamel was removed under running water using a model trimmer to expose a flat dentin surface which was examined under a dissecting microscope to ensure that no enamel remnants remained. The dentin surfaces were wet ground with 600-grit silicon carbide paper for 60 seconds to create a uniform surface and smear layer.

Four dentin bonding agents were evaluated in this study: Single Bond (3M Dental Products Division, St Paul, MN 55144, USA) and Stae (Southern Dental Industries, Bayswater, Victoria, Australia), both are one-bottle dentin bonding systems; Clearfil SE Bond (Kuraray Co Ltd, Osaka, Japan), a self-etching primer system and PermaQuik (Ultradent Products, Inc, South Jordan, UT 84095, USA), a conventional three-

Table 1: *Materials, Manufacturers, Batch Numbers and Compositions*

Adhesive system	Etchant (Batch #)	Primer (Batch #)	Resin (Batch #)	Manufacturer
Single Bond	35% phosphoric acid (7HB)	Priming resin Bis-GMA, HEMA, polyalkenoic acid copolymer, water, ethanol, dimethacrylates (7AR)		3M Dental Products Division, St Paul, MN
Stae	37% phosphoric acid (990331)	TEGDMA, HEMA, UDMA, acetone, water, camphorquinone, butylated hydroxytoluene, *Special monomer (proprietary) (990321)		Southern Dental Industries, Bayswater, Victoria, Australia
Clearfil SE Bond	Self-etching primer MDP, HEMA, hydrophilic dimethacrylate, dl-camphorquinone,N,N-diethanol-p-toluidine, water (00105A)		MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-camphorquinone, N,N-diethanol-p-toluidine, silanated colloidal silica (00032A)	Kuraray Co, Ltd, Osaka, Japan
PermaQuik	35% phosphoric acid (S337 LOT 2XC2)	Canadian balsam, methacrylic acid, HEMA, camphorquinone, phosphate monomer, ethanol (S337 LOT 2XC2)	Bis-GMA, TEGDMA, diluent monomer, tertiary amine, camphorquinone, proprietary releasing fluoride fillers (S337 LOT 2XC2)	Ultradent Products Inc, South Jordan, UT

HEMA=hydroxyethylmethacrylate; Bis-GMA=bisphenol glycidyl dimethacrylate; UDMA=urethane dimethacrylate; MDP=10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA=triethylene glycoldimethacrylate

Table 2: *Bonding Procedures*	
Single Bond	Etching: Apply 15 seconds, rinse 10 seconds, dry gently Bonding: Brush on two consecutive coats of priming resin, air dry gently 2-5 seconds, light cure 10 seconds
Stae	Etching: Apply 20 seconds, rinse thoroughly, dry gently Bonding: Apply two layers of Stae, leave undisturbed 20 seconds, air dry gently, light cure 20 seconds
Clearfil SE Bond	Self-etching primer: Apply with a brush, leave undisturbed for 20 seconds, air dry mildly Bond: Apply with a brush, air thin gently, light cure 10 seconds
PermaQuik	Etching: Apply 15 seconds, rinse 5 seconds, dry gently Priming: Rub firmly 15 seconds, air-thin 1-3 seconds, air-dry 10 seconds, light cure 20 seconds Bonding: Rub with moderate pressure 15 seconds, gently air thin, light cure 20 seconds

step system (Table 1). The 80 teeth were divided randomly into the four dentin bonding treatment groups and bonded with one of the dentin bonding agents following manufacturers' instructions (Table 2). The bonded flat surface was covered with a layer of Silux Plus resin composite (3M Dental Products) less than 1 mm thick and light cured for 40 seconds. The specimens in each dentin bonding group were randomly assigned to four sub-groups that were kept in phosphate buffered saline solution (pH 7.4) containing 0.01% sodium azide (which had a stable pH and minimized bacterial growth) at 37°C for 24 hours, three months, six months or 12 months. The prepared storage solution was stored at 4°C before use, and heated to 37°C prior to changing it every two weeks, to avoid thermo-cycling specimens. All bonded specimen margins were finished and polished with Sof-Lex disks (3M Dental Products) 24 hours after manufacture. The specimens were checked with a dissecting microscope to ensure that there was no flash at

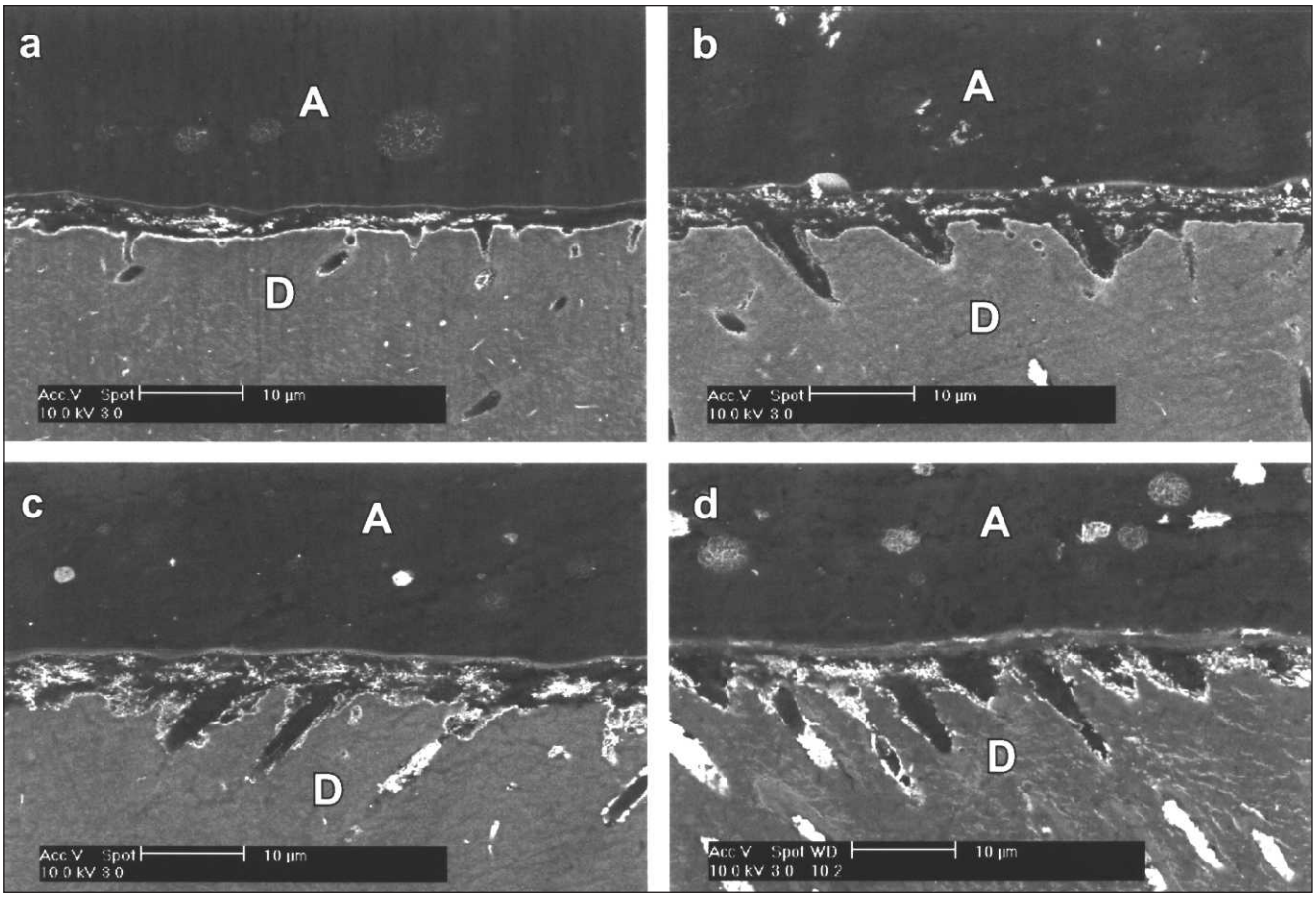

Figure 1. *Backscattered SEM images of the interface bonded with Single Bond to dentin (x1000): a. 24-hour storage, b. three-month storage, c. six-month storage, d. 12-month storage. Six and 12-month storage show more silver deposition within the hybrid layer and adhesive resin (A, adhesive resin; D, demineralized dentin).*

