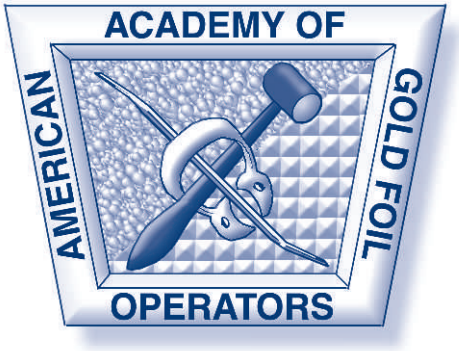
OPERATIVE DENTISTRY



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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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In Memoriam

Dr Ralph J Werner

The Academy of Operative Dentistry lost a dear friend, a trusted mentor and a large piece of its history with the passing of Dr Ralph J Werner on January 4, 2005. Ralph was not only a founding member of our organization, but was instrumental in the incorporation of the Academy and the formalization of its IRS status. He served as secretary/treasurer for 17 years and continued as treasurer for seven more. His love and financial wizardry carried the Academy from its lean beginnings to the international organization that exists today.

Ralph Werner was born on September 26, 1922 to Jacob and Amelia Werner. He was a native of Bloomer, Wisconsin and graduated from the University of Minnesota College of Dentistry in 1945. After marrying his beloved wife Bonna in 1947, Ralph served a tour of duty in the US Navy and then established his dental practice in Menomonie, Wisconsin. While maintaining a private practice, Dr Werner entered the world of academia. He taught as a clinical professor of Operative Dentistry for 27 years at the University of Minnesota and 11 years at Northwestern University in Chicago. He was a consummate craftsman and mentor, and his passion for both learning and teaching inspired his own children and hundreds of students and colleagues over the years.

Ralph's professional and personal focus throughout his life was always on excellence. This, coupled with his generosity of spirit, unswerving sense of duty, enduring sense of humor and enviable drive and work ethic, earned him the respect and admiration of his friends, patients and colleagues around the world. He had an international reputation but never lost touch with his "small town" values. Ralph was equally comfortable providing care in his dental office, presenting information in the lecture hall or classroom, arranging a dental meeting for several hundred peers, serving as bank president in Bloomer or simply enjoying the beauty of nature while walking through the Wisconsin countryside. He was a teacher who learned from everyone and everything around him and a leader who was not afraid to listen and follow when necessary.

Ralph J Werner
1922-2005

Dr Werner's interests were amazingly diverse. His activity in professional organizations included the Academy of Operative Dentistry, the American Academy of Gold Foil Operators, the American Dental Association, the University of Minnesota School of Dentistry Century Club and Dental Alumni Society, the Minnesota Prosthodontic Society, the Northwest District Dental Society of the Wisconsin Dental Association, the Minnesota Academy of Gnathological Research, the GV Black Dental Study Club, the American Association of Dental Schools, the American Academy of Restorative Dentistry, the Association of Dentistry International and the Dental Insultants. In addition, he was a fellow of both the American and International College of Dentists and the Midwest

Academy of Prosthodontics. Ralph also contributed greatly to his community. He was a member of the Masonic Temple and Lions Club and maintained a seat on the Board of Directors of the Peoples State Bank in Bloomer, Wisconsin for more than 30 years, serving as chairman from 1981 to 2004. Somehow, Ralph also found time to travel, golf, snow and water ski, enjoy boating and fishing, maintain an active interest in history and raise a family with care and love.

It is impossible to sum up the life of the unique individual that was Ralph Werner in any number of words. However, in the eulogy presented by Ralph's son-in-law, James Winnefeld, I found the following to be particularly appropriate:

"Ralph was honest, decent, patriotic, generous, determined, smart and very tough. He was a man who always, unselfishly did his duty and gave of his personal resources whenever he felt justice was not served. Ralph did not care about who you were, he cared about what you did. He was not an elitist, and believed every man deserved a chance. His first instinct was to be inclusive rather than exclusive. He and Bonna together are a wonderful example of doing well by doing good."

Ralph Werner leaves a legacy of what can be accomplished both professionally and personally by "doing good." His passing is a great loss, but the accomplishments of his life, the inspiration he provided, the memories he has left us and his spirit will live on in his progeny, his peers and the Academy of Operative Dentistry that he loved.

Dr Ralph Werner is survived by his wife of 57 years, Bonna, three children, Warren Werner, Sarah (Becker) Werner and Mary Winnefeld and two grandchildren, James and Jonathan Winnefeld. Memorials in honor of Dr Werner can be sent to the University of Minnesota Dental Student Scholarship/Activity Fund, University of Minnesota Development Office, 15-226 Moos Tower, 515 Delaware Street, SE, Minneapolis, Minnesota 55455 or Peace Lutheran Church Sunday School Program, 917 7th Street, Menomonie, Wisconsin, 54751.

Michael A Cochran, Editor

I would like to thank the Werner family for providing much of the information presented in this In Memoriam. Our prayers are with them all.

MAC

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Clinical Research

Polishing Occlusal Surfaces of Direct Class II Composite Restorations *In Vivo*

M Jung • K Hornung • J Klimek

Clinical Relevance

Under clinical conditions, the four polishing methods under consideration had similar smoothing effects on occlusal composite surfaces. The use of Occlubrush was of limited efficiency with respect to achieving rounded occlusal contours.

SUMMARY

This study evaluated the effects of four polishing methods on the occlusal surfaces of direct Class II composite restorations under clinical conditions.

Forty premolars and 40 molars were treated with direct Class II restorations using the hybrid composite Herculite XRV (Kerr). After placement of the restorations, all of which were on occlusal surfaces, they were finished with a sequence of 30 µm diamonds and tungsten carbide instruments. Twenty restorations each, consisting of 10 premolars and 10 molars, were polished with one of the four following methods: (1) Diafix-oral (Mueller-Dental), (2) MPS gel (Premier), (3) P 403-W (Dentsply) and (4) Occlubrush (KerrHawe). Selection of the polishing methods followed a randomized protocol. Replicas of the restored teeth were fabricated and the occlusal surfaces were evaluated quantitatively for roughness with the help of profilometry. Qualitative assessment of the surfaces by SEM was done with respect to roundness of contours and surface roughness. The results were analyzed statistically by two-way ANOVA, chi-squared test for crosstables and Kruskal-Wallis test.

Analysis of the quantitative data showed that there was no significant effect of the polishing methods on occlusal surface roughness ($p>0.05$). Localization of the restoration in premolars or molars had no effect on surface roughness ($p>0.05$). With respect to occlusal relief, SEM examination revealed that the use of the Occlubrush resulted in significantly more edged contours compared to the other polishing methods ($p=0.008$). Qualitative roughness evaluation showed that there were no significant differences among the four polishing methods ($p>0.05$).

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INTRODUCTION

Finishing and polishing is an essential component leading to the quality and success of direct Class I and II composite restorations. After placement of the com-

posite material, finishing by rotary instrumentation is necessary. The removal of overhangs and occlusal adjustment causes roughening of surfaces, which subsequently must be polished. Occlusal surfaces are characterized by the presence of cusps and fissures. Flexible discs have proven to be efficient for finishing and polishing plain and convex surfaces (Nagem Filho & others, 2003; Özgünaltay, Yazici & Görücü, 2003; Wilson, Heath & Watts, 1990). For anatomically structured occlusal surfaces, rigid rotary instruments such as diamonds or tungsten carbide burs are recommended with respect to finishing (Kaplan & others, 1996; Lutz, Setcos & Phillips, 1983).

For polishing, a wide variety of techniques are available, including rubber polishers, felt wheels, polishing gels and abrasive brushes. The effects of these techniques on the surface texture of composite specimens have been the subject of several *in vitro* investigations. Finishing diamonds have a great cutting efficiency (Westland, 1980). They turned out to be useful for removing excess composite material (Lutz & others, 1983), but at the same time caused rough surfaces, depending on diamond particle size (Herrgott, Ziemiecki & Dennison, 1989). With respect to tungsten carbide instruments, some studies reported a good smoothing capacity, but the cutting efficiency and durability seems to be limited compared to diamonds (Kaplan & others, 1996; Siegel & Von Fraunhofer, 1996; Whitehead & Wilson, 1989). Rubber polishers represent a very heterogeneous group of polishing devices, depending on the type and particle size of the abrasive and the number of application steps. Therefore, the polishing results of these instruments range from good to poor, depending on the individual product and type of composite used (Goldstein & Waknine, 1989; Jung, Bruegger & Klimek, 2003; Northeast & van Noort, 1988; Reis & others, 2003; Tjan & Chan, 1989). Felt wheels achieved good results on composite surfaces, but the quality of polishing was strongly affected by the initial finishing protocol (Jung & others, 2003). In general, the polishing quality of different gels depended on the nature of the abrasive and its grain size. Compared to a variety of other polishing systems, gels frequently achieved good polishing results; polishing gels based on diamond particles were more efficient than those containing aluminum oxides (Hondrum & Fernandez, 1997; Kaplan & others, 1996; Reis & others, 2003). Abrasive brushes have turned out to be well suited for polishing resin composite surfaces (Krejci, Lutz & Boretti, 1999; Schmidlin, Sener & Lutz, 2002).

Most of the studies evaluating finishing and polishing methods were carried out under *in vitro* conditions using composite specimens with plain surfaces. The extent to which these results can be transferred to clinical conditions is unknown. The use of polishing techniques on structured occlusal surfaces of Class I and II restorations provides an additional challenge to the instruments used. Some studies tried to overcome these problems by simulating clinical conditions. Wirz, Jäger and Schmidli (1987) fabricated specimens with a standardized occlusal relief; others used extracted posterior teeth and restored them with inlays for evaluation of the destructiveness of finishing and polishing systems (Schmid, Krejci & Lutz, 1991). Recently, the effect of finishing and polishing techniques on margins and surfaces of composite and ceramic inlays has been evaluated *in vivo* (Jung, Wehlen & Klimek, 2004). Clinical studies with respect to finishing and polishing of direct Class I or II composite restorations have not yet been performed.

Therefore, this study evaluated the surface quality of Class II composite restorations after polishing the occlusal surfaces with four different methods under clinical conditions.

METHODS AND MATERIALS

Fifty-four patients participated in the study; 37 male and 17 female. The average age was 35 years; the youngest patient was age 11, the oldest was 64 years of age.

In total, 80 posterior permanent teeth with Class II cavities were filled with direct composite restorations; half were premolars (n=40), the other half molars (n=40). All teeth were sensitive to thermal stimulation at the start of treatment. The cavities were completely surrounded by enamel margins. After the removal of insufficient old restorations and/or the excavation of caries, a subbase material (Kerr-Life, Kerr, Orange, CA, USA) was applied in case of pronounced lesion depth. Glass-ionomer cement (Ketac-Fil, 3M ESPE Dental Products, St Paul, MN, USA) was inserted as base material. Cavity design was box-shaped in the occlusal and proximal parts; there was no beveling of margins. After application of the rubber dam, plastic matrices were inserted proximally (Lucifix Matrix System, KerrHawe, Bioggio, Switzerland) and fixed with wooden wedges (KerrHawe). The cavity walls were etched with Dental Etching Gel (Pulpdent Corporation, Watertown, MA, USA) for 40 seconds and subsequently conditioned using Optibond Solo Plus (Kerr). The cavities were filled with the hybrid composite Herculite XRV (Kerr) in a multi-layer technique. The occlusal relief was shaped using hand instruments Composite PFI 18 and PFI 179 (Dentsply Ash Instruments, Addlestone, Surrey KT 15 2SE, GB). Each layer was cured for 40 seconds using a polymerization unit Polylux (Satelec, Mettmann, Germany).

All restorations were finished occlusally using a sequence of 30 µm diamonds followed by tungsten carbide finishing instruments. The burs were mounted in a new red-ring handpiece (TE 200, Sirona Dental

Table 1: *Specification of the Finishing and Polishing Instruments*

Type of Instrument	Manufacturer	Order-#	Abrasive Particle	Particle Size/ # of Blades	Shape	RPM	Water-Cooling
Finishing							
Diamond bur	Brasseler, Savannah, GA, USA	806 314 001 514 023	diamond	24-40 µm	spherical	50,000	water-cooling
	Brasseler	806 314 537 514 012	diamond	24-40 µm	chamfer	50,000	water-cooling
	Brasseler	806 314 540 514 009	diamond	24-40 µm	flame	50,000	water-cooling
Tungsten carbude bur	Brasseler	500 314 001 071 023	tungsten	20	spherical	50,000	water-cooling
	Brasseler	500 314 537 072 012	tungsten carbide	8	chamfer	50,000	water-cooling
	Brasseler	500 314 496 071 009	tungsten carbide	12	flame	50,000	water-cooling
Polishing							
Diafix-oral felt wheel	Mueller-Dental, Lindlar, Germany	80099	diamond	3-5 µm	wheel	3,200	no cooling
Two-Striper MPS gel	Premier Dental Products, Norristown, PA, USA	14101	diamond	4-6 µm/<1 µm (2 steps)	pointed tip	6,000	no cooling
Rubber polisher	Dentsply, Konstanz, Germany	P 403-W	silicon carbide	3-5 µm	pointed tip	5,000	water-cooling
Occlubrush	KerrHawe, Bioggio, Switzerland	2503	silicon carbide	5 µm	bristle cup	10,000	water-cooling

Systems, Bensheim, Germany) and used at 50,000 rpm under water-cooling. The diamond and tungsten carbide instruments were applied in three different shapes (Table 1). Finishing diamonds were used to remove excess composite material, contour cusps and fissures and adjust occlusal interferences. The corresponding tungsten carbide instruments were applied for initial smoothing of the occlusal surfaces.

For polishing, the restorations were randomly assigned to four groups of 20 restorations each; half of each group were premolars (n=10), the other half molars (n=10). The occlusal surfaces were polished with diamond-impregnated felt wheels (Diafix-oral), a gel based on diamond particles (Two Striper MPS), rubber polishers (P 403-W) or abrasive brushes (Occlubrush). The polishing instruments were mounted in an unused blue-ring handpiece (TE 40, Sirona Dental Systems) and applied according to the manufacturer's recommendations (Table 1). For each restoration, a new polishing instrument was used.

Both placement of the composite restorations and finishing and polishing were done by one experienced dentist. Selection of the polishing methods followed a randomized protocol.

After polishing, an impression of the restoration was taken using a silicon elastomer in two different consistencies (Blend-a-scon, Procter & Gamble, Schwalbach, Germany). Resin replicas were fabricated using Stycast 1266 (Grace NV Specialty Polymers, Westerlo, Belgium).

The occlusal surfaces of the replicas were evaluated quantitatively and qualitatively. Quantitative examination was done with mechanical profilometry using a diamond stylus FRW 750 (Mahr, Goettingen, Germany) with a tip radius of 10 µm, a stylus angle of 90° and a measuring force of 0.6 mN. The surface data were collected and processed in the central unit S8P (Mahr). Roughness measurements were performed on an air-damped platform (VW-3036-OPT-0330, Newport, Fountain Valley, CA, USA). Each occlusal surface was scanned by five parallel tracings from buccal to oral with a distance of 0.5 mm between two tracings. The measuring conditions were:

Transverse length (L_T) = 1.75 mm

Evaluation length (L_N) = 1.25 mm

Sampling length (L_S; 1/5 of L_N) = 0.25 mm

Vertical band width = 625 µm

Profile filter cut-off λc = 0.25 mm (Gauss-filter)

Evaluated area = 1.25 x 2 mm

Surface roughness was described by the arithmetic mean of the absolute ordinate values (average roughness, R_a [ISO-Standards, 1997]) and the average maximum peak to valley height of five consecutive sampling lengths within the evaluation length (R_z [DIN-Normen, 1995]). The R_a and R_z values were distributed normally; statistical analysis of the roughness data was done with two-way ANOVA (SPSS for Windows, version 11.5.1).

Qualitative evaluation was performed with the scanning electron microscope PSEM 500 (Philips Electronics, Eindhoven, Netherlands). The resin replicas were gold coated with the sputtering device SCD 040 (Bal-Tec, Balzers, Liechtenstein). The working tension of the PSEM 500 was set at 25 kV. Photomicrographs at an original magnification of 10x (overview) and 80x (details) were made of the occlusal surfaces of each restoration. Photoprints 16 x 12 cm were used for the evaluation.

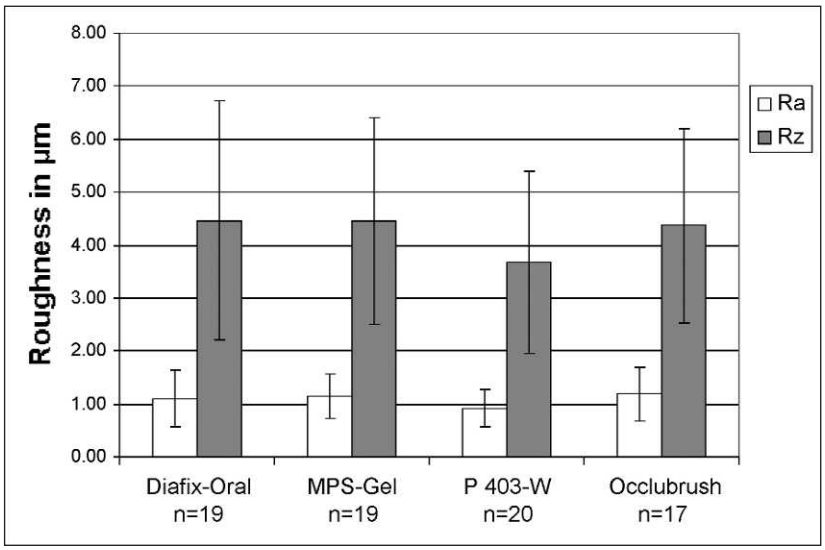

Figure 1. *Occlusal surface roughness (R_a, R_z; mean ± SD) of direct Class II composite restorations after polishing by four different methods.*

Overviews were assessed in three categories with respect to roundness of the occlusal relief:

- Smooth rounding
- Few edged contours
- Predominantly edged contours

The photoprints of the occlusal details were subdivided into 48 squares, with each square being assessed separately with respect to surface roughness using three gradings:

- Smooth surfaces
- Minor roughness
- Severe roughness

The results of the qualitative examination were statistically evaluated with chi-squared test for crosstables (SEM overviews) and the non-parametrical Kruskal-Wallis test for independent samples (SEM details).

RESULTS

Quantitative Evaluation

There was no significant effect from the four different polishing methods on the roughness of the occlusal composite surfaces (p=0.325 for R_a and p=0.547 for R_z). The rubber polisher P 403-W achieved the lowest roughness values (R_a = 0.93 ± 0.36 µm and R_z = 3.67 ± 1.71 µm, Figure 1); the surface roughness caused by the other three polishing systems was only slightly greater and ranged from R_a = 1.11 ± 0.52 µm (Diafix-oral) to 1.19 ± 0.51 µm (Occlubrush). Localization of the composite restorations in premolars or molars had no significant effect on surface roughness (p=0.593 for R_a and p=0.880 for R_z). The occlusal composite surfaces of premolars (R_a = 1.06 µm and R_z = 4.19 µm) were only slightly smoother than molars (R_a = 1.11 µm and R_z = 4.26 µm).

Qualitative Evaluation

Overview photoprints showing examples of the three gradings for assessment of occlusal relief are given in Figure 2 a-c. “Smooth rounding” was the predominant quality after application of the rubber polisher P 403-W (70.6%) and Diafix-oral (55%; Figure 3). After application of the MPS gel, about half of the occlusal surfaces (52.9%) were graded as having “few edged contours” and 41.2% had smooth rounding.

Use of the Occlubrush achieved only 22.2% smooth rounding, whereas 50% of the restorations had few edged contours and 27.8% were graded as predominantly edged. Compared to the other three polishing methods, the differences with respect to the quality of the occlusal relief were significant (p=0.008).

Examples of the qualitative roughness gradings are given in Figure 4 a-c. The roughness evaluation in SEM corroborated the quantitative results. Overall, there was no significant effect of the polishing methods on occlusal surface roughness. This was true for the three categories “smooth surfaces” (p=0.652), “minor roughness” (p=0.910) and “severe roughness” (p=0.361). “Smooth surfaces” were the predominant roughness quality and were observed in 50.7%–59.4% of the occlusal surfaces. “Minor roughness” (25.4%–33.7%) and “severe roughness” (13.6–23.9) were found less frequently (Figure 5).

In SEM, there were no signs of surface destruction by any of the polishing methods used.

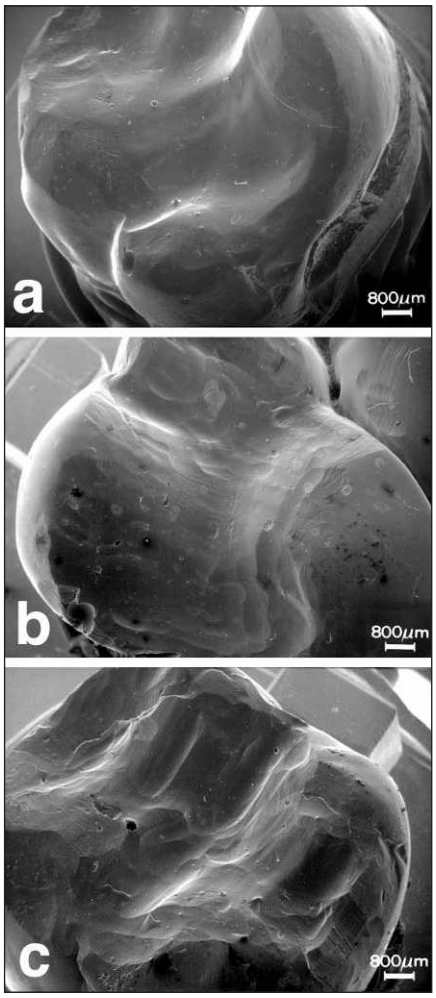

Figure 2. *Qualitative evaluation of the occlusal relief.*
(a) "smooth rounding" achieved by the rubber polisher P 403-W
(b) "few edged contours" after application of the Occlubrush
(c) "predominantly edged contours" after use of the Occlubrush.

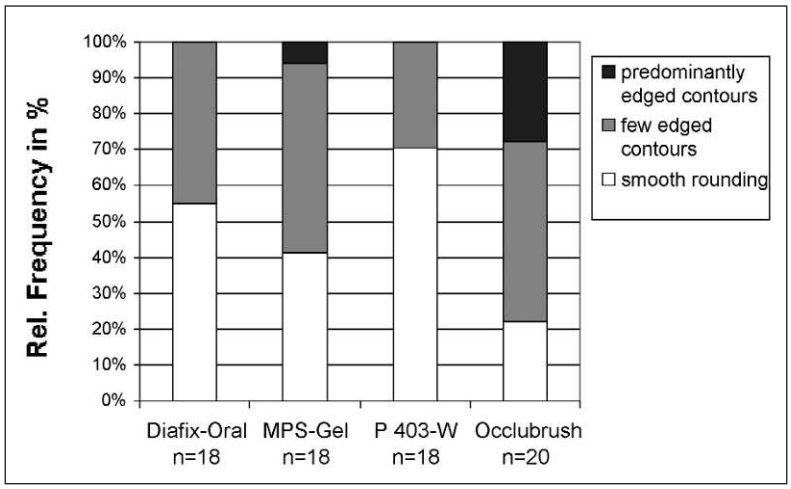

Figure 3. *The quality of occlusal relief of direct composite restorations after finishing and polishing.*

DISCUSSION

Profilometry is a widely used method for roughness evaluation. Problems associated with this method arise from the fact that the corresponding pick-up systems only have a limited vertical dislocation capacity. On the one hand, a small stylus-tip diameter provides precise exactness of measurements, but on the other hand, it limits its corresponding dislocation capacity. A large stylus-tip permits a greater vertical dislocation but compromises exactness of profile tracing (Joniot & others, 2000; Whitehead & others, 1999). This is no problem, however, when specimens with plain or slightly convex shaped surfaces are used for profilometry. In this study, we made an attempt to measure roughness on occlusal surfaces. It was therefore necessary to select a comparatively crude pick-up system with a stylus radius of 10 µm in order to be able to perform quantitative profilometry.

The parameters R_a and R_z, which had been selected for this study, are vertical parameters. They describe roughness by profile amplitudes. R_a is a representative roughness parameter and was chosen in order to compare these results with other studies. One problem associated with R_a is that it gives little information about the actual depth of the assessed surface irregularities. Therefore, R_z was chosen as an additional parameter in order to provide a better estimate of the height of the profile elements.

It has been pointed out that in order to describe the texture of a surface adequately, more than one roughness parameter should be appropriate (Whitehead & others, 1995). In this study, it was not possible to use a hybrid parameter, such as the profile-length ratio. The advantage of such a parameter is that it describes roughness by its vertical (height of profile) and horizontal (number of irregularities) dimensions (Jung, 2002). In this case, the relationship between the large vertical difference with respect to cusps and fissures, on the one hand, and the comparatively short evaluation length and profile depth, on the other, made it impossible to calculate this alternative parameter.

Surface quality is an important factor contributing to the behavior of composite materials. Rough surfaces accumulate more plaque and plaque components compared to smooth surfaces (Kawai & Urano, 2001). The surface state affects fracture resistance of composites (Graf & others, 1998). Rough restorations are more abrasive toward antagonistic surfaces and show less wear resistance (Tjan & Chan, 1989). The hardness of composites is influenced by finishing and polishing methods (Yap, Lye & Sau, 1997). Smooth surfaces are less susceptible to staining (Dietschi & others, 1994) and enhance the patient's comfort with respect to sen-

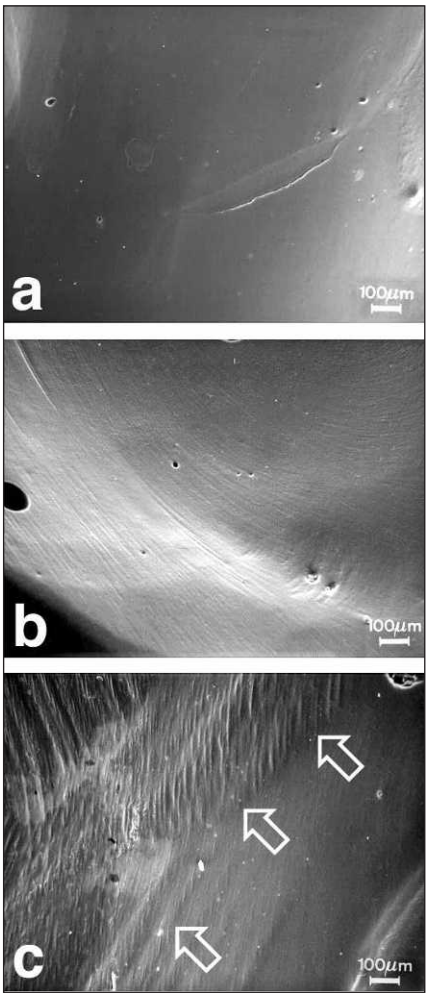

Figure 4. *Qualitative roughness evaluation of occlusal composite surfaces*
(a) "smooth surfaces" after application of Diafix-oral
(b) "minor roughness" after use of the MPS gel
(c) "severe roughness" on the left part of the surface (↖) after application of the MPS gel.

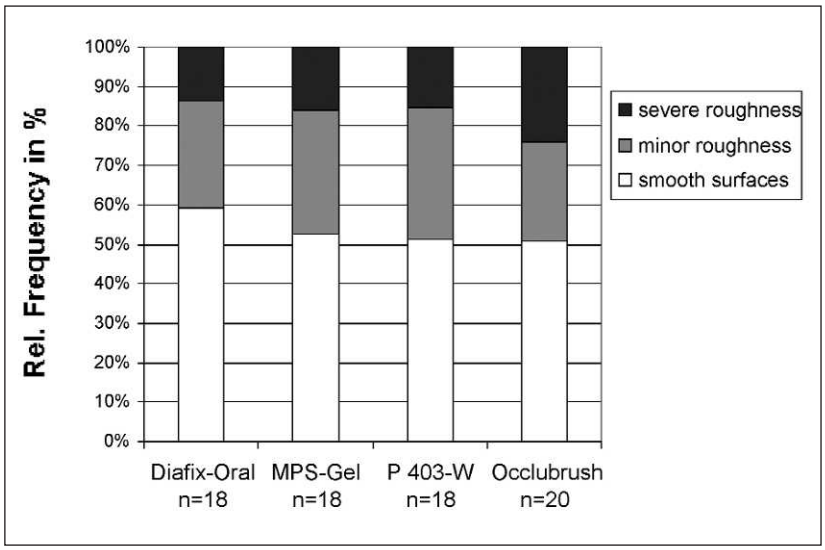

Figure 5. *Qualitative roughness evaluation of occlusal composite surfaces after polishing by four different methods.*

sation of surface irregularities (Van Noort & Davis, 1984).

The occlusal surfaces of all composite restorations were finished by a sequence of 30 µm diamonds and tungsten carbide instruments. *In vitro* studies have shown that this ensures excellent cutting efficiency and a good pre-smoothing of composite surfaces (Lutz & others, 1983). This can be of particular significance when using one-step methods for subsequent polishing (Jung, 2002; Jung & others, 2003).

There are a great variety of techniques used for polishing composite surfaces. The selection of polishing methods in this study arose from the intention to include four different application modes commonly used in dental practice (impregnated felt wheels, gels, rubber polishers and abrasive brushes). These methods represent different approaches to reaching the irregularities of structured occlusal surfaces. Another purpose was to include two types of abrasive particles—diamond and silicon carbide. Furthermore, the number of application steps to accomplish finishing and polishing should not exceed three or four.

Experimental investigations concerned with the finishing and polishing of restorative materials frequently use plain specimens. In these cases, surface quality primarily is a result of the interaction of the rotating instrument and surface under ideal conditions. In contrast, surface quality in this study is influenced by two completely different factors. One is the effect of the abrasive particles on the composite surface; the other is the accessibility of the individually shaped instruments to the structure of the occlusal surface. With respect to quantitative and qualitative roughness, there was no difference among the four methods used for polishing. This demonstrates that, under clinical conditions, different polishing approaches and abrasive particles can achieve a similar smoothing effect on structured hybrid composite surfaces.

The results of the SEM evaluation, with respect to roundness of contours, emphasize the importance of a qualitative examination, in addition to quantitative roughness measurements. After the use of Occlubrush, the occlusal surface had significantly more edged contours than after application of the other polishing methods. Thus, under clinical conditions, the combination of silicon carbide abrasive particles and resin bristles was unable to remove edges sufficiently, which remained after finishing. This finding might have been caused by

the type or size of the abrasive particles, the way the particles are embedded in the resin bristles or the shape of the Occlubrush.

Except for rubber polisher P 403-W, the other polishing systems have been the subject of experimental investigations. Abrasive bristle brushes were recommended for cleaning and polishing fine and coarse hybrid resin composites (Schmidlin & others, 2002). Using the Occlubrush on hybrid composite specimens, Krejci and others (1999) achieved an average roughness R_a of 0.3 µm. The corresponding roughness values of this study are about four times greater.

The *in vitro* application of Diafix-oral on hybrid composite specimens after the same finishing protocol as that used in this study achieved R_a values ranging from 0.25 µm - 0.6 µm (Jung, 2002; Jung & others, 2003). In contrast to the results of the study with Diafix-oral, there is considerable difference, yielding an R_a of 1.1 µm.

The effect of MPS gel has also been the subject of *in vitro* studies. After finishing with a diamond and tungsten carbide instrument, the average roughness of hybrid composite specimens was reduced to 0.47 µm (Jung, 2002). Hondrum and Fernandez (1997) used the MFS/MPS finishing and polishing system on Prisma composite specimens and reported an R_a of 0.61 µm. Compared to the results of this clinical study, there is again a considerable difference in roughness values. Only one experimental study was performed that showed greater R_a values after application of the MPS gel, ranging from 1.5 to 10.3 µm (Kaplan & others, 1996).

In a clinical study concerned with the finishing and polishing of composite inlays, no differences were found between Diafix-oral and MPS gel with respect to the roundness of occlusal contours (Jung & others, 2004). This is in accordance with the results of the current study. The gradings "smooth rounding" and "few edged contours" were the prevailing category with respect to inlays and direct restorations. A total of 8.3% of all direct composite restorations and 5% of composite inlays were classified as "predominantly edged."

Qualitative roughness evaluation after applying Diafix-oral and MPS gel on occlusal composite inlay surfaces demonstrated that, in accordance with this study, there were no significant differences between the two polishing methods (Jung & others, 2004). Smooth surfaces prevailed both on composite inlays and direct restorations; only the frequency of smooth surfaces on inlays (65–80%) was greater than on direct restorations (50-60%). Severe roughness was infrequent, both on inlay (5-12%) and direct restorations (12-22%). These minor differences might be attributed to the fact that inlays have an excellent surface quality prior to insertion and not all inlay surfaces necessarily require finishing and polishing after insertion.

The results of this study demonstrate that there are considerable differences between finishing and polishing of composite surfaces under experimental and clinical conditions with respect to surface roughness. Compared with surface quality after *in vitro* studies, difficulties associated with access to posterior teeth and problems arising from the complex structure of occlusal surfaces are explanations for the increase in roughness after finishing and polishing *in vivo*. Nevertheless, experimental studies on finishing and polishing should continue to be performed. Owing to their great sensitivity, *in vitro* studies can contribute valuable information about the interaction of finishing or polishing instruments and a composite surface under ideal conditions. Additional clinical studies must show whether the theoretical smoothing capacity of a rotating instrument will work under the conditions of the oral cavity.

CONCLUSIONS

Under clinical conditions, different polishing techniques such as diamond impregnated felt wheels, a diamond gel and silicon carbide rubber polishers were similar in their effect on the quality of occlusal composite surfaces. Compared to the other polishing methods, abrasive brushes were less efficient with respect to removing edged contours.

Diafix-oral, MPS gel and the rubber polisher P 403-W can be recommended for use on occlusal composite surfaces if an appropriate initial finishing protocol is provided. Due to the fixation of diamond particles with the help of wax, Diafix-oral cannot be re-used; from a practical point of view, this is a disadvantage.

In the case of a complicated surface geometry, the results of *in vitro* investigations on finishing and polishing may not be reproducible under clinical conditions.

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Response of Human Pulp Capped with a Bonding Agent After Bleeding Control with Hemostatic Agents

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A Reis • A Muench • VC Araújo

Clinical Relevance

Calcium hydroxide should be used as the material of choice for pulp capping. The use of dentin bonding agents in vital pulp capping even after successful hemostasis is contraindicated.

SUMMARY

Purpose: This study evaluated the response of human pulps capped with a bonding agent after bleeding control with different hemostatic agents. Material and Methods: Twenty-five Class II cavities were prepared in 25 caries-free human premolars scheduled for extraction due to orthodontic treatment. The pulp exposures were performed on the occlusal floor. The teeth were randomly divided into five groups. Groups 1-4 were capped with an adhesive system after hemostasis with different agents: Group 1—saline solution; 2—ferric sulfate; 3—2.5% NaOCl; 4—$Ca(OH)_2$ solution. In Group 5, after hemostasis with saline solution, the pulp was capped with calcium hydroxide (control group). Then, ScotchBond Multi Purpose Plus was applied and the resin composite Z-100 placed incrementally according to the manufacturers' directions. After 60 days, the teeth were extracted and processed for light microscopic examination (HE) and the groups were categorized in a histological score system. The data were subjected to a non-parametric test (α=0.05). Results: Overall, the histological features showed that the pulp response from Groups 1 through 4 was inferior to the response from Group 5, where dentin bridging occurred. In all groups, where the adhesive system was used for capping, the pulp response varied from an acute inflammatory, with varying degrees, to

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necrosis. No dentin bridge was formed after adhesive capping.

INTRODUCTION

In recent years, interest in the possibility of direct pulp capping with dentin bonding materials has increased. The rationale behind the use of dentin bonding materials instead of the commonly used calcium hydroxide compounds has been that prevention of leakage, bacterial contamination and toxin ingress may be more critical for the healing processes than the material used for capping (Cox & others, 1987, 1996).

A criterion for healing the exposed pulp has normally included the formation of a hard tissue barrier across the wound area and the absence of inflammation in the subjacent tissue. Although this hard tissue, also called a dentin bridge, is often perforated by tunnels and cell inclusions (Tsuneda & others, 1995; Cox & others, 1996), its formation is considered to better protect the pulp against bacterial penetration than no hard tissue formation (Pameijer & Stanley, 1998; Bergenholtz, 2000).

Many studies have shown pulp healing and dentin formation when human pulp is capped with different formulations of calcium hydroxide. Opposite conclusions were drawn when human teeth were capped with bonding agents (Gwinnett & Tay, 1998; Hebling, Giro & Costa, 1999; Pereira, Segala & Costa, 2000; Costa & others, 2001a). In these studies, bonding agents applied on pulp exposures, following acid etching, elicited a moderate inflammatory response at short-term evaluation, which sometimes leads to necrosis. Over time, a persistent mild inflammatory pulp response mediated by macrophages and giant cells was demonstrated adjacent to the pulp exposure site. This chronic inflammatory response seems to play a role in the lack of complete dentin bridge formation. In the long-term evaluation, the lack of a dentin bridge was a common histological finding (Pereira & others, 2000; Costa & others, 2001a).

However, there are authors reporting that, as long as cavity margins are sealed, bonding agents can be used as capping agents (Cox & others, 1998, 2001). According to these authors, pulp capping with bonding agents is an extremely sensitive technique, since bleeding can cause failures in the sealing provided by the adhesive systems to cavity margin, consequently, promoting microleakage that may lead to bacterial contamination and/or ingress of toxins (Cox & others, 1998, 2001). Therefore, pulp hemorrhage control is considered an important issue for the achievement of clinical success after pulp exposure and adhesive capping (Tsuneda & others, 1995; Cox & others, 1998; Akimoto & others, 1998; Kitasako, Inokoshi & Tagami, 1999; Hafez & others, 2002).

Several hemostatic agents have been employed for hemorrhage control, such as NaOCl at different concentrations, chlorhexedine and calcium hydroxide solutions (Katoh, Kidokoro & Kuroso, 1978; Cox & others, 1998; Hafez & others, 2002; Pameijer & Stanley, 1998; Horsted-Bindslev, Vilkinis & Sidlauskas, 2003). Another hemostatic agent is ferric sulfate (Fuks & others, 1997). To date, no study has evaluated the response of human pulp capped with a bonding agent after hemorrhage control with different hemostastic solutions, which was the aim of this investigation.

METHODS AND MATERIALS

Twenty-five healthy human premolars scheduled for extraction for orthodontic reasons were selected from patients ranging from 15 to 25 years of age. All the premolars were examined clinically and radiographically to assure the absence of proximal caries and periapical lesions. The patients and, when appropriate, their parents signed consent forms after receiving a thorough explanation about the experimental rationale, clinical procedures and possible risks, allowing the clinical procedure. Both the consent form and the research protocol were obtained according to the Human Subject Review Committee from the University of São Paulo—Brazil.

For thermal testing, ENDO-ICE frozen gas (Coltène/Whaledent Inc, Mahwah, NJ, USA) was applied for five seconds on the buccal surface of the premolars scheduled for pulp therapy, as well as the adjacent teeth. After local anesthesia (Citanest 3%; Merrel Lepetit, São Paulo, Brazil), rubber dam isolation was implemented and each tooth was pumiced with rubber cup at low speed. Mesio-occlusal cavities, with 0.5 mm beyond the cementum-enamel junction were prepared via sterile diamond burs (#1095, KG Sorensen, Barueri, São Paulo, Brazil) at high speed under water/spray coolant. The dimensions of the cavity were approximately as follows: occlusal depth, 3.0±0.2 mm; axial depth, 4.0±0.5 mm; proximal faciolingual width, 3.0±0.2 mm. Pulp exposure was performed in the center of the pulpal floor by means of a round diamond bur under water cooling (#1014, ϕ 1.2, KG Sorensen). One bur was used for each cavity. The teeth were then divided

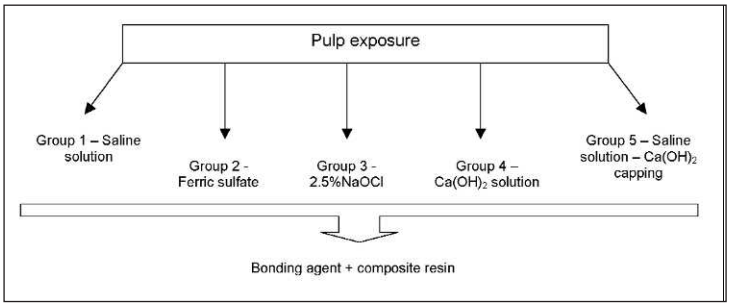

Figure 1. *Experimental design.*

into five experimental groups (n=5) as shown in Figure 1. All materials employed are described in Table 1.

In Group 1 (saline solution), the bleeding was only controlled by abundant irrigation with saline solution followed by application of a damp cotton pellet embedded in saline solution, which was held in place for one minute. In Groups 2 through 4 (ferric sulfate, NaOCl and $Ca(OH)_2$ solution), hemostasis was performed according to one of the hemostatic agents presented in Figure 1. Each solution was applied to sterile cotton pellets, which, in turn, were maintained in place for one minute. The cavity was then irrigated with saline solution.

In Group 5, a calcium hydroxide powder was applied on pulp exposure, via an amalgam carrier, after homeostasis as described with Group 1. Calcium hydroxide cement (Dycal, Dentsply, Petropólis, RJ, Brazil) was applied over the powder on the occlusal floor.

A metal matrix band was installed and both the enamel and dentin were conditioned with 35% phosphoric acid for 20 seconds. The acidic agent was rinsed and slightly dried in such way that the dentin stayed visibly moist with a shiny surface. One coat of primer was applied and air dried for 20 seconds. The bonding resin was, subsequently, applied and light cured for 10 seconds at 450mW/cm^2 (Ultralux Electronic, Dabi Atlante, Ribeirão Preto, SP, Brazil). Increments of Z-100 (3M ESPE, St Paul, MN, USA) were used to restore the cavities. Each increment (± 2 mm) was light cured for 40 seconds at 450mW/cm^2 (Ultralux Electronic, SP, Brazil). A radiometer (Model 100P—Demetron Research Corp, Kerr, Danbury, CT, USA) was used to check the light intensity immediately before each clinical appointment. When necessary, excess material was removed using an ultra-fine diamond bur at high speed under water cooling (KG Sorensen).

After 60 days, the patients were asked about the presence of post-operative sensitivity throughout the study period. Then, the teeth were extracted under local anesthesia and their roots sectioned to about 2 mm in

Table 1: *Products, Commercial Name and Composition*

Product/Commercial Name	Composition
ScotchBond Multi Purpose Plus (3M ESPE, St Paul, MN, USA)	1. Etchant: 35% phosphoric acid 2. Primer: water (40%), HEMA (47%) and polialkenoic acid copolymer (13%) 3. Adhesive: Bis-GMA (65%), HEMA (34%) and initiators/accelerators (1%)
Z-100 (3M ESPE, St Paul, MN, USA)	Bis-GMA, TEGDMA and silica/zirconium filler
Astringedent (Ultradent, South Jordan, Utah, USA)	15.5% ferric sulfate solution
Sodium hypochlorite (Fórmula e Ação, São Paulo, SP, Brazil)	2.5% NaOCl
Calcium hydroxide solution (Fórmula e Ação, São Paulo, SP, Brazil)	20g $Ca(OH)_2$ mixed in 200 ml of distilled water
Calcium hydroxide powder (Labrynth Prod, Diadema, SP, Brazil)	$Ca(OH)_2$
Saline solution (Labrynth Prod, Diadema, SP, Brazil)	2% NaCl
Dycal (Dentsply, Petropólis, RJ, Brazil)	1. Base paste: ester glycol salicylate, Calcium phosphate, Ca tungstate and ZnO 2. Catalyst paste: ethylene toluene sulfon amide, $Ca(OH)_2$, ZnO, Ti_2O and Zn stearate

Table 2: *Scores Used During the Histological Exams*

Scores	Inflammatory Cell Response
1	None or a few scattered inflammatory cells present in the pulp beneath the exposure site
2	Polymorphonuclear leukocytes (acute) or mononuclear lymphocytes (chronic) in an inflammatory lesion
3	Severe inflammatory lesion appearing as an abscess or dense infiltrate involving one-third or more of the coronal pulp
4	Completely necrotic pulp
Scores	**Soft Tissue Organization**
1	Normal or almost normal tissue morphology below the exposure site and throughout the pulp
2	Lack of normal tissue morphology below the exposure site, with deeper pulp tissue appearing normal
3	Loss of general pulp morphology and cellular organization below the exposure site
4	Necrosis in at least the coronal third of the pulp
Scores	**Dentin Bridge Formation**
1	New barrier tissue directly adjacent to some portion of the restorative material
2	New dentin bridge some distance from the material interface
3	No evidence of any dentin tissue formation in any of the tissue sections

order to facilitate fixation in 10% buffered formalin solution for 72 hours. The teeth were decalcified in 20% formic acid for six to eight weeks, prepared according to normal histological techniques and embedded in paraffin. Six micron-thick sections were cut with a microtome parallel to the main vertical axis of the tooth. The sections, mounted on glass slides, were stained with hematoxylin and eosin (H/E). The sections were blindly evaluated by an experienced pathologist according to the criteria described in Table 2. The multiple sections were used to achieve an overall assessment for each tooth. Therefore, in each experimental condition, there were five statistical units (n=5).

The scores attributed to each group were subjected to non-parametric Kruskal-Wallis analysis. This test was performed separately for each histological exam (inflammatory cell response, soft tissue organization and dentin bridge formation). The comparisons between averages were performed by comparing the ranks with appropriately computed critical values (α=0.05) (Conover, 1980). A Spearman correlation test (α=0.05) was also performed between ranks of pain frequency versus histological tests (inflammatory cell response, soft tissue organization and dentin bridge formation).

RESULTS

Table 3 shows the percentage of scores observed for each group. Overall, the histological features showed that the pulp response from Groups 1 through 4 (saline solution, ferric sulfate, NaOCl and $Ca(OH)_2$ solution) were inferior to the response from Group 5 ($p<0.05$). No significant correlation between the histological scores and pain was observed ($p>0.05$).

Histological Feature (Figures 2 through 6)

Saline Solution: Twenty percent of the specimens exhibited normal pulp tissue; however, no dentin bridge formation was observed. In 80% of the cases, an intense and chronic inflammatory infiltrate was observed throughout the pulp tissue, reaching the apical region with different extensions. In 20% of the cases, patients reported pain.

Ferric Sulfate: In all specimens where ferric sulfate was used, an intense mononuclear inflammatory infiltrate was observed. Odontoblasts were aspirated into the dentinal tubules and a high retraction of pulp tissue towards dentin walls was observed, with a cavity formation. This finding was restricted to the coronal region of the pulp. In 60% of the cases, patients reported sensitivity to cold during the 60 days prior to extraction.

NaOCl: Sixty percent of the specimens showed a chronic inflammatory infiltrate, with several extension rates and all over the pulp tissue. In 40% of the cases, partial coagulation necrosis occurred. The pulp tissue was also retracted but to a lesser extent than the anterior group. No evidence of odontoblast aspiration into dentinal tubules was found. No report of pain was recorded for this group.

$Ca(OH)_2$ Solution: In 80% of the cases, a chronic inflammatory infiltrate with several extension rates was observed. In some cases, pulp retraction was noted; however, no evidence of odontoblast cell aspiration was found. In 20% of the cases, a partial coagu-

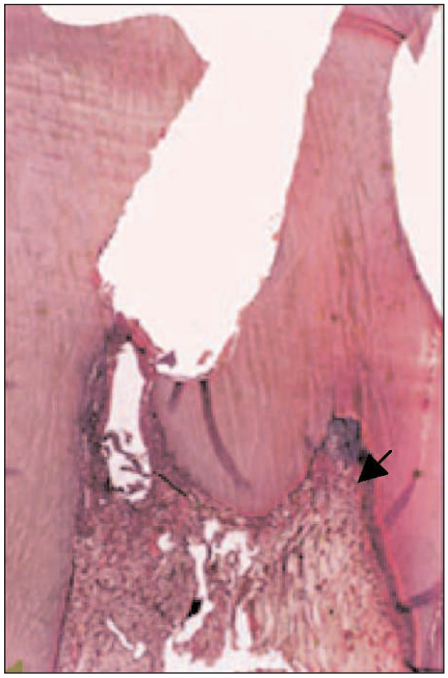

Figure 2. *Hemostasis performed with saline solution before adhesive capping (Group 1). Observe the great area of chronic inflammation adjacent to the exposure site after 60 days (black arrow) (HE, original magnification - 25.6x).*

Table 3: *Percentage of Scores Attributed for Each Group in Each Criteria and Multiple Comparisons and Frequency of Pain (%) and Correlation (***)*

Groups (*)	Inflammatory Cell Response (ICR)				(**)	Soft Tissue Organization (STO)				(**)	Dentin Bridge Formation (DBF)			(**)	Pain (%)
	1	2	3	4		1	2	3	4		1	2	3		(***)
1- Saline solution	20	80	0	0	b	20	80	0	0	e	0	0	100	h	20
2- Ferric sulfate	0	100	0	0	b,c	0	100	0	0	e,f	0	0	100	h	60
3- NaOCl	0	60	0	40	c	0	60	0	40	f	0	0	100	h	0
4- $Ca(OH)_2$ solution	0	80	0	20	b,c	0	80	0	20	e,f	0	0	100	h	20
5- Calcium hydroxide	100	0	0	0	a	100	0	0	0	d	100	0	0	g	0

(*) Groups 1 to 4 were capped with adhesive systems; (**) Different letters indicate statistical differences (p<0.05); (***) Spearman's correlation between ranks of pain versus average ranks of ICR, STO and DBF were not significant (p>0.05).

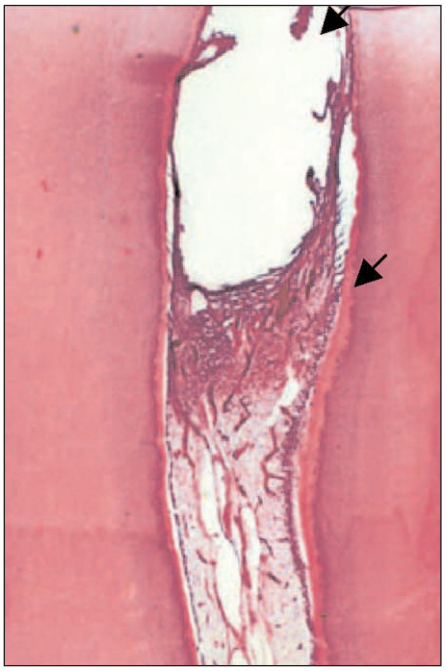

Figure 3. *Hemostasis performed with ferric sulphate before adhesive capping (Group 2). Note the pulp retraction below the exposure site after 60 days (delimited by two black arrows). A chronic inflammatory response with mononuclear cells and odontoblasts aspiration into dentinal tubules can be seen near the lower arrow (HE, original magnification - 25.6x).*

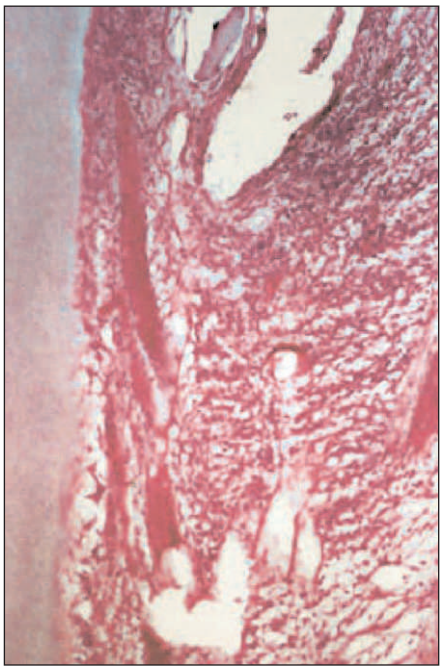

Figure 4. *Hemostasis performed with NaOCl before adhesive capping (Group 3). An intense inflammatory chronic infiltrate can be seen near the exposure site after 60 days (HE, original magnification—100x).*

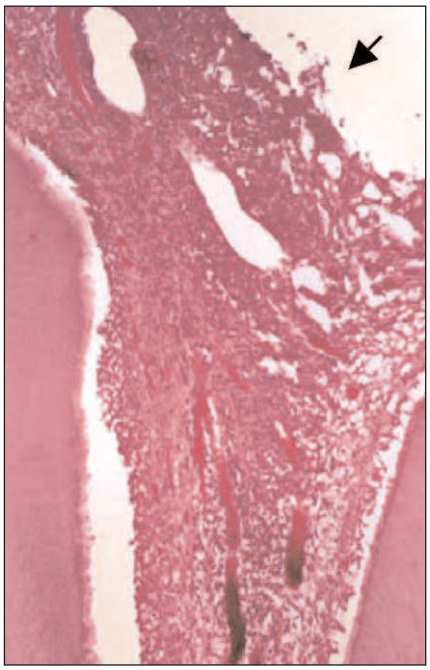

Figure 5. *Hemostasis performed with $Ca(OH)_2$ before adhesive capping after 60 days (Group 4). Observe the chronic inflammatory infiltrate near the exposure site days (black arrow) (HE, original magnification—25.6x).*

lation necrosis was observed. In this case, the patient complained about pain that slowly disappeared over time. In 20% of the cases, the patient reported pain.

Calcium Hydroxide: Contrary to the previous groups, all specimens showed an amorphous dentin bridge formation with irregular contouring and varied mineralization degrees. Inclusion of pulp cells was also observed within the dentin bridge. The pulp exhibited a loose connective tissue, with several blood vessels delineated by an odontoblastic layer in the predentin interface. A similar odontoblastic layer was also observed along with the dentin bridge. No patient complained of pain.

DISCUSSION

In recent years, several researchers have indicated the use of adhesive systems for pulp capping, instead of the commonly used $Ca(OH)_2$ therapy (Kopel, 1997; Cox & others, 1998; Schuurs, Gruythuysen & Wesselink, 2000). They supported the concept that microleakage of bacteria and its products around composite restorations, rather than around the composites, is responsible for pulpal inflammation (Brännström & Nyborg, 1972; Cox & others, 1987).

Testing several adhesive systems over exposed monkey's pulp, Cox and others (1998) demonstrated that no systems caused pulp irritation to non-exposed and exposed pulps, showing pulp responses similar to those treated with $Ca(OH)_2$. The authors attributed the clinical success of adhesive capping to proper hemorrhage control with 2.5% NaOCl before adhesive placement.

Our data is in direct contrast with the findings of Cox and others (1998). The use of 2.5% NaOCl for hemorrhage control (Group 3) did not improve the pulp response of exposed pulp from human teeth. In fact, the histological findings from Group 3 (NaOCl) were worse than Group 1 (saline solution), where no specific hemostatic agent, apart from saline solution, was employed. This means that hemostasis did not improve pulp response. One may suppose that the lack of pulp healing could be due to the toxic effects of sodium hypochlorite to exposed dentin. Actually, NaOCl is a well-known, nonspecific proteolytic agent capable of breaking organic material. Evidence-based literature has indicated that a 6% NaOCl solution and other agents such as 3% H_2O_2 or the bonding agent Syntac Sprint are more aggressive to pulp than a calcium hydroxide-saline solution (5g of $Ca[OH]_2$ in 10 ml of sterile distilled water) or phosphate buffered saline. These substances have been shown to depress the mitochondrial enzyme response by 97.7% (6% NaOCl solution), 97.3% (3% H_2O_2) and

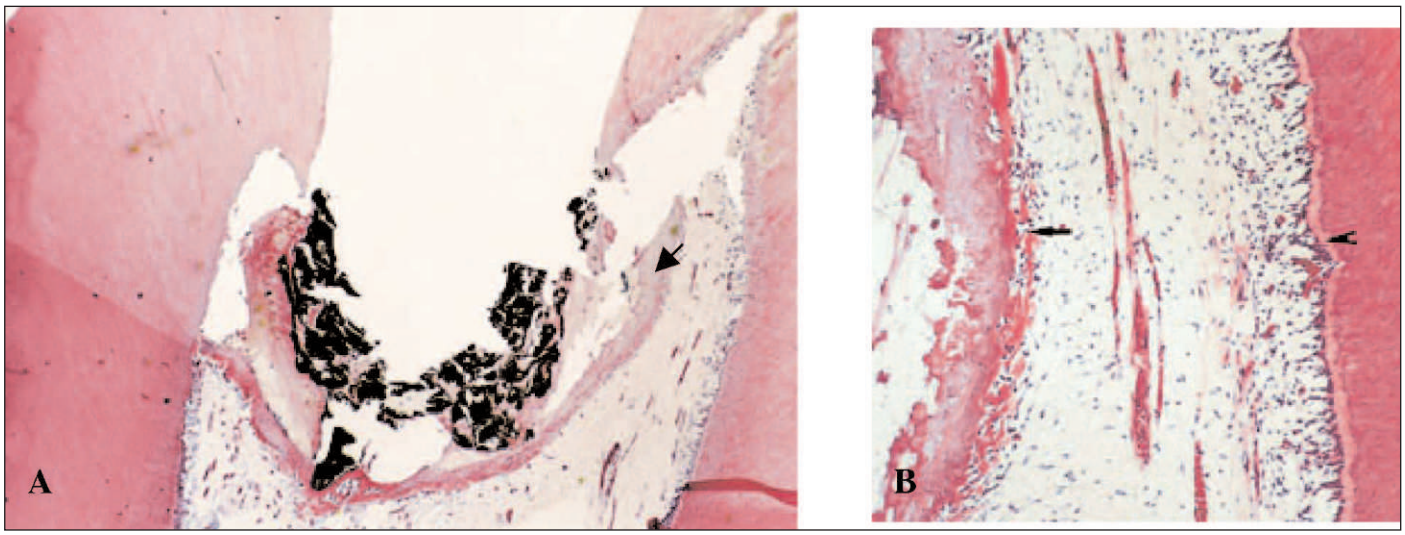

Figure 6. *Calcium hydroxide capping after 60 days (Group 5). In A, observe the dentin bridge formation (black arrow) and adjacent pulp tissue with normal features (HE, original magnification—25x). In B, a higher magnification of the dentin bridge (HE, original magnification—100x). It is clear that the dentin bridge is composed by na amorphous material with a varied degree of mineralization. Odontoblast-like cells can be seen (black arrow in the right). Adjacent loose connective tissue and odontoblasts-like cells seen near the pre-dentin (black arrow in the left).*

95.0% (the bonding agent Syntac Sprint) and promote dramatic changes in cell morphology (Costa, Edwards & Hanks, 2001b). On the other hand, $Ca(OH)_2$ solution only depressed the metabolic activity of cells by 5% and both $Ca(OH)_2$ solution and phosphate buffered saline have not caused any dramatic alteration in cell morphology (Costa & others, 2001b).