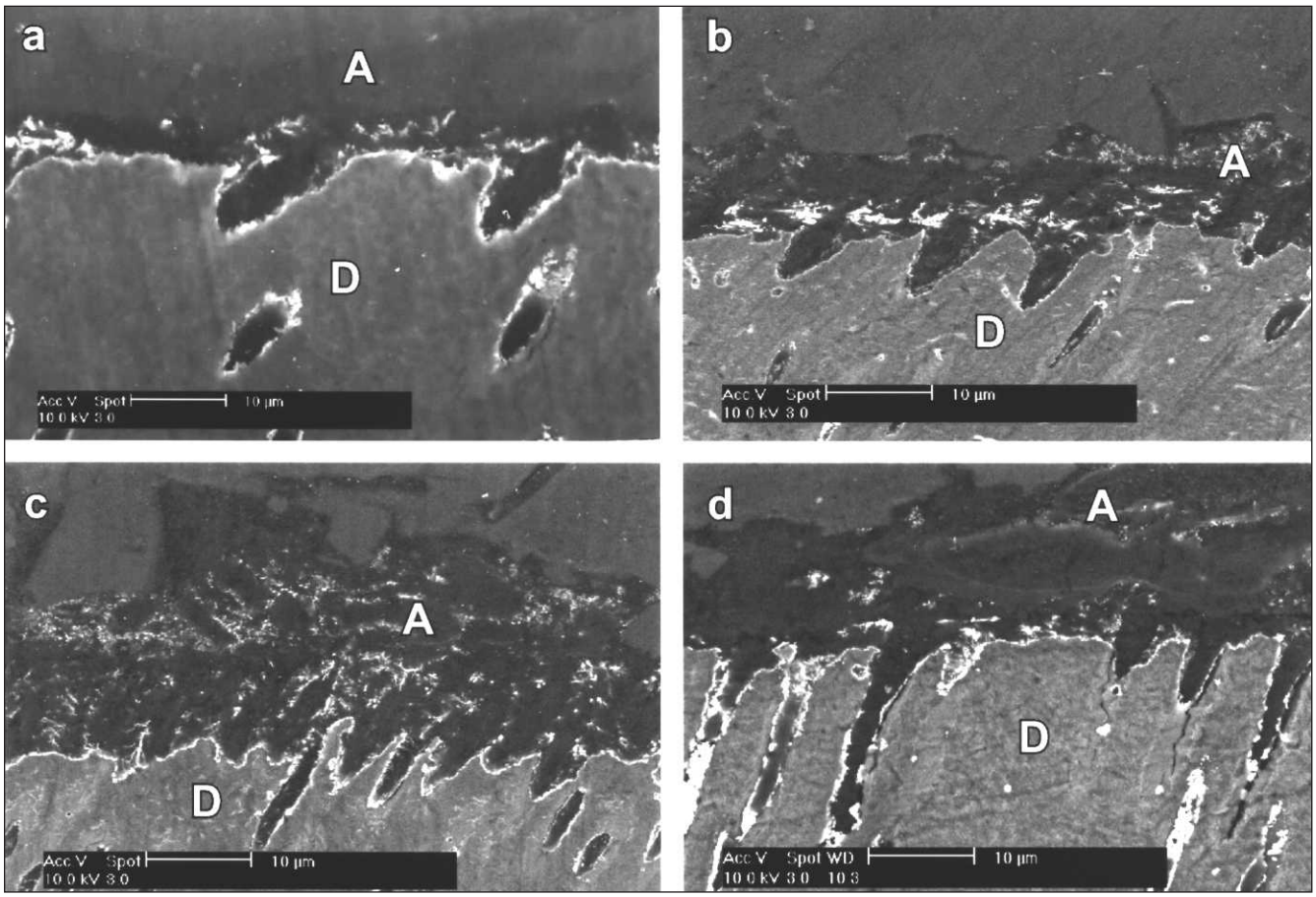

Figure 2. *Backscattered SEM images of the interface bonded with Stae to dentin (x1000): a. 24-hour storage, b. three-month storage, c. six-month storage, d. 12-month storage. Six and 12-month storage show more silver deposition within the hybrid layer and in the adhesive resin (A, adhesive resin; D, demineralized dentin).*

the margins of the bonded surfaces. At the end of each storage period, the root apices of each tooth were sealed with Silux Plus and the entire tooth, except for an area 1 mm adjacent to the bonded interface, was coated with two layers of nail varnish. The teeth were placed in a 50% silver nitrate solution in total darkness for 24 hours, rinsed in running water for five minutes, immersed in photo-developing solution and exposed to a fluorescent light for eight hours to reduce silver ions to metallic silver. After removal from the developing solution, the teeth were rinsed thoroughly in running water for five minutes (Sano & others, 1995 a,b).

Three sections were cut longitudinally and bucco-lingually across each bonded tooth, resulting in a total of 15 disks for each sub-group. All the cut surfaces were polished with increasingly fine diamond pastes (6, 3, 1 µm; Buehler Ltd, Lake Bluff, IL 60044, USA). The specimens were ultrasonically cleaned, air-dried, mounted on aluminum stubs and kept in a desiccator for 24 hours, followed by coating with carbon and observation under scanning electron microscope (SEM) using the backscattered electron mode.

RESULTS

Single Bond (Figure 1) stored for 24 hours showed silver deposition at the base of the hybrid layer, and patches of silver were intermittently deposited within the hybrid layer. A line of silver deposition occurred at the top of the hybrid layer. Silver particles were also seen in the adhesive resin layer. Most tubules took up silver just on the tubule walls; few tubules were filled with silver deposits. After three months storage, the leakage pattern revealed a similar pattern to that at 24 hours. The specimens at six months showed slightly more silver deposition compared with the 24-hour group. However, specimens after 12-months storage had greater silver deposition at the base and within the hybrid layer, and more tubules were filled with silver particles. The line of silver deposition at the top of the hybrid layer was thicker than the 24-hour specimen. Silver particles were evident in the adhesive layer.

Stae (Figure 2) showed a pattern similar to Single Bond, but with greater silver deposition in the adhesive layer, which was thin. Specimens at 24 hours and three months were similar in silver deposition. Specimens at