The major difference between Group 1 (saline solution) and other groups is that saline solution is not aggressive to pulp when used for hemorrhage control. Although no dentin bridge formation occurred in Groups 1 through 4 (saline solution, ferric sulfate, NaOCl and $Ca(OH)_2$ solution), 20% of the specimens from Group 1 exhibited normal connective tissue and no pulp retraction. In Group 4, where a $Ca(OH)_2$ solution was employed for hemostasis, only 20% of the cases showed necrosis, a rate that was inferior to Group 3 (NaOCl). This is in agreement with the findings of Costa and others (2001b), who showed that, apart from buffered saline, the $Ca(OH)_2$ solution provided the lowest metabolic alterations. Thus, based on such findings, and in the small differences in histological features among adhesive system capped groups, there is likely a negative synergism between aggressive hemostatic agents and the application of bonding resins over pulp exposures.

The aspiration of odontoblasts into dentinal tubules and a severe pulp retraction observed in those teeth treated with ferric sulfate may have precluded the bonding agent to contact or diffuse into the pulp. This lack of contact could be responsible for the lack of necrosis observed in this specific group. The high number of patients complaining of pain (60%) could be attributed to this pulp retraction and the presence of an intense chronic inflammation in the coronary pulp. Ferric sulfate has been used to control bleeding in endodontic surgery and promote gingival retraction before impression taking (Fischer, 1987; Fei, Udin & Johnson, 1991). Even though the mechanism of hemostatic action for ferric sulfate is still debated, it seems that the agglutination of blood proteins results from the reaction of blood with ferric and sulfate ions and with the acidic pH of the solution. The agglutinated proteins form plugs that occlude the capillary orifices (Lemon, Steele & Jeansonne, 1993). In a study where 15.5% ferric sulfate solution was used for pulpotomies, only 60% of the pulps treated were normal and 40% had severe inflammation, with some presenting abscess formation (Fuks & others, 1997). In another study, internal resorption was a common radiographic finding (Papagiannoulis, 2002). We do not encourage clinicians to use this solution for hemorrhage control, since this was the only group in which patients complained of pain throughout the study.

However, we cannot rule out the fact that the components from the adhesive systems or even the acid etching played a significant role in the lack of pulp healing observed in Groups 1 through 4 (saline solution and ferric sulfate, NaOCl and $Ca(OH)_2$ solution). Pameijer and Stanley (1998), in a study in sub-human primates, showed that the use of 2% chlorexedine solution (Consepsis) as a hemostatic agent was only effective when the pulp capping procedure was performed with calcium hydroxide. Cox and others (1998) argued that the lack of pulp healing of teeth capped with adhesive systems in the study by Pameijer and Stanley (1998) could be due to the fact Consepsis is an irritant solution to the exposed pulps, which compromises healing and

bridging. However, it is fair to suppose that if the statement by Cox and others (1998) were true, even in those cases where calcium hydroxide was used after Consepsis hemostasis, its aggressive effects would have equally compromised the pulp healing, which was not the case.

Therefore, the lack of pulp healing after adhesive capping shown in this investigation and in other published studies seems to be due to the application of cytotoxic components over the pulp. Even when hemorrhage control is performed, no dental bridge is formed after adhesive capping (Pameijer & Stanley 1998; Gwinnett & Tay, 1998; Hebling & others, 1999; Pereira & others, 2000; Costa & others, 2001a; Horsted-Bindslev & others, 2003). It is interesting to note that most of the articles reporting acceptable biocompatibility of adhesive agents over exposed pulps were conducted in monkeys or rats (Akimoto & others, 1999; Kitasako & others, 1999; Cox & others, 1998, Costa, Mesas & Hebling, 2000b); however, these findings were not reproducible in human teeth (Gwinnett & Tay, 1998; Hebling & others, 1999; Pereira & others, 2000; Costa & others, 2001a; Horsted-Bindslev & others, 2003). Possibly, resin components produce more immunosuppression of pulpal immunocompetent cells in humans than they do in monkeys or other animals (Jontell & others, 1995; Carvalho & others, 1999). Thus, care should be taken when extrapolating the results obtained from animal teeth to human dentition, since they exhibit specific pulp responses (Costa, Hebling & Hanks, 2000a). It is worth emphasizing that, 10 years ago, Cox and others (1987) also showed successful treatment of pulp exposures when NaOCl was not used for chemical lavage of pulp exposures.

Acid etching of deep dentin causes a dramatic increase in dental permeability, increasing the outward flow of dentinal fluids due to the enlargement of dentinal tubules, removal of the smear layer and smear plugs and the hypertonic property of acid gel (Pashley & others, 1993). The large amount of dentinal fluid that microscopically pools on the surface following dentin conditioning of the pulp floor of cavities seems to interfere with the complete polymerization of primer and/or adhesive resin, reducing its resin-dentin bond strength (Pereira & others, 1999). Under such overwetting conditions, phase separation of the hydrophobic and hydrophilic moieties and water dilution of resin monomers (Tay, Twinnett & Wei, 1996, 1998a,b) causes a decrease in the conversion degree of monomers (Jacobsen & Soderhölm, 1995; Paul & others, 1999).

While fluid-filled channels of dentin are responsible for hydrodynamic fluid shifts across dentin in response to painful stimuli, they also permit the diffusion of irritating substances such as bacteria and/or their by-products and unpolymerized resin to pulp, thus initiating detrimental reactions. This phenomenon is likely to occur when adhesives are applied directly over wet and vital pulp tissue. Recently, this was demonstrated by the structural features of pulpal responses following application of All-Bond2 to acid-conditioned human pulp tissue. An irreversible injury to odontoblasts closest to the site of cavity preparations and the resultant death of these cells was observed. Resin particulates in the form of globules could be seen within the dentin-pulp complex, which have appeared to trigger a foreign body response characterized by the presence of mononuclear infiltrate and the appearance of multinuclear giant cells (Gwinnett & Tay, 1998). It seems that the chronic, unresolved inflammatory response creates an inadequate pulpal environment for odontoblast-like cell differentiation subjacent to pulp exposures (Hebling & others, 1999; Costa & others, 2001a), which explains the lack of calcified dentin bridge formation.

In Class II cavities, the bonding of adhesive systems to cavity walls can be reduced in case blood or fluid contamination occurs (Abdalla & Davidson, 1998; Kaneshima & others, 2000; Dietrich & others, 2000); this can reduce cavity sealing and, therefore, protect against microleakage and bacterial invasion. Excellent hemostasis can be accomplished with the use of hemostastic agents. However, the subsequent application of acid may cause bleeding that is difficult to control and makes placement of pulp capping in a dry field difficult, besides the likely contamination of the cavity walls (Pameijer & Stanley, 1998).

This investigation only showed pulp healing with bridge formation when calcium hydroxide powder, followed by calcium hydroxide paste, was used for pulp capping. This finding is in agreement with other published studies (Hebling & others, 1999; Pereira & others, 2000; Costa & others, 2001a; Horsted-Bindslev & others, 2003). For instance, Pereira and others (2000) exposed human premolar pulps and either capped them with an adhesive system or calcium hydroxide. The teeth were extracted after two evaluation periods (9-12 and 92-175 days). The histological analysis showed early signs of pulp repair (dentin bridging) for calcium hydroxide groups, which was consolidated at the longest evaluation period. Pulps capped with the adhesive system showed moderate inflammation with hyalinization of the extracellular matrix and hydropic degeneration of cells at the earliest period. Vasodilatation and hyperemia combined with a mild, persistent inflammation was detected at later periods and no evidence of complete calcified dentin bridging was found.

Criticism of the calcified dentin bridges induced by calcium hydroxide rely upon the fact that they are porous and contain tunnels which may serve as a pathway for bacteria and/or its toxins to contaminate the pulp

(Tsuneda & others, 1995; Cox & others, 1996). Some authors generally report that calcium hydroxide "disappears" with time (Barnes & Kidd, 1979; Heitmann & Unterbrink, 1995). However, by the time this occurs, the calcium hydroxide will have already stimulated pulp healing.

As reported by Carvalho and others (1999), dentin bridges are not intended to "restore" the cavity. If bacteria can reach the dentin bridge and invade it to reach the pulp, it means that the material and technique used to restore the cavity was not able to seal the margins. When the cavity margins are placed in dentin or cementum, microleakage cannot be avoided by the sole use of adhesive systems and resin composites (Loguercio & others, 2004). Therefore, in case bacteria contamination occurs along the adhesive interface, the presence of an antibacterial agent such as calcium hydroxide may avoid it reaching the pulp tissue (Stuart & others, 1991). The bacterial properties of calcium hydroxide compounds are related to high pH, since most microorganisms are destroyed at pH 9.5 (Foreman & Barnes, 1990).

Authors in favor of adhesive pulp capping believe that adhesive restorations can provide a long-lasting hermetic seal of cavity margins. This, in fact, may be true when the entire margins of the cavities are in enamel; otherwise, it is a pretentious assumption (Loguercio & others, 2004). Therefore, in categorizing success and failure in pulp capping, many other factors need to be considered other than just the presence or absence of sealed margins. In addition, studies on adhesion have shown that adhesive interfaces are very prone to hydrolytic degradation over time (Sano & others, 1999; Hashimoto & others, 2000) and even under ideal adhesive conditions (Reis & others, 2004).

CONCLUSIONS

Calcium hydroxide should be used as the material of choice for pulp capping and the use of the total etch technique and dentin bonding agents, in vital pulp capping, even after successful hemostasis is contraindicated.

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Efficacy, Side-effects and Patients' Acceptance of Different Bleaching Techniques (OTC, in-office, at-home)

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Clinical Relevance

The outcome of this clinical study suggests that the three evaluated bleaching techniques resulted in the desired whitening of teeth within the recommended application time periods. Each method was also well accepted by the patients. The side effects that occurred were reversible and none of the products tested resulted in detectable changes in the enamel surface.

SUMMARY

This clinical study compared the efficacy of three different bleaching techniques with respect to the bleaching times required in order to achieve six grades of whitening in human teeth. Any side effects that were noted and the patients' acceptance of the method were recorded by a visual analog scale ranging from 0 to 10. Moreover, epoxy casts from the study teeth were analyzed by scanning electron microscopy in order to detect any potential changes in the enamel surface due to treatments.

Thirty-nine volunteers participated in the study and were allocated randomly to one of three different bleaching treatments: Group A (n=13) used Whitestrips (over-the-counter technique; one cycle=30 minutes), Group B (n=13) used Opalescence PF 10% (at-home bleaching technique; one cycle=8 hours) and Group C (n=13) used Opalescence Xtra Boost (in-office bleaching technique; one cycle=15 minutes) until a defined whitening of six tabs compared to the baseline were reached (assessed by the VITA shade guide).

All three methods achieved six grades of whitening. The mean treatment time required to reach the defined level of whitening was 31.85 ± 6.63 cycles in Group A, 7.15 ± 1.86 cycles in Group B and 3.15 ± 0.55 cycles in Group C. All products differed significantly from each other in terms of treatment cycles and required treatment time ($p<0.001$ by ANOVA and Mann-Whitney-U-test). Using the VA scale, side effects noted within the three groups were minimal. Tooth hypersensitivity ranged from 2.62 (Whitestrips) to 3.38 (Opalescence PF), and gingival irritation ranged

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between 0.23 (Opalescence Xtra Boost) and 0.85 (Whitestrips). The most accepted method was the at-home bleaching technique. None of the teeth studied showed detectable enamel surface changes in the subsequent SEM analysis using 200x and 2000x magnification.

INTRODUCTION

Tooth bleaching has been described in the literature as early as 1889. Many of these early attempts were not very successful (Kirk, 1889). The most effective methods involve the use of hydrogen peroxide (Haywood, 1992). This bleaching agent enables the treatment to be efficient at removing intrinsic staining, and most of the current vital bleaching materials contain hydrogen peroxide in some form, either as carbamide peroxide or hydrogen peroxide per se (Fasanaro, 1992). Carbamide peroxide solutions are unstable and dissociate into their constituent parts on contact with tissue or saliva (Haywood, 1992; Haywood & Heymann, 1989). Carbamide peroxide solution ($CH_6N_2O_3$) breaks down into hydrogen peroxide (H_2O_2) and urea ($Ca[NH_2]_2$), after which the urea degrades into ammonia (NH_3) and carbon dioxide (CO_2). The active agent (H_2O_2) has to be in contact with the outer enamel surface for a period of time in order to develop its bleaching potential. Hydrogen peroxide breaks down into oxygen and water, which then penetrate the tooth and liberate the pigment molecules.

The most common methods used to remove discoloration from teeth consist of two clinical and a non-clinical technique. At-home bleaching is a method where the patient fills a custom-designed tray with bleaching material (10% to 20% carbamide peroxide resulting in 3.35-7% hydrogen peroxide) that is then worn for several hours. Since its introduction by Haywood and Heymann (1989), the original technique has undergone some modifications. For example, there have been changes in tray material, tray design and the use of reservoir and ingredient concentration (Leonard, Sharma & Haywood, 1998; Matis & others, 2002). This technique has become an efficient, safe method for lightening discolored teeth. Over-the-counter (OTC) bleaching products (5.3% to 5.6% hydrogen peroxide) are sold as cosmetics and are freely available through stores, pharmacies and the Internet. They can be sold, for example, as either strip or varnish systems and may cause patients problems, because a dentist does not monitor the bleaching procedure. The efficacy and structural side effects of this system have not been fully studied (White & others, 2003). In-office bleaching is useful for removing stains by using a high concentration of hydrogen peroxide (35% to 38%). The dentist is in complete control of the process throughout the treatment. This provides the advantage of being able to terminate the discoloring process at any time. Studies have shown that higher concentration materials may bleach teeth faster (Leonard & others, 1998). They usually work so rapidly that visible results can be observed after only a single visit.

Each of the described techniques has certain advantages and disadvantages (Leonard & others, 2001b; Dahl & Pallesen, 2003). A common clinical side effect is thermal sensitivity of individual teeth. This may occur during the bleaching procedure and usually stops when treatment is suspended. Gingival irritation caused by bleaching agents has also been reported. With the in-office technique, making use of a dam and with the at-home bleaching technique, using an individually designed guard, the agent only has minimal contact with soft tissue. Additionally, several studies have evaluated such adverse events as carcinogenicity (Dadoun & Bartlett, 2003) and effects on restorative materials (Langsten & others, 2002; Turker & Biskin, 2003). *In vitro* scanning electron microscopic evaluations of the surface texture of dentin (de Freitas & others, 2002) and enamel treated with different bleaching agents showed little to no changes (Haywood, Houck & Heymann, 1991; Leonard & others, 2001a; Auschill & others, 2002).

Because of the different techniques available and their varying peroxide concentrations, individual exposure times are necessary in order to achieve the same level of whitening. This allows for individually tailoring the bleaching program for each patient, but which technique is the best? Which technique does the patient prefer/accept most? Because the observations regarding effectiveness and adverse effects are still controversial and only a few studies have been carried out under intraoral conditions (Leonard & others, 2001b; Zekonis & others, 2003), there is a need for additional research on the impact of currently available bleaching techniques.

Thus, the aim of this study was to evaluate the efficacy of the three bleaching techniques *in vivo*, possible side effects such as tooth sensitivity and gingival irritations, patients' acceptance and any effects on enamel surface texture studied by scanning electron microscopy (SEM).

METHODS AND MATERIALS

This randomized, examiner blind clinical study used a parallel group design. It assessed intrinsic stain removal and the occurrence of intraoral adverse effects after bleaching with three different bleaching techniques *in vivo*. In addition, their effect on enamel surface morphology was investigated by SEM.

Thirty-nine human subjects in good health were recruited for this study. Subjects with poor general or dental health, fixed orthodontic appliances or known hypersensitivity were not permitted to participate. An

Table 1: *The Vita Value-oriented Shade Guide with 16 Shades Ranked From the Lightest Color on the Left to the Darkest Color on the Right*

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

Table 2: *Side Effects and Patients' Acceptance Evaluated by Visual-analog Scale*

	Group A	Group B	Group C
Tooth sensitivity	2.62 ± 1.46	3.38 ± 1.66	2.85 ± 1.41
Gingival irritations	0.85 ± 1.82	0.38 ± 0.87	0.23 ± 0.44
Patients acceptance	2.31 ± 1.93	1.46 ± 1.33*	3.31 ± 1.75*

**: statistically significantly different from each other (p<0.01; Mann-Whitney-U-test).*

inclusion criterium was the presence of the right unrestored upper canine, which was initially of grade A3 or darker according to the VITA shade guide (VITAPAN classical, VITA Zahnfabrik, Bad Säckingen, Germany). The degree of color change was evaluated by using color slide photography. All subjects gave their written consent and completed a medical history form.

The subjects received a professional tooth cleaning prior to the start of the study and were asked to brush their teeth twice daily with the allocated toothbrush (elmex inter X, GABA International AG, Basel, CH) and two toothpastes (aronal/elmex, GABA International AG, Basel, CH) in order to standardize tooth cleaning during the study.

Two trained, qualified examiners, who were blind as to treatment assignment and period, measured the baseline tooth color by using the VITA shade guide (VITAPAN classical, VITA Zahnfabrik, Bad Säckingen, Germany) on the facial surface of the right upper canine. Prior to starting the study, a calibrating session was held to review shade matching using the VITA system.

The tabs of the shade guide were arranged from B1 to C4, corresponding to a grade of whitening from 1 to 16 (Table 1) (Leonard & others, 2001b; Pohjola & others, 2002; Auschill & others, 2002), in which a smaller number means the tooth is lighter.

Participants were randomly assigned to three groups of 13 volunteers each (n=13 upper right canine). Group A was treated with the over-the-counter technique, Group B with the at-home bleaching technique and Group C with the in-office technique. The three groups were screened, then treated according to the specific bleaching technique:

For Group A, the strips (Whitestrips, 5.3% hydrogen peroxide, Procter & Gamble Technical Centres Ltd, Egham, UK) were distributed and their application demonstrated. The recommended wearing regimen was 30 minutes twice a day, and the participants were asked to rinse their mouth with water after wearing in order to remove any remaining gel from the teeth.

For Group B, a maxillary alginate impression was taken from the subjects and a model was cast for fabrication of the whitening tray. A 1-mm buccal reservoir from the right to the left upper canine was formed using block-out composite (LC Block-Out Resin, Ultradent Products, Inc, South Jordan, UT, USA). An ethyl-vinyl-acetate-tray (Sof-Tray, Ultradent) was made with the press-down machine. The participants were asked to wear their tray filled with the bleaching solution (Opalescence PF, 10% carbamide peroxide, Ultradent) for eight hours per night.

In Group C, the teeth to be bleached were isolated using a conventional dam (FlexiDam, Coltene, Langenau, Germany). With a syringe-to-syringe mixing process, the bleaching agent (Opalescence Xtra Boost, 38% hydrogen peroxide, Ultradent) was activated and a 1-mm thick layer of the material was applied on the labial surface of each tooth. The gel was removed 15 minutes after application (one cycle per appointment). This product contains hydrogen peroxide, which is chemically activated when mixed and does not need light activation.

Within each group, the whiteness of the study teeth from every subject was inspected 24 hours after the bleaching session, and the individual bleaching steps were repeated until the expected result was reached; in the case of this study, six tabs lighter than the baseline value. The examiner scored the shade of each test tooth by selecting the closest matching shade tab on the guide. If the examiners disagreed during the session, differences were discussed and an agreement reached. Intraoral color slides were taken in order to record the tooth shade for documentation of the baseline shade and to compare whether a change in shade occurred. Shade determinations were always performed under the same conditions (for example, no lipstick, same light source).

For safety and acceptance monitoring of gingival-irritations and tooth-hypersensitivity, participants were asked to record the total hours of wear daily and any intraoral adverse events in their teeth and gingiva. Subjects completed their questionnaires seven days after treatment, recording their personal response to the above mentioned side effects and their overall impression of the treatment (patients' acceptance) (Zekonis & others, 2003). Table 2 shows abnormalities

not present at baseline or which worsened during the bleaching process.

At baseline and after reaching the defined level of whiteness, impressions (Dimension Garant L, ESPE, Seefeld, Germany) of the upper right canine were taken. They were rinsed, dried and epoxy resin casts (Blue Star, Girrbach Dental GmbH, Pforzheim, Germany) made. The cast was removed from the impression, trimmed, dried for 24 hours, fastened on a carrier, sputter coated with gold palladium and examined under scanning electron microscope SEM (REM Leo 435 VP, LEO Electron Microscopy Ltd, Cambridge, GB) at 15kV. SEM pictures at baseline and after treatment of the upper canine at 200x and 2000x magnification were obtained in order to evaluate enamel texture changes. SEM photographs of each replica were taken 4-mm labially from the incisal edge and half mesiodistally in order to ensure the same location on the tooth was used. Three examiners compared each of the picture-pairs to find out whether a difference between the photographs evaluating enamel texture could be seen.

Statistical Analysis

Although the shade tab used was not linear between the different tabs, a statistical analysis could still be performed since the baseline data were similar and did not differ significantly from one another ($p > 0.05$; by ANOVA).

The average values of the bleaching cycles, required time for the individual products and the visual analogue scale were calculated using the statistical program SPSS 11.0. First, the data records were checked for normal distribution using the Kolmogorow Smirnov test. Since they were not normally distributed and significant differences between the products were found using ANOVA (analysis of variance), the Mann-Whitney-U test for independent samples was applied for statistical comparison among the three groups.

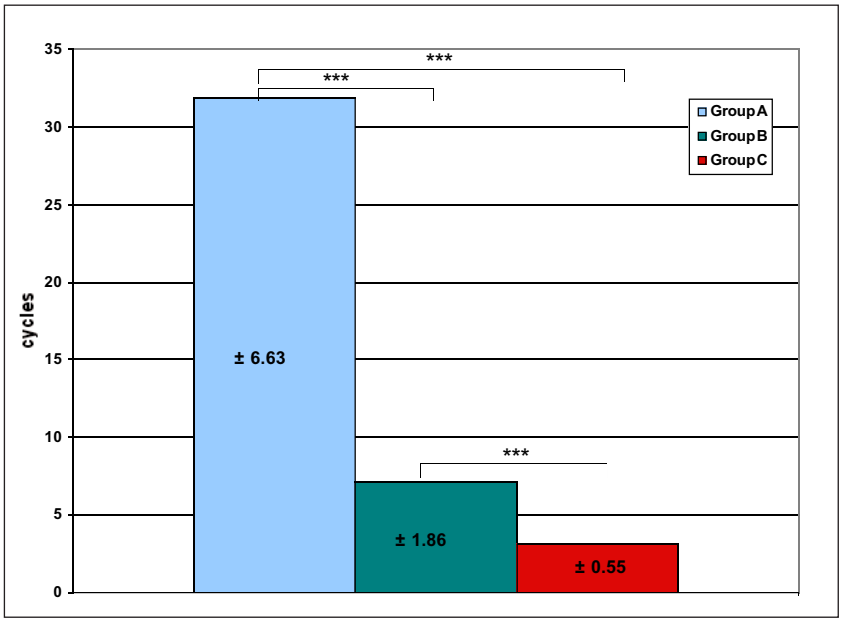

Figure 1. *Mean number of cycles (± standard deviations in parenthesis) for the various bleaching treatments and results of the statistical analysis (*:*p ≤ *0.05; **:*p ≤ *0.01; ***:* p ≤ *0.001 using Mann-Whitney-U test).*

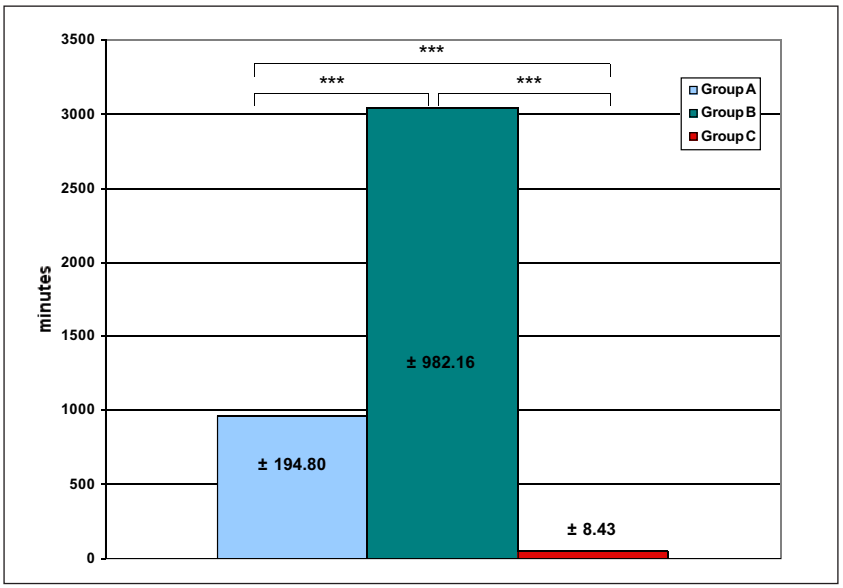

Figure 2. *Mean application time (in minutes) of the different groups after the various bleaching treatments and results of the statistical analysis (*:*p ≤ *0.05; **:*p ≤ *0.01; ***:* p ≤ *0.001 using Mann-Whitney-U test).*

RESULTS

All 39 participants completed the study. The volunteers ranged in age from 21 to 68 years, with the average age being 29.82 years. The participants were randomized into three groups. There were no statistically significant differences in the mean baseline shade, age or gender of the participants.

Efficacy of Shade Change

The shades of the three groups did not differ significantly at baseline. Their values ranged from grade 9-15, with a mean value of 11.2±1.8 in Group A, 11.5±2.0 in Group B and 11.4±2.1 in Group C ($p > 0.05$ by ANOVA). All three techniques proved to be effective at whitening. The mean treatment time required to achieve the defined shade was 31.85 ± 6.63 cycles (= 958.46 ±

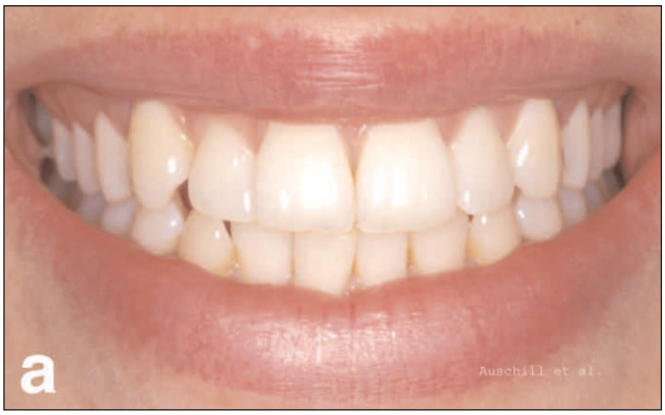

Figure 3a. *Clinical photograph before over-the-counter treatment.*

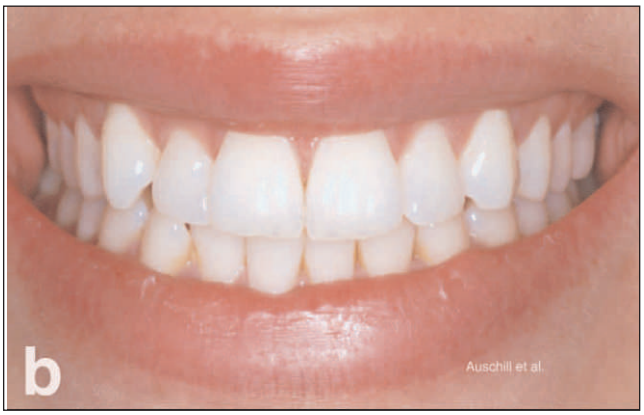

Figure 3b. *Clinical photograph after 16 days of over-the-counter treatment.*

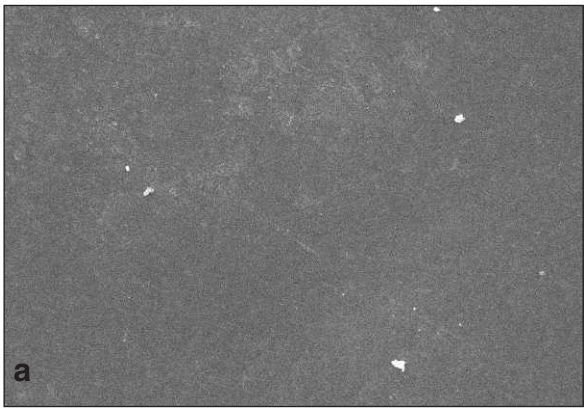

Figure 4a: *SEM photograph of enamel before over-the-counter treatment (2000x).*

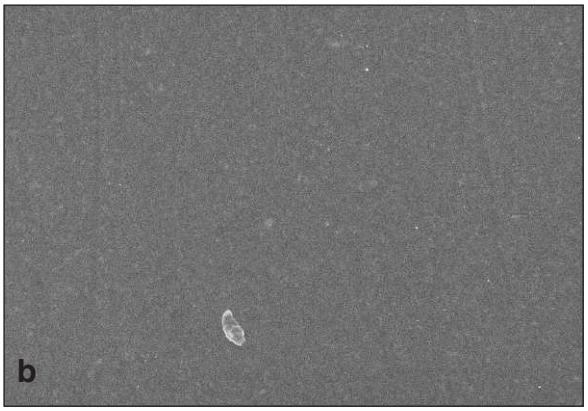

Figure 4b: *SEM photograph of enamel after over-the-counter treatment (2000x).*

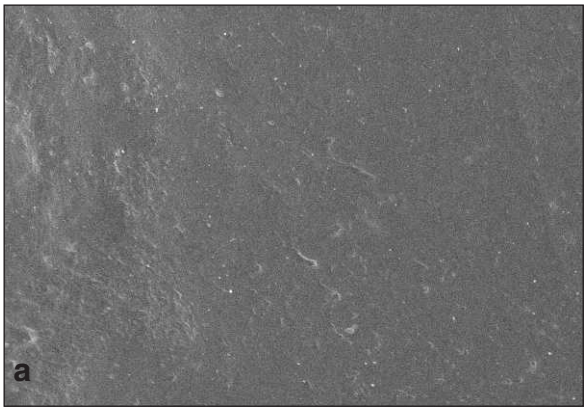

Figure 5a: *SEM photograph of enamel before at-home treatment (2000x).*

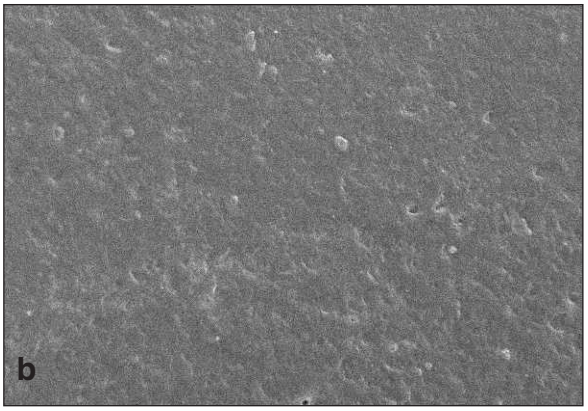

Figure 5b: *SEM photograph of enamel after at-home treatment (2000x)*

194.80 minutes) in Group A, 7.15 ± 1.86 cycles (= 3043.85 ± 982.16 minutes) in Group B and 3.15 ± 0.55 cycles (= 47.08 ± 8.43 minutes) in Group C (Figures 1 and 2). Photographs of one participant (representing the pre- and post-bleaching situations) are shown in Figure 3a and b.

Side-effects and Patients' Acceptance

In order to record side effects and patients' acceptance, a visual analogue scale was used in which the extremes represented the limits of pain and acceptance. One end was labeled "no discomfort" or "best acceptance" (0), whereas, the other end was labeled "severe discomfort" or "no acceptance" (10). Subjects were asked to mark

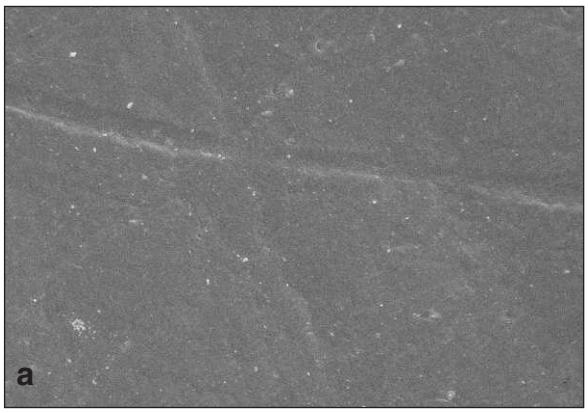

Figure 6a: *SEM photograph of enamel before in-office treatment (2000x.)*

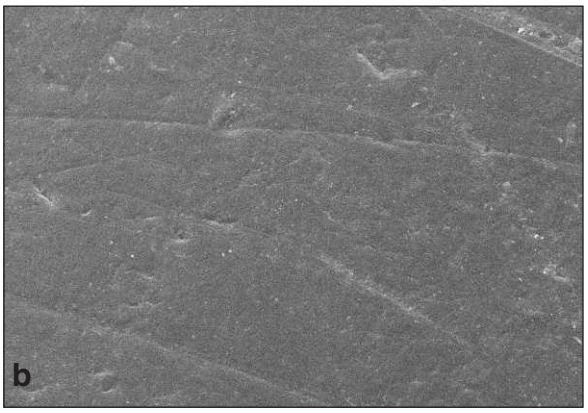

Figure 6b: *SEM photograph of enamel after in-office treatment (2000x).*

the position that best indicated their current opinion. All products yielded minimal side effects in the volunteers. Transient problems documented by the patients were initial gingival irritation and slight thermal tooth sensitivity (Table 2). These symptoms were mild, transient and reversible. Additionally, Table 2 shows the average values of patients' acceptance.

Statistical Analysis of these data demonstrated that there was only one significant difference between Opalescence PF 10% and Opalescence Xtra Boost concerning acceptance ($p \leq 0.05$, by Mann-Whitney-U-Test). All other comparisons detected no significant differences ($p > 0.05$ by ANOVA).

SEM Analysis

The second part of the study showed that teeth treated with bleaching agents had no observable enamel surface texture changes when evaluated by the three examiners. No differences outside normal tooth variations were visible when the texture of the epoxy resin replica surface at baseline was compared with its corresponding cast after bleaching, regardless of whether 200x or 2000x magnification was used. Figures 4 through 6 present pre- and post-treatment SEM photographs of the enamel surface of each group.

DISCUSSION

To date, several studies have been performed in order to observe the whitening effect of some products following a defined bleaching time. In contrast, this study examined the time required to achieve a defined bleaching result. Thus, this study design was chosen to take into account the patients' claims of achieving visible whitening rather than using a product for a defined time.

It is difficult to compare the results of this study with data from the literature, because of significant variations in study design (*in vivo* vs *in vitro*), concentration of active agents or length of exposure time. On the other hand, to the best of our knowledge, there are no published studies available where these three techniques are compared with one another.

Efficacy

The precondition was to bleach six grades lighter than baseline value, which was achieved in each of the 39 study teeth (Figures 3a and 3b). Thus, the clinical efficacy rate for the volunteers in the groups was 100%. Different methods of determining this tooth shade change can be used (colorimeter, shade guide). In this study, the VITA shade guide was used, because it is still the most commonly used method and is predictable when whitening teeth (Freedman, 1997).

Side Effects

An additional objective of this study was to determine possible side effects. Teeth and gum sensitivity were self-evaluated by the volunteers. Penetration of bleaching agents into tooth hard tissue results in different changes in vital teeth. Numerous studies have shown that pulpal reactions to bleaching agents are reversible (Cohen, 1979; Robertson & Melfi, 1980). They reported no histological changes in the treated teeth when compared with the controls and concluded that vital bleaching was harmless to pulpal tissues. A study by Seale, McIntosh and Taylor (1981) showed that treatment in dogs with 33% hydrogen peroxide alone or with heat caused obliteration of odontoblasts, hemorrhage, resorption and inflammatory infiltration, while heat alone was not detrimental. Pulpal changes demonstrated evidence of reversibility after 60 days.

In this study, none of the three tested products needed light for activation of the bleaching process. This may explain the low sensitivity values. The at-home treatment caused slightly higher tooth sensitivity compared to the over-the-counter and in-office treatments but this was not significant and had no consequence in patients' acceptance. This higher value could be explained by the longer application time (in minutes)

(Figure 2). Similar general observations could be made regarding gingival irritation. The over-the-counter treatment caused higher gingival irritation compared to the at-home or in-office treatments, but again, no significant differences existed between the groups (Li & others, 2003). These higher irritations could be due to the fact that there was no monitoring of the over-the-counter technique by a dentist. The strips were more irritating to the gingiva than the in-office treatment, which used a dam, and the at-home treatment, with its individually designed guard. However, it should be kept in mind that the irritation was mild and reversible in each case and none of the volunteers had to resign. Additionally, visual inspection by the examiners showed no signs of gingival inflammation or necrosis after clinical treatment. The fact that, for many years, carbamide peroxide and hydrogen peroxide solutions have been investigated and used clinically to bleach vital teeth without incurring pulpal and gingival damage is an indication of its safety to these tissues.

Patients' Acceptance

The at-home bleaching treatment was significantly more accepted by patients compared with the in-office method. When asked for reasons, the volunteers indicated that the at-home technique required less chair-time despite the in-office method being under the dentist's control. It should be emphasized that values between zero and five indicate wide acceptance for all three techniques. Thus, in principle, all products are recommendable.

SEM

The second part of the study examined the effect of tooth bleaching agents on enamel surface. These observations were based on scanning electron microscopic evaluations of epoxy casts of replicas made of the study teeth (Leonard & others, 2001a). All evaluations were performed by three examiners blinded to the status of the tooth. After the respective treatment times in each group (16 days of active treatment in Group A, seven days in Group B and one day in Group C), the surface morphology showed no noticeable changes compared to baseline (Figures 4-6). Leonard and others (2001a) demonstrated that a regimen using a 10% carbamide peroxide solution had minimal to no effect on the enamel surface. This is in accordance with other SEM findings (Haywood & others, 1991; Spalding, Taveira & de Assis, 2003; White & others, 2003), where bleaching was considered to be safe for enamel. In contrast, Bitter (1992) and McGuckin, Babin and Meyer (1992) found some grooves on the enamel surface in their studies. Similar results were presented by Hegedüs and others (1999), where atomic force microscopy pictures showed that several grooves present in the enamel surface of untreated teeth became deeper after the bleaching procedure. The increase in depth of the grooves was more pronounced in the case of the higher concentrated solution (30% HP) after 28 hours of treatment. It was presumed that the differences in groove depth after treatment were caused by the difference in hydrogen peroxide concentration. However, in this study, the treatment with 38% hydrogen peroxide took 60 minutes in order to reach the desired result. Thus, in addition to concentration, the major difference among the study-designs was the time of application of the active agent. In summary, the SEM results of this study showed that no differences between the tested bleaching methods could be observed with regards to surface texture changes. However, it should be kept in mind that peroxide could have not only affected the surface but also the inner structure of the tooth. Without penetrating through hard tooth tissues, it would be impossible to treat the intrinsic discolorations (Seale & others, 1981). Chemical release of calcium and other minerals was observed (Mc Cracken & Haywood, 1996), whereas, the clinical significance of this small amount was assumed not to be significant. It was not possible in the course of this *in vivo* study to evaluate these internal areas and more investigations are needed on this topic.

The results of this study indicate that each of the treatments has certain advantages and disadvantages. The dentist should be familiar with all of them in order to serve patients best. The patient can be treated with a single technique or a combination. For example, the patient can start with the in-office technique to receive immediate results and continue with one of the others to save or extend the whitening.

CONCLUSIONS

It can be concluded that all three tested techniques are effective in removing intrinsic staining. In principle, the higher the concentration of the active ingredient, the faster tooth lightening occurs (in minutes). Following the manufacturers' instructions (cycles), it took an average of 16 days with the over-the-counter bleaching technique, seven days when using the at-home bleaching technique and, with the in-office bleaching technique, the result may be achieved in one day. The side effects that appeared were reversible, none of the volunteers had to resign and there were no statistically significant differences among the groups. All techniques were well accepted, with a slight preference for the home bleaching method. Additionally, the tested products had no evident effect on the surface morphology of teeth when viewed under SEM at 200x and 2000x magnification.

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Laboratory Research

Microleakage of Compomer Restorations in Primary Teeth After Preparation with Bur or Air Abrasion

Ö Aysegül • Ö Nurhan
B Haluk • T Dilek

Clinical Relevance

Where the cavity had been prepared conventionally or with air abrasion, acid etching may be eliminated for compomer restorations.

SUMMARY

This study compared the degree of marginal leakage of a compomer in Class V cavities of human primary molars prepared by a conventional dental bur and air abrasion with or without acid etching.

Fifty-six non-carious extracted primary molars were randomly divided into four groups (n=14) to be prepared by four techniques: Group-1: Bur followed by acid etching: Class V cavity preparations were placed on the buccal surfaces of each tooth using a high-speed handpiece. The preparations were 1.5-mm deep, 3-mm long and 2-mm wide, with the occlusal margin in enamel and the cervical margin extending 0.5 mm below the cementoenamel junction. The preparations were acid etched with 37% phosphoric acid starting at the enamel margins for 30 seconds and rinsed with water for 20 seconds. The preparations were then restored with Compoglass F. 2-Group 2: Bur: The preparations and the treatment procedures were the same as in Group 1, with the exception of 37% phosphoric acid application. Group 3: Air abrasion followed by acid etching: Class V cavity preparations were placed on the buccal surfaces of each tooth using a handpiece of an air-abrasive system (PrepStart, Danville Engineering). The system was supplied with dry compressed air at 80 psi. In all tests, the air-abrasion system was operated with an 80˚-angle handpiece tip and 50-mm aluminum oxide particles. A tip with a 0.38-mm inner diameter was used at a 2-mm distance. The treatment procedures were the same as in Groups 1 and 2. Group 4: Air abrasion:The preparations and treatment procedures were the same as in Group 3, with the exception of 37% phosphoric acid. After finishing the restorations, the teeth were stored in distilled water at 37˚C

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for 24 hours. The samples were thermocycled for 500 cycles between 5˚C and 55˚C with a dwell time of 30 seconds. The samples were then immersed in 0.5 percent basic fuchsin dye for 24 hours at 37˚C. The surface-adhered dye was then rinsed in tap water and the teeth were embedded in a chemically-activated acrylic resin and bisected longitudinally in a mesiodistal direction with a low speed diamond disk. Each section was examined under a stereomicroscope (Nikon, Tokyo, Japan) at 20x magnification. The data were analyzed statistically by Kruskal-Wallis analysis of variance to determine any statistical significant differences in microleakage scores among the groups at a *p*-value of 0.05. Also, the enamel versus cementum-dentin microleakage scores of each group were compared using z-test at the 0.05 significance level. There was no statistically significant difference among the groups ($p>0.05$), but a statistical difference between enamel and cementum-dentin surfaces was evaluated ($p<0.05$).

INTRODUCTION

In recent decades, dental research has produced improved restorative techniques and materials to reproduce the characteristics and appearance of lost dental tissue. The development of adhesive resin restorative systems has minimized the need for classical Black retentive cavity preparation (Horiguchi & others, 1998). Newer methods, such as aluminum oxide air abrasion, have become widespread. However, while air abrasion has offered some remarkable advantages, final cavity preparation still required mechanical instrumentation. This shortcoming, coupled with the development of high-speed air turbine handpieces, caused air abrasion technology to be dismissed and only reintroduced in the last decade (Rinaudo, Cochran & Moore, 1997). Modern air abrasion units employ a high-speed stream of purified aluminum oxide particles propelled by air pressure. The technique has been proposed for potential dental applications, including modification of tooth surfaces and cavity preparation (Laurell, Carpenter & Beck, 1994; Goldstein & Parkins, 1994, 1995; Myers, 1994; Nikaido & others, 1996; Horiguchi & others, 1998; Chan & others, 1999; Murdoch-Kinch & McLean, 2003).

As the cavity preparation by air abrasion provides a surface roughening with a groove width of 1-20 µm (Laurell & Hess, 1995), the abraded surface is suitable for direct bonding techniques. Although bond strengths to air abraded enamel were comparable to that achieved with acid-etched surfaces, higher bond strengths to dentin were found with abraded compared to etched surfaces (Laurell, Lord & Beck, 1993; Keen, Von Fraunhofer & Parkins, 1994). In contrast, satisfactory bond strengths are only achieved with abraded surfaces when they are also etched (Brockmann, Scott & Eick, 1989; Roeder & others, 1995; Brown & Barkmeier, 1996; Öztas, Alaçam & Bardakçy, 2003).

The loss of seal integrity manifested as microleakage is a primary cause of secondary caries, post-operative sensitivity and staining. Reducing or eliminating microleakage around restorations is important in clinical practice (Wu & others, 1983; Von Fraunhofer & Hammer, 1984; Dumscha & Biron, 1984; Kanca, 1989; Fortin & others, 1994). The relationship between air abrasion and microleakage has received limited attention. There are relatively few studies on microleakage occurring with restorations placed in cavities prepared by air abrasion (Corona & others, 2001; Von Fraunhofer & others, 2000; Guirguis, Lee & Conry, 1999; Setien & others, 2001; Fu & Hannig, 1999).

Although manufacturers of air abrasive devices claim that their use reduces or eliminates the need for acid etching, the degree of microleakage that may result following air abrasion tooth preparation with or without acid etching is unclear.

Guirguis and others (1999) measured and compared microleakage at preventive resin restorations where the cavity had been prepared conventionally or with air abrasion with or without acid etching. This study's findings did not support the manufacturer's claims that stated the use of air abrasion reduced or eliminated the need for acid etching.

Von Fraunhofer and others (2000) reported that air abrasion alone did not induce sufficient surface roughening of hard tooth tissue to avoid or prevent microleakage, and they claimed that hard dental tissue should always be acid etched prior to any bonding procedure irrespective of cavity preparation method. Hannig and Fu (2001) reported that the use of air abrasion, in combination with self-etching priming agents, could not prevent microleakage at the dentin-resin interface of Class V resin composite restorations.

However, Corona and others (2001) investigated the degree of marginal leakage of a resin composite in Class V cavities prepared by high speed dental bur, air abrasion or Er:Yag laser and concluded that conventional dental bur preparation with acid etching resulted in the best overall seal.

There is no reported research concerning the effects of air abrasion on primary teeth and, consequently, the marginal seal of cavities prepared by air abrasion or conventional dental bur.

For the restoration of permanent teeth, composites offer advantages over compomers and glass ionomers in terms of wear resistance and esthetic stability. However, requirements may differ for primary teeth. In addition, caries rates are likely to be high in children

with proximal lesions, so fluoride release may be helpful. Manufacturers have packed compomers with the recommendation that a separate phosphoric acid-etching step is not required, because the acidity of the bonding agents results in some dentin etching and produces acceptable marginal leakage, measured *in vitro*. Since children may be uncooperative in terms of lengthy etching and bonding procedures, compomers may provide a better alternative to resin composite. The use of compomer one-bottle adhesive systems without a phosphoric acid pretreatment is controversial, as several trials have demonstrated lower rates in bond strength and marginal adaptation (El Kalla, 1999). Also, three clinical studies have demonstrated improved clinical performance of compomers when the total etch technique is employed (Cadenaro, DiLenarda & Cernaz, 1998; Ferrari & others, 1998; Attin & others, 2001).

This study compared the degree of marginal leakage of a compomer in Class V cavities of human primary molars prepared by a conventional dental bur and air abrasion with or without acid etching.

METHODS AND MATERIALS

Fifty-six non-carious extracted primary molars stored in distilled water at room temperature for up to three months were used in the study. After surface debridment with a hand-scaling instrument and cleaning with a rubber cup and slurry of pumice, the teeth were randomly divided into four groups for preparation by four techniques: 1-Bur followed by acid etching, 2-Bur, 3-Air abrasion followed by acid etching and 4-Air abrasion.

Group 1: Bur followed by acid etching:

Class V cavity preparations were placed on the buccal surfaces of each tooth using a high-speed handpiece. The preparations were 1.5-mm deep, 3-mm long and 2-mm wide, with the occlusal margin in enamel and the cervical margin extending 0.5 mm below the cementoenamel junction. The preparations were acid etched with 37% phosphoric acid starting at the enamel margins for 30 seconds, rinsed with water for 20 seconds and gently air dried to leave the surfaces wet (Ferrari & others, 1998; García-Godoy, 2000; Attin & others, 2001). Syntac Single Component (Vivadent, Schaan, Liechtenstein) was applied to the moist surfaces with a brush, left undisturbed for 20 seconds and the excess solvent removed with a gentle stream of air and light cured with a visible light-curing unit (Translux EC, Kulzer, Germany) for 20 seconds. A second coat was applied, air dried and light cured for 20 seconds. The preparations were then restored with Compoglass F (Vivadent, Schaan, Liechtenstein) in one increment and light cured for 40 seconds.

Group 2: Bur:

The preparations and treatment procedures were the same as in Group 1, with the exception of 37% phosphoric acid application.

Group 3: Air abrasion followed by acid etching:

Class V cavity preparations were placed on the buccal surfaces of each tooth using a handpiece of an air-abrasive system (PrepStart, Danville Engineering). The system was supplied with dry compressed air at 80 psi. In all tests, the air-abrasion system was operated with an 80˚ angle handpiece tip and 50-mm aluminum oxide particles (Peruchi & others, 2002). According to Kotlow (1996), this particle size is recommended for sealants and the preparation of small primary teeth. The tip, with a 0.38-mm inner diameter, is used at a 2-mm distance (Peruchi & others, 2002). The treatment procedures were the same as in Groups 1 and 2.

Group 4: Air abrasion:

The preparations and treatment procedures were the same as in Group 3, with the exception of 37% phosphoric acid.

All samples were finished with Sof-Lex discs (3M ESPE, St Paul, MN, USA) immediately after restoration and stored in distilled water at 37˚C for 24 hours. The samples were thermocycled for 500 cycles between 5˚C and 55˚C, with a dwell time of 30 seconds. The restored areas and root apices of the remaining samples were sealed with Vitrebond (3M ESPE). Two coats of nail varnish were applied on the tooth, 1-mm short of the margins to be exposed to dye. The samples were then immersed in 0.5 percent basic fuchsin dye for 24 hours at 37˚C. The surface-adhered dye was then rinsed in tap water, the teeth embedded in a chemically-activated acrylic resin and bisected longitudinally in a mesio-distal direction with a low speed diamond disk. Each section was examined under a stereomicroscope (Nikon, Tokyo, Japan) at 20x magnification. The following scoring scale was used to assess the extent of the linear dye penetration at the tooth-restoration interface: 0= no dye penetration; 1= dye penetration up to or less than half of the occlusal or gingival wall; 2= dye penetration up to the axial wall; 3= dye penetration along the axial wall. Each section was independently evaluated by three evaluators as described in the other studies (García-Godoy, 2000; Toledano & others, 1999; Puckett & others, 1995).

The data were analyzed statistically by Kruskal-Wallis analysis of variance to determine any statistically significant differences in microleakage scores among the groups at a p-value of 0.05. Also, the enamel versus cementum-dentin microleakage scores of each group were compared using z-test at the 0.05 significance level.

Table 1: *Enamel Microleakage Scores*

	Microleakage Scores*			
Group	**0**	**1**	**2**	**3**
1(n=14)	8	0	6	0
2(n=14)	10	2	2	0
3(n=14)	12	2	0	0
4(n=14)	10	0	4	0

**No significant difference among all groups (p>0.05).*

Table 2: *Cementum-dentin Microleakage Scores*

	Microleakage Scores*			
Group	**0**	**1**	**2**	**3**
1(n=14)	2	0	10	2
2(n=14)	2	2	4	6
3(n=14)	2	2	4	6
4(n=14)	4	0	6	4

**No significant difference among all groups (p>0.05).*

RESULTS

Tables 1 and 2 show the results. There was no statistically significant difference among the groups ($p>0.05$); however, a statistical difference between the enamel and cementum-dentin surfaces was found ($p<0.05$).

DISCUSSION

Air abrasive tooth cutting has been accepted relatively well by a small segment of the profession. Its use is growing slowly and is expected to continue to grow in popularity. Devices are becoming more refined and lower-cost air abrasion units are now available. The use of air abrasion tooth-cutting for smaller restorative situations is relatively painless, fast, effective and non-invasive compared with rotary cutting. It has been postulated that cutting with air abrasion systems results in minimal loss of tooth structure (Goldstein & Parkins, 1994; Banerjee & Watson 2002).

Some investigators (Guirguis & others, 1999; Von Fraunhofer & others, 2000; Hannig & Fu, 2001) claimed that acid etching after air abrasion is necessary for restorations where the cavity had been prepared conventionally or with air abrasion, with or without acid etching. Although higher bond strengths are only obtained with abraded surfaces when they are also etched, Laurell and others (1993) and Keen and others (1994) found higher bond strengths to dentin with abraded compared to etched surfaces. However, in all of these studies, permanent teeth were investigated. As there was no any report including air abrasion in primary teeth, we could not compare our results to the others.

There was no statistical difference between Group 3 (air abrasion followed by acid etching) and Group 4 (air abrasion). So the elimination of acid-etching following air abrasion or a conventional preparation may be considered for compomer restorations of primary teeth.

Parameters for the safe and precise use of an air-abrasion system for cavity preparation on primary teeth may reproduce the desired results. Peruchi and others (2002) evaluated the influence of diameter, tip-to-tooth distance and time of application of an air-abrasive system (Prep-start) on the cavity patterns produced in primary teeth and found that removal of enamel in primary teeth is best accomplished when a tip with a 0.38 mm inner diameter was used at a 2-mm distance. In this study, the same air-abrasive system was used with a handpiece with an 80˚ angle nozzle, 50 µm abrasive particle size and 80 psi air pressure. The tip diameter and the distance of the tip from the enamel surface were selected according to Peruchi and others' study. The successful results obtained in this study may be related to the correct use of the air abrasive system in primary teeth.

A number of reports have been published that phosphoric acid pre-treatment resulted in strong micromechanical interlocking to both enamel and dentin, better demineralization depth and hybrid layer thickness (El Kalla, 1999; Ferrari & others, 1999; Attin, Buchalla & Hellwig, 1996).

Manufacturers often advocate etching when supplying a dentin adhesive with a resin composite material but recommend against it when supplying the same adhesive with a compomer. The acid-etching procedure used in this study was designed according to the other studies (Ferrari & others, 1998; García-Godoy, 2000; Brackett & others, 2001).