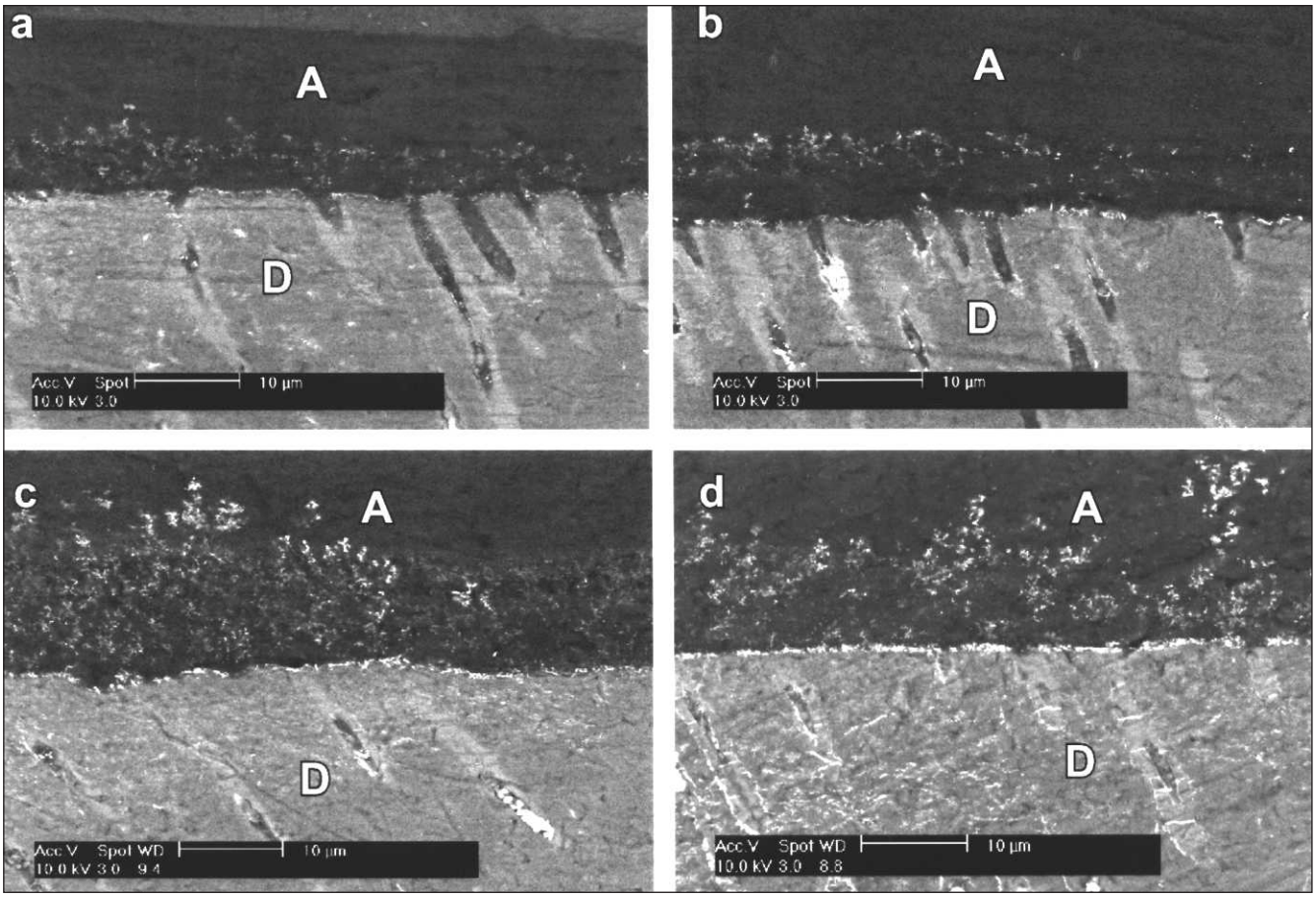

Figure 3. *Backscattered SEM images of the interface bonded with Clearfil SE Bond to dentin (x1000): a. 24-hour storage, b. three-month storage, c. six-month storage, d. 12-month storage. Six- and 12-month storage show more silver deposition in the adhesive resin and within the hybrid layer (A, adhesive resin; D, demineralized dentin).*

six and 12 months showed greater silver deposition in the tubules, whereas silver deposits within the hybrid layer were the similar to the 24-hour specimens.

Clearfil SE Bond (Figure 3) showed a different type of silver deposition pattern compared with the other three adhesive systems. There were intermittent, diffuse silver deposits within the hybrid layer, and dentinal tubules rarely took up silver. The adhesive layer also showed some silver deposition. Specimens at six months and 12 months showed more silver in the adhesive layer, and the specimens after 12-months storage revealed denser silver deposition compared with the 24-hour specimens.

PermaQuik (Figure 4) demonstrated a leakage pattern where silver deposition formed a thin line at the base of the hybrid layer with a diffuse distribution of particles within the hybrid layer; the tubule walls took up small amounts of silver, but no filler particles were noted in the adhesive layer. All the samples at the different storage periods showed similar silver deposition patterns except for 12 months, which had a line of silver deposition between the hybrid layer and adhesive resin.

DISCUSSION

It is important for a long-term investigation of adhesion to tooth substrates to ensure that the pH stability and bacterial growth are prevented, otherwise these changes may affect the resin structure and the dentin, thus possibly producing invalid results (Burrow & others, 1993). In this study, phosphate buffered saline containing 0.01% sodium azide, used as the storage solution, was changed every two weeks, which maintained a pH of 7.4 and minimized bacterial growth. However, it has to be pointed out that laboratory systems are not truly reflective of the oral environment in which restorations exist, with positive dentinal fluid pressure and a myriad of bacteria and their subsequent by-products. Such factors as these might affect the results of the investigation.

In general, all systems showed increased nanoleakage within the hybrid layer and/or adhesive resin to some extent over 12-months storage. This may be a consequence of incomplete penetration of the adhesive resin into the demineralized dentin, leaving the collagen unenveloped by resin, or it may result from poly-

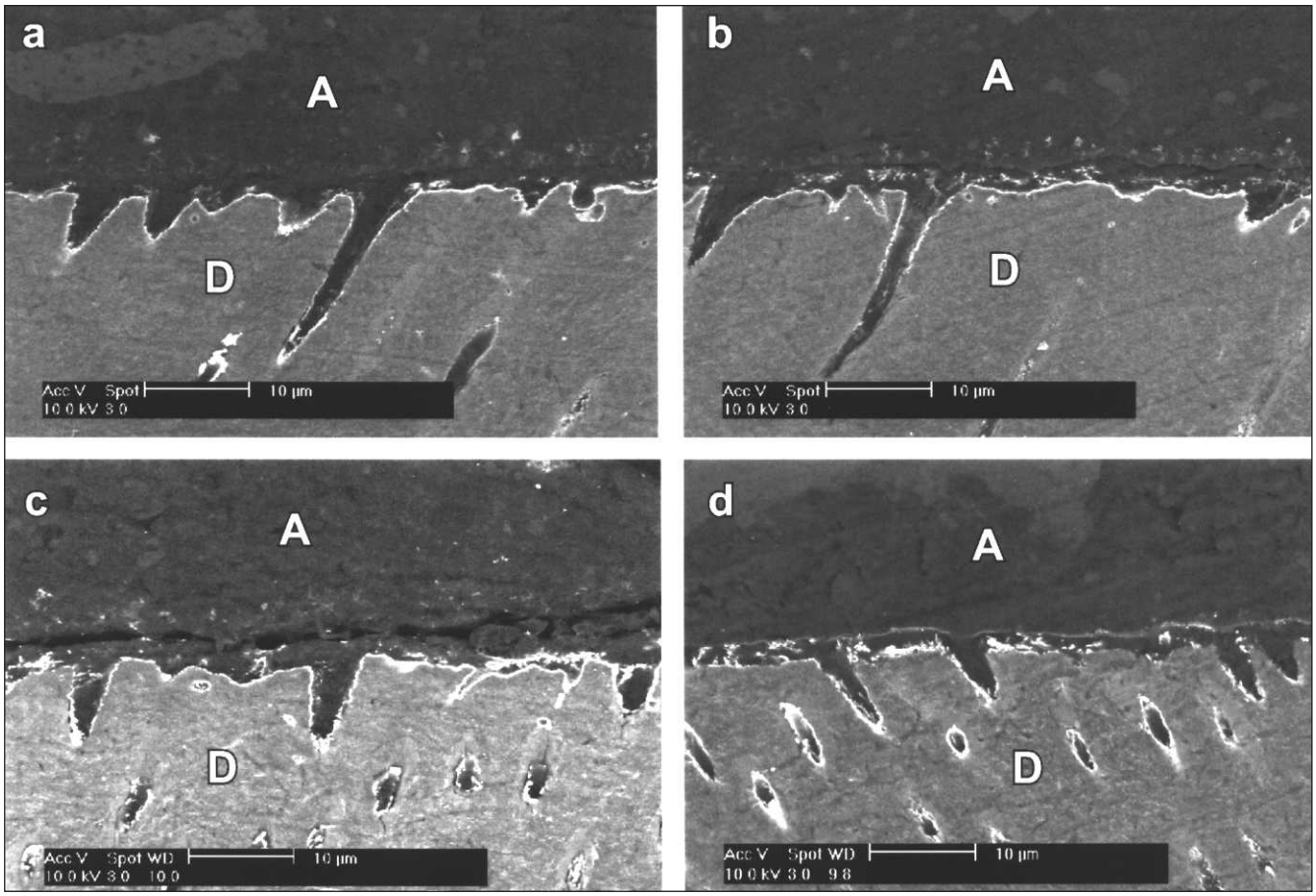

Figure 4. *Backscattered SEM images of the interface bonded with PermaQuik to dentin (x1000): a. 24-hour storage, b. three-month storage, c. six-month storage, d. 12-month storage. All the figures showed the similar leakage pattern and d had an extra silver line at the top of the hybrid layer (A, adhesive resin; D, demineralized dentin).*

merization shrinkage (Sano & others, 1995a,b; Spencer & others, 2000). The nanoleakage pathway may be located within the adhesive resin, within the hybrid layer and within partially- or fully-demineralized dentin (Sano & others, 1999). Porosity in the hybrid layer not infiltrated by adhesive resin is the most important pathway for nanoleakage, since solutions can penetrate into these micrometer-sized spaces and hydrolyze the exposed collagen and adhesive resin, influencing the durability of the bond (Sano & others, 1995a,b; Burrow & others, 1996).

During long-term storage, water sorption inevitably occurs, and the consequent swelling of the resin may result in the closure of any space between the bonding resin and the dentin surface (Burrow & others, 1996; Burrow, Inokoshi & Tagami, 1999). Conversely, stresses may simultaneously be induced at the bonding resin-dentin surface, which may pull the collagen fibers into the hybrid layer and resin, leading to tearing along the bonded interface as the collagen fibers become weaker over time from hydrolysis (Burrow & others, 1999). Incomplete curing of the resin may cause certain components of the resin to leach into the water, leaving minute spaces within the polymer matrix that would subsequently be replaced by water (Burrow & others, 1999). This provides one explanation as to why some adhesive resins showed greater silver deposits after 12 months of storage.

Paul & others (1999) found that using an air stream may not effectively remove residual water from a HEMA/water primer, with remaining water interrupting the formation of an adequate polymer network. Acetone or alcohol contained in the adhesive resin of one-bottle systems may help displace water from the moist surface. All three systems that showed silver deposition in the adhesive contain water and HEMA, while PermaQuik, which contains ethanol and HEMA, showed no silver in the adhesive layer. Thus, it was believed that water that remained in the primed and bonded dentin may not have been evaporated completely and was subsequently filled with silver particles. Van Meerbeek & others (1999) reported that solvent differences and the way of applying primer/adhesive, that is, scrubbing or not, may account for the dif-

ference in the effectiveness of hybridization. Clinically, dentinal fluid, which contains hydrolytic enzymes and calcium (Eick & others, 1997) could fill any voids within the hybrid layer or the adhesive resin. It is reported that demineralized or new mineralization may occur (Clarkson & others, 1991; Tatsumi & others, 1992). Thus, it is of concern that if the demineralized collagen fibers at the base of the hybrid layer may not be enveloped by resin, they can resist hydrolytic attack if that region is not remineralized (Eick & others, 1997). Tatsumi & others (1992) observed remineralization of demineralized dentin within four months following restoration with adhesive resin in monkey teeth. However, Sano & others (1999) reported in a clinical study that porosity within the hybrid layer increased after placement of 12-month restoration, indicating hydrolytic damage of the adhesive interface might occur via the nanoleakage pathway. In a laboratory study, Burrow & others (1996) found that bond failures increased at the base of the hybrid layer at three years due to hydrolytic degradation, and they proposed that this was the weak link in durable adhesive bonding. In the current study, all systems using phosphoric acid as an etchant showed more silver deposition at the base of the hybrid layer.

With the exception of Clearfil SE Bond, the other three systems used phosphoric acid as the demineralizing agent, and all formed a line of silver deposition at the base of the hybrid layer. This is believed to result from incomplete penetration of adhesive resin into the whole thickness of the demineralized dentin. Spencer & others (2000) recently reported that the percentage of Single Bond adhesive penetrating the demineralized dentin dropped from approximately 70% at the resin-dentin interface to approximately 20% at the base of the hybrid layer, suggesting that the contribution from the Single Bond adhesive is less than 50% throughout , which is nearly half of the demineralized dentin. Clearfil SE Bond simultaneously etches and primes the dentin, allowing the adhesive resin to fully penetrate the demineralized dentin surface, and thus minimal silver deposition occurred at the base of the hybrid layer. However, all systems could not ensure complete penetration of adhesive resin into the inter-fibrillar collagen spaces, otherwise, silver would not appear within the hybrid layer.

The conventional three-step dentin bonding system PermaQuik stored for 24 hours showed a similar leakage pattern to that previously reported (Li & others, 2000a,b), and had the least change in nanoleakage over 12-months storage. In Figure 4c, a gap in the six-month sample occurred after the silver staining procedure or/and during preparation for SEM observation; otherwise, a greater amount of silver would fill up the gap. Only a slight increase in silver deposition occurred within the hybrid layer and tubule walls after 12-months. Silver deposition at the base of the hybrid layer appeared the same for all the storage periods. For Clearfil SE Bond, the adhesive layer showed more silver deposition over time. Both single-bottle systems (Single Bond and Stae) demonstrated greater silver deposition at the base of the hybrid layer and in the tubules, and Single Bond stored for 24 hours showed a similar leakage pattern to that previously reported (Li & others, 2000a,b). This may suggest that different dentin bonding systems may contain a weak link in different bonding zones, such as at the top, middle or the base of the hybrid layer, or in the adhesive resin itself. These differences could be related to the different compositions of the dentin bonding systems. For example, the degree of hydrophobicity/hydrophilicity of the bonding resins may prevent or allow penetration of solution through the resin and hybrid layer. Burrow & others (1999) found that Clearfil Liner Bond II, containing hydrophilic HEMA and 10-methacryloyloxydecyl dihydrogen phosphate (MDP), showed greater water sorption. This may allow for greater penetration of water into the adhesive layer, subsequently translating into increased silver deposition.

CONCLUSIONS

The dentin bonding systems observed in this study showed an increase in nanoleakage during the 12-months storage period. The conventional three-step system, PermaQuick, was least affected. While the clinical retention rate of restorations is a valuable objective criterion to evaluate the clinical performance of dentin bonding systems, clinical microleakage may better aid in differentiating modern adhesive systems, since extensive leakage may have occurred around a restoration while it remains without complete debonding. Clinically, nanoleakage may also be important especially for modern dentin bonding systems that can achieve gap-free margins.

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Porosity in Manually and Machine Mixed Resin-Modified Glass Ionomer Cements

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Clinical Relevance

Hand-mixed resin-modified glass ionomer cement has greater porosity and weaker shear force resistance than machine mixed cement.

SUMMARY

The powder and liquid components of resin-modified glass ionomer cements are available in manual and machine mixed forms. This study quantified the effect mixing methods have on the porosity and shear strength of a resin-modified glass ionomer cement (RMGIC).