Compomer adhesive systems, such as for Dyract (Dentsply Detrey, Konstanz, Germany), Compoglass (Vivadent, Schaan, Liechtenstein) and F2000 (3M ESPE, St Paul, MN,USA), are based on a "one bottle" bonding procedure as self-etching/priming/adhesive agents to be applied to enamel and dentin. There are contrary reports regarding the etching performance of self-etching primer/adhesive systems without phosphoric acid pre-treatment (Hse & Wei, 1997; Andersson-Wenckert, Folkesson & van Dijken, 1997; Roeters & others, 1998; Papagiannoulis & others, 1999; Hse, Leung & Wei, 1999; Tate, You & Powers, 1999, 2000; Marks & others, 1999; Luo & others, 2000). In a clinical study, Attin and others (2001) evaluated the three-year success rate of Class II restorations in primary molars performed with Compoglass (compomer) and TPH-Spectrum (hybrid composite). The primary molars were restored with TPH-spectrum using the total-etching technique and Compoglass without acid etching prior to application of the bonding adhesive and found that both demonstrated acceptable clinical results. In this study, the effect of phosphoric acid etching for reducing microleakage was not significantly

different. This finding may be related to the varying mineral content and sclerotic changes of primary dentin and therefore may limit the expected effects of acid pre-treatment (El Kalla & García-Godoy, 1998). Acid probably acts differently on primary teeth because of differences in the microstructure of primary and permanent teeth (Rontani & others, 2000). Of the self-etching primer/adhesive systems used in the study, Syntac Single Component contains modified polyacrylic acid and maleic acid. Self-etching primer adhesive systems can affect enamel even though the etching pattern was found to be shallower than conventional acid etching (Hse & others, 1999; Luo & others, 2000).

Glass-ionomer cement requires a prolonged maturation time and should not be challenged with dehydration within six months of placement (Mount, 1990). Preparation of the specimens for this investigation may have led to some dehydration of the restorations, especially during application/drying of the nail varnish. Bouschlicher, Vargas and Denehy (1996) showed that inadvertently dessicating the restoration prior to dye immersion increased microleakage scores with some resin-modified glass ionomers, glass ionomer cements and microfil resins. If contraction under desiccating conditions disrupts the bond at the cervical margin or is far greater than the expansion by water absorbtion (Wilson & Paddon, 1993), there could be a resultant increase in microleakage. It should be remembered that materials evaluated in this study were likely to be more technique sensitive, perhaps leading to greater differences.

There was a statistically significant difference between the enamel and cementum/dentin microleakage scores. As the cervical margin was extended below the cementoenamel junction, higher microleakage results obtained on cementum margins for bur or air abrasion may be considered normal. The leakage scores at cementum margins may not be clinically relevant due to the relatively short life span of the primary dentition.

CONCLUSIONS

1. Enamel margins provided better marginal sealing than dentin/cementum margins.
2. Surface treatment had no significant effect on microleakage between the tooth structure and compomer restorations of primary teeth.

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Bond Strength and SEM Observation of CO_2 Laser Irradiated Dentin, Bonded with Simplified-step Adhesives

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Clinical Relevance

Dentin surface irradiated by CO_2 laser could be an adherent only when the carbonized dentin layer on the surface to be bonded was mechanically removed, although long-term durability of the interface is still to be studied.

SUMMARY

This study investigated, mechanically and morphologically, whether the dentin surface irradiated by CO_2 laser could be a possible adherent when bonded with simplified-step adhesives. Buccal enamel and cementum of extracted human premolars were removed to expose a flat dentin surface. The dentin surfaces were irradiated continuously with CO_2 laser at 1.0 W. Before bonding with either a single-bottle adhesive (Single Bond) or a self-etching priming system (Mega Bond), the irradiated dentin surface was treated as follows: no treatment, $NaHCO_3$ powder abrasion and wet-grinding with 600-grit SiC paper. The treated dentin surfaces were bonded to resin composite with either of the two adhesives. Non-irradiated dentin surfaces were also used as control. Resin bonded specimens were stored in water at 37°C for 24 hours and subjected to microtensile bond test. Additionally, to observe the resin/irradiated dentin interface, resin-bonded specimens were similarly prepared, sectioned into slabs, embedded in epoxy resin, polished with diamond pastes, sputter coated Au-Pd and examined with scanning electron microscopy (SEM). After SEM observation, the specimens were further polished with diamond paste to remove the Au-Pd sputter-coat, immersed in HCL and NaOCl and finally observed by SEM again.

In the presence of carbonized dentin, microtensile bond strength drastically decreased but recovered to the control value by removing the carbonized dentin layer visually with SiC paper for both adhesive systems. However, the laser-affected dentin that remained on the bonded interface was easily dissolved with NaOCl and HCl.

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INTRODUCTION

Laser is well known as an abbreviation for light amplification by stimulated emission of radiation and the laser beam is widely used in various fields. Shortly after the laser had been invented (Maiman, 1960), application of the laser beam to tooth substrates had started to be investigated in the field of dentistry. The laser has been reported to change the surface characteristics of tooth tissue and thus is supposed to have several possibilities (Goldman & others, 1965) such as serving as an effective tool for cavity preparation for restorations or a conditioning instrument for enamel to increase acid-resistance (Goldman & others, 1965; Stern, 1974; Featherstone & others, 1998; Hsu & others, 2000). It is also reported that dentin irradiated with CO_2 laser is sterilized and acquires acid resistance (Nammour, Renneboog-Squilbin & Nyssen-Behets, 1992; Hossain & others, 1999; Le Goff & others, 1999). These phenomena are of importance in treating dental caries and preventing subsequent recurrent caries.

In general, when bonding resinous materials to dentin, three steps are required to achieve an effective bond. Acid-etching is first performed to decalcify the dentin surface, followed by a priming step and the application of adhesive resins, resulting in micro-mechanical retention through the development of resin-tags and/or a so-called "hybrid layer" (Nakabayashi, Kojima & Masuhara, 1982). Most recent developments have focused on simplification of the multi-step bonding process using one of two different approaches, namely "total-etch" and "self-etch" concepts (Van Meerbeek & others, 2001; Inoue & others, 2000). Simplified "two-step total-etch" adhesives combine the primer and adhesive resin into one application. Its underlying mechanism of adhesion to dentin is similar to the conventional three-step total-etch adhesives. "Two-step self-etch" adhesives are based on the use of non-rinsing acidic monomers that simultaneously condition and prime dentin. The second step, application of the adhesive resin, then follows. The bonding mechanism of "self-etch" adhesives to dentin is also based on hybridization, the difference being that only submicron hybrid layers are formed and resin-tag formation is less pronounced (Van Meerbeek & others, 2001).

When the laser is irradiated onto dentin, localized melting and re-crystallization occurs and fungi-form projections are produced on dentin surfaces (Cooper & others, 1988). Many studies have described the measurements of bond strength to laser-irradiated dentin and the morphological changes to lasered dentin surfaces (Silberman & others, 1994; Moritz & others, 1998; Kataumi & others, 1998; Ceballo & others, 2002; Kameyama & others, 2000). According to them, the bond strength of adhesive resin to laser-irradiated dentin is lower than to acid-etched dentin. However, no report is available on the detailed mechanism of how adhesive agents react with laser-irradiated dentin, especially when simplified-step adhesives are used. This study investigated mechanically and morphologically whether the dentin surface irradiated by CO_2 laser could be a possible adherent when bonded with simplified-step adhesives, that is, a single-bottle adhesive and a self-etching priming system.

METHODS AND MATERIALS

Tooth

Twenty-eight non-caries human premolars extracted for orthodontic reasons with an informed consent protocol approved by the Commission for Medical Ethics of Hokkaido University Dental Hospital were used within two months of extraction. Eighteen teeth were used for the microtensile bond test and 10 teeth were used for scanning electron microscopic observation of the bonded interface as stated below.

Specimen Preparation and Bonding Procedures

The buccal enamel and cementum of 18 teeth were removed using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water coolant, to expose a flat mid-coronal dentin surface. Each surface was ground with 600-grit silicon carbide paper under running water for 30 seconds. Four surfaces were used as the non-irradiated control (Group 1). Fourteen dentin surfaces out of 18 were irradiated continuously with CO_2 laser (Opelaser 03-S, Yoshida, Tokyo, Japan) at an energy level of 1.0 W. The irradiation was defocused, because it is difficult in clinical situations to irradiate by focus setting. Then, the irradiated dentin surfaces were subdivided into three groups (Groups 2-4). The irradiated surfaces of Group 2 (4 surfaces) were not treated further. In order to remove the carbonized surface dentin visually, Group 3 (5 surfaces) were treated with an abrasion system using $NaHCO_3$ powders (Polaris powder, Osada, Tokyo, Japan) and Group 4 (5 surfaces) were wet ground with a 600-grift SiC paper.

Then, 18 specimens were subjected to bonding treatment using either of the two adhesive systems. Table 1 lists the components and chemical formulations of the adhesives used. For Single Bond, the prepared dentin surfaces of Groups 1 through 4 (2 surfaces each) were acid-etched for 15 seconds and thoroughly rinsed using water spray. Excess water was blot dried from the surface with a cotton pellet, leaving the surface visibly moist (wet bonding). The bonding resin was applied with at least two consecutive coats and light cured for 10 seconds with a light-curing unit (XL 3000, 3M, St Paul, MN, USA) with a light output of not less than 550 mW/cm^2. For Clearfil Mega Bond, a self-etching primer was applied onto the prepared dentin surfaces of Groups 1 and 2 (2 surfaces each) and Groups 3 and 4 (3 surfaces each) and left undisturbed for 20 seconds.

Table 1: *Components and Chemical Formulations of the Adhesives Investigated*

Adhesive	Code	Manufacturer	Components (Lot #)
Single Bond	SIB	3M	Etch (0EA): phosphoric acid Resin (0FA): Bis-GMA, HEMA, water, dimethacrylate, polyalkenoic acid copolymer
Clearfil Mega Bond	MB	Kuraray	Primer (00135A): MDP, HEMA, dimethacrylate Bond (0008A): MDP, HEMA, dimethacrylate, filler, photoinitiator

Bis-GMA = bis-phenol-A-diglycidylmethacrylate, HEMA = 2-hydroxyethylmethacrylate, MDP = 10-methacryloyloxydecyl dihydrogen phosphate

Table 2: *Microtensile Bond Strength*

Adhesive	Group			
	1	2	3	4
SIB	57.2 (4.5)[a]	0 (0)*	12.8 (14.0)	50.0 (14.2)[a]
	(0/8)	(8/8)	(4/8)	(0/8)
MB	41.1 (10.0)[b]	4.8 (6.7)	23.5 (11.0)[b]	25.7 (13.7)[b]
	(0/8)	(5/8)	(0/11)	(0/10)

Data are shown as mean (SD) in MPa. Values in bottom row show the number of pre-testing failures/total number of specimens. Bond strength values with the same superscript indicate no significant difference (Kruskal Wallis and Mann-Whitney test).

**: All specimens were broken during specimen preparation.*

Bonding resin was then applied and light cured for 20 seconds. After the bonding treatment, four layers of resin composite (Clearfil AP-X, Kuraray, Osaka, Japan) were built up incrementally to a height of 3-5 mm and light cured for 40 seconds each. The bonded specimens were stored in water at 37°C for 24 hours and subjected to microtensile bond testing.

Microtensile Bond Test

The specimens were sectioned into slabs, approximately 0.7-mm thick, perpendicular to the bonded surface, using a low speed diamond saw under water coolant. These slabs were trimmed into an hourglass shape using a superfine-grit diamond bur (c-16ff, GC, Tokyo, Japan), ensuring the narrowest portion was located at the bonding interface. The width of the narrowest portion was approximately 1.4 mm, resulting in an interface area approximately 1 mm^2. The specimens were then attached to a Ciucchi jig (Paul & others, 1999) with a cyanoacrylate adhesive (Model Repair II Blue, Dentsply/Sankin, Otahara, Japan), and subjected to microtensile bond strength testing in a desktop material testing machine (EZ test, Shimadzu, Kyoto, Japan) at a cross-head speed of 1-mm/minute.

The bond strength was expressed in MPa derived from dividing the load at failure (in N) by the bonded area (in mm^2). The bond strength of the specimens debonded during specimen preparation was calculated as zero. The mean bond strength was statistically analyzed by Kruskal Wallis test and Mann-Whitney test at a 95% level of significance, because the Levene test for homogeneity of variance revealed significant differences between groups. In Group 2 of Single Bond, all specimens were debonded during specimen preparation. This group was eliminated from statistical analysis.

Failure Mode Analysis

After the microtensile bond test, in order to determine the failure patterns of the interface, the fractured surfaces of all specimens were examined using a light microscope (B202, OLYMPUS OPTICAL, Tokyo, Japan). We assigned the failure modes to one of the following groups: (I) cohesive failure in lased dentin and (II) mixed failure (adhesively failed between dentin and bonding resin and cohesively in resin composite)

SEM Observation of the Bonded Interface

In the same manner of preparing specimens for the microtensile bond test, four kinds of dentin surfaces (Groups 1 through 4: one tooth each) were bonded to a resin composite with one of two adhesive systems. The resin bonded specimens were sectioned into slabs, approximately 0.7-mm thick, perpendicular to the adhesive interface using a low speed diamond saw under water coolant. The slabs were embedded in epoxy resin (SpeciFix-20, Struers, Tokyo, Japan). The embedded specimens were polished with 6, 3 and 1 µm diamond pastes (DP-Paste, P, Struers, Tokyo, Japan) under water lubrication, dried for 24 hours at room temperature, sputter-coated with Au-Pd and examined with SEM (S-4000, Hitachi, Tokyo, Japan). After SEM observation, the specimens were detached from aluminum stubs and polished with 1 µm diamond paste to remove the Au-Pd coat. The specimens were then immersed in 6N HCl (Wako Pure Chemical Industries, Osaka, Japan) for 30 seconds and 1% NaOCl (Wako Pure Chemical Industries) for 10 minutes (Kameyama & others, 2000). They were ultrasonically rinsed in distilled water for 30 seconds. Finally, the specimens were again placed on an aluminum stub, sputter-coated with Au-Pd and observed by SEM.

RESULTS

Table 2 summarizes the results of the microtensile bond strength (MTBS) test.

For Single Bond, the MTBS for the control group (Group 1, 57.2 MPa) decreased to a level that could not

Table 3: *Failure Mode Analysis*

Adhesive	Failure Mode	Group			
		1	2	3	4
SIB	Cohesive failure in dentin	0 (0%)	7 (87.5%)	6 (75.0%)	0 (0%)
	Mixed failure (adhesive-cohesively in CR)	8 (100%)	1 (12.5%)	2 (25.0%)	8 (100%)
MB	Cohesive failure in dentin	0 (0%)	8 (100%)	2 (18.2%)	2 (20.0%)
	Mixed failure (adhesive-cohesively in CR)	8 (100%)	0 (0%)	9 (81.8%)	8 (80.0%)

CR = resin composite

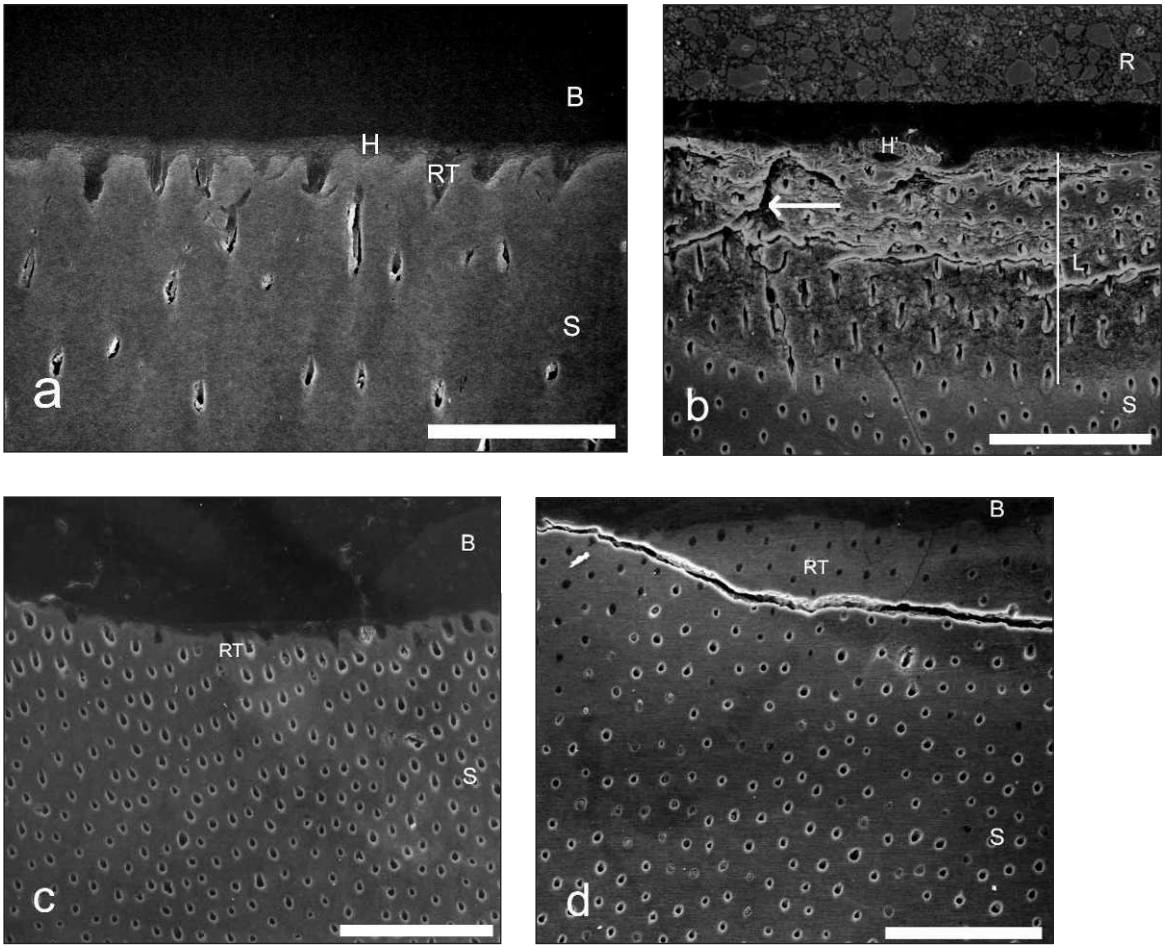

Figure 1. *FE-SEM evaluations of adhesive interface produced by Single Bond. Specimens were only polished with diamond paste. Bar = 45 µm. a. SEM image of the interface of resin and sound (non-laser-irradiated) dentin (Group 1). Hybrid layer of approximately 3-5 µm and resin-tags within dentinal tubules were clearly observed. B = Bonding resin, H = Hybrid layer, RT = Resin-tag, S = Sound dentin. b. SEM image of interface of resin and laser-irradiated dentin that was not treated further after irradiation (Group 2). Dentin structure was drastically affected by laser irradiation. Though typical hybrid layer formation and resin penetration into dentinal tubules was hardly seen, hybrid layer-like structure was observed. At the interface, gaps (white arrows) were formed. R = Resin composite, H' = Hybrid layer-like structure, L and narrow white bar = Laser affected dentin zone, S = Sound dentin. c. SEM image of interface of resin and laser-irradiated dentin that was treated with $NaHCO_3$ powder abrasion to remove the carbonized dentin visually (Group 3). Carbonized dentin was not observed. Hybrid layer was not clearly seen, and resin-tag formation occurred to some extent. B = Bonding resin, RT = Resin-tag, S = Sound dentin. d. SEM image of interface of resin and laser-irradiated dentin that was treated with SiC paper to remove the carbonized dentin visually (Group 4). The interface was similar to that of Group 3. Crack was observed that was thought to be an artifact because of specimen preparation procedure, the dehydration procedure and/or high vacuum. B = Bonding resin, RT = Resin-tag, S = Sound dentin.*

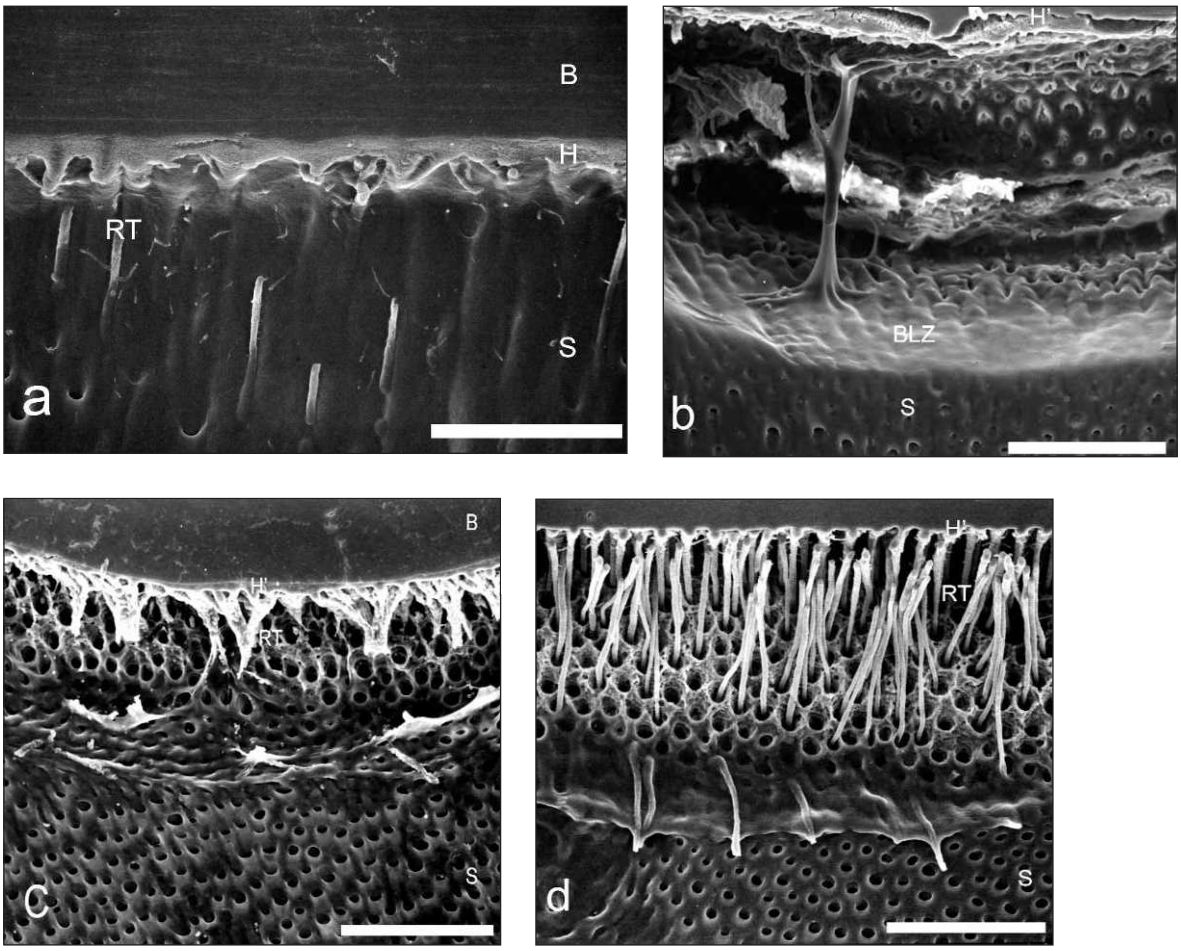

Figure 2. *FE-SEM evaluations of adhesive interface produced by Single Bond. Specimens were treated with NaOCl and HCl after polishing with diamond paste. Bar = 45 μm.*
a. SEM image of the interface of resin and sound (non-laser-irradiated) dentin (Group 1). Hybrid layer of approximately 3-5 μm and resin-tags penetrating deeply into dentinal tubules were clearly observed. B = Bonding resin, H = Hybrid layer, RT = Resin-tag, S = Sound dentin. b. SEM image of interface of resin and laser-irradiated dentin that was not treated further after irradiation (Group 2). Dentin structure was drastically affected by laser irradiation. Hybrid layer-like structure of a few microns width was observed, but resin penetration into dentinal tubules was hardly seen. Chemical treatment dissolved the lased dentin, resulting in a wide crater-like gap of 100 μm width. H' = Hybrid layer-like structure; BLZ = Bottom of laser affected dentin zone; S = Sound dentin. c. SEM image of interface of resin and laser-irradiated dentin that was treated with $NaHCO_3$ powder abrasion (Group 3). Hybrid layer-like structure and shallow resin-tags were exposed. There exists the laser-affected dentin zone right above sound dentin. B = Bonding resin, H' = Hybrid layer-like structure, RT = Resin-tag, S = Sound dentin. d. SEM image of interface of resin and laser-irradiated dentin that was treated with SiC paper (Group 4). Typical hybrid layer was not seen, but longer and more resin-tags were exposed as compared to Group 3. H' = Hybrid layer-like structure, RT = Resin-tag, S = Sound dentin.

be measured after laser irradiation was performed to the dentin surface (Group 2), because all specimens in Group 2 were broken during specimen preparation. When the carbonized surface dentin was removed with $NaHCO_3$ powders prior to bonding (Group 3), MTBS was 12.8 MPa, which was significantly lower than the control value, though half of the specimens in Group 3 failed prior to testing. When SiC paper polishing was used to remove the carbonized dentin (Group 4), MTBS (50.0 MPa) was not significantly different from the control. For Mega Bond, laser irradiation significantly decreased MTBS from 41.4 MPa in the control (Group 1) to 4.8 MPa in Group 2, having five pre-testing failures out of eight specimens. Groups 3 and 4 showed similar MTBS values (23.5 and 25.7 MPa, respectively), which were not significantly different from the control.

Table 3 shows the failure mode analysis of the fractured specimens after the microtensile bond test. For both adhesives in Groups 1 and 4, almost all fractured specimens showed mixed failures that were broken in bonding resin and resin composite. In Group 2, cohesive failures in the lased dentin were predominant for both adhesives. In Group 3, Single Bond mainly showed cohesive failures in lased dentin, whereas, failures occurred mostly adhesive-cohesively in Mega Bond specimens.

SEM observation of the interface of resin and sound dentin revealed typical morphology, with the hybrid

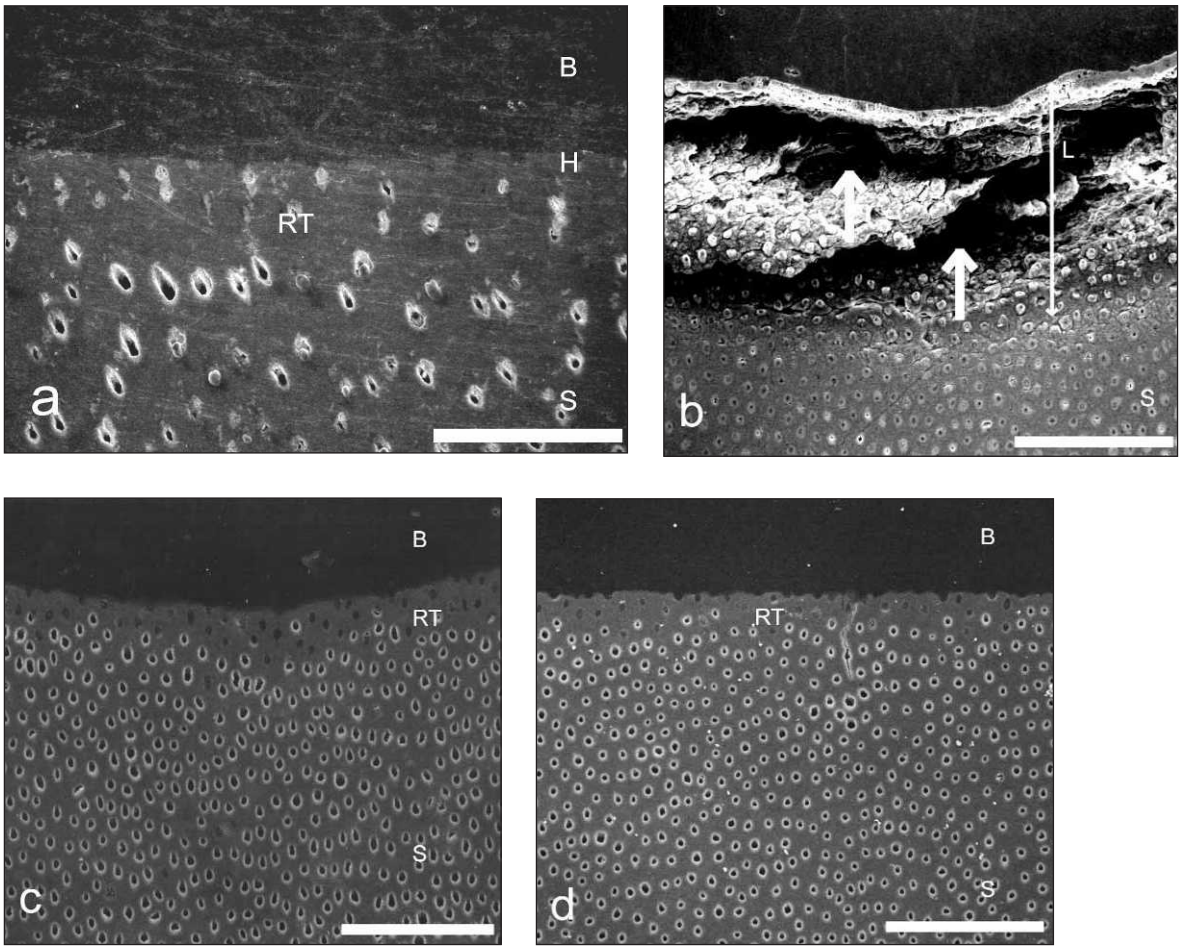

Figure 3. *FE-SEM evaluations of adhesive interface produced by Mega Bond. Specimens were only polished with diamond paste. Bar = 45 μm. a. SEM image of the interface of resin and sound (non-laser-irradiated) dentin (Group 1). Hybrid layer of approximately 1 μm and resin-tags within dentinal tubules were observed, but were less pronounced as compared to Single Bond. B = Bonding resin, H = Hybrid layer, RT = Resin-tag, S = Sound dentin. b. SEM image of interface of resin and laser-irradiated dentin that was not treated further after irradiation (Group 2). Dentin structure was drastically affected by laser irradiation. Though typical hybrid layer formation and resin penetration into dentinal tubules was hardly seen, hybrid layer-like structure was observed, as similar to Single Bond. At the interface, gaps (wide white arrows) were formed. R = Resin composite, H' = Hybrid layer-like structure, L and narrow white arrow = Laser affected dentin zone, S = Sound dentin. c. SEM image of interface of resin and laser-irradiated dentin that was treated with $NaHCO_3$ powder abrasion (Group 3). Carbonized dentin was not observed. Hybrid layer was not clearly seen, and resin-tag formation occurred to some extent. B = Bonding resin, RT = Resin-tag, S = Sound dentin. d. SEM image of interface of resin and laser-irradiated dentin that was treated with SiC paper (Group 4). The interface was similar to that of Group 3. B = Bonding resin, RT = Resin-tag, S = Sound dentin.*

layer and resin-tag formation within dentinal tubules, although the width of the hybrid layer varied in each adhesive (Figures 1a and 3a). After hyper-dissolution treatment, similar morphology was observed with the hybrid layer and resin-tag formation within dentinal tubules (Figures 2a and 4a).

For Single Bond, laser irradiation (Group 2) drastically affected the dentin surface morphology, and resin penetration into the underlying dentinal tubules was hardly observed (Figure 1b). Though the typical hybrid layer was not formed along the interface in Group 2, the hybrid-like structure (H') was observed, which was more clearly observed by hyper-dissolution treatment (Figure 2b). Also, hyper-dissolution treatment easily dissolved the lased dentin, resulting in a wide gap of 100 μm (Figure 2b). In Group 3, $NaHCO_3$ powder removed the carbonized dentin, the typical hybrid layer was not distinguished and resin-tag formation was seen to some extent (Figure 1c). The hyper-dissolution treatment exposed the hybrid layer-like structure and shallow resin-tags and showed the existence of a laser-affected dentin zone above sound dentin (Figure 2c). Group 4 (Figure 1d) exhibited a similar image as Group 3, except for longer resin-tag formation (Figure 2d).

For Mega Bond, representative images are shown in Figures 3 and 4. Each group showed similar interface morphology as the corresponding group of Single Bond, except that the width of the hybrid layer-like structure and length of the resin-tags were less than those seen in the Single Bond.

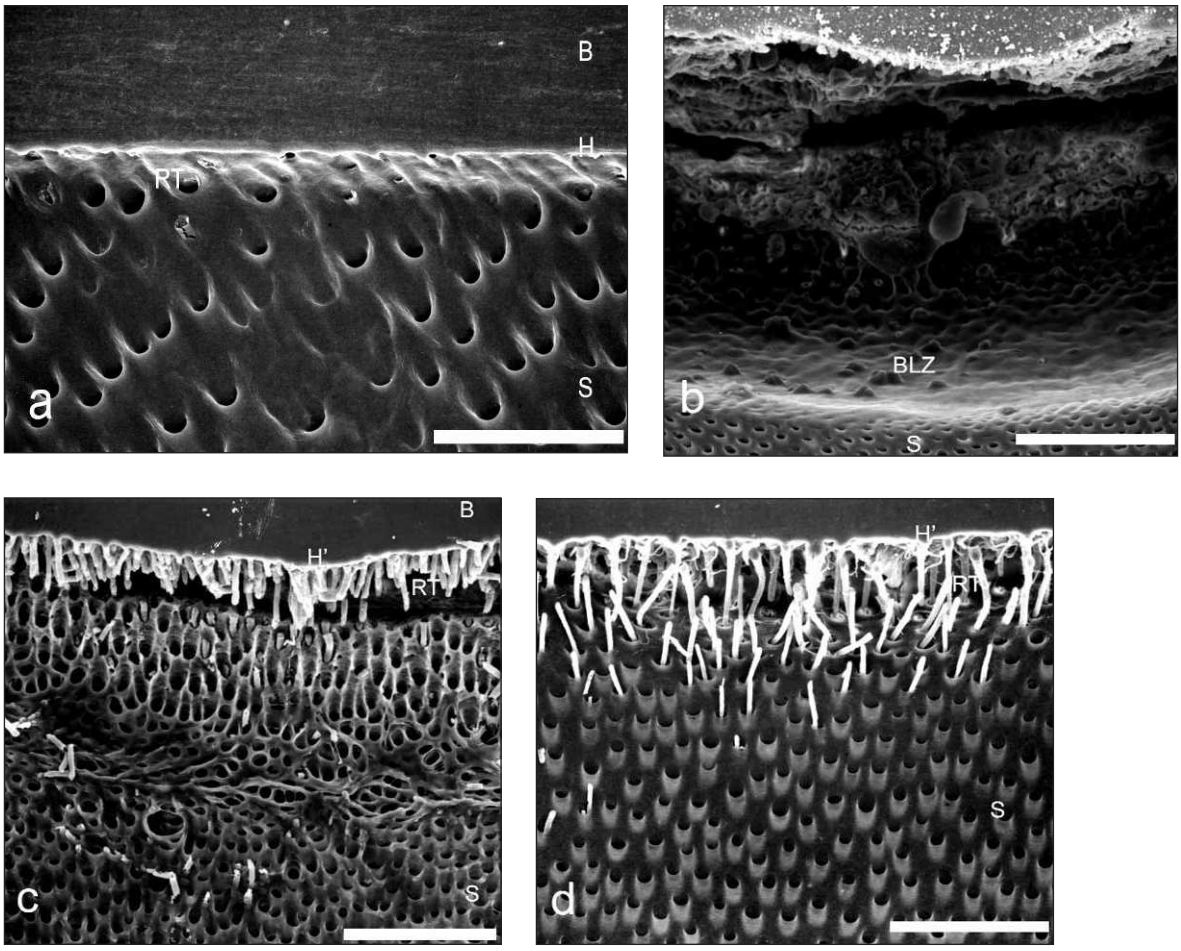

Figure 4. *FE-SEM evaluations of adhesive interface produced by Mega Bond. Specimens were treated with NaOCl and HCl after polishing with diamond paste. Bar = 45 μm.*
a. SEM image of the interface of resin and sound (non-laser-irradiated) dentin (Group 1). Hybrid layer of approximately 1 μm and resin-tags penetrating into dentinal tubules were clearly observed. B = Bonding resin, H = Hybrid layer, RT = Resin-tag, S = Sound dentin. b. SEM image of interface of resin and laser-irradiated dentin that was not treated further after irradiation (Group 2). Dentin structure was drastically affected by laser irradiation. Resin penetration into dentinal tubules was hardly seen. HCl and NaOCl treatment dissolved the lased dentin, exposing wide crater-like gap of 100 μm width. BLZ = Bottom of laser affected dentin zone; S = Sound dentin. c. SEM image of interface of resin and laser-irradiated dentin that was treated with $NaHCO_3$ powder abrasion (Group 3). Thin hybrid layer-like structure and short resin-tags were exposed. There exists the laser-affected dentin zone right above sound dentin. B = Bonding resin, H' = Hybrid layer-like structure, RT = Resin-tag, S = Sound dentin. d. SEM image of interface of resin and laser-irradiated dentin that was treated with SiC paper (Group 4). Typical hybrid layer was not seen, but longer and more resin-tags were exposed as compared to Group 3. H' = Hybrid layer-like structure, RT = Resin-tag, S = Sound dentin.

DISCUSSION

There are several advantages to bonding resin composite to laser-irradiated dentin (Goldman & others, 1965; Stern, 1974; Featherstone & others, 1998). Thus, it was thought that the lased dentin surface might be more desirable for bonding resinous restorative materials (Cooper & others, 1988). When dentin is treated by laser, localized melting and re-crystallization produce fungi-form projections on the dentin surface (Nammour & others, 1992; Hossain & others, 1999; Le Goff & others, 1999). These changes are thought to be induced by thermo-mechanical abrasion. The irradiated tooth surface spot is immediately heated to a temperature over 1000°C (Holcomb & Young, 1980) and carbonized to black. From SEM observations of the lased dentin surface in this study, spherical structures of unknown origin were observed on the area of intertubular dentin at the bottom of the impacted spot (Figures 5a and 5b). This may be due to the presence of the smear layer dehydrated by heat generated on the surface by absorbed CO_2 laser energy. The carbonized area was surrounded by a dehydrated smear layer resistant to acid attack. It appears that laser irradiation, depending on the energy density used, leaves a carbonized surface on which either an acid resistant smear layer or melted dentin is found. These phenomena may become the total or partial obstruction of micromechanical attachment with adhesive systems, mini-

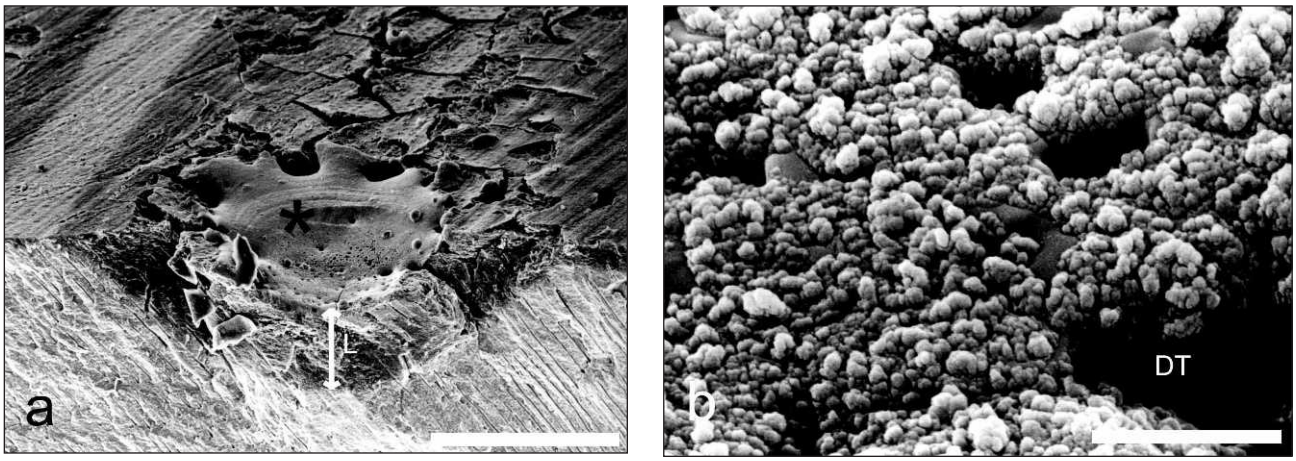

Figure 5. *FE-SEM photomicrographs of dentin irradiated with CO_2 laser.*
a. Vertical and horizontal view of a laser impacted spot (asterisk). Bottom half of the image represents the dentin surface to be exposed by the laser irradiation. The depth of the spot was 100 µm (white arrows and L). Cracks (white arrows) that started from the dentinal tubules in the impacted spot were observed. Bar = 180 µm b. High magnification of the center of laser impact zone (Figure 5a). Some dentinal tubules (DT) were observed. At the area of inter-tubular dentin, spherical structures of unknown origin were deposited. Bar = 1.2 µm.
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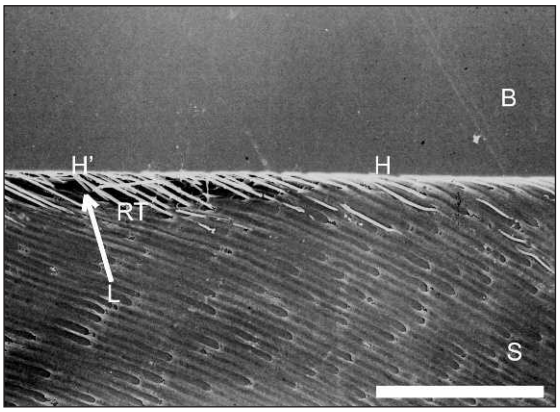

Figure 6. *SEM image of interface of resin and laser-irradiated dentin that was treated with SiC paper (Group 4), that is, another region of Figure 4d. Interface of the left side of this image represented the similar morphology as Figure 4d, whereas that of the right side showed the typical hybrid layer and resin-tags formation between resin and sound dentin as in Figure 3a. H = Hybrid layer, H' = Hybrid layer-like structure, RT = Resin-tag, S = Sound dentin. L = Laser affected dentin; S = Sound dentin; B = Bonding resin, Bar = 90 µm.*

mizing possible formation of the hybrid layer (Silberman & others, 1994) because the lased dentin surface is resistant to acid-etching, which is essential for the hybrid layer formation and, therefore, making it difficult for the bonding agent to penetrate into dentin. Thus, in this study, bond strength to various dentin surfaces with and without the carbonized layer after irradiation was evaluated.

In addition, we observed the resin/dentin interface after hyper-dissolution treatment of the dentin structure by HCl and NaOCl, because there is a report that heated dentin was easier to dissolve in NaOCl solution than non-heated dentin (Tonami & others, 1999). The tertiary structure of the heated dentin may have been changed through laser irradiation. The heated dentin that remained at the interface would then be of clinical importance, because it might be easily influenced by various stresses in the oral environment through nanoleakage (Sano & others, 1995), such as water and enzymes, resulting in interfacial degradation after a long period.

According to the results of this study, the control group showed relatively high bond strength (Table 2) and typical morphology of the resin/dentin interface for both adhesives, as reported previously (Inoue & others, 2001) (Figures 1a, 2a, 3a and 4a). Once the dentin surface is treated by CO_2 laser (Group 2), MTBS significantly decreased in specimens, irrespective of the adhesives used. Especially for the specimen bonded with Single Bond, all specimens failed prior to bond strength testing and thus the true MTBS were not able to be measured, undoubtedly indicating extremely low bond strength. From the cross-sectional SEM images of the interface (Figures 1b and 2b), resin penetration into the underlying dentinal tubules was hardly observed in both systems. Though the typical hybrid layer was not formed along the interface in Group 2, a hybrid-like structure (H') was observed (Figures 1b, 2b, 3b and 4b). It seemed that the acid-resistant carbonized layer was not etched enough and became an obstacle for resin penetration into the lased dentin. Hyper-dissolution treatment dissolved the lased dentin, resulting in a wide gap (Figures 2b and 4b). The width of the gap was approximately 100 µm. Since no laser energy is thought to penetrate beyond 100 µm, this treatment might have removed the lased dentin, thus producing the wide gap.

The fracture mode analysis showed that fractures mostly occurred in lased dentin in both adhesive systems (Table 3). It seemed that dehydration due to laser irradiation made the dentin brittle enough to fail cohesively in the dentin of the laser-impacted zone. The laser-induced difference of the interfacial morphology between a single bottle and a self etching-priming adhesive system was not clarified within the limits of this study.

In order to remove the carbonized layer, either $NaHCO_3$ powder or SiC paper was used in this study. $NaHCO_3$ powder is often used clinically with pressurized air clinically as a part of the air-abrasion system, to remove the exogenous deposition of tarnish. Its particle size is approximately 50x50x100 μm (manufacturer's information). This powder could visually remove the black carbonized layer on the lased dentin surface. SiC paper is not used in daily clinic but is widely used in *in vitro* experiments to create a standard smear layer (Pashley & others, 1995), in other words, to simulate the clinical situation of caries removal by rotary instrument. In this study, SiC paper removed the carbonized layer visually and thus might produce a certain amount of the smear layer.

For Single Bond, when $NaHCO_3$ powder was used (Group 3), MTBS increased compared to Group 2, though it was significantly different from Group 1. According to the SEM image (Figure 1c), the carbonized dentin that was removed was seen in the interface and seemed to serve as the barrier against resin penetration. Figure 2c, the SEM image of the interface after hyper-dissolution treatment, confirmed the removal of the carbonized layer by powder abrasion, because resin tag formation was observed, although the length of the resin tags were shorter than expected. Figure 2c also showed a wide gap, such as a crater. This was caused by the hyper-dissolution effect to laser-affected dentin. Failure mode analysis showed that, in the case of this group, breakage of the specimens mostly occurred cohesively in dentin. This also means that laser-affected dentin still remained and may negatively influence bond strength. Next, when SiC paper was used to remove the carbonized layer, MTBS increased to a value similar to Group 1. The carbonized dentin was also removed (Figure 1d) and resin tag formation was clearly observed (Figure 2d). Aggressive acid treatment using a phosphoric acid prior to bonding might remove the smear layer and smear plug produced by the use of SiC paper, resulting in high bond strength. Another possible reason for high bond strength is the sound dentin left unlased in the resin/dentin interface. Laser impacts dentin spot-by-spot (Figure 5a) and no laser energy is thought to penetrate beyond 100 μm. When removing the carbonized dentin layer by SiC paper, non-irradiated sound dentin may be exposed in the surface to be bonded. This kind of dentin may lead to producing the typical hybrid layer formation that is thought to be essential for good bonding (Figure 6) (Nakabayashi & Pashley, 1998). Although the ratio of the lased and sound dentin exposed is unknown, the high values of standard deviation in bond strength data of Groups 3 and 4 may imply the possibility of the existence of sound dentin left unlased in the resin/dentin interface.

For Mega Bond, the mean MTBS in Group 3 tended to be lower than that in Group 1, but there was no significant difference between them. The SEM image (Figure 3c) showed almost the same morphology as Figure 3a, indicating that the carbonized dentin was removed. After hyper-dissolution treatment (Figure 4c), resin-tag formation was relatively obvious, but its length was also shorter than expected. The hybrid-like layer (H') produced by Mega Bond was thinner than Single Bond. This might be due to the difference in acidity of the primer solution. In the case of Single Bond, the strong acid of a phosphoric acid was used, whereas, in the case of Mega Bond, the self-etching primer, of which acidity was mild, was used to decalcify the dentin surface (Inoue & others, 2000, 2001; Van Meerbeek & others, 2001). Failure mode analysis showed that most fractures occurred adhesive-cohesively in bonding and/or resin composite. Thus, laser affected dentin might be modified by Mega Bond, resulting in relatively high bond strength. In addition, because the functional monomer in Mega Bond, namely MDP, is reported to bond chemically with calcium from hydroxyapatite that remained around the collagen fibrils (Yoshida & others, 2003), the possibility of a chemical bond may be one of the reasons for the high bond strength. When SiC paper was used to remove the carbonized layer, the MTBS of Group 4 of Mega Bond was similar to Group 3, which was not significantly different from Group 1 (Table 2). The SEM image of the interface of Group 4 (Figure 3d) showed the same morphology as Group 3 (Figure 3c) and the control (Figure 3a). Failure mode analysis showed that breakage of the specimens mostly occurred adhesive-cohesively in bonding resin and/or resin composite. This also means that laser-affected dentin might be modified by Mega Bond.

In this study, we used the hyper-dissolution method to observe the resin/dentin interface. For both adhesives, this method revealed that laser-affected dentin was easily dissolved by NaOCl and HCl and resulted in a crater-like wide gap, although the sound dentin was hardly dissolved. This may be due to the fact that the heated dentin was easier to dissolve in NaOCl solution than non-heated dentin (Tonami & others, 1999).

Further study is needed to determine a better method for laser irradiation, when the CO_2 laser is used on dentin in clinical situations.

CONCLUSIONS

From the results of this study, it is concluded that bonding to laser-irradiated dentin is different from sound dentin. In the presence of carbonized dentin, MTBS drastically decreased but recovered to the control value by removing the carbonized dentin layer visually with SiC paper for both a single bottle and a self-etching adhesive system. However, the laser-affected dentin that remained on the bonded interface was easily dissolved with NaOCl and HCl.

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Effect of Prophylaxis Regimens on Surface Roughness of Glass Ionomer Cements

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S Chelvan • ESF Tan

Clinical Relevance

The effect of prophylaxis regimens on glass ionomer cements was material dependent. Glass ionomer restorations may require re-polishing after exposure to some prophylaxis regimens.

SUMMARY

This study investigated the surface roughness of conventional (Fuji II Capsulated [FC], GC Corporation, Tokyo, Japan), resin-modified (Fuji II LC [FL], GC Corporation) [FL] and highly viscous (Fuji IX GP Fast [FN], GC Corporation) glass ionomer cements [GICs] after exposure to five prophylaxis regimes. The surface roughness obtained was compared to untreated polished specimens (control). The prophylaxis regimes evaluated were rotating brush with pumice-water slurry [PB]; rotating rubber cup with pumice-water slurry [PC]; rotating rubber cup with prophylaxis paste [PP]; rotating rubber cup with prophylaxis gel [PG] and air-powder polishing [PJ]. Forty-eight specimens (3-mm long x 3-mm wide x 2-mm deep) were made for each material. The specimens were stored in distilled water at 37°C for one month, polished with 1200 grit sandpaper using a lapping device and randomly divided into six groups (n=8). They were then stored for an additional two months in distilled water at 37°C prior to exposure to the various prophylaxis regimens. The mean surface roughness value (Ra; µm) was measured with a profilometer. Data was subjected to ANOVA/Scheffe's tests at significance level 0.05. Mean Ra ranged from 0.30 to 1.70 µm for FC, 0.40 to 2.52 µm for FL and 0.36 to 1.79 µm for FN. Regardless of the type of glass ionomer, treatment with PJ resulted in significantly rougher surfaces when compared to the control group. For FC and FN, a significant increase in roughness was observed after treatment with PB and PP, respectively. Glass ionomer restorations may require re-polishing after exposure to some prophylaxis regimens.

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INTRODUCTION

The importance of smooth surfaces to the success of dental restorations has been well-documented (Fruits & others, 1996; Goldstein, 1989). The esthetics and longevity of tooth-colored restorative materials is heavily dependent on the quality of surface finish, as the presence of irregularities may influence appearance, surface discoloration, plaque retention and gingival irritation (Shintani & others, 1985; Dunkin & Chambers, 1983; Chan, Fuller & Hormati, 1980; Weitman & Eames, 1975; Larato, 1972). In addition, restorations are more easily maintained when they are smooth (Strassler & Bauman, 1993; Weitman & Eames, 1975). Bollen, Lambrechts and Quirynen (1997) reported a critical threshold surface roughness for bacteria adhesion of 0.2 µm. While no further reduction in bacteria accumulation is expected below this threshold value, any increase in surface roughness above 0.2 µm results in a simultaneous increase in plaque accumulation and increases the risk for caries and periodontal inflammation (Bollen & others, 1997).

Glass ionomer cements were first introduced to the dental profession in the early 1970s (Wilson & Kent, 1972). Their favorable adhesive and fluoride-releasing properties have led to their widespread use as luting, lining and restorative materials (Sidhu & Watson, 1995). Disadvantages of conventional glass ionomers include early moisture sensitivity, low initial mechanical properties and inferior translucency compared to resin composites. Resin-modified materials that combine glass ionomer and resin composite technology were subsequently developed to help overcome some of these problems. Highly viscous glass ionomers, which are essentially high powder:liquid ratio cements, were recently introduced as an alternative to amalgam for posterior preventive restorations. Conventional, resin-modified and highly viscous glass ionomer cements are commonly used for restoring cervical cavities. As heavy plaque depositions and stains are common near gingival tissues, cervical restorations are inevitably exposed to prophylaxis procedures during maintenance therapy.

Different types of prophylaxis regimes are available. These procedures are usually performed using a variety of prophylaxis agents with varying abrasiveness and rotary rubber cups or brushes as carriers. Air-powered devices have also been introduced into clinical practice. With these devices, sodium bicarbonate particles are propelled by air jet and combined with a small stream of water, creating a slurry that is directed onto the tooth surface (Reel & others, 1989). Although the effects of prophylaxis procedures on composite restoratives have been relatively well studied (Warren & others, 2002; Carr & others, 2002), their effects on glass ionomer cements have not been widely investigated. The objective of this study was to investigate the effect of five different prophylaxis regimes on the surface roughness of conventional, resin-modified and highly viscous glass ionomer cements.

METHODS AND MATERIALS

Table 1 shows the technical profiles of the glass ionomer cements investigated. All the cements were supplied in capsulated form and activated/mixed according to the manufacturer's instructions. The cements were injected into the square recesses (3-mm long x 3-mm wide x 2-mm deep) of customized acrylic molds and covered with matrix strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide was then placed over them and pressure applied to extrude excess material. The resin-modified cement was light polymerized for 20 seconds, while the conventional and highly viscous cement was allowed to set for 10 minutes. Light curing was done using a commercial light-curing unit (Polylux II; Kavo Dental, Warthausen, Germany) with an output of 500mW/cm^2. Fuji Coat (GC Corporation, Tokyo, Japan) was subsequently applied on all specimens and light cured for 10 seconds. Forty-eight specimens of each material were made and stored in distilled water at 37 ± 1°C for one month. The specimens were then subjected to polishing with ANSI (American National

Table 1: *The Glass Ionomer Cements Investiged*

Material	Manufacturer	Components	Mean Particle Size (µm)
Fuji II LC (Lot #0201245) Resin-modified	GC Corporation, Tokyo, Japan	*Powder:* Alumino silicate glass, pigments *Liquid:* Polyacrylic acid, distilled water, HEMA (17%), dimethacrylate monomer, camphoroquinone	4.5
GC Fuji IX GP Fast (Lot #0109083) Highly viscous	GC Corporation, Tokyo, Japan	*Powder:* Alumino silicate glass, pigments *Liquid:* Polyacrylic acid, distilled water	7.0
GC Fuji II Capsulated (Lot #0012277) Conventional	GC Corporation, Tokyo, Japan	*Powder:* Alumino silicate glass, pigments *Liquid:* Polyacrylic acid, distilled water	4.5

Standards Institute) 1200-grit sandpaper (Carbimet disks; Wirtz-Buehler, Dosseldorf, Germany) at 2,000 rpm for four minutes using a lapping device (Phoenix Beta; Wirtz-Buehler, Dusseldorf, Germany). This sandpaper grit had a similar Ra value (0.23 µm) as fine Sof-Lex contouring and polishing disks (3M Dental Products, St Paul, MN, USA). The specimens were randomly divided into six groups of eight and stored in distilled water at 37°C ± 1°C for an additional two months prior to treatment with the following prophylaxis regimes: Group 1—rotating brush with pumice-water slurry (Casone, Noceto-parma, Italy) [PB]; Group 2—rotating rubber cup with pumice-water slurry (Casone, Noceto-parma, Italy) [PC]; Group 3—rotating rubber cup with prophylaxis paste (Zircon F; Henry Schein, Middlesex, UK) [PP]; Group 4—rotating rubber cup with prophylaxis gel (PTC Paste Regular & Fine, GC Corporation, Tokyo, Japan) [PG] and Group 5—air-powder polishing (AirFlow S1; EMS, Nyon, Switzerland) [PJ]. Group 6, which was not exposed to any prophylaxis treatment, served as the control group.

For the PJ group, the test specimens were subjected to 12 seconds of air polishing, with the jet tip located 10 mm away from the center of each specimen. For the PG group, regular paste was used for the first six seconds and fine paste for the subsequent six seconds. Prophylaxis agents were replaced every six seconds for the remaining groups. All prophylaxis regimes were carried out using a contra-angle slow speed handpiece at a speed of 2,000 rpm. New rotary brushes and cups were used for each material-treatment combination. To minimize the effect of operator variability, all prophylaxis regimes were conducted by one operator. Mean surface roughness (µm) of the specimens after exposure to the various prophylaxis regimens was measured using a profilometer (Surftest SV-400; Mitutoyo, Kanagawa, Japan). Readings were taken at the center of each specimen and four sampling lengths of 0.25 mm were used. All statistical analysis was carried out at significance level 0.05. Two-way ANOVA was used to determine significant interactions between materials and prophylaxis regimes. One-way ANOVA and Scheffe's post-hoc tests were used to compare the surface roughness associated with the different prophylaxis regimes and as inter-material comparison.

RESULTS

Table 2 shows the mean surface roughness observed with the different prophylaxis regimes. Results of statistical analysis are reflected in Tables 3 and 4. Figure 1 shows the mean surface roughness of the materials with reference to the critical threshold surface roughness for bacterial adhesion (0.2 µm).

Mean Ra ranged from 0.30 to 1.70 µm for FC, 0.40 to 2.52 µm for FL and 0.36 to 1.79 µm for FN. For all materials, the roughest surfaces were observed with the use of PJ. Two-way ANOVA revealed significant interaction between materials and prophylaxis regimes. The effect of prophylaxis regimen on surface roughness was therefore material dependent. Regardless of the type of glass ionomer, treatment with PJ resulted in significantly rougher surfaces when compared to the control group. For FC and FN, a significant increase in roughness was also observed after treatment with PB and PP, respectively. With the exception of PJ, FL was not significantly affected by exposure to any of the prophylaxis regimes.

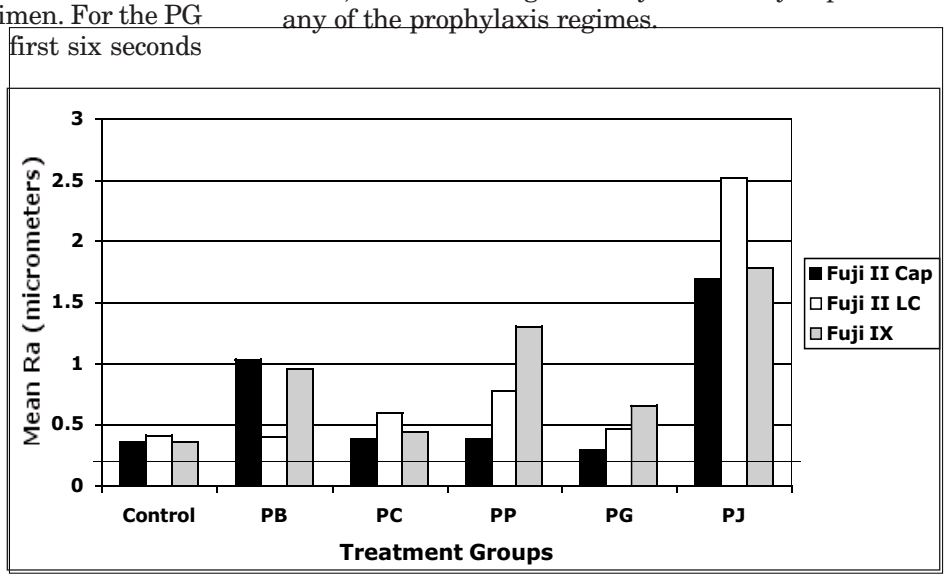

Figure 1. *Mean surface roughness of the materials with reference to the critical threshold surface roughness (0.2 µm) for bacterial adhesion.*

Table 2: *Mean Surface Roughness Observed with the Different Prophylaxis Regimes*

Treatment Groups	Fuji II Capsulated [FC]	Fuji II LC [FL]	Fuji IX GP Fast [FN]
Group 1 (PB)	1.03 (0.11)	0.40 (0.09)	0.96 (0.34)
Group 2 (PC)	0.39 (0.13)	0.59 (0.22)	0.44 (0.17)
Group 3 (PP)	0.39 (0.04)	0.78 (0.33)	1.31 (0.39)
Group 4 (PG)	0.30 (0.05)	0.47 (0.09)	0.66 (0.22)
Group 5 (PJ)	1.70 (0.08)	2.52 (0.70)	1.79 (0.81)
Group 6 (Control)	0.36 (0.09)	0.41 (0.10)	0.36 (0.12)

Standard deviations in parentheses.

Table 3: *Comparison of Mean Surface Roughness Among Different Prophylaxis Regimes*

Materials	Differences
Fuji II Capsulated [FC]	PJ > PB > PC & PP, control, PG
Fuji II LC [FL]	PJ > PP, PC, PG, control, PB
Fuji IX GP Fast [FN]	PJ > PB, PG, PC, control PP > PC, control

> denotes statistically significant difference. Results of one-way ANOVA/Scheffe's test (p<0.05).

Table 4: *Comparison of Ra Values Among Materials*

Treatment Groups	Differences
Group 1 (PB)	FC, FN > FL
Group 2 (PC)	No significant differences
Group 3 (PP)	FN > FL > FC
Group 4 (PG)	FN > FC
Group 5 (PJ)	No significant differences
Group 6 (Control)	No significant differences

> denotes statistically significant difference. Results of one-way ANOVA/Scheffe's test (p<0.05).

No significant difference in surface roughness was observed among the three glass ionomer materials for the PJ, PC and control groups. For the PB group, both FC and FN were significantly rougher than FL but no significant difference in Ra values was noted between FC and FN. With the PP and PG groups, FN was significantly rougher than FC.

DISCUSSION

The materials were made and stored for one month to allow the acid base reaction to mature (Yap & others, 2002; Eliades, Kakaboura & Palaghias, 1998). Although restoratives cured against a matrix represent the smoothest surface possible (Yap, Lye & Sau, 1997), some degree of finishing and polishing is often required clinically. The control group therefore simulated glass ionomer restorations finished with a fine Sof-Lex contouring and polishing disk. A lapping device was utilized instead of abrasive disks mounted on slow speed handpieces, as this approach was less operator dependent and resulted in a more consistent surface finish. A total aging period of three months was necessary to replicate a practical recall period employed for maintenance therapy. Efforts were also made to standardize the prophylaxis procedures, which were designed according to common clinical practices in terms of contact duration, handpiece speed and change of prophylaxis agents.

Glass ionomers are heterogeneous and biphasic in nature. The set material consists of unreacted glass particles embedded in a polysalt/resin matrix. During abrasive procedures (finishing, polishing and prophylaxis), each particle of abrasive acts as a fine tool, cutting a groove in the surface of the dental restoration. The softer matrix phases of the cements are preferentially removed, leaving the harder, unreacted glass particles protruding from the surface. This accounts for the significant increase in Ra values observed after most prophylaxis treatments. The effect of prophylaxis procedures was found to be material dependent. All glass ionomer materials were significantly roughened by air-powder polishing. This was in agreement with previous studies (Reel & others, 1989; Gutmann, Marker & Gutmann, 1993). Resin-modified glass ionomer cement (FL) was not significantly affected by all prophylaxis regimes, with the exception of air polishing. This may be due to the fact that resin incorporation results in the improvement of physical properties, such as abrasion and wear resistance (Shabanian & Richards, 2002).

Conventional glass ionomer cement (FC) was significantly roughened by pumice water slurry with brush. This could be attributed to the abrasive nature of rotary brushes. Highly viscous glass ionomer cement (FN) was not as affected due its better wear resistance. The latter may contribute in part to the use of significantly larger glass particles (Yap, Pek & Cheang, 2003). FN was, however, significantly roughened by prophylaxis paste with cup. The fine zirconium particles present in prophylaxis paste may abrade the glass ionomer matrix and dislodge the large glass particles. The use of prophylaxis gel or pumice slurry with cup is recommended for the removal of plaque and stains near glass ionomer cements, as these prophylaxis regimes did not significantly roughen the surface of the glass ionomer cements. Fine PTC gel may also provide a polishing effect on glass ionomer materials. Glass ionomer restoratives may require re-polishing after treatment with some prophylaxis regimes. This is especially critical as glass ionomers are inherently rough and exceed the critical threshold surface roughness for bacteria adhesion after finishing and polishing procedures.

CONCLUSIONS

Within the limitations of this *in vitro* study, we concluded that:

1. The effect of prophylaxis regimes on the surface roughness of glass ionomer cements was material dependent.
2. Conventional glass ionomer cement (Fuji II Cap) was significantly roughened by the use of air polishing and pumice with brush.
3. Resin-modified glass ionomer cement (Fuji II LC) was not significantly affected by all prophylaxis regimes, with the exception of air polishing.
4. Highly viscous glass ionomer cement (Fuji IX) was significantly roughened by the use of air polishing and prophylaxis paste with cup.

5. The use of prophylaxis gel or pumice slurry with cup is recommended for the removal of plaque and stains near glass ionomer cements.

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Curing Depth of a Resin-modified Glass Ionomer and Two Resin-based Luting Agents

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Clinical Relevance

Chemical cure of dual-cure luting agents was unable to provide a high degree of conversion for the dual-cured composites.

SUMMARY

The degree of conversion of resin-based luting agents used for retention of prefabricated posts has been questioned due to the difficulty of light penetration into the resin-filled root canal. This study evaluated the depth of cure of a resin-modified glass ionomer cement (Rely X–3M ESPE) and two resin-based luting agents (Rely X ARC—3M ESPE and Enforce–Dentsply). Twenty-four 14x2x2mm³ specimens were prepared in a Teflon split mold with the three luting agents (n=8). After preparation, the specimens were stored at 37°C in a dark box for 24 hours prior to microhardness testing. Measurements of Knoop hardness were performed at three different depths: superficial, medium and deep thirds. The results (KHN) were statistically analyzed by repeated measures ANOVA and Tukey test (0.05), which showed that resin-based luting agents presented the highest Knoop hardness values within the superficial third. Within the medium third, there were no significant differences among luting materials. However, within the deep third, Rely X presented the highest values. KHN values of resin-based luting agents decreased remarkably as depth increased.

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INTRODUCTION

In an attempt to overcome the inherent problems of zinc phosphate cements and provide better handling and esthetic properties, resin-based luting agents were introduced and indicated for retention of indirect restorations and posts (Mendoza & Eakle, 1994). The use of dentin bonding agents in association with resin-based materials can provide reinforcement of the remaining tooth structure (Saupe, Gluskin & Radke, 1996; Morgano & Brackett, 1999). Moreover, the composite layer can also provide a buffer zone that con-

tributes to uniform stress distribution between the post and canal wall during function in the oral environment due to its favorable mechanical properties (Nash, 1998).

According to their mode of polymerization, resin-based luting agents can be classified into self-cured, light-cured and dual-cured. The use of dual-cured luting composites in combination with adhesive systems has been proposed for cementation of prefabricated posts (Cohen & others, 2000; Hofmann & others, 2001; Ferrari, Vichi & Grandini, 2001). These materials polymerize chemically upon the mixing of a base and catalyst components (self-curing) and also when subjected to light from a light-curing unit. During post fixation, exposed marginal areas can benefit largely from both self- and light-curing, as they are readily accessible to the curing light. However, as light is irradiated, a significant reduction in intensity occurs due to light scattering within the luting composite and shadowing that might be produced by both post and tooth structure. In this way, the portions which are not directly irradiated by light rely extensively on the self-curing component of the polymerization system (Rueggeberg & Caughman, 1993; El-Mowafy, Rubo & El-Badrawy, 1999).

Thus, the degree of cure and depth of polymerization of resin-based luting agents has been questioned due to the difficulty of light penetration through the canal and the inability of their chemical cure to compensate for the absence of visible light activation (El-Mowafy & others, 1999). This study tested the null hypothesis that the degree of cure of two resin-based and a resin-modified glass ionomer luting agent used for the retention of posts would not be altered within the extension of the root canal by means of Knoop Hardness testing.

METHODS AND MATERIALS

Two dual-cured resin-based luting agents and a glass-ionomer cement were used in this study: Rely X ARC, Enforce and Rely X, respectively. Table 1 lists the composition, batch number and manufacturers of the luting agents.

Eight specimens of each luting cement were prepared in a polytetrafluorethylene split mold with a cross-section of 2 mm x 2 mm and 14 mm depth. The mold consisted of three parts that were affixed with a clamp prior to insertion of the luting agents: two 30 x 10 x 20 mm^3 blocks, and a 2-mm thick central spacer (Figure 1). The luting agents were mixed following manufacturers' instructions and applied into the mold with a lentulo drill. The resin-based luting cements were irradiated from the top surface for 40 seconds using a visible light curing unit (XL–3000—3M ESPE, St Paul, MN, USA) with a power output of 600 mW/cm^2 and the resin-modified glass ionomer cement was allowed to chemically cure for 10 minutes.

After preparation, the clamp was opened and the specimens removed from the molds and stored at 37°C in a dark box for 24 hours prior to microhardness testing. The specimens were positioned transversally beneath the indentor of a digital microhardness tester (FM–1E, Future Tech, Tokyo, Japan) to assess Knoop Hardness (KHN) at three different depths from the top surface: superficial (2.3 mm), medium (7.0 mm) and deep (11.4 mm). Three microhardness measurements were made at each depth for each specimen. A 25g load was applied through the indentor with a dwell time of 10 seconds. The results (KHN) were statistically analyzed by repeated measures ANOVA and Tukey test at the 0.05 confidence level.

RESULTS

The mean KHN values are displayed in Table 2. Repeated measures ANOVA revealed that there were statistically significant differences for the factors "luting agent" (p=0.00538) and "depth" (p=0.00001) and for the interaction between factors (p=0.00001). The Tukey test showed significant differences among luting agents at different depths (p<0.05).

For the superficial third, Enforce and Rely X ARC presented statistically similar KHN values, which were significantly higher than Rely X. For the medium third, there was no significant difference among materials. However, in the deep third, Rely X presented the highest KHN values, statistically different from Rely X ARC. Enforce presented intermediary KHN values, with no significant difference either from Rely X or from Rely X ARC.

Resin-based luting agents presented a progressive decrease in KHN with increased depth. The Tukey test evidenced significant differences for both materials among superficial, medium and deep thirds. On the

Table 1: *Luting Agents Composition, Batch Number, Curing Mode and Manufacturers*

Material (Shade)	Composition	Batch #	Curing Mode	Manufacturer
Enforce (A2)	Bis-GMA, TEGDMA, BDMA, EDAB, BHT DHEPT, bore silicate glass, barium, silica, titanium dioxide, camphorquinone, benzoyl-peroxide	65597	Dual	Dentsply/Caulk, Milford, DE, USA
Rely X ARC (A1)	Bis-GMA, TEGDMA, 67.5 wt% of 1.5 mm zirconium/ silica filler particles, camphorquinone, benzoyl-peroxide	BPBP	Dual	3M ESPE, St Paul, MN, USA
Rely X (A2)	fluoroaluminosilicate glass, modified polyalkenoic acid and HEMA	OBR	Chemical	3M ESPE, St Paul, MN, USA

other hand, Rely X mean KHN for the superficial third was significantly lower than for the medium and deep thirds, which were not significantly different from each other.

DISCUSSION

Depth of cure of light activated dental composites has often been evaluated indirectly by measurement of the hardness of the material at specific depths (DeWald & Ferracane, 1987; Chung & Greener, 1990; Blackman, Barghi & Duke, 1990). A relationship between the degree of conversion and hardness has been demonstrated (Asmussen, 1982; Rueggeberg & Craig, 1988).

The durability and quality of indirect restorations depend on the luting agents' mechanical properties (Chan, Harcourt & Brockhurst, 1993). A high degree of conversion often results in the improved physical and mechanical properties of resin composites (Silikas, Eliades & Watts, 2000). The resin-based luting agents used in this study are dual-cured resins, which means that monomers are polymerized by means of chemical and physical activation. The chemical activation is obtained through the reaction of benzoyl-peroxide with amines. The mixing of these components generates free radicals that will break the carbon double bonds (C=C) that begin the polymerization process. The initiator system of physical activation is based on camphorquinone, which, when exposed to the visible light energy in the wavelength between 400 and 500nm, absorbs energy and combines with a tertiary amine to form an excited state complex that breaks down into reactive free radicals (Ruyter & Øysæd, 1982).

The results of this investigation revealed significant differences among luting materials and depths. For the factor "depth," resin cements presented the highest Knoop hardness values on the superficial third, which decreased significantly along the medium and deep thirds (Figure 2). It suggests that the luting agents were in fact inefficiently cured by chemical cure at deeper regions. Our results are consistent with those found by Blackman and others (1990), Hasegawa, Boyer and Chan (1991), Rueggeberg and Caughman (1993) and El-Mowafy and others (1999), all whom investigated the degree of hardening achieved through

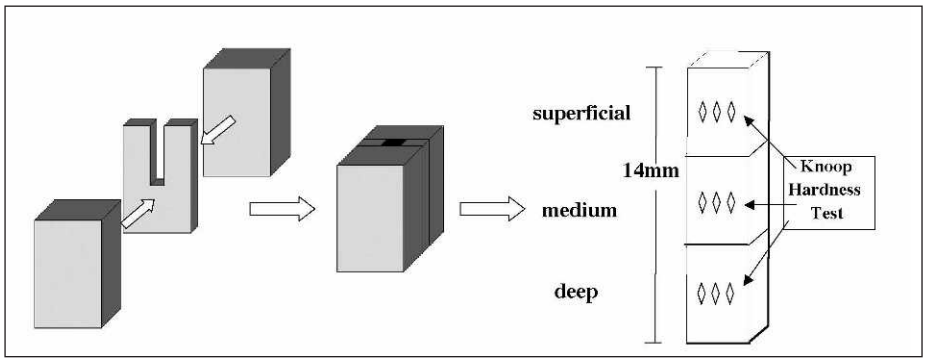

Figure 1. *Schematic representation of specimen preparation with a polytetrafluorethylene split mold and microhardness testing.*

Table 2: *Means (SD) of Knoop Hardness for the Luting Materials at Different Depths*

	Enforce	Rely X ARC	Rely X
Superficial	33.95(5.72)Aa	32.66(3.40)Aa	12.88(5.84)Bb
Medium	22.83(3.73)Ba	20.83(4.20)Ba	18.13(6.46)Aa
Deep	12.92(5.63)Cab	7.05(1.64)Cb	18.34(5.53)Aa

Means followed by different letters (capital letter–column, lower case–row) differ among them by Tukey test at the 0.05 level of significance.

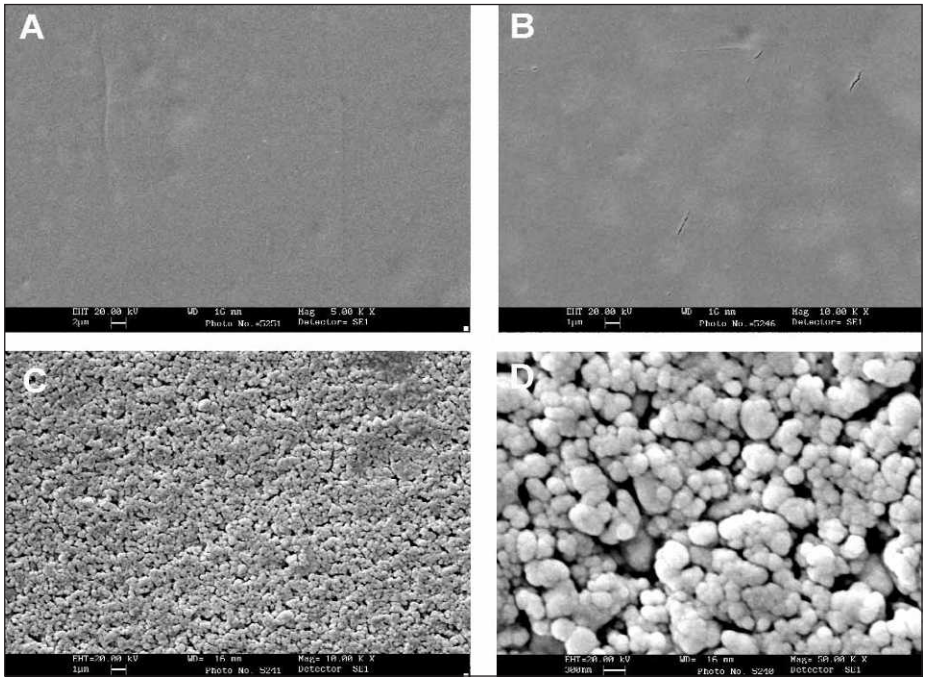

Figure 2. *Scanning electron micrograph of Rely X ARC at superficial (A), middle (B) and deep (C and D) regions. Superficial (A) and middle (B) regions showed similar morphological aspects with smooth surfaces. However, at deeper areas non-uniform globular structures were observed, which might have resulted from poor polymerization (C and D). A–original magnification 5000x, B and C–10,000x and D–50,000x.*

self-curing only and through dual curing. It was concluded that self-curing alone was not adequate to achieve sufficient hardening. Thus, it can be suggested that the amount of chemical initiator was not able to provide a high degree of conversion for the dual-cured composites tested in this study at increased depths. It seems that manufacturers keep the amount of chemical activator to a limited proportion in order to increase working time.

The resin-based luting agents used in this study showed no significant differences in KHN. However, at the deep third, even though no statistical difference was detected, Enforce presented greater KHN values than Rely X ARC. This result may be attributed to differences in monomer composition (Asmussen, 1982), in the amount of chemical activator and in their different filler types and content (Chung & Greener, 1990). Both resin-based luting agents presented features at the deep third that were different from those observed at the medium and superficial thirds (Figure 2). It can be speculated that the irregularities observed at the Rely X ARC deep third are filler particles and resin oligomers that were not able to form longer polymeric chains and/or crosslinks, resulting in non-uniform globular structures (Figures 2C and 2D). However, it is not easy to distinguish among them using Scanning Electron Microscopy.

The resin-modified glass ionomer cement had the lowest hardness in the superficial third, significantly lower than resin-based luting agents. This result may be attributed to the fact that the specimens were not protected at the surface to avoid loss of water. Since resin-modified glass ionomer cements are water-based materials, if water is lost from the cement by desiccation during setting, the chemical curing reaction will stop. Therefore, dehydration of the cement probably resulted in the lowest hardness in the superficial third. On the other hand, the resin-modified glass ionomer cement presented the highest KHN values within the deep third when compared to the resin luting agents, because the cure reaction is not based on light activation. In addition, both the medium and deep thirds were less susceptible to water loss, resulting in higher KHN values than the superficial third. However, despite this desirable property, glass-ionomer cements still lack some physical and mechanical properties presented by resin composites (Davidson & Mjör, 1999).

CONCLUSIONS

The results of this study showed that resin-based luting agents presented the highest hardness values on the superficial third, where marginal areas can benefit largely from both self- and light curing, as they are readily accessible to the curing light. However, the portions that are not directly irradiated by light rely on the self-curing component of the polymerization system, resulting in decreased KHN values. At the deep third, Rely X presented the highest KHN means. Further research is necessary for the development of luting materials with improved mechanical and handling properties.

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The Assessment of Unaided Visual Examination, Intraoral Camera and Operating Microscope for the Detection of Occlusal Caries Lesions

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Ö Uzun • E Baspinar

Clinical Relevance

The use of systems providing magnification aided occlusal caries diagnosis according to a ranked visual scoring system (ERK).

SUMMARY

This study compared the efficiency of unaided visual examination, intraoral camera and operating microscope according to a visual scoring system (ERK) at occlusal caries detection.

A total of 84 extracted human molars were mounted to create mouth models with a premolar in contact on both sides. The models were examined in a phantom head simulating clinical conditions by four observers using the three techniques: unaided visual examination, an intraoral camera and on operating microscope according to the ERK scale. The teeth were than sectioned in a mesio-distal direction and examined under a stereomicroscope with 10x magnification for histological validation.

The sensitivity, specificity, positive predictive and negative predictive values were calculated for the four observers with three techniques and statistical analyses were performed using Friedman and DUNN tests, while strength of agreement was determined by calculating Kappa values.

From the data, mean sensitivity values were calculated as 0.26, 0.43, 0.49 and mean specificity values as 0.87, 0.80 and 0.73 for unaided visual examination, intraoral camera and operating microscope, respectively. The Kappa values ranged between 0.187 and 0.301 for visual examination,

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0.328 and 0.459 for intraoral camera and 0.363 and 0.516 for operating microscope.

As a result, the use of an intraoral camera and operating microscope improved occlusal caries detection according to the ERK scale.

INTRODUCTION

Some occlusal carious lesions may progress into dentin without microscopically visible enamel breakdown. This has partly been explained as the result of an increased use of topical fluorides (Sawle & Andlaw, 1988), as they build up and maintain a relatively well-mineralized surface enamel zone deep to which the mineral loss may gradually progress. One recent study reported that 15% of clinically non-cavitated teeth had occlusal caries penetrating deep into dentin (Wenzel, Larsen & Fejerskov, 1991).

The ERK scale, introduced by Ekstrand, Ricketts and Kidd (1997), helps dentists decide whether teeth are sound or have occlusal caries based on their clinical appearance. When there is a carious lesion, the scale also helps to predict the depth of the lesion. Based on this scale, the lesion is scored as opacity or discoloration hardly visible on the wet surface, but distinctly visible after air drying, opacity or discoloration distinctly visible without air drying, localized enamel breakdown in opaque or discolored enamel and/or grayish discoloration from the underlying dentin and cavitations in opaque or discolored enamel exposing the dentin. The combination of visual inspection with light, probe and dental mirror is accepted as a standard examination procedure in occlusal caries diagnosis (Lussi, 1991). However, forceful probing may result in damage to the fissures and micro-organisms may progress into the deeper parts of underlying substance (Ekstrand, Qvist & Thylstrup, 1987); therefore, alternative diagnostic methods for occlusal caries detection have been developed.

One promising new method uses magnifying visual aids. Magnification is applicable to all tooth surfaces and the usefulness of the technique has been reported (Whitehead & Wilson, 1992; Forgie & others, 1999). Magnification loops (Forgie, Pine & Pitts, 2002), intraoral camera (Forgie, Pine & Pitts, 2003) and operating microscopes (Haak & others, 2002) are being used in caries detection but there is still the need for research on their efficiency in routine dental examinations and clinical acceptance.

This study assessed the efficiency of unaided visual examination, intraoral camera and operating microscope for the detection of occlusal caries in extracted human molars according to the ERK scoring system.

METHODS AND MATERIALS

Out of a stock of extracted human teeth kept in a 10% buffered formalin solution, teeth with no signs of demineralization to those with varying degrees of demineralization on the central fossa were selected following examination under a stereomicroscope. After the teeth were rinsed under running water, they were cleaned with a toothbrush. Eighty-four molars were mounted to the mouth models with one premolar on each side, simulating contacts. The mouth models were fixed into a phantom head adjusted to the dental unit during examination periods.

Occlusal surfaces of each tooth were examined by four observers. These were an associate professor, an assistant professor and one research assistant in the Operative Dentistry and Endodontics Department, having 13, 10 and 4 years of experience, respectively, and a research assistant in the Oral Diagnosis, Oral Medicine and Radiology Department with four years of experience. All of the observers were dentists.

The first examination was performed by unaided visual examination using a dental unit light, compressed air and water from the unit air-water syringe and a standard dental mirror without magnification. A maximum of 20 seconds was allowed to examine one

Table 1: *Criteria Used in Visual Examination (ERK visual, Ekstrand & others, 1997)*

Score	Criteria
0	No detectable change of indicative caries.
1	Opacity or discoloration hardly visible on the wet surface, but distinctly visible after air drying.
2	Opacity or discoloration distinctly visible without air drying.
3	Localized enamel breakdown in opaque or discolored enamel and/or grayish discoloration from the underlying dentin.
4	Cavitation in opaque or discolored enamel exposing the dentin.

Table 2: *Criteria Used in the Histological Evaluation (ERK histological, Ekstrand & others, 1997)*

Score	Criteria
0	No enamel demineralization or a narrow surface zone of opacity (edge phenomena).
1	Enamel demineralization limited to the outer 50% of the enamel layer.
2	Demineralization involving between 50% of the enamel and outer third of dentin.
3	Demineralization involving the middle third of the dentin.
4	Demineralization involving the inner third of dentin.

tooth. The available surfaces were assessed independently by four observers according to the conventional visual scoring system (ERK) (Table 1).

Two weeks after the unaided visual examination, the four observers independently assessed the teeth again according to the ERK scale using an intraoral camera (Rydalmere NSW 2116, Australia) at normal magnification setting, taking up to 20 seconds to examine each tooth. The pictures captured with the camera were stored in the computer for re-examination.

Approximately one week later, the same teeth were again examined independently by the four observers using an operating microscope (Moller-Wedel, Dento 300, and Germany) according to the ERK scale with 16x magnification, taking no more than 20 seconds to examine each tooth. The occlusal surfaces of the teeth were kept wet during examination by each observer and moisturized again for the remaining examiners during examination sessions using the three methods.

Approximately three weeks later, all observers repeated their examinations, starting with the unaided visual examination. Two weeks later, the same teeth were examined with an intraoral camera, and one week subsequent with the operating microscope similar to the first round.

After the second round of examinations were completed, the teeth were removed from the mouth models and hemi-sectioned in a mesio-distal direction through the selected investigation side with a diamond saw (Hp: 915S/220 Risa Dental, Germany). The sections were viewed under a stereomicroscope with 10x magnification by two observers having experience in determining sound surfaces and caries depth under a stereomicroscope (Olympus SZ-PT, Japan) according to the criteria shown in Table 2. The depth of enamel demineralization was assessed as the area showing the greatest extension of demineralization along the direction of rods. The depth of dentin demineralization was assessed where the color changed from brownish/yellow to gray along a line at right angles to the dentoenamel junction towards the pulp (Kidd & others, 2003). Any discrepancies in the histological scores were corrected by consensus after reviewing the sections.

Statistical analyses were performed by calculating the sensitivity, specificity, positive and negative predictive values for unaided visual examination, intraoral camera and operating microscope. Sensitivity is defined as the percentage of correctly identified carious lesions, and specificity is defined as the percentage of correctly identified sound teeth with a diagnositic task. Freidman and DUNN tests were used to establish the relation with observer's diagnosis with the three methods and strength of agreement was assessed by calculating Kappa values. When the value was found as <0.00, strength of agreement was said to be poor, between 0.00-0.20 as slight, 0.21-0.40 as fair, 0.41-0.60 as moderate, 0.61-0.80 as substantial and 0.81-1.00 as almost perfect.

RESULTS

Based on histological examination, 31 teeth were non-carious, 8 had caries limited to the 50% of the outer enamel, 26 had lesions between 50% of the enamel and the outer third of dentin, 17 had lesions localized at the middle third of the dentin and 2 had lesions localized at the inner third of dentin. Table 3 shows the sensitivity and specificity values for the four observers and three techniques. From the mean sensitivity values, it was found that unaided visual examination had the lowest

Table 3: *Four Observers' Sensitivity and Specificity Values for Unaided Visual Examination, Intraoral Camera and Operating Microscope According to ERK Scale*

	Unaided Visual Examination		Intraoral Camera		Operating Microscope	
	Sensitivity	Specificity	Sensitivity	Specificity	Sensitivity	Specificity
1st observer	0.11	0.97	0.38	0.81	0.43	0.74
2nd observer	0.32	0.81	0.45	0.74	0.51	0.61
3rd observer	0.30	0.84	0.38	0.81	0.47	0.71
4th observer	0.30	0.87	0.49	0.84	0.55	0.87
Mean value	0.26	0.87	0.43	0.80	0.49	0.73

Table 4: *Four Observers + Predictive and – Predictive Values for Unaided Visual Examination, Intraoral Camera and Operating Microscope According to ERK Scale*

	Unaided Visual Examination		Intraoral Camera		Operating Microscope	
	+ predictive	-predictive	+ predictive	-predictive	+ predictive	-predictive
1st observer	86%	39%	77%	43%	74%	43%
2nd observer	74%	41%	75%	44%	69%	42%
3rd observer	76%	41%	77%	43%	73%	44%
4th observer	80%	42%	84%	49%	88%	53%

value (0.26) and operating microscope had the highest value (0.49). On the other hand, when specificity values were discussed, the operating microscope had the lowest (0.73) value and unaided visual examination had the highest value (0.87). The predictive values (positive, negative) for four observers and three techniques can be seen in Table 4. According to the Friedman test, there was a significant difference between the score medians of all observers in the three techniques, therefore, the DUNN test was used to determine which group was different according to the sum of ranks. For the first, second and third observer, the difference between the sum of ranks for unaided visual examination-intraoral camera; unaided visual examination-operating microscope and intraoral camera-operating microscope was significant ($p<0.05$), while for the fourth observer, the difference between unaided visual examination and operating microscope was significant but the difference between the unaided visual examination-intraoral camera and intraoral camera-operating microscope was not significant ($p>0.05$) (Table 5).

Table 5: *Sum of Ranks According to Friedman Test for Four Observers and Three Techniques*

	Sum of Ranks		
Observers	**V E**	**I O C**	**O M**
1	118.5	163.5	204.0
2	133.0	157.5	195.5
3	136.5	160.5	189.5
4	142.0	162.0	182.0

V E: Visual Examination, I O C: Intraoral Camera, O M: Operating Microscope

Table 6: *Strength of Agreement According to Kappa Values for Each Observer and Three Techniques*

	Sum of Ranks		
Observers	**V E**	**I O C**	**O M**
1	0.187	0.328	0.384
2	0.301	0.377	0.363
3	0.299	0.376	0.370
4	0.280	0.459	0.516

V E: Visual Examination, I O C: Intraoral Camera, O M: Operating Microscope

Kappa value <0.00: poor, 0.00-0.20: slight, 0.21-0.40: fair, 0.41-0.60: moderate, 0.61-0.80: substantial, 0.81-1.00: almost perfect.

Kappa values ranged from 0.187 to 0.301 for visual examination, 0.328 to 0.459 for intraoral camera and 0.363 to 0.516 for operating microscope. Details are shown in Table 6.

DISCUSSION

The ERK scale is a visual ranked scoring system that can be used to detect occlusal carious lesions. It claims to help dentists decide whether teeth are sound or have occlusal caries and predicts lesion depth according to clinical appearance (Ekstrand & others, 1997). It can be easily used in routine dental examinations, because it does not require extra equipment or extra time. Ricketts and others (2002) have reported that it can be used to predict the level of infection of dentin. Therefore, we aimed to evaluate the use of the ERK scoring system with unaided visual examination, intraoral camera and operating microscope.

Since sensitivity value is defined as the percentage of correctly identified carious lesions, a good diagnostic task should have a high sensitivity value. If the diagnostic task has a low value, the percentage of correct diagnosis of carious lesions is expected to be low. The dentist should keep this in mind. The specificity value, described as the percentage of correctly identified sound surfaces, is preferred to be high for a caries detection method, too. A low value causes sound surfaces to be incorrectly diagnosed as carious and results with overtreatment.

The mean sensitivity value for unaided visual examination was 0.26 and increased to 0.43-0.49 when teeth were examined with the intraoral camera and operating microscope, respectively. The correct diagnosis number was nearly doubled with the two methods providing magnification. The increase in the number of correctly defined lesions helps dentists more effectively identify preventive measures. On the other hand, sound surfaces that were correctly identified decreased, compared to unaided visual examination at both techniques. The greatest drop was achieved with the operating microscope, which provided the highest magnification rate in the study (16x). Therefore, it could be concluded that, with the increase in magnification, there was an increase in false positive diagnosis. This could result in over treatment; the clinician should keep this in mind. Forgie and others (2003) reported that, compared to the unaided visual examination, the use of an intraoral video camera significantly increased the number of occlusal caries detected with a concurrent rise in false positive scores. These results are consistent with this study.

In this study, sensitivity and specificity values according to the ERK scoring scale were used to assess the efficiency of unaided visual examination, intraoral camera and operating microscope at occlusal caries detection. The ERK scale divides the carious lesion, as demineralization limited to the outer 50% of the enamel layer, demineralization involving between 50% of the enamel and outer third of dentin, demineralization involving the middle third of dentin and demineralization involving the inner third of dentin according to the lesions clinical appearance. Sensitivity values were found to be low

for all methods. One of the reasons for the low values could be related to the scale, as it divides the lesions into four areas as mentioned above. If the lesions were assessed according to different scales, sensitivity values might have been higher. More studies are needed to evaluate this point.

The first, second and third observers were from the Department of Operative Dentistry and Endodontics, having 13, 10 and 4 years of experience, respectively, and the fourth observer was from the Department of Oral Diagnosis Oral Medicine and Radiology, having four years of experience. All observers had been calibrated for occlusal caries detection in epidemiologic studies. The difference among the sum of ranks for unaided visual examination-intraoral camera, unaided visual examination-operating microscope and intraoral camera-operating microscope was significant for the three restorative dentists, while for the fourth observer, the difference between unaided visual examination and operating microscope was significant but the difference between unaided visual examination-intraoral camera and intraoral camera-operating microscope was not. The strength of agreement ranged among the four observers from slight to fair for the ERK scale when used with unaided visual examination, fair to moderate for the intraoral camera and fair to moderate for the operating microscope. All observers' Kappa values increased for both the intraoral camera and operating microscope compared to the unaided visual examination.

The operating microscope produces a magnified image (16x in this study) of the occlusal surface, which is seen on a television screen connected to the microscope. The operating microscope and intraoral video camera systems are expensive and the operator needs proper training. In this study, an experienced observer took images with the two systems, and optimal quality images were simultaneously displayed on the television/computer monitor and independently assessed by the four observers. With this, each observer examined the occlusal surfaces of the teeth under optimized standard conditions. The pictures captured with the intraoral camera were displayed on a 14-inch computer monitor and displayed on a 21-inch television. There was also a difference between the size of screen that displayed the images. Further investigations should be made to research whether or not the size of the screen affects the use of systems at occlusal caries detection.

As a result, when depth assessment was made according to the ERK scale, compared to the unaided visual examination, the use of an intraoral camera and operating microscope proved the correct diagnosis, with an increase in false positive diagnosis.

CONCLUSIONS

In respect to this study, it can be concluded that the intraoral camera and operating microscope help dentists attain a higher degree of correct diagnosis according to the ERK scale but with a drop in correct sound surface diagnosis.

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Microtensile Bond Strengths of One-step and Self-etching Adhesive Systems

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Clinical Relevance

In this investigation, conventional one-step adhesives showed significantly higher microtensile bond strengths than self-etching adhesives.

SUMMARY

The microtensile bond strength of resin composite bonded to human enamel was evaluated utilizing four light-cure bonding agents. Human third molars were embedded in auto-cure acrylic and the buccal surfaces were sequentially abraded to 400 grit. Resin composite cylinders were then bonded using the four bonding systems according to the manufacturer's specifications. Each bonded tooth produced three to four longitudinal sections which were then laterally notched to give a square bond area (~2.25 mm²). Specimens (n=10) were assigned to two groups: Group I was stored in distilled water at 37° ± 2°C for seven days. Group II was stored in distilled water at 37° ± 2°C for seven days, during which time it was thermocycled in hot and cold water baths for 1,000 cycles. In addition, a water sorption test was performed on three of the four adhesive systems. The microtensile bond strength of the conventional adhesive Optibond Solo Plus was significantly greater than that of the self-etching adhesives Tyrian SPE and Prompt L-Pop. Adhesive systems that were more hydrophilic tended to show lower bond strengths, especially after thermocycling.

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INTRODUCTION

It has been nearly 50 years since Buonocore proposed chemically treating enamel with an aqueous acid in order to improve the adhesion of resinous restorative materials (Buonocore, 1955). Later studies showed that the principal mechanism for increased adhesion to enamel was through the formation of resin tags that penetrated microporosities within the etched enamel surface (Buonocore, Matsui & Gwinnett, 1968). Since that time, the practice of etching enamel with aqueous phosphoric acid has become a standard technique.

First and second enamel and dentin resin adhesive systems were formulated primarily to increase the bond

to dentin (Bowen, 1965). They usually contained adhesive monomers that were the phosphoric acid esters of glycerol dimethacrylate or Bis-GMA. These bifunctional molecules were believed to chemically chelate to the calcium ions within the dentin and enamel hydroxyappetite in addition to polymerizing into the resin matrix of the low viscosity adhesive (Bowen, 1965).

Enamel and dentin bonding agents have since progressed from the more complicated multi-bottle third and fourth generation adhesive systems which required a conditioner (etchant), primer and low viscosity resin adhesive to the more simplified "one step" fifth and sixth generation adhesives (American Dental Association, 1987; Nakabayashi, 1985; Nakabayashi, Nakamura & Yasuda, 1991; Ferrari, Goracci & García-Godoy, 1997). These "one bottle" systems combined both primer and resin adhesives but still required a separate step to etch the enamel and condition the dentin (Ferrari & others, 1997).

More recently, "all in one" self-etching adhesives have been introduced which combine the conditioner or etchant, primer and adhesive into one application and, therefore, do not require rinsing (Gordan & others, 1997). These new adhesive systems, however, contain a higher concentration of acidic monomers, compared to conventional bonding agents, in order to obtain an etched enamel surface (Tay & Pashley, 2001). Previous studies have found that bonding adhesives that contain a large number of acidic groups are susceptible to excess water sorption (Tanaka & others, 1999). Therefore, because of their hydrophilic nature, there is concern as to the long-term durability of these self-etching adhesives in the oral environment, especially in the presence of plaque. Therefore, the purpose of this investigation was to evaluate the microtensile bond strength of resin composite bonded to human enamel utilizing two light-cure self-etching and two light-cure conventional "one step" bonding agents.

METHODS AND MATERIALS

Microtensile Bond Test

Freshly extracted non-carious human third molars were debrided of tissue and stored in 0.5% chloramine T at 4°C for up to one month prior to embedment. The roots were then removed and the buccal enamel was ground to a flat surface 0.5-mm deep with a diamond bur using a high-speed handpiece and water spray. Each tooth was then embedded in auto-cure acrylic so that the buccal surface was exposed. The buccal enamel was then sequentially fine sanded with silicon paper and water to 400 grit. The area of prepared enamel surface averaged 8 mm x 4 mm. The embedded teeth were then randomly distributed into four groups of seven teeth each. One of the adhesive systems to be tested was then applied to the teeth of each group according to manufacturer specifications (Table 1). Resin composite cylinders (4 mm x 10 mm [diameter]) were built up incrementally in 1-mm layers utilizing a clear lucite plastic ring (Figure 1). Each layer was light cured for 30 seconds using a Demetron 401 halogen curing light (Demetron Research, Danbury, CT, USA) calibrated with a radiometer at 600 mW cm^{-2}. Specimens were stored in distilled water at 37° ± 2°C for 24 hours. Each bonded specimen was mounted on a low-speed diamond saw cutting machine and serially sectioned perpendicular to the enamel surface to produce three to four 1.5-mm thick longitudinal sections. Each longitudinal section was then side cut along the resin/enamel interfacial bond with a cylindrical (1.0 mm diameter) fine diamond bur in a high-speed handpiece under copious air-water spray. This procedure resulted in a 1.5-mm square bond area approximately 2.25 mm^2 for each bonded specimen. Specimens with noticeable defects or voids were discarded. Twenty bonded sections remained for each of the four adhesive systems.

Specimens from each adhesive system were then equally divided into two groups (n=10):

Group I (non-thermocycled): Bonded specimens were stored in distilled water for seven days.

Group II (thermocycled): Specimens were stored in distilled water at 37° ± 2°C for seven days during which time they were thermocycled in water baths at 5°C and 55°C for 30 seconds dwell time per bath and 1000 cycles.

Delrin plastic strips were then cyanoacrylated to the enamel and composite sides of each specimen perpendicular and away from the bond area, as depicted in Figures 2 and 3. Each bonded specimen with attached plastic strips was mounted in a custom microtensile testing apparatus on a universal testing machine (Instron, Canton, MA, USA) (Figure 4). The bonded specimens were then fractured in tensile mode at a crosshead speed of 0.5 mm/minute and the maximum load at failure was recorded. After testing, the cross-sectional dimensions of each fractured specimen were determined using digital calipers. Fractured test specimens were then examined under low-power microscope (20x) in order to determine the bond failure mode. Bond failure was characterized according to the area of resin remaining on the enamel surface.

Water Sorption Test

Disc-shaped specimens (15 mm [diameter] x 0.5 mm) of three of the bonding adhesive systems (n=3) were light polymerized (total exposure 60 seconds) utilizing a polypropylene mold compressed between two glass plates at a constant load (5 kg). Tyrian SPE specimens were not prepared because the self-etching primer and adhesive resin were separated into a two-bottle system. The specimens were placed in a dessicator at 37°C, con-

Table 1: *One-step and Self-etching Adhesive Systems*

Adhesive	Manufacturer	Type	Composition	Procedures
Optibond Solo Plus	Kerr Corp, Orange, CA, USA	5th generation "one step"; requires separate etch step	*Etchant**: 35% phosphoric acid. *Adhesive:* BisGMA, HEMA, GPDM, ethanol, barium aluminum borosilicate glass, fumed silica, sodium hexafluoro-silicate, photoinitiator.	Etch 15 seconds; rinse 10 seconds with air/water spray; dry lightly with air; apply adhesive 15 seconds (active); air thin; glossy appearance; VLC 20 seconds.
Prime & Bond NT	Dentsply/Caulk, Milford, DE, USA	5th generation "one step"; requires separate etch step	*Etchant:* 35% phosphoric acid. *Adhesive:* PENTA, silicone dioxide nanofillers, tri and dimethacrylates, cetylamine hydrofluoride, acetone, photoinitiators.	Etch 15 seconds; rinse 10 seconds with air/water spray; blot; apply adhesive 20 seconds (active); air thin; glossy appearance; VLC 20 seconds.
Prompt L-Pop	ESPE, Seefeld, Germany	Self-etching	*Adhesive:* Water, methacrylated phosphoric acid esters, phosphine oxide, fluoride complex with zinc, photoinitiators.	Clean enamel surface with non-fluoride pumice paste; rinse and air dry; apply adhesive 15 seconds (active); air thin; glossy appearance; VLC 10 seconds.
Tyrian SPE	BISCO, Schaumburg, IL, USA	Self-etching	Self-etching primer: 2-acrylamido-2-methyl propanesulfonic acid, Bis (methacryloyloxy ethyl), phosphate, ethanol. *Adhesive:* Biphenyl dimethacrylate, EMA, acetone, glass filler, photoinitiators.	Clean enamel surface with non-fluoride pumice paste; rinse and air dry; apply primer 10 seconds (active); blot excess primer until purple color disappears; apply two coats adhesive; air dry 10 seconds; glossy appearance; VLC 10 seconds.

**Ultra-etch (Ultradent Products, South Jordan, UT, USA).*

ditioned to a constant mass, then weighed (m_1). The dried specimens were placed in distilled water at 37°C ± 2°C for seven days, weighed (m_2) and reconditioned to a constant mass in a dessicator (m_3). The diameter and thickness of each specimen was then recorded with a digital micrometer. Using these measurements, the volume (v) of each specimen was calculated in cubic millimeters. The water sorption (Wsp) was then determined using the following formula:

$$\text{Wsp} = \frac{m_2 - m_3}{v}$$

Two-way analysis of variance (ANOVA) procedure followed by the Ryan-Einot-Gabriel-Welsh (REGW) multiple range test were performed on all data from both the microtensile and water sorption data.

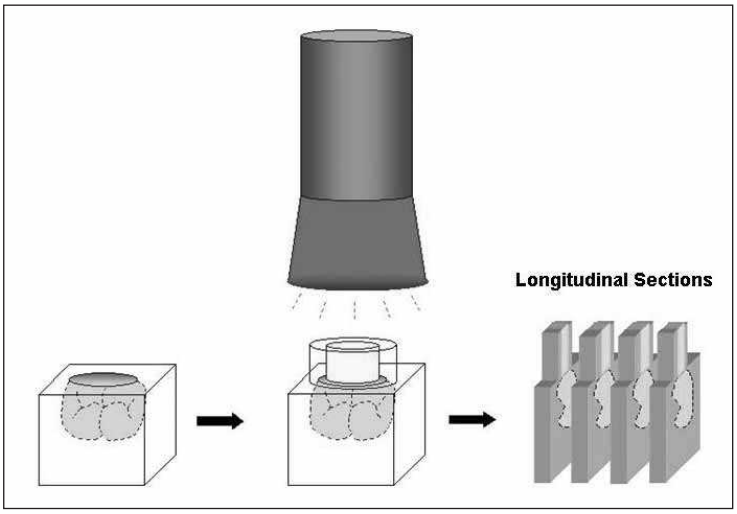

Figure 1. *Schematic representation of resin composite light-cured to buccal surface of embedded tooth with subsequent serial sectioning.*

RESULTS

The results of the microtensile bond strength test are presented in Figure 5. The general linear model (p>0.001) and REGW Multiple Range Test (p>0.05) showed significant differences among several of the adhesives tested with or without thermocycling. The mean microtensile bond strength of the conventional one-step adhesive Optibond Solo Plus was nearly 50% greater than the self-etching adhesive Tyrian SPE for both testing conditions. A significant reduction in microtensile bond strength was observed for the one-step adhesive Prime & Bond NT after thermocycling compared to specimens that were not thermocycled. No significant difference was noted between the mean microtensile bond strength value of the thermocycled and non-thermocycled specimens of Optibond Solo Plus. Prime & Bond NT and Prompt L-Pop exhibited significantly higher water sorption than Optibond Solo Plus (Table 3).

The results of the bond failure mode analysis for thermocycled and non-thermocycled specimens are presented in Table 2. Analysis of bond failure showed a greater number of adhesive failures with Tyrian SPE for both

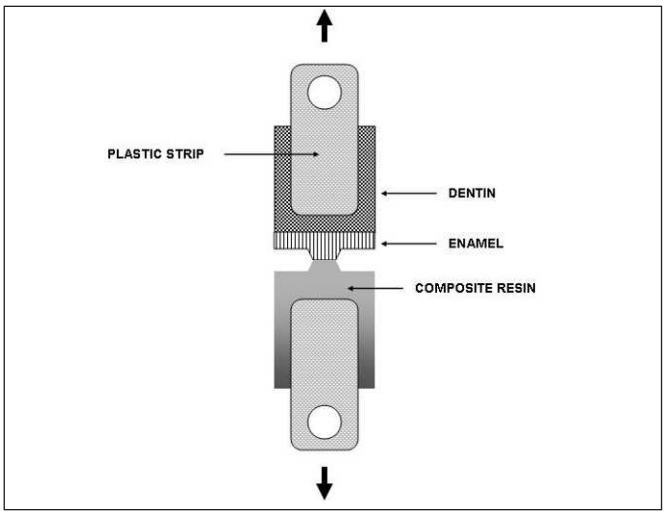

Figure 2. *Schematic representation of bonded longitudinal section with side cuts and glued plastic strips (frontal view).*

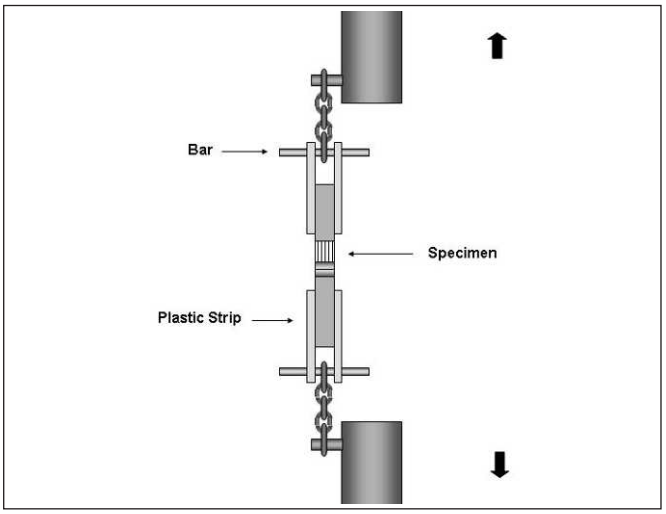

Figure 3. *Bonded specimen attached to load arms within microtensile testing jig (side view).*

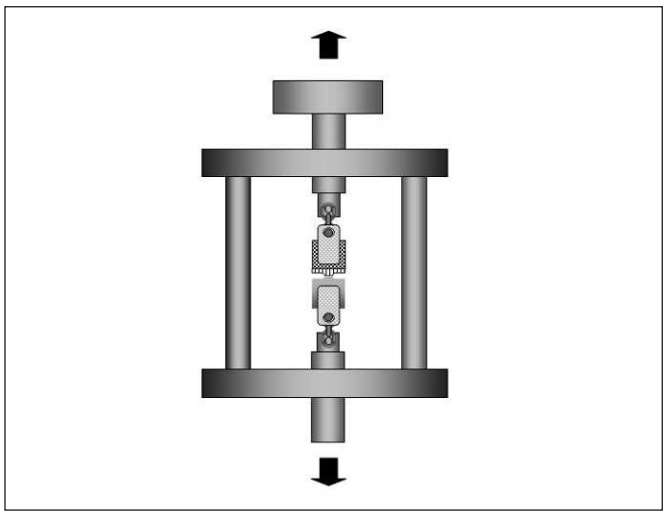

Figure 4. *Schematic representation of custom microtensile testing apparatus with attached bonded specimen.*

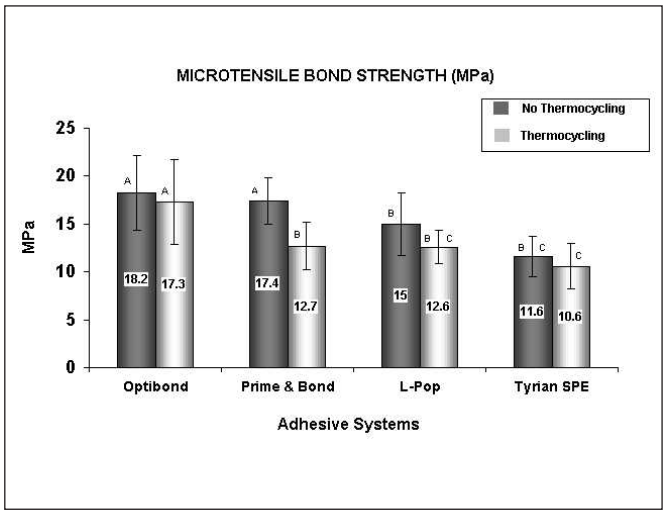

Figure 5. *Mean microtensile bond strength values in MPa with standard deviations for both testing conditions. Groups with the same letter denote no significant difference (α=0.05).*

testing conditions. Optibond showed slightly more cohesive failures than Prime & Bond NT or Prompt L-Pop for both testing conditions. Nearly all cohesive/adhesive and cohesive failures occurred through the adhesive and/or resin composite matrix.

DISCUSSION

Previous investigations have shown the microtensile bond strength of light cured resin composite to acid conditioned ground enamel to be in the range of 28.4 to 41.9 (Kanemura, Sano & Tagami, 1999; Sano & others, 1994; Frankenberger & others, 2002). Bond strength values obtained from the non-thermocycled specimens in this investigation were in the general range of 11.6-18.2 MPa. There may be several reasons for the generally lower microtensile bond strength values reported in this investigation. Studies have shown an inverse relationship between microtensile bond strength and bond surface area (Phrukkanon, Burrow & Tyas, 1998). Microtensile bond strength values tend to increase with decreasing interfacial bond area. This may be the result of a more uniform stress distribution across a smaller bond area during tensile testing. A bond area of no more than 1.6-1.8 mm^2 has been suggested in order to reduce the intra-group bond strength value variation and raise the probability that failure will occur within the adhesive layer instead of the enamel (Phrukkanon & others, 1998). In addition, specimens with a smaller bond area are less likely to exhibit internal defects, which may act as a nidus for crack propagation.

The bond length to bond area ratio of a microtensile specimen with a square interfacial bond area of 1.6 mm^2 is nearly three times that of a tensile bond specimen

with a circular bond area of 4 mm diameter (#4 gel capsule). This increased ratio would tend to expose a greater portion of the interfacial bond area to hydrolytic degradation during water storage. Adhesive systems which are more susceptible to water sorption may be more likely to show lower microtensile bond strength values (Tanaka & others, 1999), especially after thermocycling. A slightly larger bond area (2.25 mm^2) was chosen for this investigation in order to decrease the likelihood of specimen failure during thermocycling in water baths at alternating hot and cold temperatures. However, this larger than ideal bond area may have also attributed to lower microtensile bond strength values.

Table 2: *Bond Failure Mode*

	Non-Thermocycled (Thermocycled)		
Adhesive Systems	Adhesive [<25%]	Cohesive/Adhesive [25-75%]	Cohesive [>75%]
Optibond Solo Plus	2 (3)	5 (6)	3 (1)
Prime & Bond NT	3 (5)	7 (5)	0 (0)
Prompt L-Pop	3 (2)	6 (7)	0 (1)
Tyrian SPE	8 (8)	1 (2)	1 (0)

[Percent of resin remaining on enamel surface of fractured test specimens]

Table 3: *Water Sorption Test*

Adhesive Systems	pH	Water Sorption	
Optibond Solo Plus	2.9	295 (18)	A
Prime & Bond NT	2.7	518 (53)	B
Prompt L-Pop	1.8	609 (36)	C

Mean water sorption values in ug/mm^3 with standard deviations ($p<0.05$) ($n=3$). Values with the same letter denote no significant difference ($\alpha=0.05$).