A RMGIC (Fuji II LC) was manually or machined mixed according to manufacturer's instructions. Thin, disc-shaped specimens (n=5) were made by compressing the cement between glass platens to a thickness of approximately 76 µm. The specimens were light cured for 120 seconds. Digital images of the specimens were recorded using a measuring microscope and slide film scanner. Digital imaging software was used to determine the number and volume of the cement's pores. Shear test specimens of manual and machine mixed cements (n=10) of each group (~800 µm thickness) were made as previously described. Shear punch tests were conducted using a 3.75 mm diameter punch mounted on a universal testing machine.

The mean number and total volume of pores in the manually mixed specimens was considerably greater than that of the machine mixed group ($p<0.05$). The shear punch test results of the machine mixed group was significantly higher than the manual mixed group, ($p<0.05$).

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INTRODUCTION

A wide selection of glass ionomer cement formulations is available for use in clinical dentistry. Glass ionomer cements for use as luting agents, cavity preparation liners and a variety of restorative materials are available. The formation of glass ionomer cement requires a chemical reaction between an acid and base reagent (Saito, Tosaki & Hirota, 1999). The fluoraluminosilicate glass powder (base) and the polycarboxylic/water (acid) must be mechanically mixed prior to use. Glass ionomer cement package options provide the practitioner with either manual or machine mixed formulations. Both mixing methods result in air inclusion into the cement mixture. Conventional glass ionomer cement porosity studies report the percent of air ranges from six to nine surface area percent (Mitchell & Douglas, 1997; Bertenshaw & Piddock, 1993). Similar effects have been observed during manual-mixing of catalyst/base

composite resin pastes (~2-18 surface area percent) (Gjerdet & Hegdahl, 1978; Jörgensen & Hisamitsu, 1983; Ogden, 1985; Valcke & Duggan, 1981) and polymethylmethacrylate powder and liquid monomer when used to produce bone cement (five to 12 surface area percent) (Jasty & others, 1990; Lindén & Gillquist, 1989; Wixson, Lautenschlager & Novak, 1987). Researchers found that air inclusion has a detrimental effect on the mechanical properties of composite resin (Nakayama & others, 1974; de Gee, 1979) and bone cement (Jasty & others, 1990; Wixson, 1992; Topoleski, Ducheyne & Cuckler, 1990; Burke, Gates & Harris, 1984).

Adding photo-polymerizing resins into glass ionomer cements, while improving physical properties, has not eliminated the mixing process.

This study quantified the population size of air pores present in manual and machine mixed resin-modified glass ionomer cement and determined the effect porosity has on shear strength.

METHODS AND MATERIALS

Porosity Sample Preparation

Glass ionomer cement specimens of resin-modified glass ionomer cement (RM-GIC) (Fuji II LC, GC America Inc, Alsip, IL 60803, USA) were fabricated using manual or machine mixing methods.

Manually Mixed

The glass ionomer powder and polyacid liquid were weighed on an analytical scale (± .01 grams) at a ratio of 3 to 1, respectively. The powder and liquid were hand spatulated for 30 seconds and placed in a syringe. The mixture was injected onto a glass microscope slide and a second slide was placed over the mixture. The glass slides were placed between the spindles of a digital disc micrometer and compressed to produce a cement disk with a thickness of 0.076 mms and approximately 20 mms in diameter. The resin-modified cement was polymerized using a visible light photo-lamp (3M Dental Products, St Paul, MN 55144, USA) for a total of 120 seconds. Five specimens were fabricated.

Machine Mixed

The encapsulated glass ionomer cement material was mixed in a triturator for 10 seconds at about 4,000 cycles per minute according to manufacturer's recommendations. The mixture was syringed directly from the capsule onto glass slides. The remaining specimen fabrication and image recording steps were identical to the manually mixed specimens.

The specimens were photographed with a 35 mm camera attached to a microscope (100x magnification). Five images of each specimen were recorded. The processed 35 mm film was optically scanned at a resolution of 300 dpi and stored in a digital format. A digital imaging software program (Sigma Scan Pro, SPSS Inc, Chicago, IL 60606, USA) was used to quantify the number and size of the pores present in the glass ionomer cement. The dimensions of the digital image's objects were calibrated using a silicon wafer with an etched grid (Ted Pella, Inc, Reddin, CA 96003, USA). The grid was photographed and optically scanned in the previously described manner. Pore volume was determined from the measured pore diameters applied to the equation for a sphere $V=4/3\ \pi r^3$.

Evaluation of the test group data included testing for distribution normality (D'Agostino) and comparison of test variables, including the number of pores per specimen (Student's *t*-test and Kolmogorov-Smirnov test) at a level of significance of 0.05.

Shear Punch Test Sample Preparation

Manual and machine mixed RM-GIC was used to fabricate shear punch test specimens (n=10). Cement mixtures were syringed into molds consisting of steel arbor shims (3/4" outside diameter x 1/2" inside diameter x 0.031" thickness). The cement-filled shims were compressed between two glass slides and the RM-GIC was photo-polymerized using a visible light photo-curing lamp (XL3000, 3M Dental Products) for 120 seconds. The specimens were stored in 37ºC deionized water for 24 hours prior to testing.

Shear tests were done with a 3.75 mm diameter punch rod mounted in a universal testing machine (Instron Corp, Canton, MA 02021, USA) using a crosshead speed of 1 mm/minute. Specimens were loaded to failure and the generated stress-displacement curves were recorded on a strip chart. The shear loads of the specimens were calculated using the expression: $ShearLoad=W/\pi DT$. where W equals the load at failure (MPa), D equals the punch diameter (mm) and T equals the specimen thickness (mm). Statistical analysis of shear load data were conducted using a two-tailed Student's *t*-test at a 0.05 level of significance.

RESULTS

Mixing the powder and liquid compounds of resin-modified glass ionomer cement resulted in the formation of numerous pores (Figures 1 and 2). The mean pore size distribution of the manual and machine mixed samples is presented in a histogram using pore diameter intervals of five microns (Figure 3). Both manual and machine samples exhibit a non-normal distribution pattern. D'Agostino normality tests verify this.

The number of pores per specimen for the manually mixed specimens is significantly greater than the machine mixed cements. Figure 4 shows a box plot representing the data. The box represents the interquartile range (IQR) or middle 50% of the data, with the top and bottom of the box at the 75th and 25th percentiles, respectively. The horizontal line within the box is located

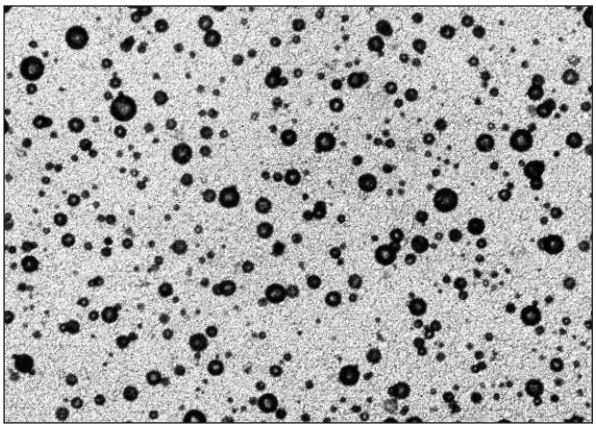

Figure 1. *Photographic image of manual mixed resin-modified glass ionomer cement.*