The mean microtensile strengths of Tyrian SPE and Prompt L-Pop adhesive systems were significantly lower than the other adhesives tested. Self-etching "no rinse" adhesives such as Prompt L-Pop and Tyrian SPE contain a higher concentration of phosphoric acid, methacrylated phosphoric acid esters and/or sulfonic acid derivatives compared to conventional bonding agents in order to obtain an enamel etch pattern amenable to micromechanical bonding (Tay & Pashley, 2001). Methacrylate adhesive monomers which contain hydrophilic moieties such as carboxylic acid, phosphoric acid and hydroxyl groups have been shown to absorb significant amounts of water when polymerized. Increased water sorption within the adhesive layer is highly correlated with lower bond strengths (Tanaka & others, 1999). Furthermore, methacrylate phosphoric acid esters within the adhesive resin matrix, which are not neutralized into insoluble calcium phosphate salts during enamel etching, would tend to make the adhesive layer highly hydrophilic.

Interestingly, the mean microtensile bond strength value of thermocycled Tyrian SPE specimens was not significantly different from the non-thermocycled specimens. This self-etching bonding system incorporates a pH indicator which undergoes a color change when most of the acid moieties have been neutralized during acid etching. This signals the operator that placing an additional layer of acidic primer is not necessary. Although this method reduces the amount of unreacted methacrylic acid esters within the adhesive layer, it does not eliminate it.

The one-step adhesive Prime & Bond NT was the only adhesive system tested that showed a significant reduction in microtensile bond strength after thermocycling when compared to non-thermocycled specimens. Components of this adhesive system, such as acetone, if not completely evaporated before polymerization, will tend to plasticize and weaken the adhesive layer. Acetone is also highly hydrophilic.

The mean microtensile bond strength value of the conventional one-step adhesive Optibond Solo Plus was significantly higher than any of the other adhesives tested after thermocycling. Optibond Solo Plus also exhibited significantly lower water sorption than Prime & Bond NT and Prompt L-Pop (Table 3). The results of the water sorption test seemed to corroborate with previous studies which show lower bond strength values with increased water sorption among the tested adhesive systems. Factors such as increased filler loading, higher pH (lower acid content) and the presence of crosslinking dimethacrylate resins may also contribute to decreased water penetration and stronger cohesion within the adhesive layer.

CONCLUSIONS

1. The microtensile bond strength of Optibond Solo Plus was significantly higher than the self-etching adhesives Prompt L-Pop and Tyrian SPE for both testing conditions.
2. A significant reduction in microtensile bond strength was observed with thermocycled Prime & Bond NT compared to specimens that were not thermocycled.
3. Adhesive systems that were more hydrophilic tended to show lower bond strengths, especially after thermocycling.
4. Further study is warranted regarding the durability of these new self-etching adhesives in the oral environment, especially in the presence of plaque.

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The *In Vitro* Morphological Effects of Some Current Pre-treatments on Dentin Surface: A SEM Evaluation

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Clinical Relevance

The first step towards a successful adhesive restorative procedure is pre-treatment of the substrate for bonding. The total-etch bonding technique involves treatment of cavities with mineral or organic acids. This treatment removes all minerals to a depth of 6-8 µm and exposes the collagenous fiber network of the matrix, making it available for adhesive resin infiltration. The role of the collagen network on dentin bonding has also been questioned, and self-etching primers have been suggested as an improved technique for bonding to dentin; they are less aggressive and promote a more uniform surface after treatment. The morphological alterations of smear layer-covered dentin promoted by these agents, evaluated by this study, are important to better understand bonding techniques.

SUMMARY

This *in vitro* study morphologically evaluated the effect of some current surface pre-treatments on dentin, using scanning electron microscopy, and related these morphological alterations to clinical implications. The labial surfaces of 30 bovine lower incisors were ground to obtain a flat dentin surface and were finished with 600-grit SiC paper to produce standardized smear layers. The teeth were randomly divided into six groups of five each. Group 1 was the control group, smear layer covered dentin; Group 2 was etched with 37% phosphoric acid (PA) for 15 seconds; Group 3, 37% PA for 15 seconds, followed by 10% NaOCl for 60 seconds; Group 4, 10% NaOCl for 60 seconds; Group 5, a self-etching primer (Clearfil SE Bond, CSEB-primer) was applied for 20 seconds; Group 6, CSEB-primer for 20 seconds, followed by NaOCl for 60 seconds. The specimens were fixed, dehydrated, dried and analyzed by SEM. Treatment with 37% PA removed the smear layer, funneled the tubules and resulted in a collagen-rich surface which appeared to have collapsed in its outermost part, producing a dense surface layer covered with silica particles. When 37% PA treatment was followed by 10% NaOCl, the collagen network was removed to reveal an eroded, rough mineral surface with numerous lateral branches and larger than normal tubular orifices. The action of 10% NaOCl on the smear layer-covered dentin showed no significant alteration in surface morphology. The treatment with CSEB-primer dissolved the smear layer but only partially dissolved the smear plugs. The tubules

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did not present the typical funnel shape seen following PA treatment. These morphological aspects on dentin surface must influence bonding results. The dentin surface alterations produced by PA appeared to be a very severe demineralization pattern, quite irregular and less permeable to monomer infiltration, while the surface provided by the self-etching primer appeared to be a more uniform, less porous surface, and the association with simultaneous monomer infiltration may reduce the occurrence of mistakes in clinical bonding procedures.

INTRODUCTION

The current concept for bonding to tooth hard tissues is based upon the infiltration of a low-viscosity resin into enamel and dentin, a process known as hybridization (Nakabayashi, Kojima & Masuhara, 1982). While bonding to enamel has been considered a reliable procedure due to its uniform composition (hydroxyapatite), bonding to dentin remains a challenge, as it contains higher water content and more organic material, mainly type 1 collagen. Dentin is an intrinsically wet tissue penetrated by a tubular network that communicates tissue fluids of the pulp chamber (Perdigão & Lopes, 1999). Furthermore, when cutting procedures are performed on tooth structure, the dentin surface becomes covered with a 1-µm thick layer of debris composed mostly of submicron particles of mineralized collagen matrix. Effective adhesion of resins to dentin begins with the treatment of this so-called smear layer (Pashley & others, 1993).

There are two different ways that the current bonding systems obtain acceptable micromechanical retention between resin and dentin. The first method is based on complete removal of the smear layer and demineralization of subsurface intact dentin, using acid etching with mineral or organic acids, leaving a collagen rich, moist surface into which resins must diffuse to form a collagen-resin interdiffusion zone first described as a hybrid layer (Nakabayashi & others, 1982). The second method uses slightly acidic monomers, the so-called self-etching primers, to partially demineralize the smear layer and underlying intact dentin, incorporating the demineralized smear layer remnants and using them as a bonding substrate (Pashley & Carvalho, 1997; Van Meerbeek & others, 1998).

Based on the methods mentioned above, successful resin bonding to dentin depends on the preparation of a surface with priming agents that renders it receptive to a bonding agent. This surface preparation must be able to alter the wetability of dentin in order to permit the diffusion of resin to the complete depth of demineralization, enveloping the entire collagen fiber network exposed by such treatment with liquid comonomers.

In the first method, the most commonly used conditioning agent is phosphoric acid in concentrations that vary from 10% to 40%. A major problem with this technique is its sensitivity (Tay, Gwinnett & Wei, 1996). Removal of the smear layer and smear plugs from dentin tubules increases the natural surface moisture of dentin (Pashley, Michelich & Kehl, 1981). Maintenance of the structural integrity of the exposed collagen fibers and interfibrillar spaces to allow permeation of adhesive monomers is totally moisture dependent, since it plays a crucial role in preventing the collapse of the collagen network (Titley & others, 1994; Tay & others, 1996; El Feninat & others, 2001). Furthermore, acidic conditioners induce considerable changes in collagen conformation, mostly associated with denaturation processes (Eliades, Palaghias & Vougiouklakis, 1997). Consequently, without adequate and precise resin-infiltration of this surface, dentinal tubules may remain unsealed, allowing for penetration of bacterial products through dentin to pulpal soft tissues, causing severe post-operative sensitivity and pulpal inflammation.

The role of the hybrid layer in dentin bonding has been constantly questioned (Guinnett, 1994; Uno & Finger, 1995; Guinnett & others, 1996). It has been shown that resin monomers do not fully diffuse through the collagen network to the depth of acid etching of the dentin. This incomplete resin-infiltration into the collagen network may produce a porous layer of collagen not protected by hydroxyapatite or encapsulated by resin. This exposed collagen is subjected to hydrolysis and degradation, resulting in increased microleakage and failure over time (Sano & others, 1994). Moreover, the theoretical benefit of avoiding an acid-exposed collagen layer is that the above mentioned problems could be prevented. The removal of collagen has been suggested as a suitable method to alter the composition of dentin, making it similar to etched enamel, that is, a more predictable and hydrophilic substrate for bonding (Sakae, Mishima & Kozawa, 1988; Tanaka & Nakai, 1993; Inaba & others, 1995).

Sodium hypochlorite (NaOCl) is a non-specific proteolytic agent that effectively removes organic compounds at room temperature (Sakae & others, 1988). Data from studies suggest that for some adhesive systems, removal of collagen fibers with NaOCl pre-treatment may actually increase bond strength (Wakabayashi & others, 1994; Chersoni & others, 1997; Boschian & others, 1997; Vargas, Cobb & Armstrong, 1997; Inai & others, 1998; Saboia, Rodrigues & Pimenta, 2000), while for other adhesive systems, collagen removal did not alter or diminish bond strength (Guinnett, 1994; Inai & others, 1998; Saboia & others, 2000; Frankenberger & others, 2000a) and did not alter or increase marginal leakage (Uno & Finger, 1995; Vichi, Ferrari & Davidson, 1997; Frankenberger & others, 2000a; Toledano & others, 2000). Scanning electron microscopy (SEM) studies of

demineralized dentin surfaces treated with NaOCl reveal an altered eroded surface appearance (Inai & others, 1998; Perdigão & others, 1999) and development of a network of secondary channels (Perdigão & others, 1999).

The composition of a smear layer is generally similar to the originating tissue; thus, it is assumed that its mineral phase is similar to normal dentin (about 50% vol), and the collagen phase (30% vol) is insoluble and cannot be rinsed away after acid etching. When acidic conditioners remove the solid mineral content of the smear layer, the residual collagen phase of the smear layer persists, thereby enriching the surface with organic material that may then partially occlude the spaces between demineralized collagen fibers, interfering with adhesive infiltration (Pashley & others, 1993). Pre-treatment of the smear layer with NaOCl might eliminate the collagen phase if it was not mineralized. SEM studies have shown no significant morphological changes when NaOCl was applied on a smear layer, suggesting only superficial collagen removal (Tanaka & Nakai, 1993; Prati, Chersoni & Pashley, 1999).

The advent of self-etching primers represents a very simple strategy to prevent exposed collagen mesh from collapsing, avoiding its unprotected exposure. When self-etching primers are applied, there is no need for etching, rinsing and drying, so the risk of over-etching, over-drying or over-moistening the dentin is eliminated (Haller, 2000). A recent morphological study classified these acidic primers into three classes, including mild, moderate and aggressive, based on their ability to penetrate dentin smear layers and their depth of demineralization into subsurface dentin. The more aggressive system completely solubilized the smear layer and smear plugs and formed hybrid layers with a thickness similar to that obtained by PA conditioned dentin, while the mild system was able to diffuse through the smear layer to the subsurface intact dentin, producing a thin but authentic 0.5 µm hybrid layer and sealed dentinal tubules with thick, resin-hybridized smear plugs (Tay & Pashley, 2001).

The morphological alterations promoted by these pre-treatments on dentin may play an important role in bonding to dentin, since they change the surface features considerably and must thereby influence diffusion of bonding resins through the substrate.

This study morphologically evaluated the effect of some current surface pre-treatments on ground dentin by SEM and related these morphological alterations to clinical implications.

Table 1: *Materials Used in this Study*

Materials	Composition	pH	Batch #	Manufacturer
Phosphoric acid	Etching gel silica-thickened 37% H_3PO_4	0.05	66561	Dentsply Ind Com Ltd, Brazil
Clearfil SE Bond (primer)	MDP, HEMA, Hydrophilic dimethacrylate, Camphorquinone, N,N-Diethanol p-toluidine, water, ethanol	2.0	00157[A]	Kuraray Co Ltd, Japan

Abbreviations: MDP: 10-methacryloyloxidecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate.

Table 2: *Treatment Groups*

Groups	Etchant	10% NaOCl
1	-	-
2	37% H_3PO_4 (15 seconds)	-
3	37% H_3PO_4 (15 seconds)	60 seconds
4	-	60 seconds
5	CSEB primer (20 seconds)	-
6	CSEB primer (20 seconds)	60 seconds

METHODS AND MATERIALS

Table 1 lists the materials, manufacturers, composition, pH and batch numbers for this study. Freshly extracted bovine lower incisors (n=30) obtained in a local abattoir and stored frozen for no longer than one month were used in this study. The teeth were ground flat on their labial surfaces on a water-irrigated grinding wheel using 320-grit silicone carbide paper (SiC) to expose the underlying dentin. Each was finished with 600-grit SiC to produce a standardized smear layer. In order to obtain rectangular shaped specimens, the roots and incisal portions were removed with a low-speed diamond saw under water, which was also used to notch the incisal and gingival edges to allow a guided fracture for longitudinal observation. The specimens were randomly divided into six groups of five each according to treatment (Table 2):

Group 1. Control Group, no treatment. The specimens were rinsed with air-water spray.

Group 2. The dentin surface was etched with 37% PA for 15 seconds and rinsed for 20 seconds with running water.

Group 3. The dentin surface was etched with 37% PA for 15 seconds, rinsed for 20 seconds with running water and a 10% NaOCl solution was lightly scrubbed on the etched surface for one minute using a disposable brush, then rinsed with running water for 20 seconds.

Group 4. A 10% NaOCl solution was lightly scrubbed on the smear layer covered dentin surface for one

minute using a disposable brush and rinsed with running water for 20 seconds.

Group 5. The self-etching primer Clearfil SE Bond (CSEB) was applied on a briefly air dried dentin surface using a disposable brush. It was left undisturbed for 20 seconds, gently air thinned, then immersed in an acetone-50% water solution for 30 seconds to remove the primer (although it is not a clinical procedure, since the primer is not rinsed in self-etching technique, it is a necessary step for the collagen fixation protocol for microscopic visualization), then rinsed for 20 seconds with running water.

Group 6. A control procedure for comparison with Group 3, the treatment was the same as for Group 5, but after the acetone-50% water solution immersion and rinsing with running water, a 10% NaOCl solution was applied to the dentin surface for one minute as in Groups 3 and 4.

The specimens were immediately immersed in a 2.5% glutaraldehyde in 0.1M sodium phosphate buffer at pH 7.2 for 12 hours at 4°C. After fixation, the specimens were rinsed with 15 ml of 0.2M sodium phosphate buffer at pH 7.2 for one hour in three baths of 20 minutes each. The specimens were dehydrated in ascending grades of ethanol in the following steps: 25% for 20 minutes, 50% for 20 minutes, 75% for 20 minutes, 95% for 30 minutes and 100% for 60 minutes. After the final ethanol step, the specimens were dried by immersion in hexamethyldisilazane (HMDS) for 10 minutes, placed on filter paper inside a covered glass vial and air dried at room temperature for 24 hours (Perdigão & others, 1995).

After drying, the specimens were fractured using a sharp chisel placed in a small notch. The specimens then provided two halves that were mounted on aluminum stubs with carbon tape, one to permit a cross-section analysis and the other for a longitudinal-section observation. The specimens were gold-sputter coated (SCD 050 Sputter Coater, BAL-TEC, Schaan, Liechtenstein) and observed by SEM (JEOL, JSM-5600LV, scanning electron microscope, Japan). The specimens were observed at several magnifications, and measurements were made directly on the microscope monitor using a multi-point measuring device.

RESULTS

Analyses of the specimens in cross-sections or longitudinal-sections showed a varied morphology that depended on the various dentin pre-treatments. These results are shown in Figures 1 through 10.

Group 1: The smear layer covered dentin observed in cross-section showed a surface completely covered by a crust of cutting debris that appeared to have little surface porosity; the orifices of the underlying tubules were completely obstructed by grinding debris that had a smoother texture than the intertubular dentin (Figure 1). The smear layer particles could not be observed individually, even in higher magnifications, since the surface appeared very compacted. The longitudinal sections revealed a thin smear layer ranging from 0.5 to 0.7 µm (Figure 2) in thickness.

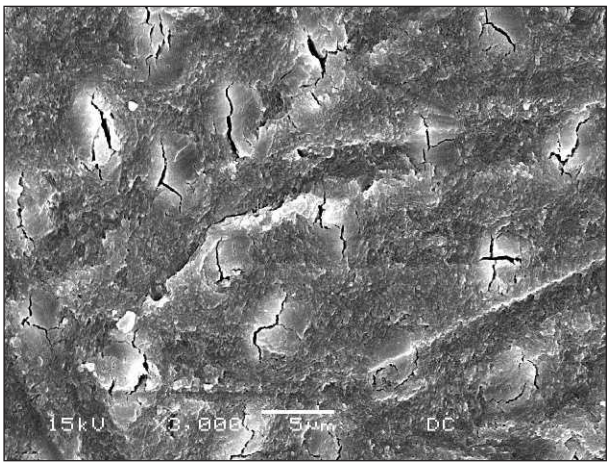

Figure 1. *SEM photomicrograph illustrating the smear layer-covered dentin. The orifices of the underlying tubules are covered by a compact crust of cutting debris (3,000x).*

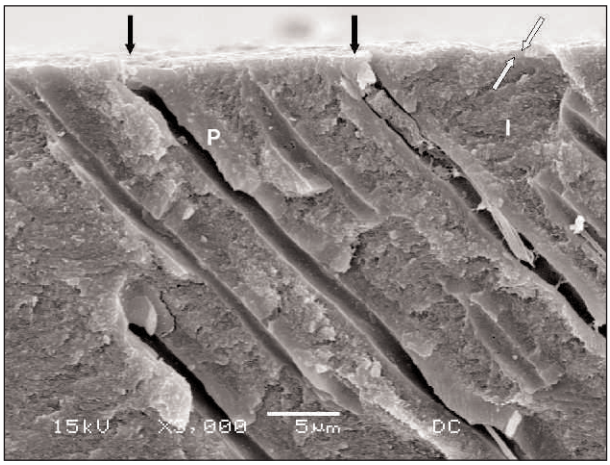

Figure 2. *SEM photomicrograph illustrating the smear layer-covered dentin in longitudinal-section. Note the thinness of smear layer (approx. 0.7 µm) indicated by the double white arrows, and the tubules occluded by smear plugs (black arrows). Peritubular dentin (P); intertubular dentin (I) (3,000x).*

Group 2: Treatment with PA resulted in a demineralized layer that appeared to have collapsed in its outermost part, producing a dense surface layer. The smear layer was completely removed and the dentin tubules opened. The diameter of the tubules ranged from 3 to 4 µm (Figure 3). A higher magnification image focused on the tubular orifice and revealed details of the peritubular collagen matrix, surrounding the tubular aperture as a dense interlacement of collagen fibers (Figure 4). Granular silica deposits from the PA thick-

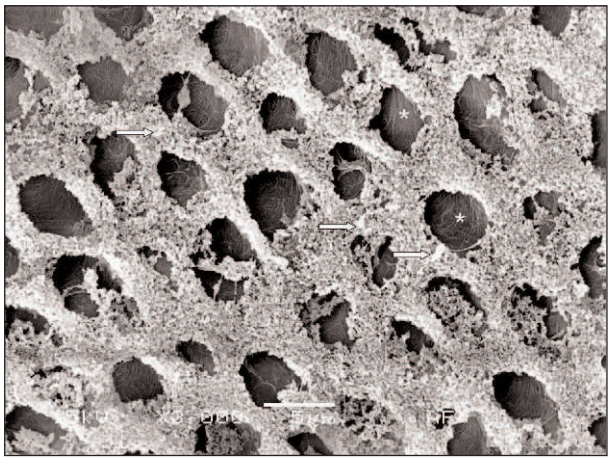

Figure 3. *SEM photomicrograph of the dentin surface treated with PA showing complete removal of smear layer, a dense collagen-rich surface and opened tubules. Observe the outer exposed peritubular fibers (*) and clusters of smaller silica particles (arrows). Silica deposits completely covered the collagen fibers, obscuring their fibrous appearance (3,000x).*

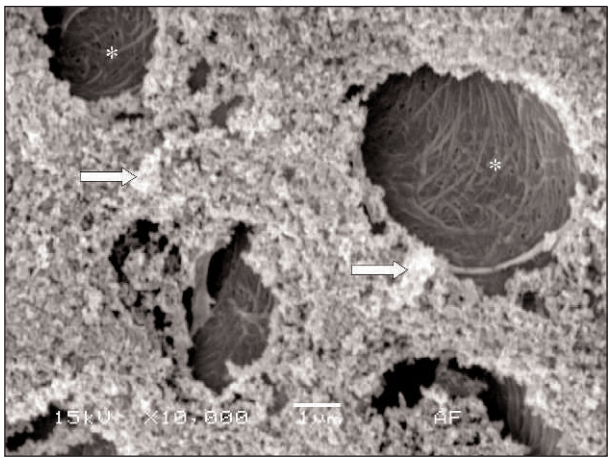

Figure 4. *Higher magnification SEM photomicrograph (10,000x) of the dentin surface treated with PA. Note the dense and appearing collapsed intertubular collagen-rich surface, covered with silica particles and clusters of smaller silica deposits (arrows). Note also inside the tubule orifice the peritubular collagen matrix as a dense tangle of fibers (*).*

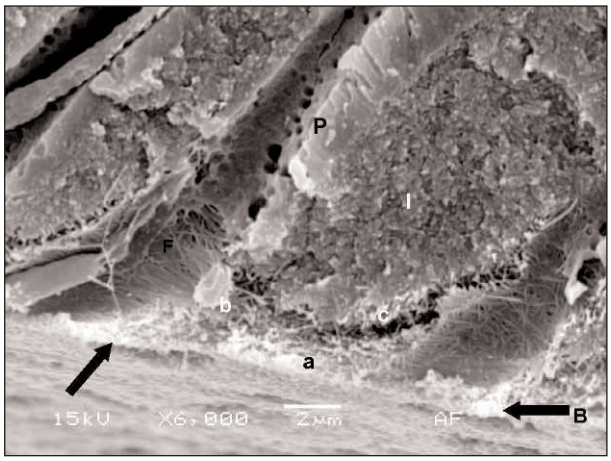

Figure 5. *SEM photomicrograph illustrating the dentin surface treated with PA in longitudinal-section view. Note the three distinct layers in the intertubular demineralized dentin zone: (a) the outmost layer of compacted fibers appearing to collapse onto the tubules lumen (arrow); (b) the more porous intermediate layer of separated collagen fibers, and (c) a zone of few collagen fibers and a hiatus observed close to unaffected intertubular dentin (I). Note also the tubules typical funnel shape and the circular arrangement of peritubular collagen fibers (B). Peritubular dentin (P) (6,000x).*

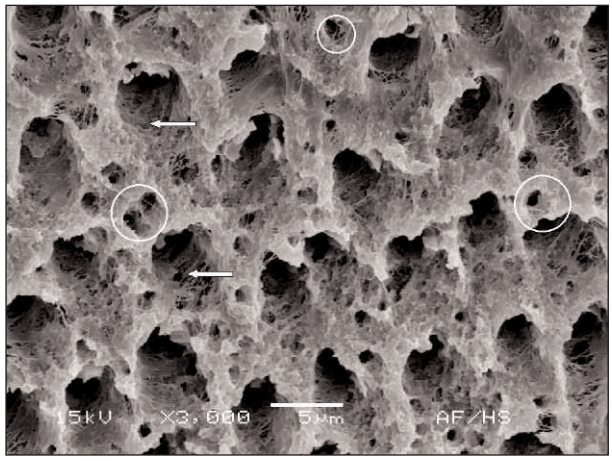

Figure 6. *SEM photomicrograph illustrating the dentin surface after acid etching with PA and deproteinization with NaOCl, in cross-section. Note the "moth-eaten" aspect and the lateral branches (circles). The tubules apertures are wider than for PA etched dentin in Figure 3. Some remnant collagen fibers from the peritubular dentin matrix could be observed (arrows) (3,000x).*

ener were also evident, covering the entire intertubular collagen surface (Figures 3 and 4). When the dentin tubules were observed longitudinally, three distinct, successive layers in the intertubular demineralized dentin zone were evident: an outermost layer of compacted collagen fibers with some remnants of the smear layer and acid etching (this outermost layer apparently collapsed onto the tubules lumen, narrowing its aperture); a second or intermediate layer of separated collagen fibers appearing as a more porous network and a third deeper layer presenting wide spaces and few collagen fibers (Figure 5). The tubules presented a funnel shape, which is attributed to a differential demineralization pattern between intertubular and peritubular dentin. The intertubular fibers presented a random distribution, while the peritubular fibers showed a circular pattern (Figure 5).

Group 3: When acid-etched dentin was followed by NaOCl deproteinization, the cross-section dentin surfaces developed an eroded or "moth-eaten" aspect. The intertubular collagen and all peritubular collagen fibers were completely removed and only a few rem-

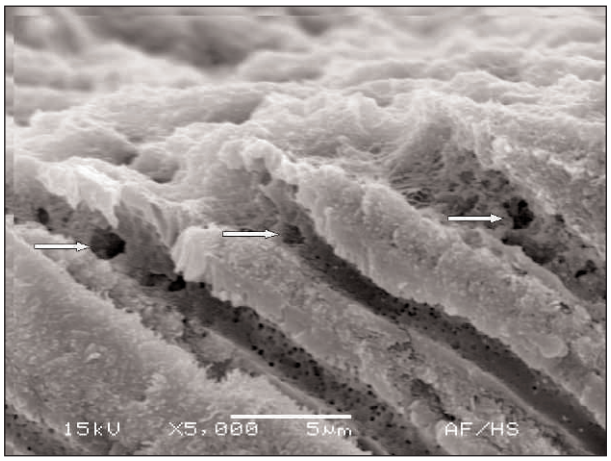

Figure 7. *SEM photomicrograph in longitudinal-section of etched and deproteinized dentin. Note the eroded surface and the funnel shape of the tubules. Several lateral branches are also evident in this image (arrows) (5,000x).*

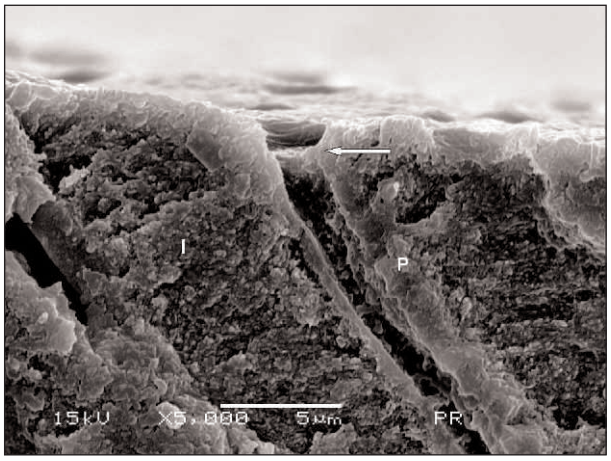

Figure 9. *SEM photomicrograph illustrating the dentin after treatment with CSEB- primer in longitudinal-section. The tubule does not present the typical funnel shape observed after PA treatment and is partially occluded by a smear plug (arrow). Peritubular dentin (P); Intertubular dentin (I) (5,000x).*

nant fibers could be seen in the peritubular collagen matrix. The tubular orifices were larger, ranging from 3 to 5 µm, and the funnel shape was more evident. The orifices of numerous lateral branches and a reduction in the intertubular dentin area were clearly observed (Figure 6). The longitudinal-sections showed funnel shaped tubules, some remnant collagen fibers from the peritubular collagen matrix and some lateral branches open in the lumen of the tubule near the dentin surface (Figure 7).

Group 4: When NaOCl was applied to the smear layer-covered dentin, there was no significant alteration in surface morphology, mainly when observed in cross-sections. The longitudinal-sections showed a light but noticeable reduction in the smear layer thick-

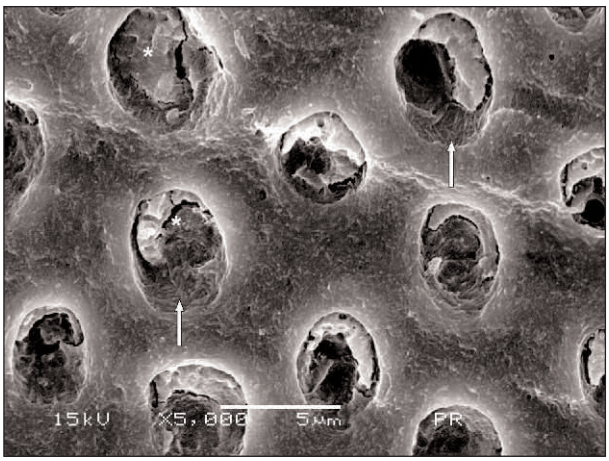

Figure 8. *SEM photomicrograph illustrating the cross-section dentin surface after treatment with CSEB-primer. Note the absence of smear layer debris and the partially dissolved smear plugs (*). Note also some exposed collagen fibers from the peritubular collagen matrix (arrows). Most of the tubules retained at least half of the circumferential peritubular dentin matrix (5,000x).*

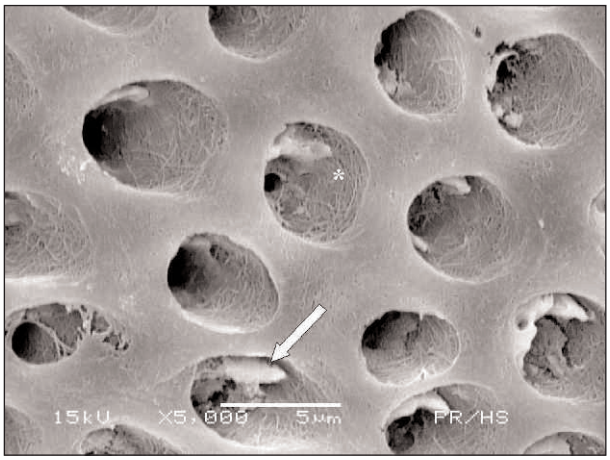

Figure 10. *SEM photomicrograph illustrating a cross-section of the dentin surface after treatment with CSEB-primer followed NaOCl deproteinization. Note that the near complete dissolution of smear plugs (arrow) which was more pronounced than for dentin only treated with CSEB-primer (Figure 8) and the very well defined tangle of peritubular collagen fibers (*) (5,000x).*

ness, ranging from 0.3 to 0.5 mm. As the images were similar to those in Group 1, they were not shown.

Group 5: Observation of the cross-section dentin surfaces showed that treatment with CSEB-primer was strong enough to dissolve the smear layer but only partially dissolved the smear plugs. The demineralization was superficial and did not show that noticeable difference between the intertubular or peritubular dentin pattern, showing parts of the mineralized peritubular dentin collar around the tubules lumen. The tubular orifices ranged from 2 to 4 µm. In addition to the partially demineralized smear plugs, some exposed

collagen fibers from the peritubular collagen matrix were also observed (Figure 8). The longitudinal-sections did not show the typical funnel shape as observed for PA specimens, with the tubules appearing partially occluded by the smear plugs (Figure 9). The lack of funneling was due to retention of the peritubular dentin matrix.

Group 6: When treatment with CSEB-primer was followed by NaOCl deproteinization, the major alterations that were noticed included further dissolution of the smear plugs, clearly observed in cross-sections. Well-defined peritubular collagen fibers could be observed through the tubular orifices (Figure 10). The longitudinal-sections observations confirmed the alterations found in the cross-sections.

DISCUSSION

There are two major problems related to dentin bonding; one relates to polymerization shrinkage of the restorative resin composites that challenge the bonded interface, and the other concerns the dentin itself as a bonding substrate. The high organic content and intrinsic wetness of dentin is due to its tubular structure and outward flow of the dentinal fluid. These factors have made dentin bonding more difficult to accomplish (Pashley & others, 1993; Pashley & Carvalho, 1997). Moreover, after cavity preparation, the dentin is covered by a layer of cutting debris with a very limited strength, low permeability (Reeder & others, 1978) and wettability (Attal, Asmussen & Degrange, 1994), the so-called smear layer. Thus, the only two options to achieve an acceptable bond strength to dentin include removal of the smear layer or development of agents that penetrate through the smear layer into the underlying dentin matrix (Pashley & Carvalho, 1997). It has been related that smear layer particles vary widely in their size, from 0.05 to 10 μm and the larger particles have a plate shape, which permits them to be compacted in a layer that is rarely thicker than 1 μm (Eick & others, 1970). In our study, the smear layer was observed in cross-sections as a compact crust, so that the particles could not be observed individually even in higher magnifications (Figure 1). The smear layer was thin, ranging from 0.5 to 0.7 μm, when observed longitudinally (Figure 2).

To remove the smear layer using the so-called total-etch technique, the most common agent used is PA in gel form and in concentrations that vary from 10% to 40%. The 37% silica-thickened PA used in this study completely removed the smear layer, funneled tubule orifices, increased intertubular porosity and produced a dense demineralized collagen-rich layer that appeared to have suffered a high degree of collapse and/or denaturation in its outermost part (Figures 3 through 5). Some possible reasons for this phenomenon include: (1) some unintended air drying after acid etching; once the water evaporates from the outer part of the collagen network, the forces of surface tension at the air-water interface tend to collapse the mesh, making the spaces around the collagen fibers smaller (Perdigão & others, 1996; Carvalho & others, 1996b), although care had been taken to keep the dentin visibly moist; (2) a surface shrinkage of the outermost exposed collagen fibers during the preparation processes for microscopy (Carvalho & others, 1996a); (3) the presence of the residual collagen phase of the smear layer, which resists acid etching and consists of small pieces of demineralized and/or denatured collagen that prevent the collagen network from being completely exposed. These particles could occlude the spaces of the outermost demineralized area, thereby making this surface more compacted and less permeable (Pashley & others, 1993) and (4) acid etching seems to induce conformational modifications on dentin collagen, characteristics of denaturation and fragmentation processes (Eliadess & others, 1997) that could also be responsible for the compacted outermost layer.

Granular silica deposits from the PA thickener are also present on the collagen fibers (Figures 3 and 4). It has been explained that these silica remnants could not be removed even with vigorous rinsing and these silica particles could influence demineralization depths and even the morphology of etched dentin, acting as a buffering agent (Perdigão & others, 1996). It has also been proposed that these adsorbed silica particles may act as a filler reinforcing agent for the unfilled bonding resins (Perdigão, 1995); however, distribution of these particles was observed to be quite random and non-uniform and could be one more obstacle to overcome. Furthermore, as these silica particles would not chemically bond to the infiltrating resin, they might increase nanoleakage and lead to failure over time.

When the acid-etched dentin surfaces were observed in cross-sections, the diameter of the tubules ranged from 3 to 4 μm (Figure 3), and in higher magnifications, the circular orientation of the collagen fibers of the peritubular matrix could be clearly seen (Figure 4). It is interesting to observe that the tubule orifices appear to be narrowed when the mineral portion is lost (Figures 3 and 4). The explanation for this narrowing is that the mineral portion that coats the collagen fibers prevents its expansion, and once it is removed by acid etching, the collagen expands and bulges out laterally, narrowing the tubule orifices (Arends & others, 1995; Van Meerbeek & others, 1992).

When the tubules were observed longitudinally, three successive layers could be distinguished: (1) a superficial compact layer, as mentioned above, composed of collapsed and/or denatured collagen and residual material that narrowed the tubules lumens, (2) a second or intermediate more fibrous and porous layer and (3) a deeper layer which was first described as a "hiatus," with wide

spaces and few collagen fibers separating the intermediate fibrous layer from the mineralized dentin, as previously described (Perdigão & others, 1995). It has been suggested that this deepest layer could be responsible for leakage within the hybrid layer due to incomplete resin infiltration through the collagen network, the so-called nanoleakage (Sano & others, 1995). However, these hiati have been recently suggested to be artifacts of desiccation procedures for SEM, and thus could not be regarded as a normal morphological entity (Agee & others, 2003). The tubules' funnel shape were also very evident after acid etching, which could be explained by the different demineralization pattern between the intertubular and more mineralized peritubular dentin. Peritubular dentin is 40% more mineralized than intertubular dentin and is formed inside the tubule with a progressive deposition that reduces the diameter of the tubule lumen over time (ten Cate, 1998).

This difference in the demineralization pattern may be explained by the structure of the two types of dentin, that is, the higher organic content of intertubular dentin helps to diminish acid diffusion through the mineral portion, while the higher mineral content of peritubular dentin allows acid diffusion from its surface into the tubules from which it is etched. This etching mechanism explains the porous matrix areas located around the tubules, suggesting they were etched from inside the tubules (Figure 5).

Resin infiltration into intertubular dentin is totally dependent on the porosity present after acid etching. Any collapse and/or denaturation of the outermost portion of the collagen matrix may reduce permeability of the demineralized zone to as little as 10% of its theoretical maximum value, in extreme cases (Pashley & others, 1993; Pashley & others, 2000). Thus, this concept of bonding to moist dentin must be considered far from perfect, as it is extremely technique sensitive, since it is completely dependent on ideal moisture and permeability, which are quite difficult to control and achieve (Tay & others, 1996; Frankenberger, Krämer & Petschelt, 2000b).

The main objective of collagen removal is to overcome technique sensitivity of the total-etch technique, facilitating access of the adhesive resins to a more permeable substrate with an increased surface area and less sensitivity to water content (Sakae & others, 1988; Tanaka & Nakai, 1993; Inaba & others, 1995), since hydroxyapatite is a high-energy substrate, while collagen has a low-energy surface (Attal & others, 1994). NaOCl solutions are widely used in various dental procedures based on their non-specific deproteinizing action and have been evaluated for their effects in dentin bonding procedures with various and controversial results (Guinnett, 1994; Wakabayashi & others, 1994; Uno & Finger, 1995; Boschian & others, 1997; Chersoni & others, 1997; Vargas & others, 1997; Vichi & others, 1997; Inai & others, 1998; Frankenberger & others, 2000a; Saboia & others, 2000; Toledano & others, 2000). The ideal NaOCl concentration and treatment time, as a dentin treatment agent, have also been determined and are considered to be 10-wt/vol% and 60 seconds, respectively (Tanaka & Nakai, 1993). This concentration and treatment time was used in this study.

The cross-section surfaces after acid etching followed by NaOCl deproteinization showed a completely eroded surface with a "moth-eaten" appearance (Figure 6), which was in agreement with previous findings (Inai & others, 1998; Perdigão & others, 1999). The use of NaOCl removed the totally demineralized collagen network and completely altered the dentin surface into a porous structure with multiple irregularities, which appears to be more compatible with bonding resins than acid etched dentin. Nevertheless, the studies of bonding to collagen-depleted dentin do not fully confirm this hypothesis (Guinnett, 1994; Uno & Finger, 1995; Vichi & others, 1997; Inai & others, 1998; Frankenberger & others, 2000a; Saboia & others, 2000; Toledano & others, 2000). Collagen removal increased the size of tubules' orifices and removed the lamina limitans, thereby exposing numerous lateral branches. One of the most interesting results of collagen depletion is that it allows an evaluation of the extent and aggressiveness of the total-etch technique after the usually recommended 15 seconds of etching time (Figures 6 and 7).

The effects of NaOCl on dentin composition have also been reported by some studies. Sakae and others (1988) reported that, from a crystallographic viewpoint, the crystals in NaOCl-treated dentin were similar to enamel crystals. Inaba and others (1995) found that dentin treatment with NaOCl solution promotes a mineral redistribution on the outermost surface. Marshall and others (2001), using nanomechanical measurements, showed a 75% reduction in the elastic modulus and hardness in dentin surface after collagen depletion with NaOCl. Recent studies (Lai & others, 2001; Yiu & others, 2002) have reported that the decline in bond strengths of some ethanol-based adhesive systems after NaOCl pretreatment is caused by a NaOCl oxidizing effect, which can be reversed using a reducing agent. These results indicate that further studies are necessary to evaluate the clinical efficacy and safety of this procedure.

The smear layer is considered the first obstacle to be surpassed for a reliable bonding to dentin. Acid etching has been shown to be quite effective at removing the smear layer, but this procedure removes only its mineral content. If the smear layer composition is similar to normal dentin, that is, 50% mineral phase and 30%

collagen phase, the residual collagen network resulting from acid etching would be enriched with these collagen remnants from the smear layer and might act as a barrier to the infiltration of bonding resins in the outermost collagen surface (Pashley & others, 1993; Spencer & others, 2001). Treatment of acid-etched dentin with NaOCl could remove this organic phase and enhance the diffusion of bonding resins. Apparently, treatment of unetched smear layers with NaOCl does not remove the organic matrix. When observed in cross-sections, the specimens' surfaces showed no significant changes when compared to smear layer covered dentin (Figure 1) and confirmed previous findings (Tanaka & Nakai, 1993; Prati & others, 1999). However, in longitudinal sections, a slight reduction in smear layer thickness was observed, which was probably due to surface collagen removal and primarily to the scrubbing action during NaOCl application. The application of NaOCl on the smear layer could also be advantageous due to its bacteriolytic effect, since bacteria in the smear layer on the ground dentin surface is one of the problems encountered clinically (Brännström, 1984). Ground dentin surface pre-treatment with NaOCl may be a useful method to improve bonding procedures with minimal risks of damage. Further studies could confirm this hypothesis.

The second strategy for bonding to dentin is based upon the use of non-rinsed acidic methacrylate monomers, the so-called self-etching primers. It has been suggested that they do not remove the smear layer completely, but infiltrate through the smear layer into smear plugs, fixing them at the tubule orifice (Perdigão, 1995). This strategy for adhesion is very attractive, because it constitutes a very simple method of preventing collapse of the collagen network, thus avoiding its unprotected exposure. Another advantage is that self-etching primers are designed to be used on dry dentin and require only one primer application with subsequent air-evaporation of the solvent (Gordan & others, 1997). Thus, this simplification in the bonding technique reduces moisture dependence by avoiding overwetting or overdrying, which can negatively influence adhesion and diminish the influence of regional differences in the substrate (Pereira & others, 1999). The first self-etching system was developed in Japan (Nishida & others, 1993), and recently, several self-etching systems have been introduced by many manufacturers. In this study, a recently introduced further simplified system was used, the Clearfil SE Bond (CSEB), which consists of one-bottle self-etching primer and a second bottle of adhesive resin.

CSEB-primer contains an unsaturated methacrylated phosphate ester, 10-methacryloyloxydecyl-dihydrogen phosphate (MDP) as the acidic monomer, combined with hydroxyethyl methacrylate, photoinitiator and accelerator into a single bottle. When this primer was applied on the dentin surface for the recommended 20 seconds and observed by SEM in cross-sections, it could be clearly observed that it was strong enough to dissolve the smear layer but it only partially dissolved the smear plugs. In addition to the partially dissolved smear plugs, the circumferential fibers that form the peritubular collagen matrix could be observed inside the tubules, which confirms demineralization through the smear layer, smear plugs and mineralized peritubular collagen matrix to the intact dentin (Figure 8). In longitudinal-sections, there was no noticeable difference in demineralization pattern between intertubular and peritubular dentin, confirmed by the absence of the typical funnel shape observed in PA-etched specimens (Figure 9). In a recent study (Tay & Pashley, 2001), CSEB-primer did not dissolve the smear layer in human dentin specimens which, as in our study, were polished with 600-grit SiC paper to produce standardized smear layers but were etched through it to demineralize the subsurface intact dentin to a depth of 0.5 µm. Another study related that the CSEB system produced a hybrid layer approximately 1-1.5 µm thick, with the same standardized smear layer produced with 600-grit SiC paper (Harada & others, 2000). The differences we found in our study were probably due to the minimal thickness of the smear layer produced during polishing of the specimens. Indeed, the smear layer thickness has been demonstrated to have a negative influence on bonding with self-etching systems (Koibuchi, Yasuda & Nakabayashi, 2001). Bovine dentin may be more easily etched by CSEB than human dentin. A study indicates that bovine dentin is less acid resistant than human dentin (Nakabayashi, Watanabe & Ikeda, 1995).

Treatment with CSEB-primer followed by NaOCl does not represent a current bonding pretreatment; however, it was designed for comparison with collagen depletion after PA treatment (Group 3). The deproteinization effect did not show any drastic surface alterations as observed with PA-etched and deproteinized specimens, which confirms the mild acid etching pattern of this primer. The most noticeable changes occurred in the partially dissolved smear plugs (Figure 10). One possible explanation for this phenomenon is twofold: the scrubbing motion during NaOCl application or the removal of organic compounds from the smear plugs, and it is probably a result of both. The presence of a well-defined peritubular collagen matrix, clearly observed through the tubules' orifices, leads to the conclusion that NaOCl action was superficial.

When the two current strategies for bonding to dentin were compared in terms of dentin surfaces after pretreatment, the differences were remarkable. The dentin surface modifications produced by PA (total-etch technique) appeared to be less permeable due to collapse of the outermost collagen layer. Moreover, the demineralization pattern appeared to be quite irregular, which

must make complete infiltration of the bonding resin through the demineralized collagen to the underlying mineralized dentin more difficult. When the collagen was removed by NaOCl deproteinization, the aggressiveness of PA etching was shown in the resultant mineralized dentin surface, which appeared to be very porous. The surface provided by the self-etching primer (CSEB-primer) rendered a more uniform and apparently less porous surface, which was confirmed when the collagen was depleted by NaOCl. Nevertheless, a recent study (Montes & others, 2003) employing these dentin pre-treatments in cavities restored with composite showed no significant difference in marginal quality irrespective of the bonding strategy, although a slight tendency for a better performance could be observed for the self-etching primer.

CONCLUSIONS

The pre-treatment of dentin surfaces for bonding still remains the initial obstacle to surpass to accomplish a safe, successful composite restoration technique. The pre-treatments evaluated by this study showed remarkable differences in the dentin substrate, which may influence the results in bonding procedures. Indeed, the surface promoted by treatment with PA (total-etch technique), which followed the most recommended protocol in concentration and treatment time (37%/15 seconds), seems to have over-etched dentin. Exposition of the collagen network brings sensitivity to the degree of dentin wetness and the possibility of weak monomer-collagen interaction, which may lead to nanoleakage and early bond degradation. Collagen depletion with NaOCl after PA treatment has not been considered a reliable procedure, since the results are not consistent for all adhesive systems; furthermore, it could be hypothesized that absence of the hybrid layer would put two rigid surfaces in contact (mineralized dentin and composite) and could, thereby, compromise the long-term bond. The ground dentin surface pre-treatment with NaOCl may minimally improve bonding procedures with minimal risks of damage. The surface provided by CSEB highlighted the uniform and mild acidic action of this primer; in fact, the advantages alleged to this bonding strategy are low technique sensitivity and adequate monomer-collagen interaction due to the simultaneous infiltration of the collagen network with resin up to the same depth of demineralization, although there is insufficient long-term clinical research to confirm this hypothesis. Further in *vitro* and *in vivo* studies should be carried out to evaluate the effects of these dentin pre-treatments for dentin bonding.

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Influence of Different Beverages on the Microhardness and Surface Roughness of Resin Composites

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Clinical Relevance

The outcomes of the reported study reveal that certain types of beverages can yield significant alterations to the mechanical properties of resin composites.

SUMMARY

This study assessed the influence of different beverages on the microhardness and surface roughness of microfilled (A110, 3M/ESPE), hybrid (Z250 3M/ESPE) and flowable (Flow, 3M/ESPE) resin composites, over time. Twenty-four disc-shaped specimens (10 mm; 2-mm thick) of each resin composite were fabricated, thereby forming three groups (n=24). Knoop microhardness and surface roughness (Ra) were analyzed at predetermined evaluation periods: 24 hours, and 7, 30 and 60 days after specimens fabrication. The 24-hour measurements were recorded after storage in artificial saliva. Next, each group (n=24) was divided into four subgroups (n=6) according to the test beverages: Coca-Cola, sugar cane spirit, coffee and artificial saliva (control). Control specimens were kept in saliva throughout the experiment (60 days). For experimental specimens, a 60-day testing cycle was carried out: specimens were initially stored in saliva for four hours, then submitted to a five-minute immersion in the beverages (Coca-Cola, sugar cane spirit, coffee) intercalated by immersions in saliva three times daily. Microhardness/roughness measurements were done at 7-, 30- and 60-day intervals. Data were submitted to three-way ANOVA and Scheffée test ($p<0.05$). It was observed that the tested beverages somewhat altered ($p<0.05$) the composites' microhardness and/or surface roughness. Knoop microhardness—for all resin composites, microhardness remained stable up to the 30-day record, decreasing significantly at the 60-day evaluation. During the interaction *beverage X evaluation period*, it was observed that the microhardness of materials immersed in coffee and Coca-Cola remained stable up to the seven-day measurement, showing a decrease at the 30-day record and a more accentuated drop at the

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60-day evaluation ($p<0.05$). Specimens immersed in sugar cane spirit exhibited no significant change in microhardness up to the seven-day measurement, increasing significantly at the 30-day record and later decreasing at the 60-day evaluation. Surface Roughness—For all resin composites, surface roughness increased at the seven-day measurement, while decreasing at the 30-day record and even more at the 60-day record. In the interaction *beverage X evaluation period*, the surface roughness of specimens immersed in test beverages increased at the seven-day measurement, showing a gradual decrease at the following records (30- and 60-day evaluations). The findings of the reported research disclosed that all beverages altered, to some degree, the microhardness and/or surface roughness of the tested resin composites. The alterations' effects ranged from slightly adverse to a markedly negative impact on the composites' microhardness and surface roughness, depending on the characteristics of the materials, type of beverage and the evaluated period. Generally, the greater number of immersions in beverages resulted in a more accentuated impact on the resins' properties.

INTRODUCTION

The outstanding development of adhesive dentistry coupled with strong esthetic demands from patients has resulted in an increasingly widespread use of resin composites in dental practice. However, despite the notable improvement in their composition and characteristics, restorative materials placed in the oral environment are subject to a great number of adverse conditions that challenge their integrity and longevity over time. Consumption of certain beverages, such as coffee, soft drinks, alcoholic beverages, tea, red wine and even water or fluoride may affect the esthetic and physical properties (microhardness, surface roughness, translucency) of the composites, thereby undermining the quality of restorations (Chan, Fuller & Hormati, 1980; Dietschi & others, 1994; Lim & others, 2001; Wiltshire & Labuschagne, 1990). The effects of these beverages may be stronger, depending on their intrinsic features, such as the chemical composition of the restorative materials (Ameye, Lambrechts & Vanherle, 1981) or external features, such as finishing/polishing of the restoration (Dietschi & others, 1994; Stober, Gilde & Lenz, 2001). Moreover, the impact of a beverage on the properties of composites may be directly related to the amount and frequency of its intake.

The chemicals in beverage formulations can lead to wear and surface degradation of composite restorations, resulting in unaesthetic external pigmentation, such as the stains produced by coffee on the surface of resin restorations (Dietschi & others, 1994; Chan & others, 1980). It has been reported (Wiltshire & Labuschagne, 1990) that coffee and red wine alter the color stability of resins to a greater extent than Coca-Cola, depending on the type of restorative material. The water sorption ability of resin composites may also be influenced by their chemical composition, which represents an important feature of the color stability and surface integrity of composites (Dietschi & others, 1994).

Earlier studies (Söderholm & others, 1984; Milleding & others, 1998) have also shown that an aqueous environment (such as the oral environment) can interfere with the characteristics of resin composites and even lead to hydrolytic degradation over time. However, it has been observed that salivary pellicle formed on the surface of restorations can act as a protective barrier and prevent (or at least minimize) the deleterious effects of some aggressive agents acting on restorative materials (Hannig & Balz, 1999).

Organic acids; acetic, propionic and lactic acids, present in the formulation of certain beverages, have been demonstrated to decrease the microhardness of resin composites (Consani & Góes, 1998). Furthermore, acidic beverages, such as soft drinks, tea, beer and orange juice may start or accelerate the development of erosive lesions on tooth surfaces, thus favoring the initiation and/or progression of the carious process (Birkhed, 1984; Bastos & Freitas, 1991; Larsen, 1991). A contemporary study (Sarrett, Coletti & Peluso, 2000) has shown that, due to its low pH, ethanol can produce erosion and alter some properties of composites, as well. For instance, wear of resin composites increases with the increase in ethanol percentage in the solution.

This study was conducted to assess the effect of microhardness and surface roughness of microfilled, hybrid and flowable resin composites, over time, of beverages widely consumed in Brazil.

METHODS AND MATERIALS

Three types of resin composites were selected for the study: a microfilled (Filtek A110, 3M/ESPE, St Paul, MN, USA), a hybrid (Filtek Z250 3M/ESPE) and a microhybrid flowable (Filtek Flow 3M/ESPE). The compositions and specifications of the tested resin composites are displayed in Table 1.

A stainless steel mold (10-mm in diameter and 2-mm thick) was used for specimen preparation (Figure 1A). The resin composites were inserted into the mold cavity in a single increment and covered with an acetate strip. To compact the material and prevent void and bubble formation, a microscopic slide with a 1,650g weight was placed over the resin/mold assembly to allow for fabrication of the specimens with smooth, highly flat surfaces. After 30 seconds, the weight was removed and

the resin composite increment light-cured through the glass slide for 40 seconds using a light-curing unit with a 450 mW/cm² output at the tip (XL 3000, 3M/ESPE) (Figure 1A). A total of 72 disc-shaped specimens were fabricated and three groups of equal size (n=24) were formed, each corresponding to one of the tested resin composites. Microhardness and surface roughness measurements of the acetate-covered surface were performed at predetermined evaluation periods: 24 hours, and 7, 30 and 60 days after specimen fabrication.

Knoop microhardness measurements were done using a micro-indentation tester (Microhardness Testers HMV-2, Shimadzu Corporation, Kyoto, Japan) with a 100 gf load applied for 30 seconds. The specimens were individually fixed in a clamping apparatus and positioned in such a way that the test surface was kept perpendicular to the tester tip. In each disc, three indentations equally spaced over a circle and not closer than 1 mm to the adjacent indentations or the margin of the specimen were taken and the average calculated. Then, at all evaluation periods, a microhardness mean value was obtained for each of the tested materials.

Surface roughness was determined using a roughness meter (Prazis RUG 03 Digital Roughness Meter, ARO-Argentina). Each specimen was individually fixed in a clamping apparatus and the meter's needle was situated at the extremity of the equipment's arm. The needle was then positioned on the specimen surface and programmed to trace a 4.8 mm course, providing the first measure in µm. Two additional measurements were accomplished by rotating the disk 90° and the average (Ra) was obtained from the three values. Then, at all evaluation periods, a surface roughness mean value was obtained for each of the tested materials.

Table 1: *Tested Resin Composites*

Resin Composites	Type	Principle Ingredients
Filtek A110	Light-curing, microfilled resin composite	BIS-GMA, TEGDMA Silica
Filtek Z250	Light-curing, hybrid resin composite oxide, Zircon/ Silica	BIS-GMA, UDMA, BIS-EMA, Aluminum
Filtek Flow	Light-curing, microhybrid flowable resin composite	BIS-GMA, UDMA, BIS-EMA, Zircon/ Silica

Table 2: *Tested Beverages*

Beverages/Solutions	Principle Ingredients
Coffee Serra da Grama	100% Arabian Coffee
Sugar Cane Spirit Cachaça Janaina	Sugar cane alcohol and sugar (Alcoholic graduation 39.00% vol)
Coca-Cola	Carbonated water, sugar, cola nut extract, caffeine, coloring, caramel
Artificial Saliva	K_2HPO_4, Sorbitol 70%, NaF, KCl, NaCl, $MgCl_2 \cdot 6H_2O$, $CaCl_2 \cdot 2H_2O$, Nipagin, Sodium benzoate, Hidroxyetilcelulose, H_2O

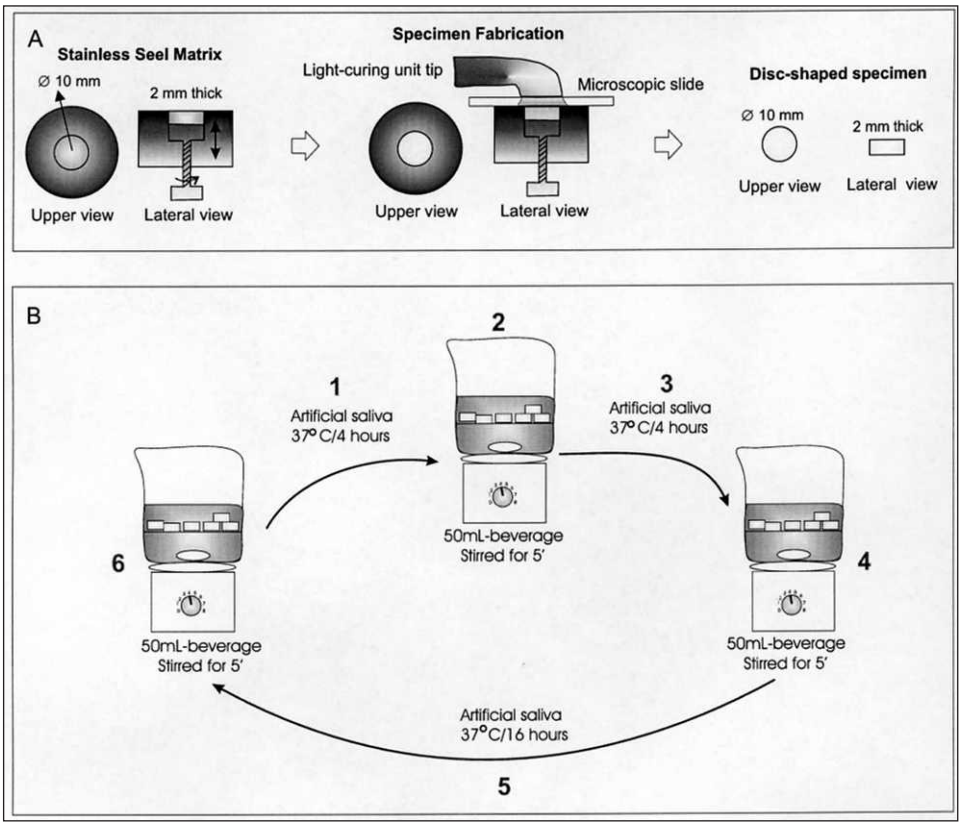

Figure 1: *Schematic illustration of specimen preparation and immersion testing cycle.*

The 24-hour measurements were recorded after the specimens were stored in artificial saliva. Next, each group (A110, Z250, Flow) was randomly divided into four subgroups of six specimens, according to beverage (Table 2): Coca-Cola (Cia de Bebidas Ipiranga, Ribeirão

Preto SP, Brazil), sugar cane spirit (Cachaça Janaina, Indústrias de Bebidas Pirassununga LTDA, Pirassununga SP, Brazil), coffee (Serra da Grama, Torrefação e Moagem de Café Serra da Grama LTDA, São Sebastião da Grama SP, Brazil) and artificial saliva (control) (Laboratory of Pharmaceutical Sciences, Pharmaceutical Sciences School, University of São Paulo [USP], Brazil). The control specimens were kept in artificial saliva throughout the experimental phase (60 days), with daily exchange of the solution. For the experimental specimens, a 60-day immersion testing cycle was carried out with the test beverages (Coca-Cola, sugar cane spirit and coffee). The following protocol was adopted to simulate high beverage intake: specimens were initially stored in artificial saliva for four hours, then immersed in 50 mL of the beverage, stirred for five minutes and returned to artificial saliva. After four hours in saliva, the specimens were immersed again in the beverage for five minutes under stirring, returned to the artificial saliva for an additional 16 hours, then submitted to a final five-minute immersion in the beverage, thus, ending the cycle (Figure 1B). Microhardness and surface roughness were re-analyzed at the remaining evaluation periods (7, 30 and 60 days).

Table 3: *Microhardness (KHN) Averages and Standard Deviations (±) of the Tested Resin Composites, Beverages and Evaluation Periods*

Beverages	Material	24 Hours	7 Days	30 Days	60 Days
Artificial Saliva (Control)	Z250	51.73 (2.49)	55.43 (2.32)	36.77 (5.64)	31.60 (1.28)
	A110	30.98 (2.25)	30.00 (2.21)	22.32 (2.27)	22.18 (0.89)
	Flow	19.92 (1.58)	21.87 (0.91)	16.77 (0.88)	17.73 (0.30)
Coca-Cola	Z250	49.30 (5.45)	52.52 (2.55)	37.85 (6.07)	36.33 (1.73)
	A110	28.97 (4.04)	29.87 (2.15)	33.68 (1.86)	21.75 (0.27)
	Flow	19.83 (3.48)	18.70 (1.42)	14.73 (1.85)	13.53 (0.92)
Sugar Cane Spirit	Z250	58.17 (4.41)	55.57 (6.76)	70.50 (3.79)	40.82 (3.58)
	A110	37.52 (2.58)	31.93 (2.69)	39.28 (2.48)	24.32 (1.16)
	Flow	24.08 (1.84)	24.92 (1.26)	28.07 (1.13)	19.62 (1.21)
Coffee	Z250	53.37 (8.29)	52.85 (3.83)	50.45 (2.87)	32.37 (2.48)
	A110	29.37 (3.15)	31.12 (2.65)	33.35 (2.42)	21.38 (2.21)
	Flow	20.50 (2.06)	24.13 (4.80)	22.40 (1.76)	15.32 (1.46)

Table 4: *Surface Roughness (mm) Averages and Standard Deviations (±) of the Tested Resin Composites, Beverages and Evaluation Periods*

Beverages	Material	24 Hours	7 Days	30 Days	60 Days
Artificial Saliva (Control)	Z250	0.83 (0.18)	0.35 (0.26)	0.24 (0.07)	0.12 (0.16)
	A110	0.81 (0.08)	1.04 (0.17)	0.21 (0.07)	0.23 (0.07)
	Flow	0.70 (0.05)	0.15 (0.01)	0.17 (0.03)	0.19 (0.06)
Coca-Cola	Z250	0.64 (0.21)	1.60 (0.74)	0.80 (0.15)	0.15 (0.04)
	A110	0.72 (0.09)	1.44 (0.41)	1.16 (0.42)	0.15 (0.05)
	Flow	0.63 (0.08)	1.33 (0.21)	0.58 (0.11)	0.16 (0.07)
Sugar Cane Spirit	Z250	0.53 (0.06)	0.72 (0.22)	0.37 (0.19)	0.25 (0.09)
	A110	0.67 (0.04)	0.90 (0.19)	0.23 (0.09)	0.12 (0.03)
	Flow	0.66 (0.08)	0.76 (0.11)	0.33 (0.15)	0.18 (0.10)
Coffee	Z250	0.28 (0.21)	1.41 (0.23)	0.25 (0.10)	0.23 (0.13)
	A110	0.34 (0.17)	1.28 (0.15)	0.36 (0.18)	0.19 (0.08)
	Flow	0.36 (0.17)	1.78 (0.29)	0.37 (0.12)	0.31 (0.06)

The coffee solution was standardized by preparing it with 5.5g of ground coffee in 100 ml of distilled boiling water. Special care was used while dispensing the Coca-Cola. In order to maintain an acceptable level of carbonic gas, a new bottle was opened for every five-minute immersion into the beverage; the unused drink that remained in the bottle was discarded. Artificial saliva was kept at 37 ± 1°C. The beverages were utilized at their usual consumption temperatures: coffee at approximately 60°C, Coca-Cola at approximately 4°C and sugar cane spirit at approximately 25°C.

Sample distribution and homogeneity were analyzed. Since a normal, homogeneous distribution was observed, data were submitted to three-way ANOVA parametric test. Multiple comparisons were done by Scheffée statistical test at 0.05 significance level.

RESULTS

The analysis of data revealed that the tested beverages altered ($p<0.05$) the microhardness and/or surface roughness of the resin composites. However, no correlation was found between roughness and microhardness (Tables 3 and 4).

Microhardness

With respect to the resin composites evaluated, regardless of the test beverages and evaluation periods, Z250 yielded the highest microhardness means ($p<0.05$), followed by A110 and Flow. Comparing the beverages

with the control (artificial saliva), sugar cane spirit and coffee were observed to increase the composites' microhardness ($p<0.05$), whereas Coca-Cola did not affect it. Considering the evaluation periods, excluding the other variables, microhardness remained stable up to the seven-day immersion, decreased after 30 days and even more after 60 days (Table 3).

In the interaction between the resin composite and the evaluation period, it was observed that for all tested materials, the microhardness remained stable up to the 30-day evaluation and decreased significantly at the 60-day evaluation. Analyzing the interaction between the beverage and the evaluation period, the microhardness of materials immersed in coffee and Coca-Cola remained stable up to the seven-day measurement, showing a decrease at the 30-day evaluation and a more accentuated drop at the 60-day evaluation ($p<0.05$). Specimens immersed in sugar cane spirit exhibited no significant change in microhardness up to the seven-day measurement, increasing significantly at the 30-day evaluation and later decreasing at the 60-day evaluation (Table 3 and Figure 2).

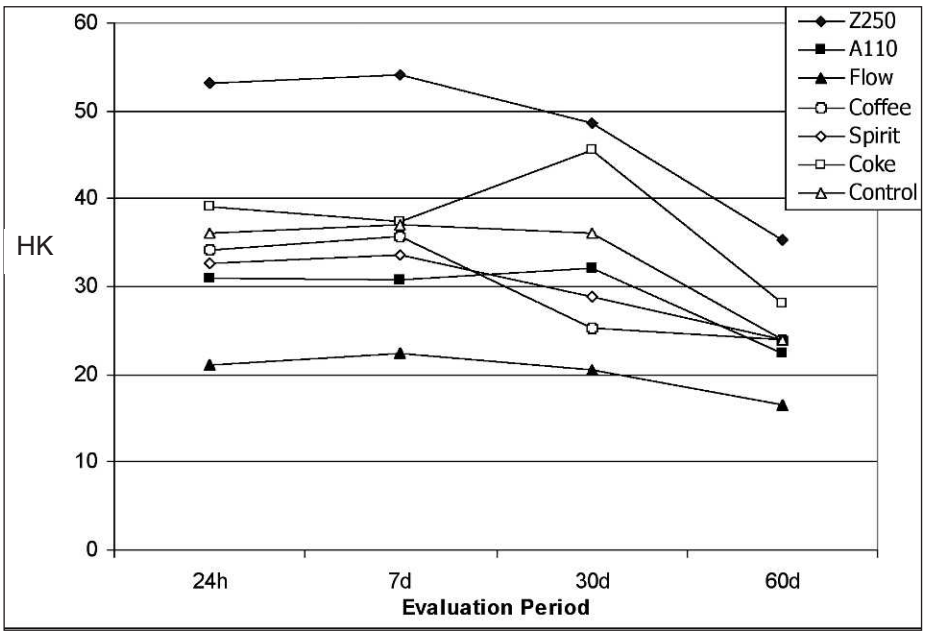

Figure 2: *Graphic representation of microhardness means for the interaction* resin composite *X* beverage*, over time.*

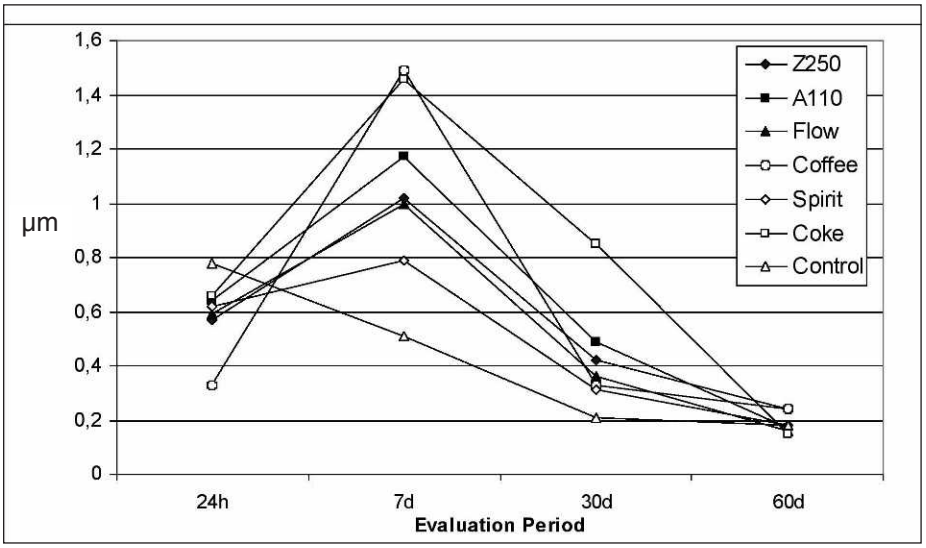

Figure 3: *Graphic representation of surface roughness means for the interaction* resin composite *X* beverage*, over time.*

Surface Roughness

Considering the resin composites, irrespective of the test beverages and evaluation periods, the highest roughness values were recorded for A110, which was statistically different from the other tested resins ($p<0.05$). Z250 and Flow yielded statistically similar results ($p>0.05$). Comparing the beverages with the control group (artificial saliva), it was found that sugar cane spirit did not alter the composite roughness, while immersion in Coca-Cola and coffee resulted in a significant increase in surface roughness ($p<0.05$). With respect to the evaluation periods, the analysis of the results showed that surface roughness increased at the seven-day measurement and decreased significantly at the following evaluations (30- and 60-day evaluations) (Table 4).

In the interaction between the resin composite and evaluation period, for all the tested materials, the surface roughness was observed to increase at the seven-day measurement, decrease at the 30-day evaluation and decrease even more at the 60-day evaluation. However, analyzing the interaction between the beverage and evaluation period, the surface roughness of specimens immersed in Coca-Cola, coffee and sugar cane spirit increased at the seven-day measurement and showed a gradual decrease at the 30- and 60-day evaluations (Table 4 and Figure 3).

DISCUSSION

The different behaviors of the tested resin composites might be ascribed to disparities in composition and filler distribution in their matrixes. Factors that affect the characteristics of composites include resin monomer, filler and coupling agents. The filler content has been correlated with depth of polymerization, color stability, hardness, compressive strength and stiffness. Increased filler loading has been shown to result in lower water absorption, thus leading to less surface degradation (Kim, Ong & Okuno, 2002; Wiltshire & Labuschagne, 1990). This could possibly explain why Z250 hybrid resin composite yielded the best overall results. Regardless, alterations may occur due to the natural, ongoing degradation of the material surface in an aqueous environment (Milleding & others, 1998). Filler/matrix cracking caused by water absorption and hydrolytic degradation of the filler surface is known to alter the mechanical properties of composites (Sarrett & others, 2000).

An earlier study of the effect of Coca-Cola on human enamel (Maupomé & others, 1998) found that the cola drink yielded significant alteration to enamel hardness, regardless of whether or not the immersion was accompanied by stirring. The authors indicated that such alteration might be attributed to carbonic acid, which is present in the beverage formulation and that its influence was further increased by the number of immersions, simulating a high number of intakes. Moreover, it has been demonstrated that, even though organic acids do not affect roughness, they cause a remarkable decrease in resin composite hardness (Consani & Góes, 1998). Therefore, it may be speculated that, in this study, the carbonic acid in Coca-Cola may have affected the microhardness of resins, particularly Z250 and A110.

Although this study did not aim to assess color alterations of the tested resins, specimens immersed in coffee, mainly those fabricated from Z250 and Flow resin composites, were strongly stained, which was evidenced after the second day of the immersion testing cycle.

Asmussen (1994) and Ferracane and Marker (1992) have demonstrated a significant decrease in the mechanical properties of composites exposed to ethanol; alcohol was thought to act as plasticizer of the polymer matrix. Cracking within the resin matrix and at the filler/matrix interface was observed by SEM after exposure to ethanol (Ferracane & Marker, 1992). It has been suggested (Sarrett & others, 2000) that plasticization of the resin matrix could aid in clinical wear resistance by making the composite more ductile and less prone to chipping. Ethanol could also have an adverse effect by making the composite too soft to withstand abrasion caused by mastication. It may be speculated that softening of the resin matrix would favor dislodgment of filler particles from the matrix upon stirring, thereby allowing for the formation of a roughened surface, which was observed in this study.

It has been experimentally demonstrated that Bis-GMA copolymer is highly susceptible to chemical softening, with a broad increasing range of solubility parameters. The extent of softening of Bis-GMA copolymer resulting from soaking in different chemicals including ethanol has been reported (Wu & McKinney, 1982). The authors (Wu & McKinney, 1982) reported that only 35% of the original hardness was retained after two weeks of immersion in ethanol concentrations above 50% volume in water, while a slight increase in hardness was observed for specimens immersed in water only. Nevertheless, it is important to highlight that most studies use an ethanol concentration (alcoholic graduation usually above 50% vol) higher than that found in most commercially available alcoholic beverages. Therefore, it is not known whether the alcohol in typical alcoholic beverages has a negative effect on certain mechanical properties of composite materials (Sarrett & others, 2000). Data from this study revealed that, even though tested sugar cane spirit (alcoholic graduation 39.00% vol) showed an overall behavior different from that of artificial saliva during the immersion testing cycle, the final microhardness means recorded for both the beverage and control solution were comparable.

The outcome of the reported research revealed that coffee yielded a remarkable increase in roughness after seven days and that the final surface roughness of coffee specimens was significantly higher than that recorded for the other beverages. Theoretically, these results could be derived from the fact that the coffee in this study was utilized at its usual consumption temperature (approximately 60°C); whereas, most of the studies that evaluated the effect of coffee on either enamel or composite surface utilized the solution at 37°C (Stober & others, 2001). Although there is no controlled study to support the suggestion, it may be speculated that extended immersion in a high-temperature solution may lead to significant alterations in certain properties of composites, such as the increase in surface roughness.

The literature has widely demonstrated the potential deleterious/adverse effects of certain beverages on enamel and dentin surfaces of both permanent and primary teeth. However, the number of reports on whether (and how) the beverages affect restorative materials, particularly resin composites, is remarkably scarce. The lack of studies focused on investigating the same properties; drinks and materials are definitely a hindrance to stating a reliable comparison

between the outcomes of the conducted research and available data.

In view of the outstanding appeal for adhesive dentistry and the growing demand for esthetic restorative materials in dental practice, further *in vitro* investigations and clinical trials are required to investigate the potential alterations of widely consumed beverages on resin composites. Studies that focus on the real impact of beverages' contents on certain properties of composites, such as microhardness and surface roughness, are of paramount importance to prompt improvements in the materials' formulation and thus, with some degree of reliability, the quality and longevity of the restorations over time.

CONCLUSIONS

The findings of this research disclosed that all beverages somewhat altered the microhardness and/or surface roughness of the tested resin composites. The effects ranged from slight to a markedly negative impact on the composite microhardness and surface roughness, depending on the characteristics of the materials, the type of beverage and the evaluated period. Generally, greater numbers of immersions in beverages resulted in a more accentuated impact on the resins' properties.

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The Influence of Fatigue Loading on the Quality of the Cement Layer and Retention Strength of Carbon Fiber Post-Resin Composite Core Restorations

HPB Bolhuis • AJ de Gee • AJ Feilzer

Clinical Relevance

For endodontic quartz coated carbon fiber posts that are used to support an adhesively bonded resin-composite core, adhesive resin-composite cements are advised.

SUMMARY

Clinical studies have shown that endodontically treated teeth restored with short posts or deficient ferrules show a high failure risk. This study evaluated the influence of fatigue loading on the quality of the cement layer between prefabricated quartz coated carbon fiber posts with restricted length and the root canal wall in maxillary premolars. Two adhesive resin composite cements, chemical-cured Panavia 21 (Group 1) and dual-cured RelyX-ARC (Group 2), and one resin-modified glass-ionomer cement, chemical-cured RelyX (Group 3), Δ were selected for this study. Post-and-core restorations were made on single-rooted human maxillary premolars from which the coronal sections were removed at the level of the proximal cemento-enamel junction (CEJ). Following endodontic treatment, a post-and-core restoration with 6-mm post length was prepared for each tooth. The posts were directly cemented into the root canal and, after applying an adhesive (Clearfil Photo Bond), they were built up with a core build-up composite (Clearfil Photo Core). For each group (n=8), half of the specimens were exposed to fatigue loading (10^6 load cycles) almost perpendicular to the axial axis (85°), while the other half were used as the control. Three parallel, transverse root sections, 1.5-mm thick, were cut from each specimen at the apical, medial and coronal location. These sections were examined by Scanning Electron Microscopy (SEM) to evaluate the integrity of the cement layer, while the retention strength of the cemented post sections was determined with the push-out test. The multivariate results of MANOVA showed that the condition main effect (fatigue or control) was not significant (p=0.059); the two other main effects, type of cement and section location, were significant (p=0.001 and p=0.008). For both the push-out strength and SEM evaluation of the cement layer integrity, the results significantly improved from RelyX to RelyX-ARC to Panavia 21 and also from apical to coronal.

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INTRODUCTION

In the past decade, adhesive luting cements that bond to root dentin have become available, together with a wide range of post and core materials. These developments revive discussion about the most effective way of restoring endodontically treated teeth. In particular, premolars, for which the anatomy is not compatible with the application of long endodontic posts (Carlsen, 1987; Raiden & others, 1999; Simon & others, 2000), could benefit from a new approach. The available length may also be limited, as after partial removal of the root canal filling, the remaining apical part of the restoration should be at least 5-6 mm (Mattison & others, 1984; Metzger & others, 2000; Nixon, Vertucci & Swindle, 1991). The use of short posts with new adhesive techniques may provide sufficient strength and preserve maximum sealing of the apical root canal filling (Abramovitz & others, 2001). However, rigid short posts can induce unfavorable stress distributions in the surrounding dentin (Holmes, Diaz-Arnold & Leary, 1996), which can cause root fractures. Therefore, premolar posts with a flexibility closer to that of tooth structure, such as quartz coated carbon fiber posts, are preferred (Martinez-Insua & others, 1998).

Besides the relevance of the flexibility of the post, the type of resin cement will be an important factor for clinical success. When cements are injected into the root canal, the anaerobic environment may, in some cases, complicate the use of resin cements. In addition, the unfavorable C-factor (Davidson, de Gee & Feilzer, 1984; Feilzer, de Gee & Davidson, 1987) for the root canal, which results in high setting shrinkage stresses, can lead to loss of marginal integrity, which is different for various types of cements. Probably, chemical-cured resin composite cements will perform better than the more abruptly setting light-initiated dual-cured composite cements (Feilzer, de Gee & Davidson, 1993). Also, mechanical properties and adhesive strength to both the post and root canal wall will generally be different between cements, for example, resin-modified glass-ionomer cements differ from resin composite cements in this respect (Almuammar, Schulman & Salama, 2001).

In many studies, post-and-core restorations are evaluated by determining fracture resistance with the cemented crown on top. However, forces applied until the restoration or the tooth fractures are frequently superior to those responsible for clinical failure. For this reason, fatigue tests were introduced for evaluating post-and-core restorations (Huysmans & others, 1992; Isidor & Brondum, 1992; Libman & Nicholls, 1995). Fatigue loading is defined as the breaking or fracturing of a material caused by cyclic or repeatedly applied loads below the yield limit, usually noticed initially as minute cracks followed by tearing and rupture (Terms, 1994).

This study evaluated the influence of fatigue loading on the quality of the cement layer between quartz coated carbon fiber posts and the root canal wall for three types of luting cements; a chemical and dual-cured resin composite cement and a resin-modified glass-ionomer cement.

METHODS AND MATERIALS

General

Two adhesive resin composite cements, chemical-cured Panavia 21 (Kuraray, Osaka, Japan) and dual-cured RelyX-ARC (3M ESPE Dental Products, St Paul, MN, USA), and one resin-modified glass-ionomer cement, chemical-cured RelyX (3M ESPE), were selected for this study. Post-and-core restorations were made on single-rooted, human maxillary premolars. The posts used were Aestheti-Plus Quartz coated Carbon fiber posts (RTD, St Egreve, France) and the core build-up material was light-cured resin composite Clearfil Photo Core (Kuraray). Half of the restorations were exposed to fatigue loading, while the other half functioned as control. The evaluation was carried out by means of SEM examination and the push-out test.

Preparation of the Teeth

Twenty-four freshly extracted caries free human maxillary single rooted premolars were used for this study. The coronal section was cut from the root at the proximal cemento-enamel-junction (CEJ) using a low-speed water-cooled saw, grit 230-270 (Buehler, Evanston, IL, USA). The root canals were instrumented with endodontic files (Dentsply-Maillefer, Ballaigues, Switzerland) to 1 mm from the apex, while regularly irrigating with 2% sodium hypochlorite, then dried with paper points and filled with gutta-percha points (Demedis, Almere, The Netherlands) and AH 26 root canal sealer (Dentsply-Maillefer, batch #0211001817) using the lateral condensation technique. A periodontal ligament was simulated by coating the root surface with a thin layer (approximately 0.3 mm) of silicone TSE 3991 (General Electric, Bergen op Zoom, The Netherlands). Finally, the teeth were embedded in acrylic resin in a standard copper tube, leaving 2 mm of the root above the acrylic.

Then, the gutta-percha was removed from the root canal with a low-speed Gates Glidden drill #3 (Dentsply-Maillefer) to a 6-mm depth as measured from the shoulder, in most cases leaving a 4-to-6 mm gutta-percha filling in the apical part.

A post space was made to the same depth with a low-speed calibrated drill provided by the manufacturer of the post system (RTD). The corresponding two-stage parallel-sided Quartz coated Carbon fiber post (RTD Aestheti-Post, batch #03101229810A) with a diameter of 1.2 mm apical and medial and 1.8 mm coronal displayed an accurate fit. Also, in the more spacious

coronal part of the root canal, the diameter (1.8 mm) was sufficient (Figure 1).

For each tooth, the root canal and dentin shoulder were cleaned with pumice and rinsed with water. After removing the water in the root canal with absorbent paper points, the tooth was air dried but not dehydrated.

Preparation of the Post-and-Core Restorations

Like a composite inlay, the post was sandblasted with 50-µm aluminum oxide (Danville Engineering, San Ramon, CA, USA) for 1-2 seconds (Nilsson & others, 2000), then cleaned with ethanol, dried and silanized for 30 seconds with Ceramic primer (3M ESPE).

The 24 prepared teeth were randomly assigned to three groups of eight teeth each. A survey of the successive steps in the preparation of the post-and-core restorations for each group is given in Table 1.

In Group 1, the dentin shoulder was etched for 30 seconds with 32% phosphoric acid (BISCO, Schaumburg, IL, USA) and thoroughly rinsed with water. After removing the water in the root canal with absorbent paper points, the tooth was air dried but not dehydrated. Self-etching ED Primer (Kuraray, batch #41128) was applied to the root canal dentin with a micro brush for 60 seconds. After removing the excess with absorbent paper points, the entrance of the root canal and post were coated with a surplus of mixed Panavia 21 (Kuraray, batch #41128) and the post was seated into the root canal. Excess cement was removed with a brush and the post was kept under occlusal finger pressure for three minutes. The cement in the entrance of the root canal, the dentin shoulder and the part of the post exterior to the root canal were covered with a thin layer of dual-cured resin bonding Clearfil Photo Bond (Kuraray, batch #0395 B), gently air-blown and light-cured for 20 seconds with an Astralis 5 (Vivadent, Schaan, Liechtenstein) curing light (Intensity 650 mW/cm^2). A standard matrix was placed around the premolar and filled with Clearfil Photocore (Kuraray, batch #1470 A) using a Centric syringe (Hawe Neos Dental, Bioggo, Switzerland) and light-cured for 60 seconds. The height of both the post and core were adjusted to 5.0 ± 0.2 mm with high-speed, coarse diamond burs type FG 142 G. 014 (Horico, Pfingst & Company, South Plainfield, NJ, USA) under profuse water spray. Finally, the opposing surfaces of the core build-up were trimmed in conformity with the shape of the tooth.

In Group 2, both the root canal and dentin shoulder were etched for 30 seconds with 32% phosphoric acid followed by rinsing with water and drying as described in Group 1. Two layers of Scotchbond 1 (in the US marketed as Single Bond) adhesive (3M ESPE, batch #20003120) were applied to the root canal dentin with a microbrush. After removing the excess with absorbent paper points, the bonding was light-cured for 20 seconds. Then, the entrance of the root canal and post were

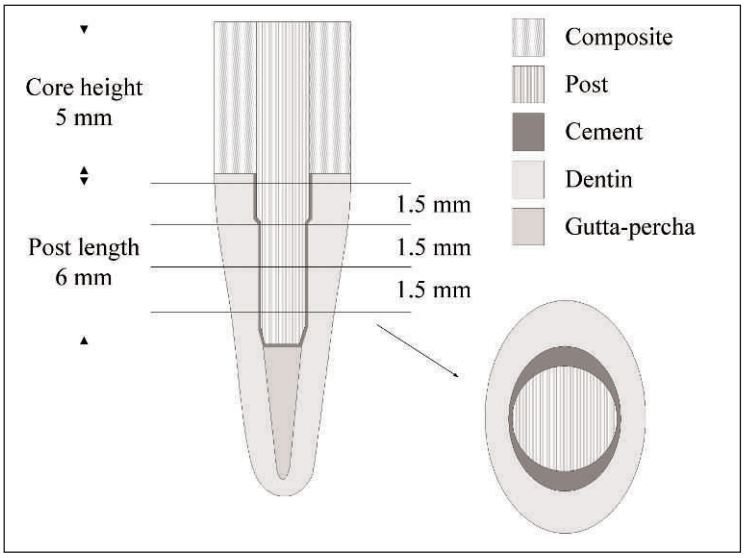

Figure 1. *Left: Schematic representation of a premolar root with post and core build-up and the levels (horizontal lines) where the root was cut to obtain 1.5-mm thick coronal (1), medial (2) and apical (3) cross-sections. Right: Cross-sectional view.*

Table 1: *Steps in Post and Core Build-Up Preparation for the Three Groups Used in This Study*

Step	Group 1	Group 2	Group 3
Dentin etching	Dentin shoulder: 32% phosphoric acid	Root canal and dentin shoulder: 32% phosphoric acid	Dentin shoulder: 32% phosphoric acid
Primer or adhesive	Root canal: ED Primer	Root canal: Scotchbond 1 adhesive[a] (LC)[b]	Na
Cement	Entrance root canal and post: Panavia 21 (CC)[b]	Entrance root canal and post: RelyX-ARC (DC)[b]	Root canal: RelyX (CC)[b]
Adhesive	Dentin shoulder and post exterior root canal: Clearfil Photo Bond (DC)[b]		
Core build-up resin composite	Clearfil Photocore (LC)[b]		

[a] *In the US, marketed as Single Bond.*
[b] *CC, LC, and DC denote chemical-cured, light-cured, and dual-cured, respectively.*

coated with a surplus of mixed RelyX-ARC (3M ESPE, batch #20003120) and the post was seated into the root canal. Excess cement was removed with a brush, and during light curing of the cement for 20 seconds, the post was held in place. The steps in preparation of the core build-up were identical to Group 1.

In Group 3, the dentin shoulder was etched for 30 seconds with 32% phosphoric acid followed by rinsing with water and drying as described in Group 1. RelyX (3M ESPE, batch #20010524) was mixed and injected into the root canal with a Lentulo Paste Carrier (Dentsply/Maillefer), then the post was seated into the root canal (Goldman, DeVitre & Tenca, 1984). Excess cement was removed with a brush and the post was kept under occlusal finger pressure for three minutes. The steps in preparation of the core build-up were identical to Group 1.

Lentulo Paste Carrier was not used for the composite cements, as this method involved the risk of an accelerated setting of the cement due to the anaerobic conditions in the apical part of the root canal. Premature setting of the cement would make it difficult to get the post in place.

During all experimental procedures throughout the investigation, the teeth were kept moist or stored in distilled water at 37°C.

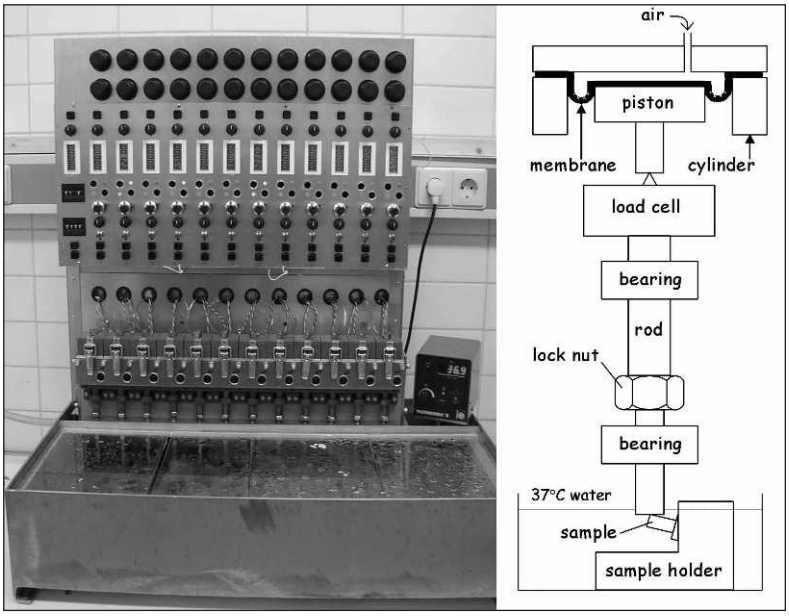

Figure 2. *Left: Twelve-station ACTA fatigue tester to test multiple samples simultaneously. Right: Schematic representation of one of the 12 stations. The cycles of each station are controlled by a pneumatic system by continuously switching the air pressure of the cylinders between two values, resulting for each second into an alternating load between 8 N (0.8 s) and 40 N (0.2 s). More details can be found at www.dentalmaterials.nl.*

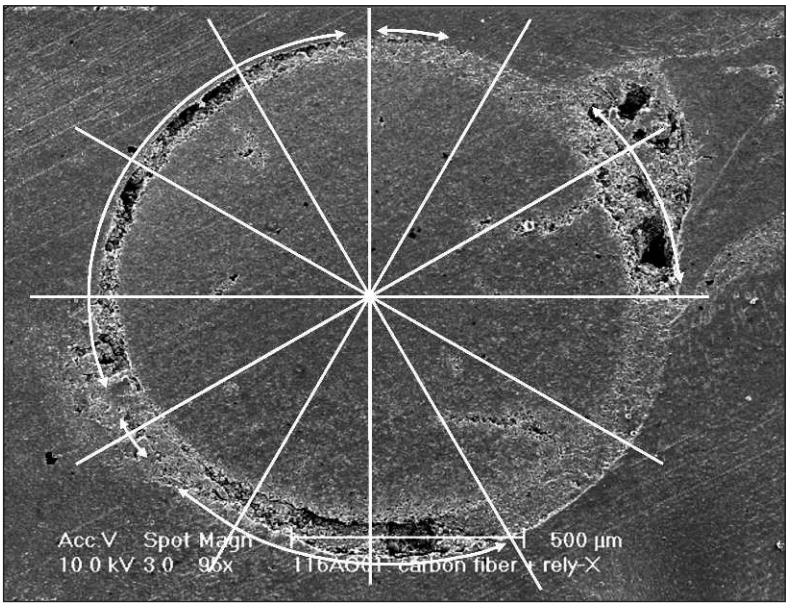

Figure 3. *SEM micrograph of the apical cross-section of a fatigued quartz coated carbon fiber post cemented with RelyX showing cracks, voids and insufficient adaptation to the post and intra-radicular dentin. The five arrows around the circumference illustrate how the irregularities were scored. As the total length of the summed arrows occupied 9/12th of the cement circumference, the score was 9 for this specimen.*

Fatigue Loading Procedure

In each group, half of the specimens (n=4) were fixed in acrylic blocks and placed in distilled water of 37°C in the ACTA fatigue machine (ACTA, Amsterdam, The Netherlands). They were loaded in a buccal-lingual direction for one million cycles (277 hours) on the axial-occlusal corner of the core at an 85° angle to the axis of the post (Outhwaite & others, 1982). With each cycle, the load alternated between 8 N (0.8 seconds) and 40 N (0.2 seconds) (Figure 2). This load is within the range of reported physiological masticatory forces (Anderson, 1956a,b; Bates, Stafford & Harrison, 1976; Helkimo & Ingervall, 1978; Libman & Nicholls, 1995; Murphy, 1965). The small displacements, which occur during loading due to the elasticity of the silicon layer between the tooth and embedding material, do not affect the adjusted maximum load, as the loading stylus returns to its upward position only if the maximum load has been fully reached. The remaining half of the specimens served as control specimens to gauge the effect of the fatiguing procedure.

Scanning Electron Microscopy (SEM) Evaluation Scores

Starting just apical from the level of the proximal CEJ, three consecutive, parallel, transverse 1.5-mm sections were cut from all specimens (Figure 1) using the low-speed, water-cooled saw previously mentioned. Impressions of the coronal surfaces of the three sections (coronal [CO], medial [MO] and apical [AO]) were made with a polyether impression material Impregum F (3M ESPE, Seefeld, Germany). The boxed impres-

sions were poured in epoxy resin Araldite D (Vantico, Wien, Austria) and kept under vacuum to remove air bubbles. After setting the epoxy, replicas were mounted on 10-mm aluminum stubs (Balzers, Liechtenstein) and gold sputter-coated (Edwards S150BE, Edwards). The epoxy replicas were then examined in a Scanning Electron Microscope XL 20 (Phillips, Eindhoven, The Netherlands) to evaluate irregularities such as cracks and voids in the cement layer, insufficient adaptation of the cement to the post or dentin and photographed at a magnification of approximately 50x (Figure 3). If no irregularities were found, a SEM evaluation score of zero was assigned. A score of one was assigned when irregularities occupied 1/12 or less (8.3% or less) of the cement circumference. The highest score level of 12 indicates irregularities occupying 91.7% to 100% of the cement circumference.

Push-out Test

Each cross-section was positioned with the coronal plane downward and the central post segment centered over a hole in a steel support that was aligned in a universal testing device (Instron, High Wycombe, UK) (Figure 4). A steel rod only in contact with the central post segment was pressed downward with a crosshead speed of 0.5 mm/minute. The load (N) required to push out the segment was divided by the area of the cylindrical root surface, enclosing the post and cement to calculate the bond strength: Bond strength (MPa) = Push-out force (N)/Perimeter (mm) x specimen thickness (mm). The perimeter of the post section was calculated from its diameter; the thickness was 1.5 mm. No distinction was made between the loss of retention between the post and cement layer or between the intra-radicular dentin and cement layer.

Statistical Analysis

The obtained data were statistically analyzed by a multiple analysis of variance (MANOVA), with the aid of the GLM subprogram of the SPSS package (Windows version 11.00, SPSS Inc, Chicago, IL, USA). Effects with a p-value not exceeding 0.05 were considered significant. Whenever an interaction of main effect was significant on a multivariate level, it was univariately examined next. Called-for effects were further explored by means of simple effects and pair wise comparisons. In this analysis, SEM evaluation scores and push-out strengths were the dependent variables. The test condition (fatigue loading or control) and type of cement (Panavia 21, RelyX-ARC or RelyX) were treated as a between subjects factor, while section location (apical, medial or coronal) was entered as a within subjects factor.

RESULTS

Fatigue loading did not cause separation of the post-and-core restorations from the roots in any of the specimens and the multivariate level was not different from the (non-fatigued) control (F=3.365, p=0.059). The multivariate results of the two other main effects, type of cement (Panavia 21, RelyX-ARC, and RelyX) and section location (apical, medial and apical), were significant (F=5.756, p=0.001 and F=5.252, p=0.008, respectively). The SEM evaluation scores and push-out strengths results are compiled in Table 2.

Univariately, the cement effect is significant for push-out strength (F=6.601, p=0.007) and SEM (F=9.578, p=0.001). For both the push-out strength and SEM, the results improved from RelyX to RelyX-

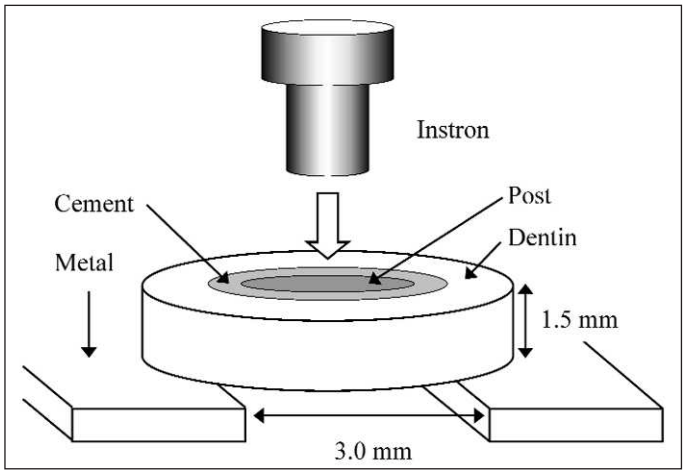

Figure 4. *Schematic representation of the push-out test. Each disk (1.5 mm) was positioned with the coronal plane downward. The pushing steel rod was only in contact with the central post segment (crosshead speed of 0.5 mm/minute).*

Table 2: *Means and Standard Deviations (between Brackets) per Cement of the Push-Out Strength and SEM Evaluation Scores of Irregularities for the Three Sections (Apical, Medial and Coronal) for Control (Non-Fatigued) and Fatigued Specimens*

Measurement	Cement	Apical Section		Medial Section		Coronal Section	
		Control	Fatigue	Control	Fatigue	Control	Fatigue
SEM evaluation (Scores 0-12)*	Panavia 21	3.5 (0.6)	5.5 (2.9)	1.8 (1.5)	4.5 (2.1)	1.5 (0.6)	2.5 (1.0)
	Rely X-ARC	4.5 (2.4)	6.0 (3.7)	3.0 (1.6)	6.3 (1.0)	3.5 (1.9)	5.0 (4.1)
	Rely X	6.3 (1.7)	9.3 (3.8)	6.5 (3.1)	9.3 (3.0)	6.3 (2.8)	8.3 (2.9)
Push-out Strength (MPa)	Panavia 21	5.5 (2.3)	4.9 (0.8)	6.1 (0.9)	5.5 (1.7)	6.4 (2.3)	6.3 (2.2)
	Rely X-ARC	2.1 (1.2)	2.1 (0.5)	6.4 (0.9)	3.4 (1.5)	6.6 (2.6)	3.9 (2.5)
	Rely X	3.2 (1.0)	3.6 (1.9)	4.2 (2.1)	4.5 (2.3)	2.4 (1.1)	2.7 (0.9)

**If no irregularities were found, a SEM evaluation score of zero was assigned. A score of one was assigned when irregularities occupied 1/12 or less (8.3% or less) of the cement circumference. The highest score level of 12 indicates irregularities occupying 91.7 to 100% of the cement circumference.*

ARC to Panavia 21; for push-out strength, the difference between Panavia 21 and RelyX is of significance (p=0.002). SEM showed significant differences between RelyX and Panavia 21 (p<0.001) and between RelyX and RelyX-ARC (p=0.014).

The location effect is also significant for push-out strength (F=6.601, p=0.009) and SEM (F=4.288, p=0.027). For push-out strength and SEM, the results improved from the apical to the coronal location. For push-out strength, there is a significant difference between the apical and medial location (p=0.001) and between the apical and coronal location (p=0.005). For SEM, significant differences were found between the coronal and apical location (p=0.022) and between the coronal and medial location (p=0.043).

DISCUSSION

As with many *in vitro* studies, it is difficult to extrapolate the results directly to the clinical situation, as it is hardly possible to simulate the complex of clinical conditions at the same time in one *in vitro* test. Where possible, this study simulated the clinical situation for post-and-core restorations by applying a "periodontal ligament" and the action of mastication (Outhwaite & others, 1982) by fatigue loading in water at 37°C. However, the magnitude and direction of the load were constant, which is not the case with chewing forces in the clinical situation, where extremely high forces can occur by impact of hard substances in food and with parafunctional loads (Bates & others, 1976; Helkimo & Ingervall, 1978; Libman & Nicholls, 1995; Murphy, 1965). The inclusion of a crown with a ferrule in the experimental set-up was deliberately omitted to exclude any external strengthening influence on the post and core. The configuration used was believed to be worse than any other with regard to resistance of the restored tooth and appeared suitable for specifically evaluating the fatigue behavior of the post in the root canal (Isidor, Brondum & Ravnholt, 1999; Nicholls, 2001; Sorensen & Engelman, 1990).

Standardization of the test specimens is another aspect that needs careful attention. In the selection of teeth, only single rooted upper premolars were chosen, however, differences in the anatomical perimeter of the teeth could not be avoided.

A limitation of the test set-up is the scoring method of irregularities in the cement layer from the SEM pictures. The different findings, summarized as irregularities, represent different causes; for example, a void is caused by the application method, while a crack or insufficient adaptation may be caused by shrinkage stresses within the luting agent or fatigue loading. Consequently, a general irregularity score does not only represent the influence of fatigue loading. The problem with scoring the mentioned irregularities is that it is not possible to examine the same specimen before and after fatigue loading. Therefore, it had to be assumed that the amount of irregularities as a result of the application method and shrinkage stresses was roughly the same in both the fatigued and non-fatigued specimens. Consequently, an increase in the amount of irregularities in the fatigued specimens was considered to result from fatigue loading.

With the current *in vitro* model, we were not able to demonstrate that fatigue loading would play a significant role in clinical failures; the results showed that the post-and-cores resisted fatigue loading after one million load cycles. This was somewhat unexpected, as the build-ups were loaded with a relatively high force of 40 N under a most unfavorable direction, nearly perpendicular to the axis of the post. Moreover, the load was applied directly on the build-up, without the support of a crown with a ferrule. In addition, the post-to-core length ratio was at the limit required for acceptable retention (Nissan, Dmitry & Assif, 2001). Under these circumstances, an effect of fatiguing could have been expected in the coronal sections, which were right below the interface between the root and build-up. An explanation for survival could be the relatively large peripheral surface and the accurate fit of the carbon fiber post, which contributed to a homogeneous distribution of stresses, keeping them well below the mechanical limits of the cement. Similar results were reported previously for well-fitting cast metal posts (Mendoza & others, 1997). One may speculate that an adequate fit in the coronal part of the root canal is of paramount importance to minimize clinical failures.

Although there was no statistical difference between the fatigue and control group, the difference was close to significance (F=3.365, p=.059), while the standard deviations for the fatigue group were higher than the control group (Table 2). This may indicate that unnoticed cracks had developed, which, in the clinical situation, could lead to leakage and, in the long term, could lead to disintegration of the cements. Because the fatigue experiment ran for only 11 days, this test did not reveal disintegration by the effect of leakage. Many of the failures observed after years of service (Bergman & others, 1989; Mentink & others, 1995; Morgano & Milot, 1993) may well be the result of a disintegrated cement from the combination of loading and long-term leakage (Libman & Nicholls, 1995). To evaluate the quality of the post-and-core build-up systems for clinical service, follow-up studies with the test set-up, where the specimens are immersed in a dye solution, could provide information about the leakage pattern, where leakage starts and how it progresses inside the root canal after load cycling.

With regard to push-out strength, the two resin composite cements Panavia 21 and RelyX-ARC performed significantly better than the resin-modified glass-ionomer cement RelyX (Table 2). This agrees with pre-

vious reports that the bond strength of resin composites is higher than that of resin-modified glass ionomers (Love & Purton, 1998). However, for all three cements, the significant variability of the push-out strength showed that the root canal remains a difficult area in which to operate. With regards to the two resin cements, the use of a Lentulo Paste Carrier is not advised, as it can accelerate the setting reaction in the anaerobic environment in the root canal. To avoid the accelerated setting, they have to be applied to the entrance of the canal, the post dipped in the cement, then inserted into position. Due to this insertion technique, flaws and voids are probably important factors influencing the push-out strength. For resin-modified glass-ionomer cement where the Lentulo Paste Carrier could be used, flaws and voids should have been diminished, but the SEM scores of RelyX showed that this technique did not contribute to a better integrity of the cement layer. An explanation could be that the integrity of all three resin cements is also influenced by the C-factor (Davidson & others, 1984; Feilzer & others, 1987).

For the root canal, the C-factor reaches values as high as 200 (Bouillaguet & others, 2003), which results in very high contraction stresses during setting. This can also create voids and, if the bond strength to either the post or intra-radicular dentin is exceeded, loss of integrity can occur. In the apical part, the situation is worse, as no cement is available to flow to the area of stress. The possibility of flow is also more restricted for the abruptly setting light-curing resin cements as compared to the slower setting chemical-cured cements (Feilzer & others, 1993), which could create more defects in the light-curing resin cement layer and weaken adhesive strength. This seems to be supported by both the SEM evaluation scores and push-out strength, which became worse from Panavia 21 (chemical-cured) to RelyX-ARC (chemical and light-cured). However, the presence of flaws, voids and other defects and the worsening of the integrity from coronal to apical, as shown by the SEM results (Table 2), are also caused by the well recognized problems of adequately delivering the cement at the apical level. Needle tubes used to inject the resin composite cements into the root canal (Fakiha, Al-Aujan & Al-Shamrani, 2001) may improve the results, but flaw and void formation as a result of the high C-factor will probably remain.

Traces of gutta-percha, which may remain in the medial and apical part of the root canal after preparation with the round calibrated burs of the post system, can also affect the final result. This most likely occurs in root canals of premolars that have an oval shape or are partially connected to a second root canal. Moreover, modern endodontic preparation techniques may leave more of the original oval root canal shape intact, which increases the risk of inclusion of gutta-percha remnants. All the causes mentioned that lead to voids and imperfect adaptation of the cements to the post and root canal wall should be minimized, as these "open spaces" are potential pathways for leakage to the apex.

The outcome of this study, in which all post-and-core specimens survived fatigue loading, is promising for the clinical situation. However, as the root canal remains a problematic area for adhesive cements, the general practitioner has to keep in mind that an adequate ferrule (Bolhuis & others, 2001; Isidor & others, 1999; Nicholls, 2001) and the preservation of tooth structure (Guzy & Nicholls, 1979; Kanca, 1988; Reeh, Messer & Douglas, 1989) are of major importance and key factors in promoting resistance to failure.

CONCLUSIONS

Within the limitations of the test set-up used in this study, it can be concluded that:

1. regardless, the choice of adhesive cement, quartz coated carbon fiber posts of 6 mm and adhesively bonded composite core build-ups can resist one million fatigue load cycles.
2. with respect to cement layer integrity and retention strength, resin composite cements perform better than resin-modified glass-ionomer cements.

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Bonded Amalgam Restorations: Microleakage and Tensile Bond Strength Evaluation

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Clinical Relevance

When a good seal and improved retention are required, the adhesive systems beneath bonding amalgam restorations should be activated by two methods (light and chemical curing). When only sealing is required, light-cured adhesives can be employed prior to amalgam condensation.

SUMMARY

Purpose: The objective of this study was to evaluate the tensile bond strength (BS) and microleakage (MI) of bonded amalgam restorations to dentin when an unfilled and a filled system are used under three application modes. Material and Methods: Seventy-two and 96 human molars, respectively, were employed for BS and MI tests. For BS, the occlusal surface of the molars was ground flat until dentin exposure. A 3-mm area was delimited for bonding. For MI, Class V cavities were prepared in the CEJ (4 mm x 4 mm x 2 mm). For each test, the molars were randomly divided into six treatment groups defined by a combination of the levels: Adhesive system (Scotchbond Multi-Purpose Plus [SBMP], Optibond dual cure [OPTB]) and Application mode (light–LC, chemical–C and combination of light and chemical curing–LCC). After adhesive application, the amalgam was condensed into a Teflon mold (BS) and into the cavities (MI). After storage in saline solution for seven days at 37°C, the specimens were subjected to the BS test at 0.5 mm/minute. For microleakage evaluation, the restorations were sealed with nail varnish, except for an area 1 mm around the restoration, immersed in 5% methylene blue solution for 24 hours and sectioned into two halves. Each half was evaluated by two trained examiners at 25x magnification in a 0-3 score system and the highest score was recorded. The BS data was evaluated by two-way ANOVA and Tukey's test (α=0.05). The MI data were analyzed by Kruskal Wallis and Mann-Whitney tests (α= 0.05). Results: The main factors were significant for the BS test: the highest BS mean was obtained using the LCC technique and the OPTB system. Regarding the MI test, only the application mode was significant: lower dye infiltration was observed for LC and LCC.

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INTRODUCTION

Since Varga, Matsumura and Masuhara (1986) and Staninec and Holt (1988) advocated the use of dentin bonding adhesives as amalgam bonding agents, this technique has become a popular clinical practice in the restoration of posterior teeth. Bonding amalgam restorations have potential advantages, including tooth reinforcement, decreased post-operative sensitivity, better marginal adaptation, decreased microleakage, reduced possibility of secondary caries and more conservative preparation (Setcos, Staninec & Wilson, 2000).

A wide variety of adhesives have been employed and there are successful reports that indicate their effectiveness as amalgam bonding agents (Adhesives, 1995; Use Survey, 1995). Nevertheless, bond strength values and sealing data vary considerably among the materials employed, such that one might suggest that the inherent features of the materials and the way they are applied may have a significant effect on the performance of bonded amalgam restorations. Some studies apply light-cured adhesive systems prior to amalgam insertion, while others use their chemical cured version (Vargas, Denehy & Ratananakin, 1994; Winkler & others, 1997; Ramos & Perdigão, 1997; Fritz & Finger, 1998; Evans & Neme, 1999; Cobb, Denehy & Vargas, 1999).

Bonded amalgam restorations have two important interfaces: the tooth-adhesive interface and adhesive-amalgam interface (Ruzickova & others, 1997). The tooth-adhesive interface is essentially the same as that formed in bonded composite restorations and remains a matter of concern in numerous studies (Eick & others, 1997), while the adhesive-amalgam interface has not been not extensively studied.

Boston (1997) has suggested that chemical coupling mechanisms and mechanical intermingling of polymer and amalgam are the bonding principles. The chemical bonding between amalgam and polymer seems to be correlated with specific monomers able to bond with metallic restorations, such as 4-META (Fritz & Finger, 1998). The mechanical intermingling of adhesives and amalgam is related to adhesive thickness and, primarily, to how thick the unpolymerized resin layer is before amalgam insertion (Vargas & others, 1994; Winkler & others, 1997; Ramos & Perdigão, 1997; Fritz & Finger, 1998; Evans & Neme, 1999; Cobb & others, 1999). Adhesive layer thickness is dependent on filler loading of the adhesive system and the way it is applied (Winkler & others, 1997, 2000a; Winkler, Rhodes & Moore, 20b).

Therefore, the objectives of this study were to evaluate the bond strength and microleakage of bonded amalgam restorations to dentin when an unfilled and filled system are applied under three application modes (light, chemical and a combination of light and chemical curing).

METHODS AND MATERIALS

Teeth Selection and Experimental Design

One hundred and sixty-two extracted non-carious third human molars were employed in this investigation. The teeth were cleaned and disinfected in 0.5% thymol solution up to six months before use. They were divided into two samples according to the test to be performed. Seventy-two and 96 teeth were employed for the bond strength and microleakage tests, respectively. For each test, the teeth were randomly divided into six experimental groups defined by a combination of the levels of Adhesive system (2 levels) and Application mode (3 levels).

Specimen Preparation

Tensile Bond Strength Test

The occlusal surface of the teeth was sectioned in a mesial to distal direction by means of a diamond saw mounted in a low-speed handpiece (KG Sorensen Ind & Com Ltda, Barueri, SP, Brazil) to expose dentin substrate. Dentin surfaces were ground wet with 240, 400 and 600 grit silicone carbide paper in order to standardize formation of the smear layer (Pashley & others, 1988). The roots of the teeth were removed similar to what is described above. The teeth were embedded in polyvinyl chlorine pipe containers with chemical cure acrylic resin (Clássico Prod Odontol, São Paulo, SP, Brazil) so that the occlusal surfaces projected well above the resin. Immediately after polymerization of the acrylic resin and before adhesive application, the area for bonding was delineated with an adhesive tape that was previously punched with a 3-mm hole (inner diameter). The margins of the adhesive tape were burnished to obtain a good seal between the adhesive tape and dentin.

After application of the adhesive (Table 1), a 5 mm Teflon mold cone (lower and higher diameter of 3 mm and 5 mm, respectively) was positioned over the dentin surface so that the demarcated dentin area lined up with the hole in the Teflon mold.

Microleakage Test

Class V cavities (4 x 4 x 2 mm) were prepared at the cementum-enamel junction (CEJ). Half of the preparation margin was in enamel and half was in cementum. The cavity was prepared by means of a carbide bur (#245, KG Sorensen Ind & Com Ltda, Barueri, SP, Brazil) mounted in a high-speed handpiece. Only one cavity was prepared per tooth.