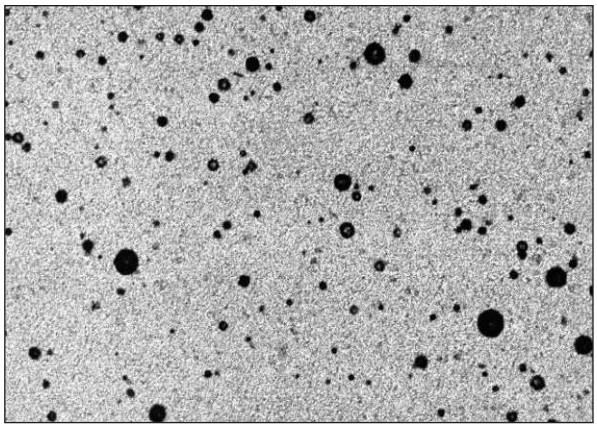

Figure 2. *Photographic image of machine mixed resin-modified glass ionomer cement.*

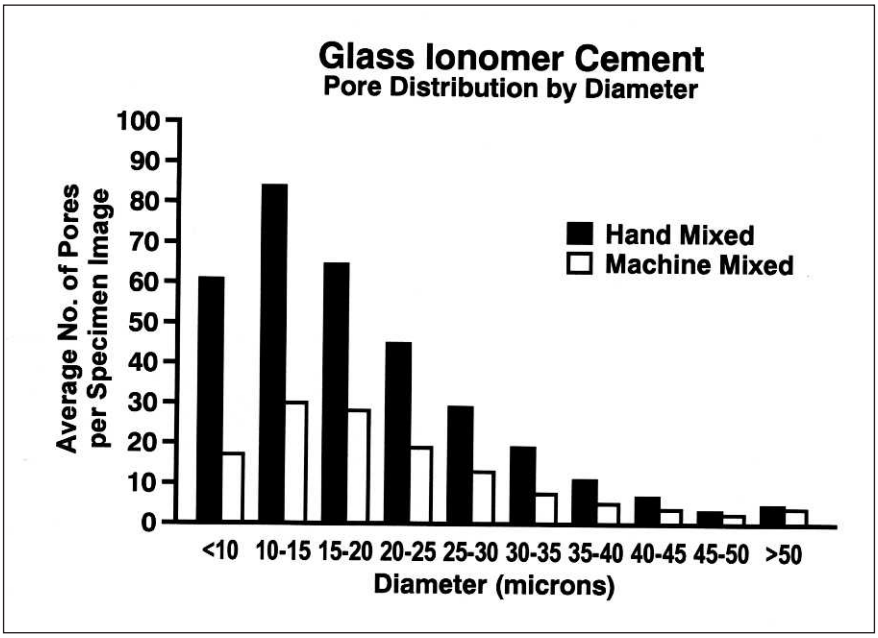

Figure 3. *Pore distribution by diameter.*

at the population median. The T-shaped lines (fence) projecting from the 75th and 25th percentiles extend to include the values within a border plus or minus 1.5 times the IQR, respectively. Data greater than the fence ranges are considered outliers and are represented as circles. A comparison of the test sample distributions using the Kolmogorov-Smirnov test resulted in a significant difference between manual and machine mixed groups ($p< 0.001$).

The percent of the specimen's volume made up of air was determined by summing the volume of the recorded pores. The pores in the manually mixed samples averaged 3.11 percent of the cement's volume; whereas, the pore volume of the machine-mixed sample had significantly less porosity, with only 1.89 percent.

Punch Test Results

Figure 5 illustrates the results of the shear punch tests. Data from both the manual and machine mixed test groups presented with a normal distribution as evaluated by the D'Agostino test for normality. A two-tailed Student's *t*-test of the shear strength values found a significant difference between the test groups ($p<0.003$). The machine mixed cement exhibited greater shear strength.

DISCUSSION

Porosity Distribution

The diameter of the pores in the manual and machine mixed specimens ranged from approximately 6.0 to 60 µms. Objects with a diameter smaller than 6 µm were excluded from analysis due to the limitations of the optical magnification and digital image resolution that made it difficult to distinguish pores from aggregates of glass particles. Similar ranges of pore diameter have been reported in studies of glass ionomer luting cements (Bertenshaw & Piddock 1993; Mitchell & Douglas, 1997 and Smales & Joyce, 1978).

The method used in this and similar studies revealed the pore size to be dependent on the selected thickness of the cement specimen. In this study an 80 µm thickness was chosen because it provided photographic images with a sufficient depth of field as well as dispersing the pores to minimize the number of overlapped pores. This made counting the number of pores and measuring their diameter possible with the least amount of error.

Specimen thickness can affect the shape of pores. Bertenshaw & Piddock (1993) noted that compressing

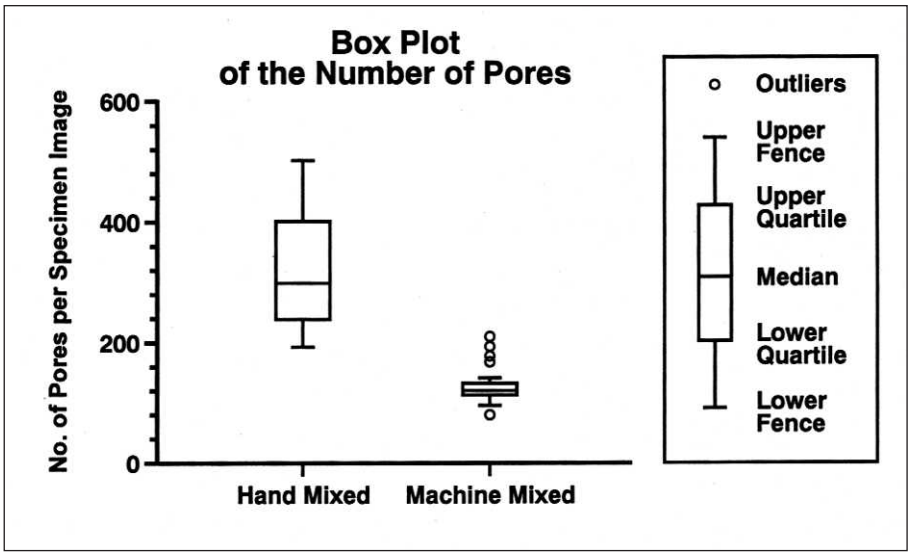

Figure 4. *Box plot of the number of pores.*

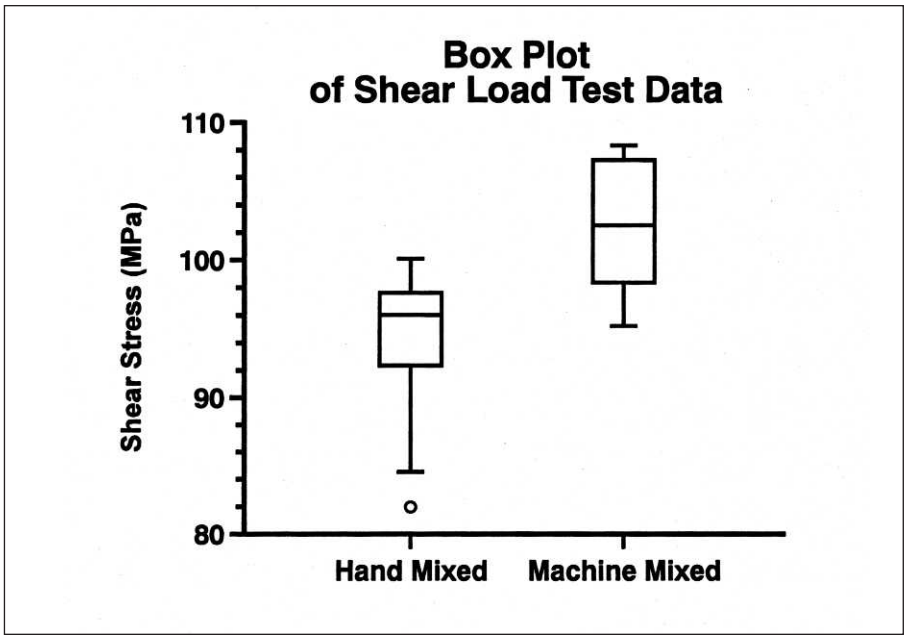

Figure 5. *Box plot of shear load test data.*

the cement film between glass plates could cause a flattening or elongation of the pores. They evaluated 50 µm and 100 µm thick specimens and found thicker specimens containing pores ranging from 9 to 46 µm were the same as thinner specimens containing pores 6 to 63 µm in diameter.