Restorative Procedure

Two total etch adhesive systems were applied: 1) ScotchBond Multi-Purpose Plus (SBMU–3M ESPE

Table 1: *Composition and Application Modes of Adhesive Systems Employed*

Adhesive Systems	Composition	Application Mode	Bonding Procedure
ScotchBond Multi-Purpose Plus (SBMP)	1 Acid—35% acid phosphoric; 1.5 Activator—ethanol based solution; sulfinic acid salt; photoinitiator; 2 Primer—HEMA, policarboxylic acid and water; 3 Adhesive—Bis-GMA, HEMA and hexafluorphosphate; 3.5 Catalyst—Bis-GMA, HEMA and peroxide (self-cure resin system).	Light-cured (LC)	a, b, c, d, e, f, g
		Chemical cured (C)	a, b, c, c_1, c_2, d, e, f_1
		Light-cured and chemical cured (LCC)	a, b, c, c_1, c_2, d, e, f, g, f_1
Optibond dual cure (OPTB)	1. Acid—38% acid phosphoric; 2. Primer—HEMA, GPDM, PMMA, water and ethanol; 3. Adhesive (A)—Bis-GMA and HEMA; Dual-cure B—HEMA, GBPD and filler (48%).	Light-cured (LC) Chemical cured (C)	a, b, c, d_1, e_1, e_2, f, g a, b, c, d_1, e_1, e_2, f_2
		Light-cured and chemical cured (LCC)	a, b, c, d_1, e_1, e_2, f, g, f_2

***a**–acid-etch (15 seconds); **b**–rinse (15 seconds); **c**–blot moist; **c1**–apply one coat of activator (10 seconds); **c2**–air-dry for 5 seconds at 20 cm; **d**–apply one coat of primer (10 seconds); **d1**–appy one coat of primer (30 seconds); **e**–air-dry for 5 seconds at 20 cm; **e1**–air-dry for 10 seconds at 20 cm; **e2**–light-cure (20 seconds–600 mW/cm²); **f**–apply one coat of adhesive; **f1**–mix one coat of adhesive and one coat of catalyst (5 seconds) and apply; **f2**–mix one coat of 3A and 3B (5 seconds) and apply; **g**–light-cure (10 seconds–600 mW/cm²).*

Dental Products, St Paul, MN, USA) and 2) Optibond dual cure (OPTB–Kerr Manufacturing, Romulus, MI, USA) according to one of the application modes described in Table 1. After adhesive application, an admixed, high copper amalgam alloy (Permite C, SDI, Bayswater, Victoria, Australia) was mixed for eight seconds in an Ultramat device (SDI, Bayswater, Victoria, Australia) and condensed either in the opening of the Teflon mode (tensile bond strength test) or inside the Class V cavities (microleakage test). When necessary (depending on the experimental group), the light curing procedure was performed with a VIP light unit (BISCO, Schaumburg, IL, USA) with a light intensity of 600 mW/cm². Thirty minutes later, the specimens were stored in saline solution at 37°C and 100% relative humidity for seven days before being tested. All restorative procedures were performed by one trained operator (Temple-Smithson, Causton & Marshall, 1992).

Tensile Bond Strength Test

The specimens were tested under tensile strength in a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil) with a cell load of 100 N at a crosshead speed of 0.5 mm/minute. The results, obtained in Newton's, were converted to MPa. The fracture mode of the specimens was analyzed under 25x magnification (Olympus SZ40, Tokyo, Japan) and classified according to the following patterns: 1) C–cohesive (failure within amalgam or dentin); 2) AAT–adhesive failure between the adhesive and tooth interface; 3) AAA–adhesive failure between adhesive and amalgam; 4) M–mixed fracture mode.

Microleakage Test

Two coats of nail polish were applied to the teeth, except for the area of restoration and up to 1 mm around its borders. The nail polish was allowed to air dry. The specimens were immersed in an aqueous 5% methylene blue solution for 24 hours. The nail polish was removed and the teeth were longitudinally sectioned in the middle of the restoration by means of a diamond saw, mounted in a high-speed handpiece, under water cooling. Both halves of the sectioned teeth were evaluated at the gingival margin by two trained examiners at 25x magnification (Olympus SZ40, Tokyo, Japan). The highest score was computed for each restoration. The dye penetration was analyzed according to a 0-3 scale (0 = no dye infiltration; 1 = dye penetration up to one half of the gingival floor; 2 = dye penetration up to more than one half of the gingival floor; 3 = dye penetration up to the axial wall) (Winkler & others, 2000a).

Statistical Analysis

Two-way ANOVA (adhesive vs application mode) and Tukey's multiple comparison tests were used to compare the tensile bond strength (α=0.05). For the microleakage data, the scores were evaluated by Kruskal-Wallis and Mann-Whitney tests (α=0.05). The agreement between examiners was evaluated by the Kappa statistics (Landis & Koch, 1977). When disagreement arose between them, a consensus had to be reached.

RESULTS

The results of the tensile bond strengths are shown in Table 2. Two-way ANOVA did not show significant effect for the interaction (p=0.67) but it did for the main factors adhesive and application mode (p=0.02 and p=0.001, respectively). For the adhesive system, OPTB had the highest bond strengths (p=0.02). Both adhesive systems performed better when they were applied according the LCC mode. The LC group provided the lowest bond strength mean, and the C group had an intermediate average (p=0.001). It is worth mentioning that six specimens from the LC group (3 from SBMP and 3 from OPTB) failed during storage and were excluded from the analysis.

In the LC group, SBMP showed 100% of AAA bond failures (between adhesive and amalgam), whereas for

OPBT, 83% were AAA and 17% of bond failures were M (mixed fracture mode). Under the C group for both systems, the percentage of failures was similar: 83% were AAT (between adhesive and dentin) and the remaining were mixed. Regarding the LCC group, most of the failures were M (91%) for both systems.

Microleakage score results are shown in Table 3. Agreement between the examiners was excellent (Cohen's Kappa = 0.89) (Landis & Koch, 1977). The Kruskal-Wallis test detected no significant difference for either the interaction (p=0.94) or the main factor adhesive system (p=0.22). Only the application mode was statistically significant (p=0.01). The LC and LCC groups were similar (p=0.38) and both techniques showed a better sealing than the C group (p=0.007 and p=0.009, respectively). These results were confirmed through a reanalysis of the data under parametric ANOVA (Neter & others, 1996) and pairwise comparisons by the Bonferroni method (Winer, 1991).

DISCUSSION

The use of adhesive systems under amalgam restoration has been used as an ordinary procedure, instead of copal varnish (Setcos & others, 2000). Based on the results of this study, it seems that not all bonded amalgam restorations are simultaneously able to provide sealing and retention of amalgam restorations.

The low bond strength mean obtained in the LC group may be explained by the fact that the adhesion of light-cured resins to amalgam apparently relies on the intermingling of the amalgam with the unpolymerized layer of adhesive that was inhibited by oxygen as it was condensed into place (Vargas & others, 1994; Winkler & others, 1997; Fritz & Finger, 1998; Evans & Neme, 1999).

This uncured resin layer hardens after it comes into contact with free radicals not consumed by oxygen (Ruyter, 1981). The thinner this layer is, the worse the micromechanical bonding. It is fair to suppose that the unpolymerized adhesive layer from the LC group is lower than the other groups and could have been much thinner in order to provide mechanical intermingling between amalgam and adhesive. In fact, this was demonstrated by Ramos and Perdigão (1997) and Staninec and others (1997) through SEM evaluation. They have shown a well-defined line distinguishing amalgam and adhesive, which evidenced that no intermingling between both materials had occurred when the adhesive was light-cured before amalgam condensation. Indeed, the LC technique demonstrated the lowest bond strength values and practically all specimens failed in the adhesive amalgam interface, indicating that both materials probably lack mechanical interlocking. It was also observed that some specimens had premature debonding during water storage.

In spite of the worst retention for the LC group, this application mode provided a lower degree of dye infiltration, indicating that the adhesive-tooth interface was well sealed. Therefore, although this technique should not be indicated in cases where an improvement in amalgam retention is desirable, light-cured adhesive systems should be used as a sealing material prior to amalgam condensation (Setcos & others, 2000).

So far, this study is in accordance with the conclusions of Ramos and Perdigão (1997), Ruzickova and others (1997) and Cobb and others (1999). These studies have shown that the bond strength of amalgam applied over light-cured adhesive systems is very low. Conversely, Winkler and others (2000a,b) have demonstrated similar performance between the LC and C groups in terms of their bond strength values.

This apparent controversy may be due to the fact that adhesive systems differ in their oxygen inhibited layer thickness (Ruyter, 1981) and the adhesive layer formed. It has already been demon-

Table 2: *Mean and Standard Deviation of the Tensile Bond Strength for Each Group*

Adhesives (*)	Application Mode (*)		
	Light-cured (α)	Chemical Cured (β)	Light and Chemical Cured (θ)
SBMP (a)	0.91 ± 0.68	3.44 ± 0.91	5.58 ± 1.59
OPTB (b)	1.24 ± 0.67	4.12 ± 0.87	6.50 ± 1.21

(*) Groups with different letters are statistically different (p<0.05)
(*) LC group (n=9 for each adhesive); C and LCC group (n=12 for each adhesive)

Table 3: *Median and Scores Frequency for Microleakage for Each Group at Gingival Margin*

Adhesive (*)	Application Mode	Scores				Median	Statistical Analyses (*)
		0	1	2	3		
SBMP (A)	**Light-cured**	06	06	02	02	0.5	α
	Chemical cured	02	06	02	06	1.5	β
	Light and chemical cured	07	06	01	02	0.5	α
OPTB (A)	**Light-cured**	14	02	00	00	0.0	α
	Chemical cured	06	10	00	00	1.0	β
	Light and chemical cured	12	04	00	00	0.0	α

(*) Groups with different letters are statistically different (p<0.05)

strated that the higher the thickness of the adhesive layer, the higher the bond strength of amalgam to tooth structure (Ramos & Perdigão, 1997; Winkler & others, 1997).

Regarding the C group, no oxygen-inhibited layer is present and, thus, its ability to couple with amalgam and improve bond strength values is related to chemical bonding of specific monomers (4-META, Fritz & Finger, 1998) and, to a lesser degree, to the mechanical interlocking between them (Scherer & others, 1992; Boston, 1997). Most of the fracture modes in this group were between the adhesive and tooth structure, which is evidence that chemical cured adhesives do not bond well to dentin.

During microleakage evaluation, the adhesive thickness in the LCC group was thicker than in the C group, which was, in turn, thicker than the LC group (unpublished data). This does not agree with the results of Winkler and others (2000b), who have demonstrated that the adhesive layer was thicker for the LC group than the C group and that LC and LCC provided higher bond strengths than the C group.

The difference in adhesive layer thickness is also dependent on the application mode employed. For instance, when SBMP is activated under the C or LCC modes, several bottles are used, which improves the adhesive layer thickness compared to the LC mode. In a similar manner, when OPTB is used under the C or LCC techniques, the use of 3B paste, which has a filler load of 48%, also increases the thickness of the adhesive layer. This fact probably did not occur with the adhesive systems employed in the study by Winkler and others (2000b).

Several other studies have demonstrated that adhesive systems show very different adhesive layer thickness (Barkmeier, Los & Triolo, 1995; Cobb & others, 1999; Winkler & others, 2000a) and we do believe that this fact plays one of the most important roles in the bond strength of bonded amalgam restorations. In an attempt to avoid other sources of variation, all restorations were performed by one trained operator (Temple-Smithson & others, 1992) and only one brush was used for each bottle (primer, adhesive, catalyst, etc), which prevented them from contamination and also guaranteed a thicker layer to be formed.

Although, theoretically, both systems employed in this study are able to form thick layers when compared with other systems presented in the market, it seems that the presence of filler load in OPTB may have accounted for the better performance of this system in the tensile bond strength test, as already demonstrated by other authors (Ruzickova & others, 1997; Ramos & Perdigão, 1997; Diefenderfer, Reinhardt & Brown, 1997; Cobb & others, 1999).

Regarding microleakage evaluation, statistical analysis did not detect any difference between the materials, which was different from the conclusions of other investigations (Brisco & others, 2002). Although differences between materials were found for the bond strength test, the microleakage evaluation failed to demonstrate this trend. Other studies were also unable to demonstrate a relationship between these two methodologies (Fritz & Finger, 1998; Neme, Evans & Maxsoon, 2000; Winkler & others, 2000b; Grobler & others, 2000), which suggests that there might be no correlation between sealing and retention.

Another concern regarding the use of a chemical-cured version of the adhesive system is the likely inclusion of resinous material within the amalgam body. Some studies have already reported reduced mechanical properties of amalgam under this situation (Charlton, Murchison & Moore, 1991; Millstein & Naguib, 1995; Boston, 1997).

Permite C alloy is an admixed, high copper that presents great net expansion upon setting when compared to similar alloys from different manufacturers (Brown & Miller, 1993). Lower microleakage values observed with this alloy were attributed to this net expansion (Ng, Hood & Purton, 1998; Mahler & Bryant, 1996). Despite this fact, differences among the adhesive application modes could be detected in this investigation and other studies (Ng & others, 1998; Brisco & others, 2002; de Morais, Rodrigues & Pimenta, 1999), which suggests that the different application modes and adhesive system employed may interfere with the sealing and retention of Permite C alloy. Unfortunately, the results of this study cannot be extrapolated for spherical high copper alloys due to the fact these alloys possess a higher trend toward higher microleakage (Mahler & Bryant, 1996).

CONCLUSIONS

Based on the results of this study, it is worth concluding that the use of a filled adhesive system applied by two methods (light and chemical curing) showed the best performance on sealing and retention and can be used when an increase in retention is a desirable feature. When cavity preparation is sufficiently retentive, light-cured adhesive systems can be used, as they provide a better seal than chemical-cured systems. Further studies must be conducted in order to evaluate the hypothesis that arose in the discussion.

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Effect of Flexural Load Cycling on Microleakage of Extended Root Caries Restorations

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Clinical Relevance

The marginal sealing ability of a flowable resin composite with dentin adhesive under a flexural cycling load was better than in other selected materials; and flowable resin composite and dentin adhesive may be suitable for the restoration of advanced root caries.

SUMMARY

This study evaluated the microleakage of resin-modified glass ionomer, flowable compomer and flowable resin composite restorations on a Class V cavity of simulated advanced root caries under a flexural load cycling condition. Thirty-six non-carious human maxillary premolars were mounted in cylindrical acrylic resin molds. The cavities were prepared in the proximal root surface, from the middle of the buccal surface to the middle of the lingual surface, approximately 1 mm below the cemento-enamel junction, 2 mm axial width and 1.2 mm in depth. The teeth were randomly assigned to one of three groups with 12 teeth in each group: Group 1: Cavity conditioner and Fuji II LC (GC America), Group 2: Prime & Bond NT and Dyract Flow (Caulk-Dentsply), Group 3: Excite and Tetric flow (Ivoclar/Vivadent). Specimens were settled laterally on a fatigue-testing machine that was adjusted to deliver a force of 60N. The specimens were load cycled at 1Hz for 5000 cycles, placed in a staining solution and sectioned to evaluate microleakage penetration. Results indicate that the coronal and gingival margins showed significant microleakage differences among the three restorations ($p<0.05$). At the coronal margin, there was no significant difference between Groups 2 and 3. At the gingival margin, there was no significant difference between Groups 1 and 2. It was concluded that the marginal sealing ability of a flowable resin composite under a flexural cycling load was better than in other selected materials and that flowable resin composite with dentin adhesive was a desirable alternative for root caries restorations extended to the proximal surface.

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INTRODUCTION

A growing population of the elderly results in a greater need for the treatment of root caries. The greater number of exposed root surfaces associated with aging increases the risk of root caries (Fejerskov, Baelum & Ostergaard, 1993). Root caries in elderly people presents dentists with some clinical difficulties. The pulpal proximity of root caris causes sensitivity and can result in pulpal necrosis. Also, it is difficult to distinguish a

carious lesion from sound dentin. Access to lesions is often compromised, because advanced lesions often wrap around and greatly weaken the tooth structure and retention is compromised because there are no "box" forms (Levy & Jensen, 1990). Also, it is not ideal to place technique sensitive materials for the restoration of root caries, because the lack of elderly patient cooperation may make it difficult to complete the procedure (Gryst & Mount, 1999).

Glass ionomer cements offer the well-recognized benefits of fluoride release, the ability to adhere directly to the tooth, the potential to re-mineralize and excellent biocompatibility. Because of these well-established attributes, glass ionomer cement should be considered the material of choice for restoring root surface caries (Billings, 1986). Recently developed resin-modified glass ionomers are approximately twice as strong. Momoi and others (1995) reported that resin-modified glass ionomers were stronger, more flexible and more resilient than conventional acid-base glass ionomers. This was in accordance with the finding that less marginal breakdown and surface deterioration occurred in a resin-modified glass ionomer than in a conventional analogue. However, its bond strength is lower than conventional dentin bonding systems (Tanumiharja, Burrow & Tyas, 2000a,b).

On the other hand, resin composite is the most aesthetic alternative for restoring root caries. Improvements in dentin bonding have progressed to the degree that leakage at the apical margin is no longer an unavoidable result of polymerization shrinkage. In particular, flowable resin composite is uniquely resilient, which may be important in lesions that exist because of abfraction forces (Grippo & Masi, 1991; Heymann & others, 1991). But the fluoride release of some flowable composites is extremely low compared to glass ionomer materials (Shay, 1997; Burgess & Gallo, 2002).

Compomers, which are resin composites containing glass ionomer filler particles, more closely resemble composites than glass ionomers in terms of polymerization shrinkage, resiliency, bond to dentin and enamel and thermal behavior. They leach fluoride at a rate that approaches 60% that of glass ionomer, have easy handling characteristics, are readily contoured and are less technique sensitive than composites (Meyer, Cattani-Lorente & Dupuis, 1998; Shay, 1997).

Yap, Lim and Neo (1995) reported that the marginal sealing ability of resin composite, compomer and resin-modified glass ionomer showed no significant difference in dentin margin under thermal cycles. However, there is no evidence to support the choice of restorative materials on teeth with advanced root caries when subjected to flexural forces.

This study evaluated the microleakage of three restorative materials placed in Class V cavities similar to those found in advanced root caries. The teeth were then placed under flexural load cycling to determine which is the best restorative material for advanced root caries affected by functional forces.

METHODS AND MATERIALS

Cavity Preparation

Thirty-six non-carious human maxillary premolars stored in deionized water were selected for the study. After surface debridement with a hand-scaling instrument, the teeth were mounted in cylindrical acrylic resin. The cavities were prepared in a U-shape using carbide burs (#303, Brasseler, Savannah, GA, USA) and finished with #702 steel fissure burs at low speed. Each cavity was prepared to simulate a proximal root surface, from the middle of the buccal surface to the middle of the lingual surface, approximately 1 mm below the cemento-enamel junction, 2 mm axial width and 1.2 mm in depth. The distance from the lower margin of each cavity to the resin molds was approximately 1.5 mm (Figures 1 and 2).

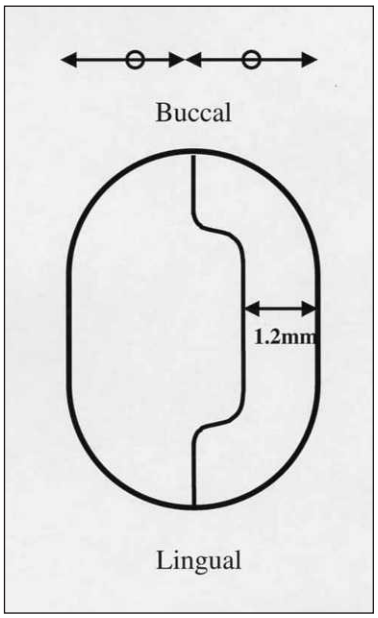

Figure 1. *Schematic horizontal cross-section of restoration: Each cavity was prepared to simulate a proximal root surface, from the middle of the buccal surface to the middle of the lingual surface, approximately 1 mm below the cemento-enamel junction, 2 mm axial width and 1.2 mm in depth.*

Restoration

The teeth were randomly assigned to one of three groups with 12 premolars in each group restored in the following manner:

Group 1: Cavity conditioner and Fuji II LC (GC America, Alsip, IL, USA), Group 2: Prime & Bond NT and Dyract Flow (Dentsply Caulk, Milford, DE, USA), Group 3: Excite and Tetric flow (Ivoclar Vivadent, Amherst, NY, USA). All materials and adhesive systems were used according to the manufacturer's instructions without matrix (Table 1). Immediate wet finishing and polishing was performed as necessary.

Flexural Load Cycling

All specimens were placed on a fatigue loading machine (Etica ESP, Sao Paulo, Brazil) and the loading probe was attached to the proximal surface that was on the same side of the restoration and was made flat in order to perpendicularly receive the force (Figure 2). The probe was

positioned 7.5 mm from the margin of the resin mount and programmed to constantly maintain contact with the surface of the tooth. The loading machine was adjusted to deliver a force of 60N. The specimens were load cycled at 1Hz for 5000 cycles. During load cycling, all specimens were kept at constant humidity.

The teeth were prepared for microleakage evaluation by coating the entire tooth with nail varnish (Hard as Nails, Sally Hansen, Farmingdale, NY, USA), except for 1 mm around the restoration margins. The specimens were placed in a solution of 2% basic fuchsin solution for 24 hours in a humidity chamber at 37°C.

Evaluation of Dye Penetration

Each specimen was sectioned vertically at the center of the proximal surface using a slow speed saw in order to evaluate the coronal and gingival margins. Dye penetration was evaluated with a stereomicroscope at a magnification of 30x (MITUTOYO, Toolmakers Microscope, Tokyo, Japan) and scored at each wall according to the following criteria:

0= No dye penetration

1= dye penetration up to 1/2 of the cavity depth

2= dye penetration greater than 1/2 of the cavity depth

3= penetration to full length of gingival or coronal or lateral wall and up to the axial wall

The data were statistically analyzed using the Kruskal-Wallis test. If differences were found, a Mann-Whitney U-test was used to identify the differences ($p<0.05$).

RESULTS

The frequency of microleakage score at the four margins—coronal, gingival, right and left—are shown in Table 2. The coronal and gingival margins showed significant differences among the three restorations ($p<0.05$). At the coronal margin, there was no significant difference between Groups 2 and 3. In the case of the gingival margin, there was no significant difference between Groups 1 and 2.

DISCUSSION

Occlusion is an oral factor that has not received much epidemiologic attention with respect to root caries but is important. Heymann and others (1991) suggested that this deformation was likely greater in older teeth, where the crown-to-root ratio was greater and there was a likelihood that restorations have replaced more of the tooth structure. Grippo and Masi (1991) reported that extracted teeth showed accelerated corrosion rates under the condition of tensile force. Jorgensen, Matono and Shimokobe (1976) reported that the decrease in cervico-occlusal diameter of empty buccal Class V cavities in maxillary first premolars was 24 µm under an axial load of 16 kg of force, and after a maximum axial load cycling of 8 Kg, all specimens showed gap discoloration caused by dye penetration. When root caries lesions are present, the lesion may spread along the cemento-enamel junction. In such cases, there is not much sound dentin left and deformation of the cavity or restoration might be greater. Therefore, the root caries restoration should have enough strength to bear the various vertical and lateral forces produced during oral function.

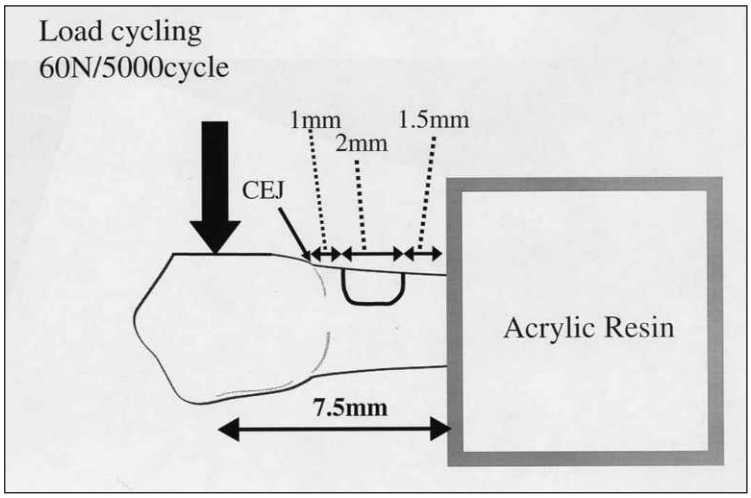

Figure 2. *The probe of the fatigue loading machine was placed 7.5 mm from the margin of the restoration on a flattened surface on the same side of the restoration. The specimens were load cycled at 1Hz for 5000 cycles.*

Table 1: *Restorative Materials, Dentin Bonding Systems and Manufacturers*

	Materials	Procedures	Manufacturer
Group 1	Cavity conditioner	apply 10 seconds, water rinse, lightly dry (not desiccate)10 seconds,	GC America
	Fuji II LC capsules	light cure 20 seconds	
Group 2	34% tooth Conditioner gel	apply 15 seconds, water rinse 10 seconds,	Dentsply/Caulk
	Prime & Bond NT	blot dry, apply 20 seconds, air blow gently, light cure 10 seconds	
	Dyract flow	light cure 20 seconds	
Group 3	Total Etch (37% Phosphoric acid	apply 15 seconds, water rinse	Vivadent
	Excite	blot dry, apply 20 seconds air blow gently, light cure 10 seconds	
	Tetric Flow	light cure 40 seconds	

Table 2: *Microleakage Scores*

Coronal Margin					
Score	**0**	**1**	**2**	**3**	
Group 1	3	1	4	4	)*) *
Group 2	6	5	1	0	)NS)
Group 3	7	2	1	1	
Gingival Margin					
Score	**0**	**1**	**2**	**3**	
Group 1	4	4	1	3	)NS) *
Group 2	3	6	1	2	)*)
Group 3	8	3	0	0	

**: p<0.05*

In this study, the loading machine was adjusted to deliver a flexural force of 60N in a transversal direction. In a pilot study, this flexural load value was determined to be the maximum force necessary not to break the specimens. Kubo and others (2001) reported that the magnitude of flexural force used in their study was 110N at the incisal point. Jang and others (2001) applied 130N on the occlusal surfaces of the specimens. The loading point of this study was positioned on the same side as the restorations and closer to the cavity than previously published studies; in addition, the cavity in this study was larger. Therefore, the aim of this study was to provide the greatest cavity distortion and generate the maximum level of tensile stresses at the margins. However, more studies might be required to estimate the effect of vertical forces and compression stresses.

Flowable resin composites have often been advocated for Class V restorations of root surface lesions, because they have lower modulus of elasticity than hybrid resin composites. The argument for this type of resin composite restoration is that because the tooth flexes during mastication, flexible restorative materials flex with the teeth. The greatest problem with resin materials is polymerization contraction, which creates stress at the marginal areas and opens the margins. In this study, Group 3 showed the lowest rate of microleakage among the three materials. Sabbagh, Vreven and Leloup (2002) reported that the static elastic modulus of Tetric Flow was 4.3 ± 0.3 Gpa. Tetric Flow had great elasticity but also great polymerization shrinkage. However, there had also been some discussion that a longer duration of the "gel" phase during polymerization of certain resins may allow for a better release and distribution of the contraction stresses (Fruits & others, 2002). Therefore, this might explain why polymerization shrinkage might not have affected the marginal sealing of Tetric Flow, as the cavosurface dentin was sound and the adhesive ability of the resin bonding system performed perfectly. The microtensile bond strength of Excite (as reported by the manufacturers and Cardoso & others, 2002) was 42.3 and 42.9 (± 7.1) MPa. Tanumiharja and others (2000a,b) reported that the microtensile bond strength of Fuji II LC with Cavity Conditioner was 18.5 ± 4.0 MPa and Prime & Bond NT was 29.9 ± 6.1 MPa. This higher bond strength may have contributed to the lower amount of microleakage of Tetric Flow.

The elastic modulus of Dyract was similar to Tetric flow (Sabbagh & others, 2002) and the microtensile bond strength of Prime & Bond NT was lower than Excite (Tanumiharja & others 2000a; Cardoso & others, 2002). The microleakage of Group 2 was similar to Group 3 at the site of the coronal margin but significantly greater at the gingival margin. Therefore, this result has shown some conflicts that require more careful analysis related to the different variables affecting microleakage.

The microleakage of Fuji II LC was greater than Dyract and Tetric flow. Polymerization shrinkage of the resin-modified glass ionomer was greater than conventional glass ionomers and the hybrid resin composite. Recently published literature has shown that resin-modified glass ionomer cement showed greater volume change than other materials during water storage (Attin & others, 1995). However, water storage might not contribute sufficient expansion to seal the margins of the restorations, because flexural load cycling was applied to the specimen almost immediately after polishing. The flexural strength of resin modified glass ionomer is smaller than compomers and flowable resin composites. The flexural strength of Fuji II LC stored in distilled water for 24 hours was 16 MPa and Dyract was 99 MPa (Meyer & others, 1998). And, the adhesive ability of a resin-modified glass ionomer seems to be smaller than a well-controlled dentin bonding system (Tanumiharja & others, 2000a,b). Therefore, it seems that, for this study, those factors affected the magnitude of microleakage of Fuji II LC. However, this fact did not indicate whether resin-modified glass ionomer is inappropriate as a restorative material for root surface caries, because its adhesive ability is not as technique-sensitive as the dentin bonding system and its fluoride release ability is greater than compomers and resin composites. Gryst and Mount (1999) reported that a resin-modified glass ionomer is a useful alternative material and has been placed with a high degree of success over a period of five years in patients with special needs. Clinical reports of Class V restorations show that glass ionomer cement can bear similar comparison with resin composite and compomer (Brackett & others, 1999, 2002; Folwaczny & others, 2001).

The results of this study suggest that a flowable resin composite and dentin adhesive may be suitable for restoring advanced root caries in the case that the proximal carious legion is large, is wrapped around the root surface and is exposed to flexural load. However, there are many factors that contribute to the success of root

caries restoration (Burgess & Gallo, 2002) and more studies are required to determine the effect of other brands in the same classes of materials and other situations in restorations and loadings.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be made:

1. The marginal sealing ability of a flowable resin composite with dentin adhesive under a flexural cycling load was better than other materials.
2. Compared to other materials in this study, a flowable resin composite with dentin adhesive is a desirable alternative for extended root caries restorations.

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Flexural Properties of Eight Flowable Light-cured Restorative Materials, in Immediate vs 24-hour Water Storage

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Clinical Relevance

After 24-hour water storage, the flexural strength and flexural moduli of flowable light-cured resin composites increased 1.5 times or more compared with the immediate condition. As a result, it is suggested that the restoration should be finished after 24 hours. In both conditions, flowable composites wear less than conventional ones.

SUMMARY

This study evaluated the flexural strength, flexural modulus, modulus of resilience and water sorption of eight flowable light-cured restorative materials compared with two conventional restoratives (as control). Forty specimens of each material were made. Twenty specimens were immediately flexural tested, while the remaining 20 were weight-measured and immersed in distilled water in a 37°C incubator. After 24 hours, the samples were weight-measured again to identify water sorption and they were flexural tested. The findings were statistically analyzed using *t*-test, one-way ANOVA, Tukey test and Pearson's Product-Moment Correlation. The results of the flexural strength test were also analyzed using Weibull statistic. All flowable light-cured restorative materials except Palfique Estelite Low Flow exhibited immediate flexural strength values between the conventional ones. All flowable light-cured restorative materials showed 24-hour flexural strength values between the conventional ones. The Weibull modulus for immediate flexural strength of the materials varied from 6.37 to 15.23, while for 24-hour flexural strength, the strength varied from 8.10 to 14.30. In both conditions, all flowable light-cured resin composites showed lower flexural moduli but higher modulus of resilience than the conventional ones. The water sorption of all resin composites was lower than the flowable light-cured compomer. There was a distinct relation (r=-0.84, *p*<0.01) between the increasing ratio in modulus of resilience and the amount of water sorption.

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INTRODUCTION

A lower type viscosity of resin composite has been introduced as a so-called "flowable" composite. This type of

composite is created by retaining the same small particle size of traditional hybrid composites but by reducing the filler content and allowing the increased resin to reduce the viscosity of the mixture (Bayne & others, 1998). This easy-to-flow characteristic was created for the purpose of using it as a liner in areas of difficult access or flow, such as irregular internal surfaces and proximal boxes of Class II preparations (Bayne & others, 1998; Chuang, Liu & Jin, 2001; Jain & Belcher, 2000; Whitters & others, 1999). It is assumed that these less viscous materials flow easily into, adapt to and fill the tooth surface, resulting in less leakage, less internal restoration voids and less post-operative sensitivity (Bayne & others, 1998). Chuang's study showed that the application of flowable composites before conventional composites in Class II cavities reduced the presence of internal restoration voids (Chuang & others, 2001). However, flowable composites were not recommended for Class IV restorations, because they were only acceptable as filling materials in low-stress applications (Bayne & others, 1998).

Fractures in the body and at the margins of restorations have been cited as a major problem regarding the failure of composites. Fracture related material properties, such as fracture resistance, elasticity and the marginal degradation of materials under stress have usually been evaluated by determining the material parameters flexural strength, flexural modulus and fracture toughness (Bouschlicher, Cobb & Boyer, 1999; Manhart & others, 2000).

Flexural strength measurement is, in a sense, a collective measurement of tensile, compressive and shear stresses simultaneously. This characteristic is very important for resin composite filling material, because it can determine whether this filling material can resist mastication forces (Anusavice, 1996; Bouschlicher & others, 1999; Manhart & others, 2000). Therefore, Weibull analysis has been recommended for the study of flexural strength, because Weibull distribution is capable of allowing for skewed data and it is also capable of predicting values within and outside the data set (Cesar, Miranda & Braga, 2001; McCabe & Carrick, 1986; Nery, McCabe & Wassell, 1995; Nomoto, Carrick & McCabe, 2001; Scherrer & others, 2001; Weibull, 1951; Whitters & others, 1999).

Flexural modulus or the modulus of elasticity is a measure of material stiffness. The higher the modulus, the stiffer the material. Marginal breakdown and loss of marginal seal are most likely to occur in products with a lower modulus of elasticity (Anusavice, 1996; Cesar & others, 2001; Reinhardt, Boyer & Stephens, 1994). The energy needed to break the material is expressed by the modulus of resilience (Anusavice, 1996; Peutzfeldt & Asmussen, 1992; Irie & Nakai, 1998).

Polymerization of composites progresses even after being light-cured (Cesar & others, 2001; Mante, Saleh & Mante, 1993). The incomplete polymerization of a resin restorative material may predispose the material to degradation; the unreacted molecules can form the walls of pores within the bulk material, which can be filled with water and cause water sorption (Anusavice, 1996). This characteristic can be advantageous to flowable light-cured composites as a partial compensation for polymerization shrinkage (Huang & others, 2002). For this reason, in addition to the flexural properties, physical behavior, such as 24-hour water sorption, would contribute to a successful restorative material (Estafan, Estafan & Leinfelder, 2000; Irie & Nakai, 1998).

Therefore, this investigation was carried out to evaluate (a) the immediate and 24-hour flexural strength, (b) the immediate and 24-hour flexural modulus, (c) the immediate and 24-hour modulus of resilience and (d) the 24-hour water sorption of flowable light-cured restorative materials. The hypothesis to be tested was that the (a), (b), (c) and (d) properties of flowable light-cured restorative materials would be significantly different compared with the conventional restorative material.

METHODS AND MATERIALS

Seven flowable light-cured resin composites color A3 (Metafil Flo, Palfique Estelite High Flow, Palfique Estelite Low Flow, Revolution Formula 2, Tetric Flow, Unifil Flow, Filtek Flow), one light-cured compomer color A3 (Dyract Flow), one microfilled light-cured resin composite (Silux Plus) color universal and one hybrid minifilled light-cured resin composite (Herculite XRV) color A3 (in this study, both will also be termed as conventional composites) as control, were used in this study. Table 1 lists the details of the materials.

A visible-light curing unit (New Light VL-II, GC, Tokyo, Japan, irradiated diameter: 10 mm) was used for light-activated materials with an irradiation time of 20, 30 or 40 seconds due to the manufacturer's recommendations. Light intensity was checked and maintained at 450 mW/cm^2 before each application of the restorative material, using a radiometer (Demetron/Kerr, Danbury, CT, USA).

Flexural Strength, Flexural Modulus and Modulus of Resilience

The prepared Teflon split mold for flexural strength measurement was 2.0 mm in depth, 2.0 mm in width and 25 mm long. A split mold was used to minimize the stresses exerted on the specimens during their retrieval. Of each material, 40 specimens were made in this mold. The mold was filled with the material, covered with a plastic strip and glass plate, then clamped. After 20 seconds, the glass plate was removed. The specimen was light-cured from the top surface in three

Table 1: *Materials Investigated. Information Provided by the Manufacturers*

Name of Product	Manufacturer	Batch #	Filler	Matrix, Monomer	Curing Time
Dyract Flow***	Dentsply Caulk, Milford, DE, USA	010514	Strontium-alumino-fluoro-silicate glass Filler content: 38 vol% (63 wt%) Filler particle size: 1.60 µm	Phosphoric acid modified polymerizable monomers, carboxylic acid modified macromonomers	20 seconds
Metafil Flo**	Sun Medical, Moriyama, Japan	VV10, VV12, EK1, EK2	Barium silica glass, colloidal silica, TMPT. Filler content: 44 vol% (65 wt%) Filler particle size: 0.01 µm colloidal silica, <1 µm: Ba-glass, 10 µm TMPT	UDMA	30 seconds
Palfique Estelite LV High Flow**	Tokuyama, Tokuyama-shi, Japan	V210Y O, V215171	Silica & silica zirconia Filler content: 49 vol% (68 wt%), Filler particle size: 0.40 µm silica, 0.10 µm silica zirconia	Bis-GMA, TEGDMA	30 seconds
Palfique Estelite LV Low Flow**	Tokuyama, Tokuyama-shi, Japan	V616171, V609YO	Silica & silica zirconia Filler content: 46 vol% (65 wt%), Filler particle size: 0.40 µm silica, 0.10 µm silica zirconia	Bis-MPEPP, TEGDMA	30 seconds
Revolution Formula 2**	Kerr, Orange, CA, USA	012819, 102504	Barium glass & synthetic silica Filler content: 46 vol% (62 wt%), Filler particle size: 1.00 µm	Bis-GMA	40 seconds
Tetric Flow**	Ivoclar Vivadent, Schaan, Liechtenstein	D08465	Inorganic filler (barium glass, ytterbium trifluoride, Ba-Al-fluoro-silicate glass, highly dispersed silicon dioxide + speroid mixed oxide) Filler content: 43.6 vol% (68.1 wt%), (Cavifil) Filler particle size: 0.04 – 3.0 µm, 0.70 µm (average)	Bis-GMA (13.1 wt%) UDMA (11.7 wt%) TEGDMA (6.3 wt%)	40 seconds
UniFil Flow**	GC Corp Tokyo, Japan	0107201	Fluoro-alumino-silicate, silica Filler content: 67 wt% (vol%: Not Available) Filler particle size: 0.7 µm	UDMA (26%) Dimethacylate (7%)	20 seconds
Filtek TM Flow**	3M, St Paul, MN, USA	OBK, 20010104	Silica & silica zirconia Filler content: 47 vol% (68 wt%), Filler particle size: 0.01 – 6.0 µm, 1.50 µm (average)	Bis-GMA, TEGDMA	20 seconds
Silux Plus*	3M, St Paul, MN, USA	1GC, 20010129	Silica Filler content: 43 vol% (55 wt%), Filler particle size: 0.04 µm	Bis-GMA, TEGDMA	40 seconds
Herculite XRV**	Kerr, Orange, CA, USA	112330	Barium silicate Filler content: 59 vol% (78.8 wt%) Filler particle size: 0.6 µm	Bis-GMA TEGDMA	40 seconds

: Conventional light-cured resin composite, **: Flowable light-cured resin composite, *: Flowable light-cured compomer.*

overlapping sections. The specimens were then removed from the mold, the excess materials taken with a silicon carbide bur, then the specimens were polished with sandpaper (#600) to obtain straight surfaces. Prior to testing, the dimensions of the specimens were measured using a digital micrometer (Mitutoyo No 293-421-20, Tokyo, Japan). The maximum accepted specimen size was 2.000 ± 0.020 mm in width and height and 25.000 ± 0.025 mm in length. Twenty specimens were flexural tested immediately (maximum five minutes from the start of light-activation), while the other 20 specimens were immersed in distilled water in a 37°C incubator for 24 hours before being flexural tested.

The flexural strength was measured using the 3-point bending method with a 20-mm span and a crosshead speed of 0.5 mm min^{-1} (Autograph DCS-2000, Shimadzu, Kyoto, Japan) outlined in ISO 9917-2 (1996) (Irie & Nakai, 1998).

A maximum 5 kgf (49 N) external force was applied to the midpoint of the test beam, then the flexural

strength of each material was calculated using the following formula:

$$\sigma = (3PL)/(2bd^2)$$

In this equation, σ is the flexural strength, P the maximum load at the point of the fracture, L the distance between supports, b the width of the specimen and d the thickness of the specimen (Anusavice, 1996; Cesar & others, 2001; EN 24049, 1997).

The flexural modulus was then calculated according to the following equation:

$$E = (P_1 l^3)/(4bd^3\delta)$$

In this equation, E is the flexural modulus, P_1 the load at a selected point of the elastic region of the stress-strain plot, l the distance between the supports, b the width of the specimen, d the thickness of the specimen and δ the deformation of the specimen at P_1 (EN 24049, 1997; Manhart & others, 2000; Reinhardt & others, 1994). The flexural modulus in MPa was subsequently converted to GPa.

Using the value of flexural strength (σ) and flexural modulus (E), the modulus of resilience (R) was computed as follows (Anusavice, 1996; Peutzfeldt & Asmussen, 1992).

$$R = \sigma^2/2E$$

All procedures except for mechanical testing were performed in an air-conditioned room, 23 ± 0.5°C and 50 ± 2% relative humidity.

Change in Weight After 24 Hour/Water Sorption

To study the degree of 24-hour water sorption before immersing in distilled water, the specimens for 24-hour flexural strength measurement were weight-measured with an electric balance (AJ 100, Mettler, Greifensee, Switzerland). The maximum accepted specimen size was 2.000 ± 0.020 mm in width and height and 25.000 ± 0.025 in length, measured using a digital micrometer. The specimens were weight-measured again after immersing in distilled water for 24 hours in a 37°C incubator and dried for one minute on a Kim Wiper. The changes in the specimen's weight immediately after being light-cured and after 24 hours in water were expressed as a percentage (Irie & Nakai, 1998).

Statistical Analysis

The results of the flexural strength test and the flexural modulus were analyzed statistically using the t-test, one-way ANOVA and the Tukey test (Bruning & Kints, 1977) at significance level 0.05. The results of the flexural strength test were also analyzed statistically using the Weibull statistic.

The equation of the Weibull two parameter distribution function used was:

$$Pf = 1 - exp\,[-(\sigma/\sigma_o)^m].$$

In this equation, Pf is the probability of failure and σ is the strength at a given Pf. σ_o is the characteristic strength and m is the Weibull modulus, a constant factor related to dispersion of the failure data. The characteristic strength or normalizing parameter value is the stress level in which 63.21% of the specimens will fail when the value of the Weibull modulus is higher than 1 (Cesar & others, 2001; McCabe & Carrick, 1986; Weibull, 1951). To get the Weibull modulus and characteristic strength, the double logarithm of 1/(1-median rank) was plotted vertically versus the logarithm of the actual data values. A straight line was fitted through the points using the median rank regression method determined by least square regression curve fitting. The exponential of intercept value was the characteristic strength, while the slope was the Weibull modulus. Then, the data were plotted once more in the form of flexural strength versus the probability of failure, and the Weibull distribution function was once more plotted in the form of a Gaussian plot.

The results of the 24-hour water sorption test were analyzed statistically using one-way ANOVA and the Tukey test, while the relationship between the flexural strength-flexural modulus and the increasing ratio in flexural strength, flexural modulus and modulus of resilience-water sorption were determined using the Pearson's Product-Moment Correlation (Bruning & Kints, 1977).

RESULTS

Flexural Strength

The results of the determination of immediate and 24-hour flexural strength of all materials tested are listed in Table 2. Statistical analysis revealed significantly higher flexural strengths for 24-hour water storage specimens compared to immediate specimens (t-test: $p<0.05$). All flowable materials except Palfique Estelite Low Flow exhibited higher immediate flexural strength compared to microfilled composite (Silux Plus), but were lower than hybrid minifilled composites (Herculite XRV). However, after 24 hours, like other flowable materials, Palfique Estelite Low Flow increased its strength and gained flexural strength value between the two conventional composites.

The Tukey statistical analysis of immediate flexural strength revealed the presence of four significant groups ($p<0.05$) in which all materials could be placed, while this statistical analysis of 24-hour flexural strength showed six group differences among the materials ($p<0.05$).

The results of the Weibull analysis for immediate and 24-hour flexural strength values are listed in Tables 3 and 4. It is shown that the Weibull moduli of flowable light-cured composites could be similar to the conventional one, but the characteristic strength values were

Table 2: *Multiple Comparisons of Immediate and 24-hour Flexural Strength Value of the Materials*

Material	Mean (SD) (MPa) Immediate Flexural Strength			24 Hour Flexural Strength			Increasing Ratio+	*p* value (t-test)
Dyract Flow***	73.18	(10.28)	a	93.62	(11.60)	C, D	1.28	<0.05
Metafil Flo**	75.69	(7.35)	a	128.12	(13.62)	A, B	1.69	<0.05
Palfique Estelite LV High Flow**	71.06	(10.21)	a	131.41	(15.71)	A, B	1.85	<0.05
Palfique Estelite LV Low Flow**	40.15	(6.04)	b	82.07	(7.62)	D, E	2.04	<0.05
Revolution**	74.79	(5.22)	a	112.01	(8.92)	C	1.50	<0.05
Tetric Flow**	75.34	(8.65)	a	130.41	(9.72)	A, B	1.73	<0.05
UniFil Flow**	71.37	(5.93)	a	118.35	(13.10)	B	1.66	<0.05
Filtek Flow**	62.42	(10.29)	c	133.12	(9.74)	A	2.13	<0.05
Silux Plus*	60.81	(7.17)	c	70.00	(8.88)	E	1.15	<0.05
Herculite XRV*	90.20	(11.28)	d	157.31	(17.07)	F	1.74	<0.05

n = 20, same letters indicate no significant difference by Tukey test (p>0.05).

** : Conventional light-cured resin composite.*

*** : Flowable light-cured resin composite.*

****: Flowable light-cured compomer.*

+ : Immediate flexural strength as baseline.

Table 3: *The Weibull Analysis of Immediate Flexural Strength of the Materials*

Material	Characteristic Strength (σ_o) (MPa)	Weibull Modulus (m)	Correlation Coefficient (r)	Stress for 1% Chance of Failure $\sigma_{0.01}$ (MPa)	Stress for 10% Chance of Failure $\sigma_{0.10}$ (MPa)	Stress for 90% Chance of Failure $\sigma_{0.90}$ (MPa)
Dyract Flow***	77.77	7.43	0.98	41.89	57.46	87.01
Metafil Flo**	79.08	10.35	0.97	50.80	63.75	85.89
Palfique Estelite LV High Flow**	75.60	7.31	0.96	40.28	55.56	84.74
Palfique Estelite LV Low Flow**	42.80	6.99	0.98	22.16	31.01	48.23
Revolution**	77.23	15.23	0.99	57.09	66.62	81.58
Tetric Flow**	79.26	9.16	0.99	47.97	61.99	86.82
UniFil Flow**	74.13	12.70	0.97	51.60	62.09	79.16
Filtek TM Flow**	66.89	6.37	0.97	32.49	46.98	76.24
Silux Plus*	64.04	8.93	0.99	38.26	49.77	70.31
Herculite XRV*	95.22	8.50	0.98	55.42	73.07	105.03

n = 20

: Conventional light-cured resin composite, ** : Flowable light-cured resin composite, * : Flowable light-cured compomer.*

significantly different. The Weibull modulus varied from 6.37 to 15.23 for immediate flexural strength, while for 24-hour flexural strength, the value varied from 8.10 to 14.19. The characteristic strength for immediate flexural strength varied from 42.80 to 79.26 MPa, and 74.08 to 138.42 MPa for 24-hour flexural strength. This statistical analysis showed coefficient of correlation (r) of the data 0.96 to 0.99.

Flexural Modulus

Table 5 lists the determination of flexural modulus values. Statistical analysis revealed significantly greater flexural moduli for the 24-hour water storage specimens compared to the immediate ones ($p<0.05$).

All flowable light-cured materials showed immediate flexural moduli values less than 2.50 GPa, which made them more flexible than the conventional ones, whose values were more than 3.50 GPa. Water storage for 24-hours in a 37°C incubator significantly increased all materials' flexural moduli ($p<0.05$). However, all flowable light-cured materials still had 24-hour mean flexural modulus values less than the conventional ones, although statistical analysis showed no significant difference between Palfique High Flow and Silux Plus ($p>0.05$).

Modulus of Resilience

The flowable light-cured resin composites, except Palfique Low Flow, listed in Table 6, showed immediate and 24-hour modulus of resilience significantly higher than flowable light-cured compomer and conventional light cure composites ($p<0.05$). The immediate modulus

Table 4: *The Weibull Analysis of 24-hour Flexural Strength of the Materials*

Material	Characteristic Strength (σ_o) (MPa)	Weibull Modulus (m)	Correlation Coefficient (r)	Stress for 1% Chance of Failure $\sigma_{0.01}$ (MPa)	Stress for 10% Chance of Failure $\sigma_{0.10}$ (MPa)	Stress for 90% Chance of Failure $\sigma_{0.90}$ (MPa)
Dyract Flow***	98.83	8.52	0.96	57.61	75.90	108.99
Metafil Flo**	134.49	9.68	0.97	83.60	106.58	146.60
Palfique Estelite LV High Flow**	138.42	8.88	0.99	82.45	107.43	152.06
Palfique Estelite LV Low Flow**	85.57	11.39	0.99	57.13	70.23	92.08
Revolution**	116.23	13.04	0.98	81.67	97.80	123.91
Tetric Flow**	134.96	14.19	0.96	97.60	115.17	143.12
UniFil Flow**	124.76	8.84	0.97	74.15	96.73	137.11
Filtek TM Flow**	137.73	14.30	0.96	99.84	117.67	146.00
Silux Plus*	74.08	8.10	0.98	41.98	56.11	82.11
Herculite XRV*	165.01	9.79	0.99	103.12	131.11	179.69

n = 20

: Conventional light-cured resin composite, ** : Flowable light-cured resin composite, * : Flowable light-cured compomer.*

Table 5: *Multiple Comparisons of Immediate and 24-hour Flexural Modulus of the Materials*

Material	Mean (SD) (GPa) Immediate Flexural Modulus	Mean (SD) (GPa) 24 Hour Flexural Modulus	Increasing Ratio+	*p* value (*t*-test)
Dyract Flow***	2.13 (0.21) a	4.52 (0.19) B, C	2.12	<0.05
Metafil Flo**	1.95 (0.22) a	4.17 (0.25) C, D	2.14	<0.05
Palfique Estelite LV High Flow**	2.05 (0.27) a	4.92 (0.68) A , B	2.40	<0.05
Palfique Estelite LV Low Flow**	1.05 (0.15) c	2.52 (0.20) F	2.40	<0.05
Revolution**	1.44 (0.19) b	3.47 (0.29) E	2.41	<0.05
Tetric Flow**	2.16 (0.31) a	4.71 (0.36) A, B	2.18	<0.05
UniFil Flow**	1.47 (0.27) b	3.74 (0.35) D, E	2.54	<0.05
Filtek TM Flow**	1.38 (0.09) b	4.22 (0.26) C	3.06	<0.05
Silux Plus*	3.88 (0.21) d	5.15 (0.33) A	1.33	<0.05
Herculite XRV*	5.62 (0.50) e	10.40 (0.81) G	1.85	<0.05

n = 20, same letters indicate no significant difference by Tukey test (p>0.05).

** : Conventional light-cured resin composite.*

***: Flowable light-cured resin composite.*

****: Flowable light-cured compomer.*

+ : Immediate flexural modulus as baseline.

of resilience values of flowable composites ranged between 0.79 and 1.98 MJ/m^3, while the 24-hour values ranged between 1.26 and 2.06 MJ/m^3.

In 24-hour water storage, almost all materials increased their modulus of resilience. However, the modulus of resilience of Dyract Flow decreased significantly, while the modulus of resilience of Revolution decreased insignificantly. Silux Plus was the exception, its modulus of resilience value remained the same.

Change in Weight After 24 Hour/Water Sorption

The values of water sorption, ranging from 0.22% to 1.10%, could be categorized into four different groups (Table 7). All flowable light-cured composites could be categorized into two groups and showed water sorption values less than 0.51%. These values were lower than the water sorption value of Silux Plus (0.77%) ($p<0.05$), but higher or not significantly different from Herculite XRV (0.26%). However, the flowable light-cured compomer (Dyract Flow) exhibited a higher water sorption value than all the resin composite materials ($p<0.05$).

DISCUSSION

In this study, flexural strength and modulus testing was based on ISO 4049, which is commonly employed in dental research (Yap & others, 2002), with the 2-mm height of the sample as the maximum dimension permissible for effective polymerization of composites. Twenty was the minimum number of samples needed for The Weibull distribution analysis (McCabe & Carrick, 1986).

It was reported that there was no correlation between the fracture toughness value and the filler content by volume of flowable composites (Bonilla, Yashar &

Table 6: *Multiple Comparisons of Immediate and 24-hour Modulus of Resilience of the Materials*

Material	Mean (SD) (MJ/m³) Immediate Modulus of Resilience	24 Hour Modulus of Resilience	Increasing Ratio+	*p* value (*t*-test)
Dyract Flow***	1.28 (0.41) c	0.81 (0.39) E	0.63	<0.05
Metafil Flo**	1.50 (0.31) b, c	1.99 (0.38) A, B	1.33	<0.05
Palfique Estelite LV High Flow**	1.31 (0.33) c	1.62 (0.44) B, C	1.24	<0.05
Palfique Estelite LV Low Flow**	0.79 (0.24) d	1.26 (0.30) C, D	1.59	<0.05
Revolution**	1.98 (0.38) a	1.91 (0.43) A	0.96	>0.05
Tetric Flow**	1.35 (0.33) c	1.87 (0.38) A, B	1.39	<0.05
UniFil Flow**	1.82 (0.37) a, b	1.97 (0.42) A, B	1.08	>0.05
Filtek TM Flow**	1.50 (0.50) b, c	2.06 (0.41) A	1.37	<0.05
Silux Plus*	0.47 (0.12) d	0.47 (0.09) E	1.00	>0.05
Herculite XRV*	0.74 (0.20) d	1.22 (0.30) D	1.65	<0.05

n = 20, same letters indicate no significant difference by Tukey test (p>0.05).

** : Conventional light-cured resin composite.*

*** : Flowable light-cured resin composite.*

****: Flowable light-cured compomer.*

+ : Immediate modulus of resilience as baseline.

Table 7: *Multiple Comparisons of 24-hour Water Sorption of the Materials*

Material	Immediate Weight	24 Hour Weight (g)	Water Sorption (%)	Tukey Test
Dyract Flow***	0.1733 (0.0022)	0.1752 (0.0022)	1.10 (0.15)	C
Metafil Flo**	0.1766 (0.0020)	0.1770 (0.0020)	0.22 (0.04)	B
Palfique Estelite LV High Flow**	0.1748 (0.0018)	0.1757 (0.0018)	0.50 (0.06)	A
Palfique Estelite LV Low Flow**	0.1682 (0.0017)	0.1687 (0.0017)	0.27 (0.04)	B
Revolution**	0.1499 (0.0017)	0.1506 (0.0017)	0.46 (0.06)	A
Tetric Flow	0.1875 (0.0027)	0.1881 (0.0027)	0.33 (0.08)	A, B
UniFil Flow**	0.1749 (0.0045)	0.1756 (0.0045)	0.38 (0.08)	A, B
Filtek TM Flow**	0.1728 (0.0017)	0.1737 (0.0017)	0.49 (0.03)	A
Silux Plus*	0.1528 (0.0039)	0.1539 (0.0039)	0.77 (0.08)	D
Herculite XRV*	0.1974 (0.0042)	0.1979 (0.0042)	0.26 (0.07)	B

( ) = SD, n = 20, same letters indicate no significant difference by Tukey test (p>0.05).

** : Conventional light-cured resin composite.*

*** : Flowable light-cured resin composite.*

****: Flowable light-cured compomer.*

Caputo, 2003). However, compared with the flexural strength mean values of BisGMA-TEGDMA (composition 3:7–7:3) experimental resin composites that vary from 140 to 152 MPa (Asmussen & Peutzfeldt, 1998), the addition of filler causes a change in the flexural strength of the composites. This condition can be observed in Palfique Estelite High Flow, Filtek Flow, Silux Plus and Herculite XRV, which have a BisGMA-TEGDMA matrix monomer.

A previous study mentioned that filler content, filler size and distribution of the filler particles of light-cured restoratives correlate with the material strength and elastic modulus (Jain & Belcher, 2000). In this study, the presence of less filler volume in Dyract Flow might cause a lower increase in flexural strength (1.28 times) compared with other flowable light-cured composites or Herculite XRV, which could increase its flexural strength 1.5 times or more in 24-hours, 37°C water storage. Although increased filler volume caused an increase in mechanical strength and flexural modulus (Manhart & others, 2000; Braem & others, 1989; Venhoven & others, 1996; Kugel, 2000), various filler contents, differences in filler particle size and stiffness made it difficult to compare the relation of the filler contents with the mechanical properties. Moreover, the shape, composition, interparticle spacing and surface treatment of the filler may have contributed to the mechanical properties of the composite materials (Manhart & others, 2000).