Relatively few pores in this study were greater than 60 µm (1% in the machine mixed and 2% in the manual mixed). Manual and machine mixed glass-ionomer cement specimens had similarly-shaped pore diameter distributions (Figure 3). However, the manually mixed group contained 2.5 times as many pores as the machine mixed specimens.

Similar pore diameter size has been reported in manual mixed-paste/paste-composite resins. Fischel, Cruickshanks-Boyd & Davies (1982) & Gjerdet & Hegdahl (1978) reported the pore's diameter as generally 50 µm or less with a mean diameter in the 20 to 25 µm ranges.

Measurement Methods

Several methods for quantifying a dental material's porosity have been reported. One method determined porosity by measuring the surface area of the pores exposed on the polished surfaces of specimens as a percentage of the analyzed site. Ogden, (1985) measured the porosity of a wide range of composite resin restorative materials. He found that two-paste formulations (catalyst/base) had a mean pore surface area of 2.0-3.3 percent, whereas the porosity of single paste, light-activated composite resins was in the range of 0.44-1.4 percent. These results are similar to those reported by Gotfredsen, Hörsted & Kragstrup, (1983).

Gjerdet & Hegdahl, (1978), Gotfredsen & others, (1983) and Medlock & others (1985) found that handling techniques also affect composite resin porosity. Use of a syringe to place composite resin into specimen molds produced less porosity than a bulk placement delivery technique.

Other researchers have used image processing of thin transparent sections of dental materials to determine the number and surface area of pores. The calculated pore surface area per specimen surface area or volume has been reported (de Gee, 1979, Alster & others, 1992, Bertenshaw & Piddock, 1993, Fano, Ortalli & Pozela, 1995).

Punch Test

The shear punch test of the machine and manual mixed specimens revealed a significant difference between the groups. The machine group exhibited a 9% increase in shear strength as measured by the shear punch test.

The punch test apparatus used in this study is similar to the device described by Roydhouse (1970). The punch test has been applied to the study of dental

materials such as dental amalgam (Roydhouse, 1969), composite resin and glass ionomer cements (Mount, Makinson & Peters, 1996), cavity preparation liners (von Fraunhofer & Chapman, 1987; Carvalho, Del'Hoyo & Suga, 1995) and tooth structure (Smith & Cooper, 1971; Carter & others, 1983; Sedgley & Messer, 1992). Mount & others (1996) cited the ease and consistency of specimen fabrication as a significant advantage of this testing method when applied to polymeric dental materials. Specimens are readily produced without special molds or the need for additional sizing or polishing. Specimens can be prepared in dimensions that can be formed from a "single mix" or clinically applicable increment of material and readily photo-cured. This is significant since glass ionomer cements are susceptible to layering effects. Anstice, Nicholson & McCabe (1992) reported that GIC specimens built up in layers were significantly weaker than single layer specimens when tested in compression.

Studies involving the effect of porosity on the strength of glass ionomer cement have not been widely reported. Studies measuring the mechanical properties of GIC generally compare different formulations from different manufacturers in addition to manual and machine mixing variations (Mitchell & others, 1994; Arcoria & others, 1992, White, 1993). Formulation of the GIC in capsular form at the time of this study was identical to the manually mixed version (personal communication, GC America).

Mitchell, Orr & Russell (1998) investigated the influence that GIC mixing methods have on the probability of survival of cemented post using pull-out tests. Commercially available bulk packaged and encapsulated glass-ionomer luting cements were prepared using manual and machine mixed methods. In general, specimens fabricated with machine mixed GIC exhibited higher probabilities of survival than manual mixed specimens.

de Gee (1979) investigated the effect of porosity on the strength of paste-based composite resin dental materials. He found that applying a partial vacuum to a machine-mixed encapsulated composite resin resulted in a 10-fold decrease in porosity and a 10% increase in diametral strength. Nakayama & others (1974) found that exposing composite resin to elevated pressure can greatly reduce porosity and increase the elastic modulus of this viscoelastic material. McCabe & Ogden (1987) found that diametral tensile and compressive strength are significantly reduced when porosity is induced into photo-cured composite resin.

Numerous studies involving the control of porosity during the preparation of bone cement have been reported (Davies & Harris, 1990, Jasty & others, 1990, Chin, Stauffer & Chao, 1990, Wixson 1992). Bone cement, polymethlymethacrylate (PMMA), is used in the fixation of total hip prostheses. PMMA mechanical properties are maximized when cured under high heat and pressure (that is, denture acrylic). However, bone cement must be autopolymerized at body temperature. This can result in a significantly weaker, more porous material. Various methods, including applying partial vacuum and/or centrifugation during mixing, have been shown to significantly improve the compressive and tensile strength of bone cement (Norman & others, 1995, Burke & others, 1984).

Topoleski & others (1990), compared the fracture surfaces of recovered *ex vivo* bone cement specimens to fracture surfaces present in bone cement found in *in vitro* static and dynamic tests. Pores within the cement function act as stress concentrators. Pores act as crack initiation sites and contribute to the propagation of crack formation. Decreasing the porosity in bone cement by centrifuging the mixture results in increased dynamic tension/compression fatigue lifespans (Davies & others, 1988). Studies by James & others (1992) found that pore size and distribution affect the crack initiation and fatigue behavior of bone cement.

Goldman (1985) determined the crack propagation resistance (K_{Ic}) and size of flaws a_o inherent in a variety of polymeric dental restorative materials including conventional glass ionomer cements. Glass ionomer cement was found to have a low K_{Ic} and high a_o values. This makes conventional GIC unsuitable for use in stress bearing restorations (that is, multi-surface posterior restorations). The role size and distribution of pores on K_{Ic} and a_o and its effect on resin-modified glass ionomer cement are not known.

CONCLUSIONS

In this study, resin-modified glass ionomer cement exhibited less porosity and greater shear strength when prepared using machine mixing methods compared to manual methods.

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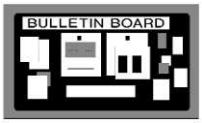
Departments

Announcements



31ST Annual Meeting of the Academy of Operative Dentistry

20-22 February 2002, Fairmont Hotel, Chicago, IL

The Academy of Operative Dentistry's 2002 Annual Meeting features an essay program that stresses the importance of the interface between the restorative dentist and various specialties to broaden both the scope and perspective of modern practice. The scientific session opens on Thursday, February 21 with a multimedia presentation by Dr Tomas Seif, a restorative dentist, and Dr Carlos Boveda, an endodontist. Their presentation "The Restorative-Endodontic Approach: A Winning Philosophy" features a teamwork approach to take advantage of the newest techniques in their fields. Dr Lars Bjorndal, the selected Buonocore Memorial Lecturer, follows with the most current science on "Dentin Caries: Progression and Management." The Academy's own Dr Frank Caughman will begin the afternoon session by "Shedding New Light on Composite Polymerization." Dr Robert Cronin follows with a review of the controversial topic "Implant Use in Growing Patients," and the afternoon program culminates with Dr Bart Johnson's "Tips and Tricks for Successful Local Anesthesia." Friday's essay program features an all morning presentation by Dr John Kois entitled "Managing the Restorative Periodontal Interface: New Paradigms for Predictable Results." Dr Richard Stevenson has organized an outstanding selection of table clinics for Friday afternoon to complete the Academy's 2002 scientific session.

Of course, the Companion Activities Program will once again provide an extremely enjoyable opportunity for spouses, family members and friends to sample some of Chicago's unique and delicious attractions. Thursday morning combines a "Continental Breakfast with a French Flair" at the Fairmont Moulin Rouge Room with an excellent program, "The Love of French Country Décor," by designer Barbara Ponzo from Calico Corners. Friday's tour and program, titled "A Bit of France in Chicago," are under the expert guidance of Art History instructor Joseph Cunniff. This package includes a viewing at the Art Institute of the largest collection of French Impressionist paintings outside of Paris and a fabulous luncheon at Cyrano's Bistro and Wine Bar.

Finally, the Gala reception on Thursday evening is an opportunity to meet with friends and colleagues in an elegant setting planned especially for members and guests of the Academy. For meeting information, please contact Dr Gregory Smith, PO Box 14996, Gainesville, FL 32604-2996; Fax 352/371-4882; e-mail: gesaod@ufl.edu. See you in Chicago!

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 $2,600.

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 2003, 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 15, 2001 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 20-22, 2002.

2002 Meeting of the Academy of Operative Dentistry European Section

The 2002 meeting of the Academy of Operative Dentistry European Section will be hosted by the University of Nijmegen College of Dental Science in Nijmegen, The Netherlands. The meeting, to be entitled "Adhesive dentistry today. Transfer of research into practice" will be held December 5th-7th, 2002. Details may be obtained from:

Dr EH Verdonschot,
University of Nijmegen College of Dental Science
PO Box 9101, NL-6500 HB
Nijmegen, The Netherlands

Tel: (31) 24 3614058/6410
Fax: (31) 24 3540265
e-mail: e.verdonschot@dent.kun.nl

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School of Dental Medicine
University at Buffalo

The Department of Restorative Dentistry at the School of Dental Medicine, University at Buffalo, is seeking applications for two (2) full-time positions for preclinical, clinical and didactic teaching in the predoctoral program in all aspects of restorative dentistry, with primary focus on direct restoratives. Responsibilities also include administration and program development in the predoctoral or advanced level (AEGD), development of independent research program and scholarly activity. Limited teaching at advanced level (AEGD) may be required. Participation in continuing education and faculty intramural practice expected. Minimal qualifications require a DDS or equivalent and eligibility for licensure in New York State, evidence of completed advanced training in a US-accredited GPR or AEGD program and practice experience in clinical restorative dentistry. Prior teaching experience preferred. For consideration at the senior level, there must be evidence of significant teaching experience, restorative program administration and scholarly activity. The University is an Affirmative Action/Equal Opportunity Employer. Letter of interest, current CV and names/addresses of three references should be sent to: Susan Kowalewski, Assistant to the Chair, Department of Restorative Dentistry, 215 Squire Hall, 3435 Main Street, School of Dental Medicine, Buffalo, NY 14214.

University of Florida College of Dentistry

The University of Florida College of Dentistry invites applications for a full-time clinical or tenure track faculty position in the Department of Operative Dentistry at the Instructor, Assistant or Associate Professor levels. A dental degree from an ADA-accredited dental school or equivalent is required. Private practice experience and teaching experience are preferred. Salary and academic rank will be commensurate with qualifications. Duties associated with the position will include clinical and/or preclinical teaching. Participation in Faculty Practice is expected. Scholarly activity is required for tenure track appointments. Women and minorities are encouraged to apply. This selection process will be conducted under Florida's Government in the Sunshine and Public Records Law. Please submit curriculum vitae and the names of three references to Dr Paul K Blaser, Chair, Search Committee, University of Florida College of Dentistry, PO Box 100415, Gainesville, FL 32610-0415 by October 15, 2001. EEO/AA/EA employer.

The University of Texas — Houston

The University of Texas Health Science Center at Houston, Dental Branch seeks applicants for a full-time, tenure/clinical educator track position in the Department of Restorative Dentistry and Biomaterials at the Assistant or Associate Professor level. Applicants must have a DDS/DMD degree with prior teaching and/or private practice experience. Advanced training in Operative Dentistry, Prosthodontics or GPR/AEGD certification is preferred. Responsibilities include clinical and preclinical teaching to undergraduate and graduate dental students, research and service. The position is available September 1, 2001. Academic rank and salary are commensurate with qualifications and experience. The University of Texas Health Science Center at Houston is an EEO/AA employer. Women and minorities are encouraged to apply. Send a letter of application, a curriculum vitae and a list of three references to: Dr William Tate, University of Texas Dental Branch, Department of Restorative Dentistry and Biomaterials, 6516 M D Anderson Blvd, Suite 493, Houston, TX 77030.

The University of British Columbia

The Faculty of Dentistry invites applications for a full-time position in the Department of Oral Health Sciences. Applicants should preferably have postgraduate training in restorative dentistry and research experience at the PhD level or the equivalent. The successful applicant will be expected to participate fully in teaching of restorative dentistry in the undergraduate and graduate dental curriculum, in research and faculty service. Preference will be given to individuals who can participate in the active research areas in the facility.

In accordance with Canadian immigration requirements, priority will be given to Canadian citizens and permanent residents of Canada. UBC hires on the basis of merit and is committed to employment equity. We encourage all qualified persons to apply. Please respond by November 15, 2001 with curriculum vitae and contact addresses of three referees to:

Dr Virginia M Diewert
Head, Department of Oral Health Sciences
Faculty of Dentistry, UBC
2199 Westbrook Mall
Vancouver, BC V6T 1Z3
See our website at: www.dentistry.ubc.ca

Indiana University — Restorative Dentistry

Full-time, tenure track faculty position in Restorative Dentistry available September 1, 2002. Activities will involve pre-clinical and clinical instruction in Operative Dentistry, as well as some didactic instruction. Research activity will be encouraged. Candidates must be graduates of an ADA-accredited DDS/DMD program. Licensure or eligibility for licensure in Indiana is required. Academic rank and salary commensurate with qualifications and experience. Opportunity for one day of private practice per week is available. Send curriculum vita, three letters of recommendation and a personal statement to Dr E Steven Duke, Chair, Department of Restorative Dentistry, 1121 West Michigan Street, Room S316, Indianapolis, IN 46202. Indiana University is an Affirmative Action/Equal Opportunity Employer.

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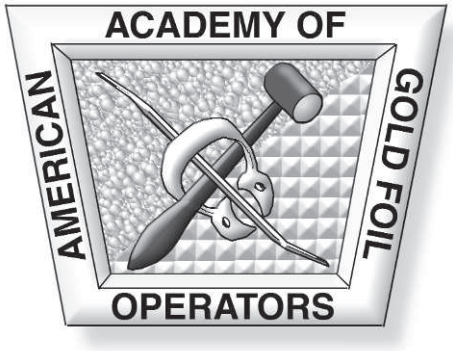

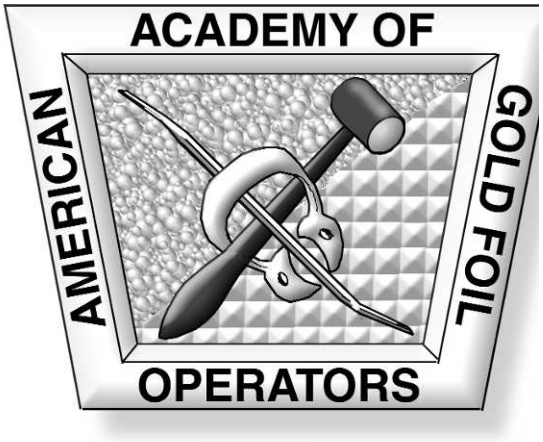

Index to Volume 26 for 2001

Index to Volume 26 for 2001

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