The Weibull distribution analysis offers the ability to predict the dependability of a material (McCabe & Carrick, 1986). The values of $\sigma_{0.01}$ and $\sigma_{0.90}$ (Tables 3 and 4) indicate the stress levels at which one would expect a 1% and 90% chance of fracture, respectively. There were wide variations in the predicted values of $\sigma_{0.01}$ and $\sigma_{0.90}$ among the 10 sample groups, as well as a variation in the values of the Weibull modulus. However, clinical failure is often judged to occur as a

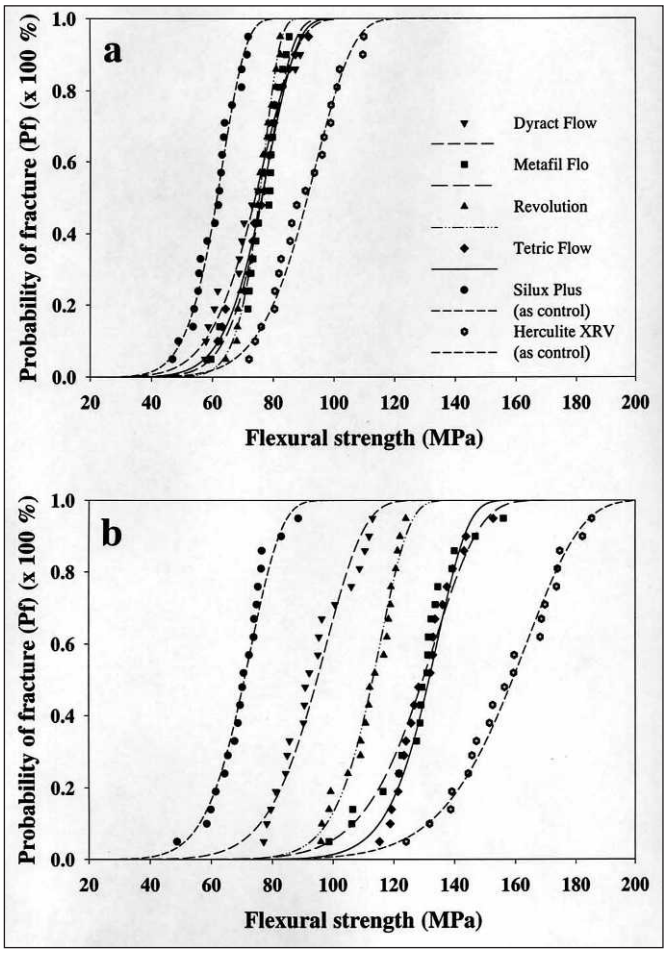

Figure 1*a. Weibull plots of the cumulative probability of fracture vs immediate flexural strength.*
1b. Weibull plots of the cumulative probability of fracture vs 24-hour flexural strength.

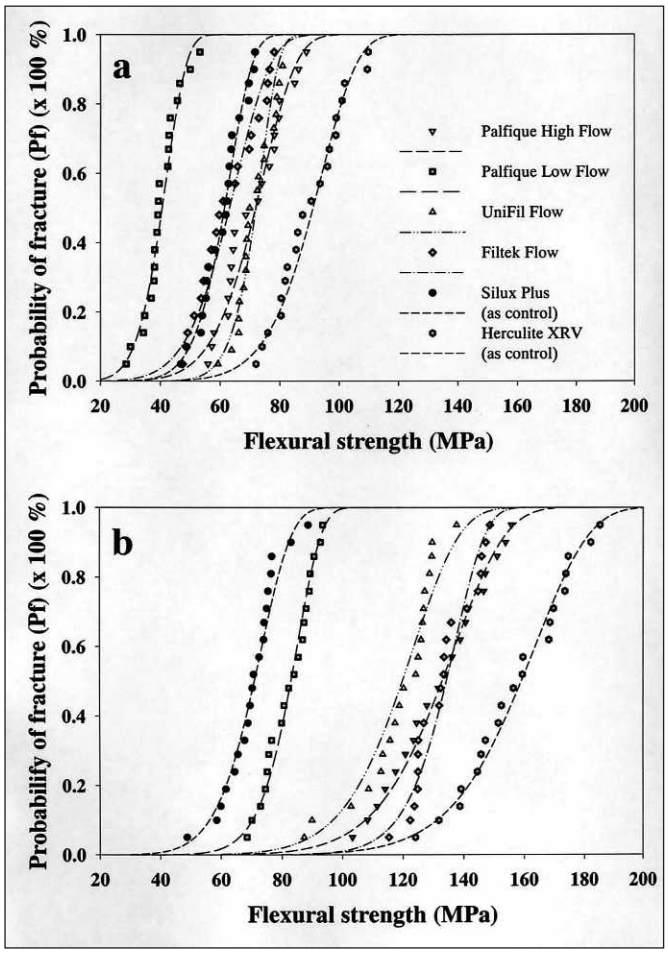

Figure 2*a. Weibull plots of the cumulative probability of fracture vs immediate flexural strength.*
2b. Weibull plots of the cumulative probability of fracture vs 24-hour flexural strength.

result of only 10% to 20% failures, and this is unlikely to be predicted by a consideration of mean values of any mechanical property. Therefore, a 10% probability of fracture caused by flexural stress was also analyzed (Whitters & others, 1999). This Weibull analysis can also be used to predict the probability of failure of these restorative materials under any level of flexural stress application (McCabe & Carrick, 1986). The Weibull cumulative Gaussian plots (Figures 1 and 2) show the prediction that after 24-hours all flowable restorative specimens and about 75% of the Herculite XRV samples will be fractured by a 170 MPa stress application, while Silux Plus specimens will be completely fractured by a 100 MPa flexural stress application. In spite of the fact that all Palfique Estelite Low Flow immediate samples will be fractured by a flexural stress of 60 MPa, the other immediate flowable restorative materials will be completely fractured by a 100 MPa flexural stress application. Although a previous study has reported that traditional composites demonstrated better performance in all mechanical property tests compared to any of the flowable composites (Bayne & others, 1998), this study showed that the flexural strength of flowable composites was in the range of the flexural strength of conventional materials.

Flexural strength and flexural modulus are mathematically connected properties and, as expected, there was a distinct relation ($r=0.74$, $p<0.01$) between the increasing ratio in flexural strength and the increasing ratio in flexural moduli (Figure 3). If the 24-hour flexural strength values were assumed to be the maximum flexural strength of the composites, immediately after being light cured, the flowable composites got only 47% to 67% of their full strength less than the flowable compomer, which got 78%, while the conventional composites got 57% and 87% of their full strength. These results showed that in 24 hours, the additional cross-linking reactions of the resin component could still be in progress, which led to an increase in flexural strength and modulus (Yap & others, 2002). The resin matrix of composites is also known to absorb an amount of water, usually a small percentage, which changes the magni-

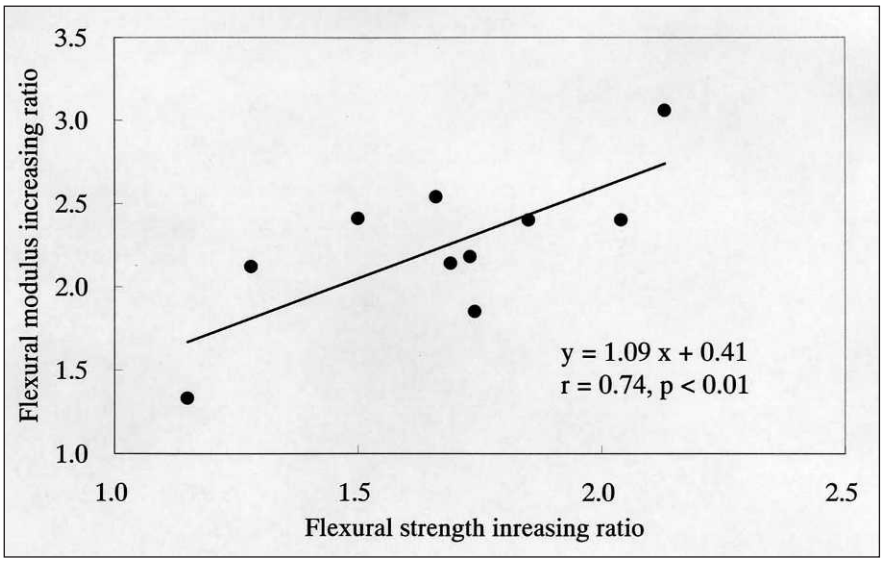

Figure 3. *Flexural strength increasing ratio vs flexural modulus increasing ratio.*

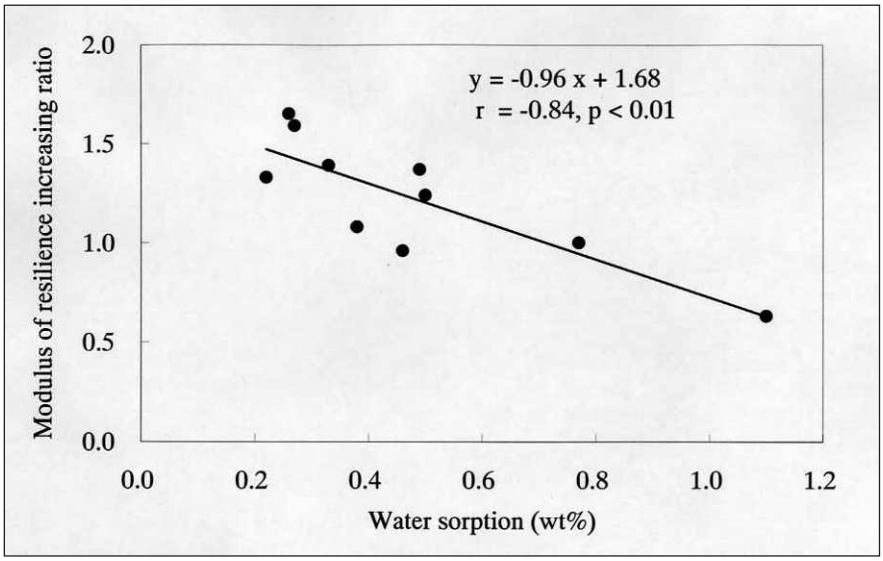

Figure 4. *Water sorption vs modulus of resilience increasing ratio.*

tude of some physical properties (Söderholm, 1984). Besides, hoop stresses also exist around the filler particles as a result of matrix shrinkage during polymerization. These hoop stresses increase the frictional forces between the filler and the resin matrix, thereby decreasing the filler pullout tendency during flexural testing (Söderholm, 1984; Yap & others, 2002). All of these polymerization progresses also lead to changes in the modulus of resilience. Except for Dyract Flow, Revolution and Silux Plus compared to the immediate condition, the materials showed a significant increase in their modulus of resilience values, which means that after 24 hours, more energy is needed to break the material. Along with the fact that all 24-hour water storage specimens showed higher flexural strengths than the immediate specimens, grinding and polishing the restoration after 24 hours is suggested, since restorative materials easily acquire surface flaws and scratches during finishing procedures (Anusavice, 1996).

As mentioned before (Peutzfeldt & Asmussen, 1992), the flexural strength and modulus of resilience were well correlated with quantitative clinical wear data. Specifically, flexural strength and modulus of resilience seem to reflect *in vivo* wear performances. If little energy is needed to break the material, it seems that cracks form more readily and, thus, result in an increased wear rate. Consequently, flowable composites that have relatively high modulus of resilience will wear less than conventional ones, which have a lower modulus of resilience. These results are in line with the fact that the fracture toughness of flowable composites is higher than packable resin composites (Bonilla & others, 2003) and the fracture toughness of packable resins is higher or has no significant difference from conventional ones (Knobloch & others, 2002), which indicates that flowable composites are more resistant to crack propagation. However, previous studies have revealed no significant wear rate for tooth abrasion between flowable and condensable composites (Bayne & others, 1998). Flowable composites have been shown to reduce the potential for void formation and microleakage under a condensable composite (Estafan & others, 2000). The higher shrinkage of these materials and the associated increase in interfacial stresses may be offset by their lower rigidity (Labella & others, 1999). However, the use of flowable composites as a restorative material in occlusal cavities is still under consideration, since the report of wear-simulator results showed more or less resistance than conventional composites (Yau, Perry & Kugel, 1997; Yu, Glace & Chadwick, 1997).

In earlier studies, it has been mentioned that water softens composites by penetrating the matrix and leaching unreacted monomer and unbound components (Cesar & others, 2001; Mante, Saleh & Mante, 1993). In this study, 24-hour water sorption seemed to influence the flexural strength (r=-0.65, $p<0.05$), but there was a more distinct relation (r=-0.84, $p<0.01$) between the increasing ratio in modulus of resilience and the amount of water sorption (Figure 4). Therefore, the

higher the water sorption, the lower the energy needed to break the material. Actually, the value of water sorption is influenced by many conditions. The particle size of flowable composite fillers, hybrid minifillers, such as Herculite XRV, might lead them to the same group water sorption value as Herculite XRV. However, the hydrophilic characteristic of the matrix monomer in Dyract Flow might cause more water sorption and therefore lead to the higher water sorption value. Besides the degree of polymerization and filler content, the difference in values of water sorption also exist with respect to the difference in chemical composition of the matrix monomer and the diffusion coefficients due to surface and grain boundary diffusion (Cattani-Lorente & others, 1999; Glasspoole, Erickson & Davidson, 2001; Kalachandra, 1989; Manhart & others, 2000; Yap & others, 2002).

CONCLUSIONS

Flowable composites were in the range of conventional composites (Silux Plus and Herculite XRV) in terms of flexural strength and water sorption. All materials in this research showed a significant increase in flexural strength and modulus value in 24-hour water storage. The modulus of resilience value of flowable composites was higher than that of conventional ones. The higher the 24-hour water sorption, the lower the energy needed to break the light cure restorative materials.

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Influence of Disinfectants on Dentin Bond Strength of Different Adhesive Systems

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U Blunck • T Attin

Clinical Relevance

Contamination of human dentin with disinfectants in the waterlines of dental units may have an influence on dentin bonding, depending on the adhesive system used.

SUMMARY

The influence of water disinfectants used in dental unit waterlines on the dentin bonding of different adhesive systems was investigated by using push-out tests. Three hundred and twenty dentin disc specimens were prepared from caries-free human molars. In each specimen, a standardized conical cavity was prepared while cooling with water from a dental unit containing one of three different disinfectants (n=80 each group; A=control: water without disinfectant, B: Alpron neutral, C: Alpron mint, D: Dentosept P). Subsequent rinsing of the cavities was performed with the respective disinfectant. The cavities were filled with the following combinations of dentin adhesives and composites, resulting in 16 subgroups (n=20): Syntac Classic/Tetric Ceram, Clearfil Liner Bond 2V/Luxacore, OptiBond FL/Prodigy and Prime&Bond NT/Spectrum. After polishing the fillings, one half of each subgroup (n=10) was stored in water (37°C) for 24 hours. The other half was stored in water (37°C) for 180 days and additionally thermocycled (2000 cycles at 5/55°C). The bond strength was then measured by push-out tests. Statistical analysis of the data was carried out using ANOVA and pairwise *t*-tests (Significance level $p \leq 0.01$). The disinfectants showed no significant influence on the loads required for debonding of Syntac Classic/Tetric Ceram, Clearfil Liner Bond 2V/Luxacore and OptiBond FL/Prodigy as compared to the controls. However, the use of disinfectants in the water supply of a dental unit decreased dentin bond strength in the specimens filled with Prime&Bond NT/Spectrum.

Disinfectants in the water of dental unit waterlines may have an influence on dentin bonding, depending on the adhesive system used.

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INTRODUCTION

Bacterial contamination and biofilm formation can occur in dental unit waterlines, which are normally connected to the public water supply. With regard to patients with immune deficiencies, such as patients with a high endocarditis risk or patients with organ transplants, there is the possible hazard of getting a bacterial infection after dental treatment due to bacteria in the waterlines. In some studies, it has been reported that antimicrobial agents are effective in preventing bacterial contamination of dental unit waterlines (Murdoch-Kinch & others, 1997; Eleazer, Schuster & Weathers, 1997; Karpay & others, 1999). It is a matter of common knowledge that proliferation of bacteria can also occur in dental unit waterlines treated with antimicrobial agents at regular intervals (daily or weekly purging with disinfectants). The permanent addition of low concentration disinfectants to waterlines is an opportunity to control bacterial contamination in some dental units with no independent water system. Some of these disinfectants have active ingredients such as alcohol, hydrogen peroxide or ethylenediaminetetra acetic acid-disodium salt (editin or EDTA). It is conceivable that these substances may have an impact on dentin structure, because it has been concluded that some dental unit waterline antimicrobial agents may adversely affect dentin bonding strength (Roberts, Karpay & Mills, 2000). The modern insertion of adhesive fillings and ceramic restorations is based on the use of bonding agents requiring a conditioning of the dentinal structure. Moreover, for some dentin adhesives, it is necessary to remove or modify the smear-layer prior to applying the adhesive. Smear-layer removal is typically performed by acid etching of the dentin with orthophosphoric acid followed by rinsing of the cavity with copious amounts of water provided by the waterline of the dental unit. In this case, it may be assumed that remnants of the disinfectant may remain on the surface of the cavity after intensive rinsing. Therefore, it is conceivable that disinfectants in the dental unit waterline may adversely influence dentin bonding of all adhesive systems independent of their application steps and the way they deal with the smear layer. The purpose of this investigation was to test whether the use of waterline disinfectants has an influence on dentin adhesion of adhesive systems with different mechanisms of adhesion.

METHODS AND MATERIALS

The set up of the experiment is presented in Figure 1. Three hundred and twenty extracted sound human wisdom teeth were stored at room temperature for less than four weeks in 0.1% thymol solution and embedded in acrylic resin (Paladur, Heraeus Kulzer GmbH & Co KG, Hanau, Germany). With a water-cooled diamond grinding machine (Micro-Grinding-System, Exakt Apparatebau, Norderstedt, Germany), 320 dentin discs 2.2-mm thick were produced by two cuts in a horizontal direction. Prior to experimental use, the specimens were stored in distilled water. With the use of a mortice dowel joint, the discs fit the test control unit exactly. Under standardized conditions, conical cavities were prepared with cooling water containing either disinfectant additives or no additives (control group). Each cavity was produced using conical diamond burs (pilot hole, preparation and burnishing instrumentation) from the Cerafil-System (Gebr Brassler/Komet, Lemgo, Germany) which were mounted in a high speed contra angle handpiece connected with the waterline of the dental unit. (Sirona C1 Titan classic, Sirona Dental Systems GmbH, Bensheim, Germany). Due to this procedure, cavities of 4° tapering with an apical diameter of 2.1

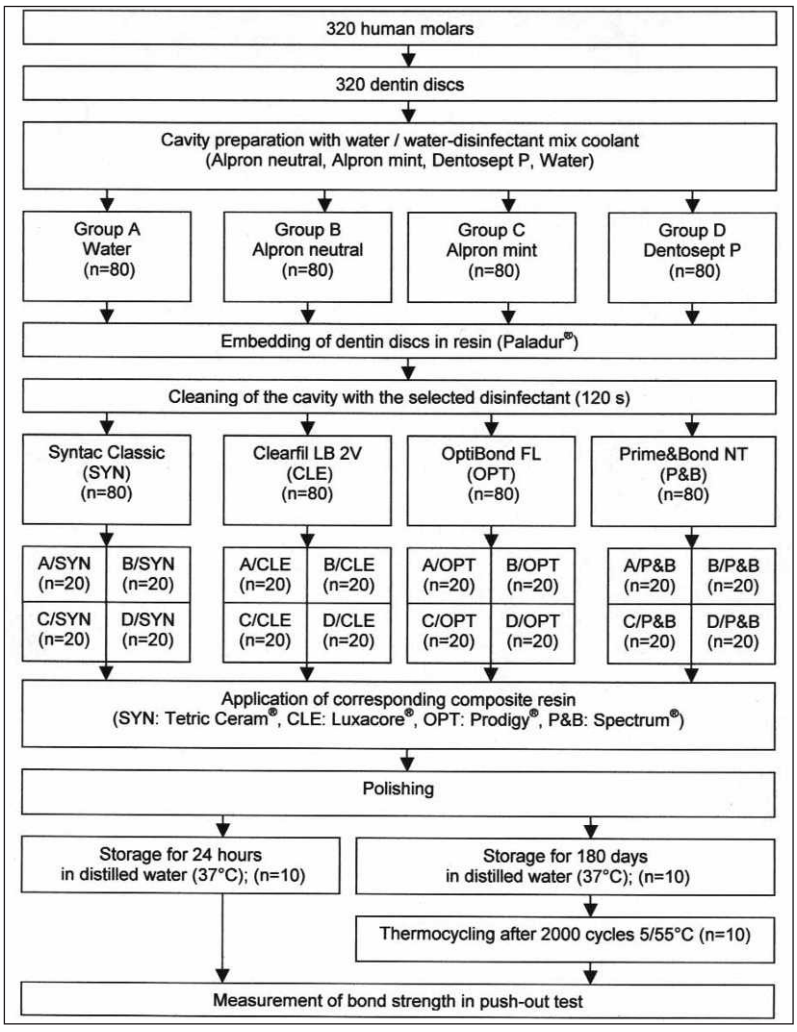

Figure 1. *Setup of the experiment.*

mm and a coronal diameter of 2.5 mm were produced (Figure 2). The dentin samples were exposed to the cooling solution for exactly 20 seconds during the drilling procedure. The cavities were rinsed for 120 seconds after preparation with the cooling solution using the multipurpose syringe of the dental unit. During preparation of the specimens, the contra angle handpiece was fixed in a special apparatus that allowed for a standardized drill vertical to the surface of the specimens. The flow rate of cooling water was adjusted to 50 ml/minute. The specimens were randomly assigned to four groups according to the three disinfectants (Alpron neutral, Alpron mint and Dentosept P) and one control group (H_2O; see Table 1). Within each group, a further random distribution was made according to the four adhesive systems applied in the study.

Fabrication of the samples and preparation of the cavities were performed on four different dental units, Type Sirona C2 (Sirona Dental Systems GmbH, Bensheim, Germany), which stood in the same room and were connected to the same water conductions. The dental units contained a built-in disinfection unit for continuous disinfection of instrument coolant and rinsing water. Prior to the experiment, the above mentioned dental units were cut off from the public water supply and all water sources were cleaned thoroughly with NaOCl for 20 minutes. The lines of the units were then rinsed with fresh water (control unit) or one of the freshly mixed water-disinfectant solutions for 30 minutes. All water-disinfectant mixes were freshly produced immediately before use. According to the manufacturers' instructions, the mixing ratio 1:100 (10 ml disinfectant : 1 l H_2O) was chosen. Application of the different adhesive systems (Syntac Classic/Tetric Ceram [SYN], Clearfil Liner Bond 2V/Luxacore [CLB 2V], OptiBond FL/Prodigy [OPT FL] and Prime&Bond NT/Spectrum [P+B NT]) was done according to the manufacturer's instructions. The 320 discs were placed on microscope glass slides and the composite materials (Tetric Ceram, Luxacore, Prodigy or Spectrum) filled the cavities (n=80 for each material) in increments using hand instruments. After applying the final increment, a microscope glass slide was put on top of the discs to achieve optimal condensation. Light curing of the composite was performed from both sides of the specimens using a polymerization lamp (Translux CL, Kulzer, Friedrichsdorf, Germany) at an energy intensity of 500 mW/cm^2 for 30 seconds for each side. The fillings were made with the self-curing material Luxacore, which was condensed with the microscope glass slide for five minutes. After polymerization, excess material was removed under a stereomicroscope (40x magnification) and the fillings were polished with flexible aluminum oxide polishing discs (Sof-Lex, 3M ESPE Dental Products, St Paul, MN, USA). The fillings were confined to the cavities only, without any overhang on the bottom and top surfaces of the dentin discs. Ten specimens of each adhesive/disinfectant combination were stored for 24 hours (37°C), with a second group of 10 specimens stored for 180 days in distilled water (37°C). Furthermore, the specimens that were stored for 180 days in water were thermocycled (2000 cycles; 5/55°C) in a microprocessor-controlled thermocycling machine (Thermo Haake GmbH, Karlsruhe, Germany). The dwell time in each bath was 30 seconds. The measurement of bond strength was performed with a push-out test as described by Kimura (1985), Haller and others (1993), Frankenberger and others (1998) and Frankenberger, Krämer and Sindel (1996). The dentin discs were placed in a holding device and fixed with an autopolymerizing resin (Palavit G, Heraeus Kulzer GmbH & Co KG, Hanau, Germany). In a universal testing machine (Zwick Universal Testing Machine No 1446, Zwick GmbH & Co KG, Ulm, Germany), a steel rod was used to apply a force vertically on the filling until

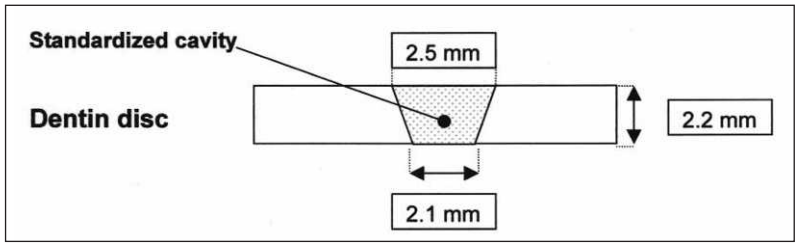

Figure 2. *Schematic drawing of the dentin disc obtained from wisdom teeth showing the dimensions of the dentinal cavity.*

Table 1: *Composition of Disinfectants*

Disinfectant	Composition
Control group	H_2O
Alpron neutral (Alpro Dental Produkte GmbH, St Georgen, Germany)	15-30% 1,2 propylenglycol 2-5% polyethyleneglycol 300 0.5-2% phenoxyethanol <0.5% polyaminopropylbiguanid phenylalanine sorbitol
Alpron mint (Alpro Dental Produkte GmbH, St Georgen, Germany)	2-5% ethylenediaminetetra acetic acid-disodium salt (EDTA) <0.5% polyaminopropybiguanid <0.2% sodium-p-toluolsulfonechloramide phenylalanine sorbitol
Dentosept P (Sirona Dental Systems GmbH, Bensheim, Germany)	1.41% hydrogen peroxide

rupture occurred. The rod was connected to the crosshead beam which moved with a speed of 0.5 mm/minute and pushed out the fillings of the dentin discs through a drilling in the holding support. The bond strength was defined as the ratio of the force needed for push-out and the adhesion area of cavity walls. Due to the special cavity design, the adhesion area had to be calculated to the following formula:

$A(M) = \pi \times S \times (R1+R2) = \pi /2 \times S \times (D1+D2)$

with:

A(M): adhesion area

S: surface line; $S=\sqrt{(R1-R2)^2+h^2}$; h=altitude of the conical cavity

R1: smaller radius of the conical cavity

R2: greater radius (base) conical cavity

D1: smaller perimeter of the conical cavity

D2: greater perimeter of the conical cavity

The bond strength or force of adhesion was calculated according to the formula:

$D = F$ (measured force)$/A(M)$ (adhesion area) $= F/\pi/2 \times \sqrt{((R2-R1)^2 + h^2)} \times (D1+D2)$.

Statistical Analysis

Statistical analysis of the data was carried out using analysis of variance (ANOVA) and pairwise *t*-tests to check the influence of disinfectants and the duration of water storage on dentin bond strength. Significance was set at $p \leq 0.01$. Pairwise comparisons were made applying the Bonferroni correction, with $p \leq 0.05$ as being significant. Due to the varying shrinkages among the resin composite materials used (linear shrinkage values according to manufacturer's specifications: Tetric ceram 2.75 vol%; Prodigy 2.8 vol%; Spectrum 2.46 vol%; Luxacore 3-4 vol%), comparisons were made within each group of the four adhesive systems only.

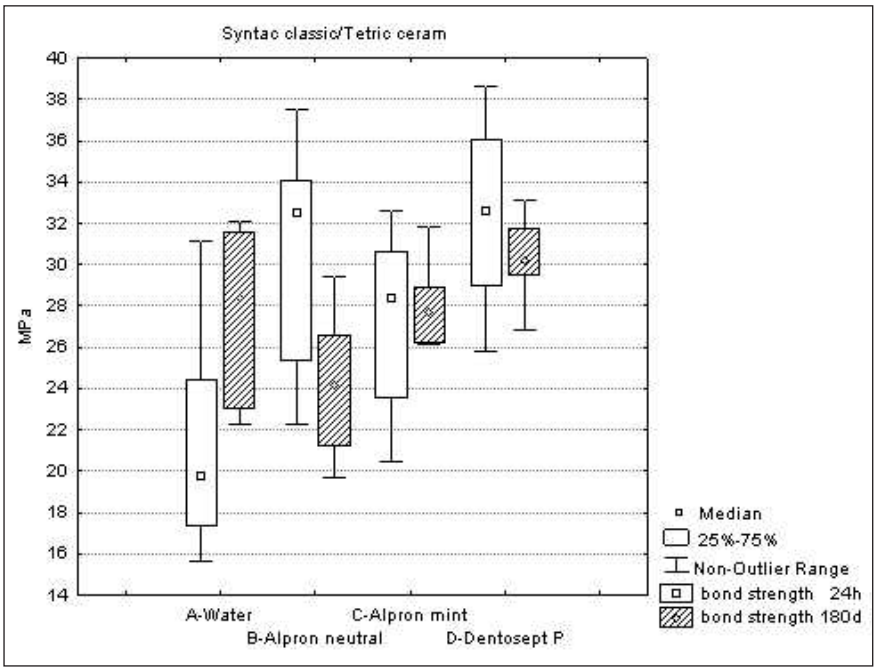

Figure 3. *Bond strength of Syntac Classic/Tetric Ceram filling.*

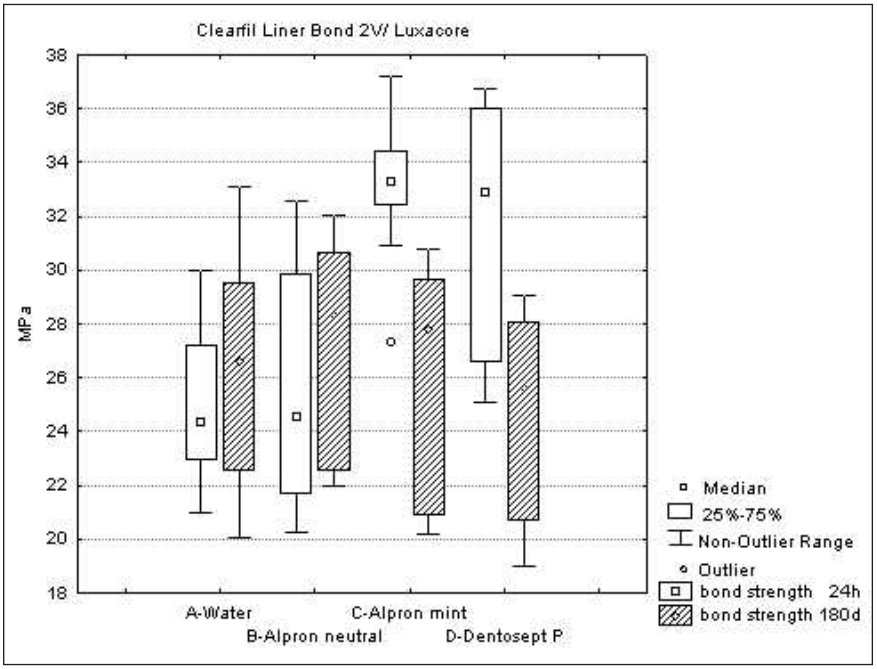

Figure 4. *Bond strength of CLB/Luxacore filling.*

RESULTS

The disinfectants did not show a significant influence ($p<0.01$) on the loads required for debonding of Syntac Classic/Tetric Ceram, Clearfil Liner Bond 2V/Luxacore and OptiBond FL/Prodigy after 180 days water storage and thermocycling treatment as compared to the controls (Figures 3 through 5).

However, the use of disinfectants in the water supply decreased dentin bond strength in the specimens filled with Prime&Bond NT/Spectrum (Figure 6). Reduced bond strength values were investigated after 180 days water storage and thermocycling for Alpron neutral ($p=0.000595$), Alpron mint ($p=0.000254$) and Dentosept P ($p=0.000137$) as compared to the controls. Additionally, after this time, significantly lower adhe-

sion values were investigated in subgroups for specimens treated with Dentosept P (p=0.000003) and Alpron neutral (p=0.000002) compared to their 24-hour water storage values.

Pairwise comparisons of adhesive system/disinfectant combinations showed singularly significant differences in adhesive strengths at different times (24 hour water storage vs 180 days water storage and thermocyling treatment) as compared to the controls (water). Specimens of the Syntac/Tetric group showed significantly higher bond strength values after 24 hours when the cavities were treated with Alpron neutral (p=0.000428) or Dentosept P (p=0.000051). In the CLB 2V/Luxacore group, significantly higher bond strength values after 24 hours were found for specimens that were treated with Alpron mint (p=0.000002) and Dentosept P (p=0.000828) compared with the control. In these groups, no significance in bond strength was investigated after 180 days water storage and thermocycling.

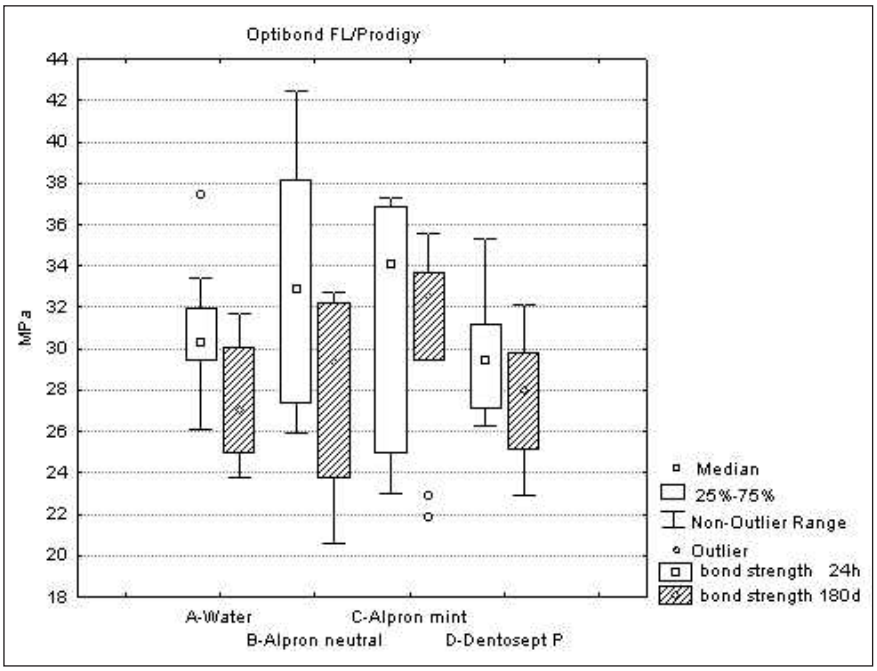

Figure 5. *Bond strength of Optibond FL/Prodigy filling.*

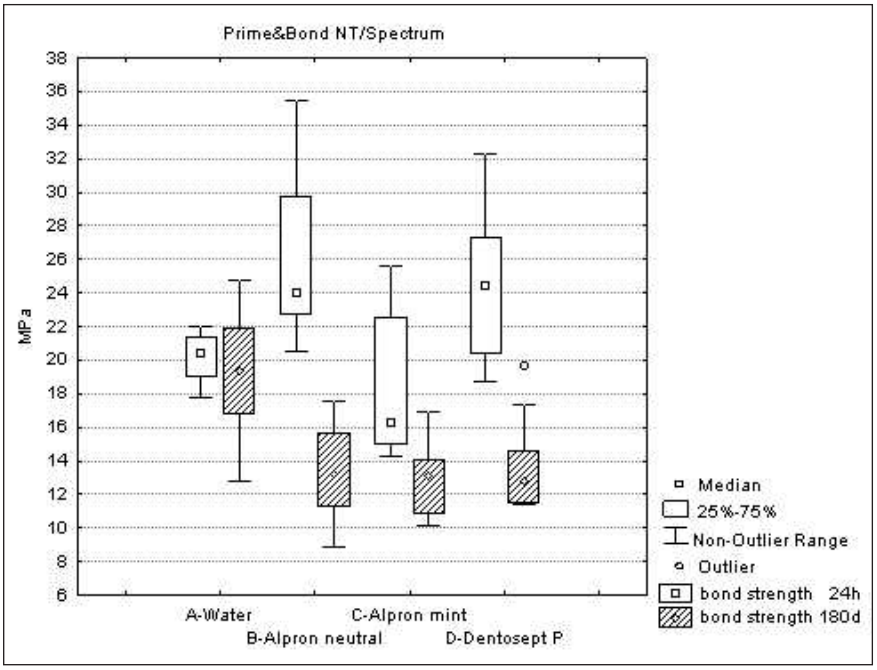

Figure 6. *Bond strength of Prime&Bond NT/Spectrum filling.*

DISCUSSION

Little is known about antimicrobial agents and how they affect adhesion strength when continuously provided in the cooling water of dental units. Various formulations of antimicrobial agents of disinfectants have been shown to be effective in reducing bacterial contamination in dental units, such as chlorhexidine gluconate (Blake, 1963; McEntegart & Clark, 1973), metallic ions (Hesselgren & Nedlich, 1979), hydrogen peroxide (Kellett & Holbrook, 1980), sodium hypochlorite (Abel & others, 1971; Fiehn & Henriksen, 1988; Pankhurst & others, 1990) and mouth rinses (Knight, Davis & McRoberts, 2001). Antimicrobial agents may reduce the enamel bond strength of composite materials (Taylor-Hardy & others, 2001). In a recent study, it was concluded that dental waterline antimicrobial agents may adversely affect dentin bond strength (Roberts & others, 2000). This research also found a negative influence of disinfectants on dentin bond strength; this effect was dependent on the adhesive system used. It has to be noted that different antimicrobial agents were used as disinfectants in contrast to the study by Roberts and others (2000). The measuring of dentin bond strength was performed with a push-out test according to former reports by Haller and others (1993). Due to this design, the effects of the disinfectants could be tested in cavities that were prepared using the disinfectants as cooling solutions. The shape of the push-out samples had a cavity-like configuration; thereby, the conditions during preparation of a cavity for restoring a tooth could be simulated. Moreover, the conical design of the push-out

samples limits frictions effects (Frankenberger & others, 1996, 1998). Therefore, the push-out test was selected as an appropriate method for measuring the adhesive potential of the various bonding systems used in this investigation. In contrast to conventional tensile shear tests, the progress of stress in the composite-dentin-interface is more homogeneous (Van Noort & others, 1989). Bending moments and fatigue strengths could be largely avoided. To minimize polymerization shrinking of the composite materials, the cavities were filled using an incremental technique.

For this study, resin-based adhesive systems with different mechanisms of adhesion have been used to investigate the disinfectant's influence. Varying formulations of light curing resin materials used lead to differences in their polymerizing shrinkages. To exclude these differences, statistical comparisons were made for each adhesive system only. Therefore, for all groups within the same adhesive system, the shrinkage was constant. Due to the design of this study, it is important to take into consideration that corresponding bond strength values may be different depending on the adhesive system used. These adhesive systems can be classified on the basis of the number of clinical application steps and their way of dealing with the smear layer (Van Meerbeek & others, 1998). Syntac Classic was selected as exemplary as a two step smear-layer modifying adhesive (multi-component dentin adhesive with self-conditioning primer, adhesive and bond). Syntac Primer partially removes or alters the smear layer using a mild acidic monomeric primer with no rinse step. On the other hand, OptiBond FL and Prime&Bond NT were selected as smear-layer removing adhesives. They completely remove the smear-layer following the total etch concept but differ in their number of application steps. OptiBond FL was selected as a three-step smear layer, removing adhesive, while Prime&Bond NT represents a one-bottle adhesive with a singular application step. Except for the different application steps, Prime&Bond NT contained acetone as a Primer solvent in contrast to alcohol/water-based OptiBond FL. Clearfil Liner Bond 2V is a self-etching adhesive system consisting of a smear-layer dissolving acidic primer and a subsequently applied adhesive.

Reduced adhesion values compared to the controls were found after water storage and thermocycling for Prime&Bond NT. A possible influence on the dentin bond strength has to be hypothesized as being due to the composition of the disinfectant solution. In the Alpron products used in this study, alcoholic components were included. In this context, polyvalent alcohol solutions are known to act as a dentin primer that affects the dentin surface and promotes the efficacy of the dentin bonding system (Ohhashi & others, 1997). Furthermore, one essential agent of Alpron mint is ethylenediamintetra acetic acid (EDTA), which has an antibacterial effect but is known as a dentin conditioning agent for removing smear layers. As is generally known, EDTA is a chelating agent that removes the smear-layer and leads to decalcification when applied to dentin. The depth of decalcification is 0.5-1 µm (Uno & Finger, 1996). In former studies, EDTA was found to be the most important conditioner for removing the smear layer and opening up the orifices of the dentinal tubules, followed by acid conditioners such as citric acid, polyacrylic acid, lactid acid and phosphoric acid (Meryon, Tobias & Jakeman, 1987). Hydrogen peroxide has been described as a substance with a potential demineralizing effect in dentin (Van Meerbeek & others, 1992).

The active substance in Dentosept P is hydrogen peroxide. Ernst, Post and Willershausen (1999) investigated the influence of cavity disinfection on the shear bond strength of dentin adhesives. They reported a significant decrease in bond strength after disinfection of the dentin with H_2O_2 and assumed collagen lysis effects. Increased pressure in the intertubular dentin due to the reactions of radicals and consecutive oxygen release may complicate penetration of adhesive resin into the intertubular dentin. In addition, oxygen in dentin might inhibit resin polymerization (McGuckin, Thurmond & Osovitz, 1992; Dishman, Covey & Baughan, 1994). Oxygenating the dentin due to the use of peroxide disinfectants could lead to the phenomena of oxygen inhibition of the free radical polymerization process.

A potential problem when using disinfectants before applying dentin bonding agents is a possible influence on the ability of the hydrophilic resin to seal the dentin (Meiers & Kresin, 1996). Therefore, we expected lower adhesion values for all the adhesive systems investigated. However, only fillings with the composite Spectrum, in combination with the acetone-based adhesive Prime&Bond NT, showed a reduced bond strength after artificial deterioration but could not be explained with the design of this study. Perhaps the tested acetone-based primer solvent is more greatly affected by the disinfectants used in this study than the tested alcohol/water based solvents. The explanation for significant differences with respect to the duration of water storage and thermocycling cannot be given due to the design of this study. The results of the study showed that further research in this area is necessary.

CONCLUSIONS

1. The tested disinfectants showed no significant influence ($p<0.01$) on the loads required for the debonding of Syntac Classic/Tetric Ceram, Clearfil Liner Bond 2V/Luxacore and OptiBond FL/Prodigy.
2. However, the use of disinfectants in the water supply decreased dentin bond strength in the specimens filled with Prime&Bond NT/Spectrum.

3. It is concluded that, within the limitations of this *in vitro* study, disinfectants in the cooling water of dental units may have an influence on dentin bonding, depending on the adhesive system used.

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Thermal Emission and Curing Efficiency of LED and Halogen Curing Lights

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A Tiba • DG Charlton

Clinical Relevance

A second-generation light-emitting diode (LED) curing light has a similar thermal emission and curing efficiency as a quartz-tungsten-halogen (QTH) curing light at similar energy densities.

SUMMARY

The purpose of this study was to compare the thermal emission and curing efficiency of LED (LEDemetron 1, SDS/Kerr) and QTH (VIP, BISCO) curing lights at maximum output and similar power, power density and energy density using the same light guide. Also, another LED curing light (Allegro, Den-Mat) and the QTH light at reduced power density were tested for comparison. Increase in temperature from the tips of the light guides was measured at 0 and 5 mm in air (23°C) using a temperature probe (Fluke Corp). Pulpal temperature increase was measured using a digital thermometer (Omega Co) and a K-type thermocouple placed on the central pulpal roof of human molars with a Class I occlusal preparation. Measurements were made over 90 seconds with an initial light activation of 40 seconds. To test curing efficiency, resin composites (Z100, A110, 3M/ESPE) were placed in a 2-mm deep and 8-mm wide plastic mold and cured with the LED and QTH curing lights at 1- and 5-mm curing distances. Knoop Hardness Numbers (KHN) were determined on the top and bottom surfaces (Leco). Bottom hardness values were expressed as a percentage of maximum top hardness. No significant differences were found in maximum thermal emission or KHN ratios between the LED (LEDemetron 1) and the QTH (VIP) at maximum output and similar energy densities (ANOVA/Tukey's; α=0.05).

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INTRODUCTION

Dental professionals have a variety of curing lights from which to choose, such as quartz-tungsten-halogen (QTH), plasma-arc (PAC), laser or light-emitting diode (LED). The relatively broad emission spectrum of QTH curing lights allows the lights to initiate the polymerization of all known resin composite materials available. The principle output from these lamps is infrared energy with the generation of high heat. Filters are used to reduce heat energy to the oral structures and provide further restriction of visible light to the narrower spectrum of photoinitiators. Finally, a silver-coated dichroic reflector passes infrared energy out the back and reflects and focuses the light forward to provide a focal area of energy at a defined distance. Ultimately, 99.5% of the original radiation is eliminated. Due to the

high operating temperatures, the QTH bulbs have a limited lifetime. The reflector, bulb and filters can breakdown over time, reducing curing effectiveness (Rueggeberg, 1999; Nagel, 1999).

LED curing lights use special semiconductors for the electroluminescence of light rather than the hot filament found in QTH lights. This reportedly provides a longer lifespan, consistent output and lower power consumption (Jandt & others, 2000). No significant ultraviolet or infrared light is emitted, thereby reducing heat and minimizing the need for a noisy fan. Since the energy is clearly defined by the semiconductor, most of the light emitted is concentrated in a narrow band around 470 nanometers, which is ideally suited for resin composites that use the photoinitiator camphoroquinone. A decreased power demand allows the use of battery-powered units (Kurachi & others, 2001).

Thermal emission from curing lights has been a matter of concern to the dental profession. Heat transfer to the tooth may cause pulpal damage (Hussey, Biagioni & Lamey, 1995; Hannig & Bott, 1999). Studies have suggested that LED curing lights, with their narrow spectral emission, generate significantly less heat from the light guide than QTH lights (Yap & Soh, 2003; Knezevic & others, 2001). Although LED curing lights emit less heat laterally from the semiconductor source, it remains unclear whether they release less heat from the tip of the light guide. First generation LED curing lights had lower power density compared to the latest generation of lights (Leonard & others, 2002; Dunn & Bush, 2002; Price, Felix & Andreou, 2003). Is the lower emitted heat from LED curing lights due to their narrow emission spectrum, the lower power density that the original LED lights were capable of producing or both? Differences in light guides, such as diameter, material or curing distance may have various effects on the power density and focusing effect (Price & others, 2000; Meyer, Ernst & Willershausen, 2002). Also, studies suggest that LED curing lights produce greater depth of cure than QTH lights (Mills, Jandt & Ashworth, 1999; Fujibayashi & others, 1998; Halvorson, Erickson & Davidson, 2004). The highly efficient, narrow emission spectrum of LED lights reportedly provides greater curing efficiency.

The primary purpose of this study was to determine the maximum thermal emission and curing efficiency of a second generation LED and a QTH curing light at similar power, power density and energy density using the same light guide. Additionally, another second generation LED curing light with a different type of light guide and higher power density was used for comparison.

METHODS AND MATERIALS

Thermal Emission

Thermal emission from two LED and one QTH curing light was measured using a temperature probe and thermocouple. The curing lights, light guides and manufacturers are listed in Table 1. All curing lights were tested at maximum power density. However, the VIP was also tested at approximately 50% of maximum output (312 mW/cm^2) to simulate a power density near minimal clinical acceptability (Rueggeberg, Caughman & Curtis, 1994). Power was measured 1 mm from the surface of the probe of a power meter (PowerMax 5200 and PM 10 probe, Molectron, Portland, OR, USA) and power density was calculated by dividing the power by the surface area of the exit end of the light guide. Three measurements were recorded per light and a mean was determined. Temperature readings were made at 0 and 5 mm from the tip of the light guide in room temperature air (23°C) using a temperature probe (Fluke Corp, Everett, WA, USA). Five millimeters is considered the average distance from the tip of a molar cusp to the pulpal floor of an average occlusal preparation (Price & others, 2000). The curing lights and temperature probe were supported with a clamp and ring stand. The curing lights were activated for 40 seconds and temperature readings were recorded every five seconds up to a total of 90 seconds. Five separate recordings were made per light.

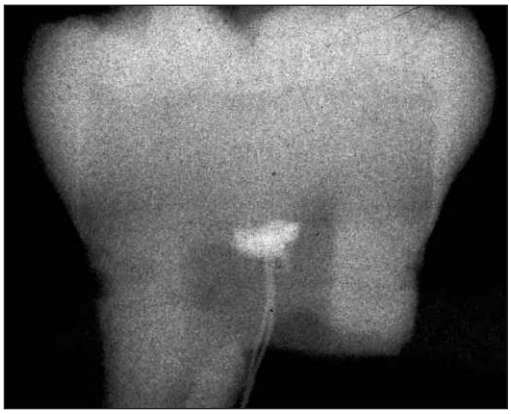

Figure 1. *Radiograph showing position of K-type thermocouple mounted on the central pulpal root with silicone compound.*

Table 1: *Curing Lights Tested*

Curing Light (Type)	Manufacturer	Type of Light Guide	Light Guide Diameters: Entrance/ exit (mm)
Allegro (LED)	Den-Mat, Santa Maria, CA, USA	Uncoated solid acrylic	14/8
LEDemetron 1 (LED)	SDS/Kerr, Danbury, CT, USA	Coated fiber optic	12/10
VIP (QTH)	BISCO, Schaumberg, IL, USA	Coated fiber optic	12/10

Pulpal temperature increase was measured in an extracted human lower molar with a Class I cavity preparation (2.5-mm depth) on the occlusal surface. One root was sectioned with a separating disc and the pulpal residues were removed with a barbed broach. The tooth was mounted in a plastic lid with a thin film of autopolymerizing acrylic resin. A thermocouple (K-type, Omega Corp, Stamford, CT, USA) was inserted into the pulp chamber and maintained on the central pulpal roof with a small layer of silicone heat-sink compound (GC Thorsen, Rockford, IL, USA). The position of the thermocouple was checked radiographically as shown in Figure 1. The mounted tooth was placed in a water-filled cup so that the roots and pulp chamber were submerged in 37°C distilled water. The mounted tooth and cup were placed in 37°C distilled water in a humidity chamber (Sabri Enterprises, Downers Grove, IL, USA). A dentin bonding agent was placed into the cavity preparation following the manufacturer's instructions (Scotchbond Multi-Purpose, 3M/ESPE, St Paul, MN, USA) and light cured. The light guides from the curing lights were positioned on the cusp tips of the molar and held with a clamp and ring stand. The lights were activated for 40 seconds and temperature readings were recorded every five seconds up to 90 seconds using a digital thermometer (Omega Corp, Stamford, CT, USA). Five separate recordings were made per light as before. Mean maximum values and standard deviations were calculated for each light under each testing condition (0 and 5 mm, pulpal) and analyzed by ANOVA and Tukey's Post-hoc tests (α=0.05). After completion of the pulpal temperature measurements, the tooth was sectioned and the remaining dentin thickness between the floor of the preparation and the roof of the pulp chamber was determined to be 3.2 millimeters.

Composite Hardness

A microfill resin composite, Filtek A110, shade A3 (3M/ESPE) and a hybrid resin composite Z-100, shade A3 (3M/ESPE) were used in this evaluation. A polytetrafluoroethylene mold 2-mm high and 8 mm in diameter was used to prepare five specimens for each test group. To prepare each specimen, the mold was placed on a glass slide and the uncured resin composite placed in the mold. The resin composite was then covered with a second glass slide to reduce the effects of air inhibition and to ensure that the exposed surface of the composite was flat and parallel to the surface of the mold. The composite was then exposed to each curing light at maximum output for 20 seconds at 1- and 5-mm curing distances. The VIP was also used at approximately 50% of maximum output (304 mW/cm^2) to simulate a power density near minimal clinical acceptability (Rueggeberg & others, 1994). The tip of the curing lights was held flush and 4 mm from the 1-mm thick glass slide to produce the 1- and 5-mm curing distances, respectively. After dry storage in a light-proof container for 24 hours, indentations were made with a hardness tester (LECO M 400 G2, St Joseph, MO, USA) using a 200-gram load and a dwell time of 10 seconds. Three measurements were made for each side of each specimen and the top and bottom mean Knoop Hardness Numbers (KHN) were calculated. Acceptable polymerization of a resin composite has been suggested to have occurred when a test specimen's bottom surface hardness is at least 80% of the top surface hardness (Pilo & Cardash, 1992; Skeeters, Timmons & Mitchell, 1983). The bottom KHN was expressed as a percentage of the maximum KHN to prevent errors when comparing groups with softer top surfaces (Vandewalle, Ferracane & Ly, 2002). A resin composite's maximum KHN was determined to be the mean KHN of the top surface of the hardest tested specimen. The percent mean maximum KHN ratios and standard deviations were calculated for each light under each testing condition. Data were analyzed by a three-way ANOVA/Tukey's (α=0.05) to evaluate the effects of composite, distance and curing light on mean KHN ratios.

The power density over distance was measured for each light using a power meter (PowerMax 5200 and PM10 probe, Molectron, Portland, OR, USA). The curing light was held in place with a clamp and ring stand and the window of the power meter probe was dropped from the light guide in 1-mm increments from 0 to 20 mm on a moveable stage. Correct distances were maintained with the use of an electronic digital caliper (Fowler Ltd, Louisville, KY, USA) mounted on the stage. Three separate recordings were made for each light. Mean power density values and standard deviations were calculated for each 1-mm increment.

The emission spectrums of the curing lights were recorded with a spectrophotometer (PR-650, Photo Research Inc, Chatsworth, CA, USA) at a distance of 1 meter from a standardized white reflecting surface. The spectral absorbance of camphoroquinone was determined in methanol using an ultraviolet-visible spectrophotometer (8452A, Hewlett Packard, Palo Alto, CA, USA). Light diffusion images were obtained by mounting each curing unit on a ring stand and projecting the light parallel across the surface of flat black paper. Images were captured with a digital camera (Nikon D1, Belmont, CA) mounted on a ring stand and maintained at identical exposure settings for each light.

RESULTS

Thermal Emission

The maximum thermal emission resulting from the different curing lights is summarized in Table 2. At 0 mm, there was no statistically significant difference between

the mean maximum temperatures from the surface of the light guide of the LEDemetron 1 or the VIP at maximum power densities (p=0.944). Allegro had significantly higher temperatures. VIP at the lower power density (312 mW/cm^2) produced significantly less heat under all conditions. There was no statistically significant difference between the mean maximum temperatures recorded at 5 mm from the light guide of the LEDemetron 1, the Allegro or the VIP at maximum power densities (p=0.897). At the central pulpal roof, there was no statistically significant difference between the mean maximum temperatures produced by the LEDemetron 1 or the VIP at maximum power densities (p=0.195). The recorded temperature values over time are shown graphically in Figures 2 through 4.

Composite Hardness

The mean KHN ratios resulting from the different curing lights are summarized in Table 3. Significant differences were found in the KHN ratios based on composite (p<0.0001), distance (p<0.0001) and curing light (p<0.0001). However, significant interactions were present. The data were analyzed by a one-way ANOVA and Tukey's Post-hoc test (α=0.05) at each curing distance. There was no overall statistically significant difference between the mean KHN ratios for the LEDemetron 1 and the VIP at maximum power densities (p>0.05) at any distance. The KHN ratios for Allegro were significantly higher at 1 mm. The VIP at the lower power density (304 mW/cm^2) produced significantly lower KHN ratios at all distances.

The emission spectra of the three curing lights are displayed in Figure 5. The LED curing lights have narrow emission spectrums that peak near the absorption peak of camphoroquinone. The percentage of maximum power density over a 20-mm distance from the tip of the light guide is shown in Figure 6. The power density of Allegro dropped dramatically over distance. Using the same fiber-optic light guide, VIP and LEDemetron 1 maintained a similar power density over the entire 20-mm distance.

DISCUSSION

With the introduction of LED curing lights to the profession just a few years ago, multiple potential advantages over conventional QTH curing lights have been suggested. Purported advantages include longer LED life with minimal decrease in output over time and a narrower spectral emission resulting in higher efficiency, less heat production, greater depth of cure and cordless operation (Althoff & Hartung, 2000). A recent study by Yap and Soh (2003) concluded that LED curing lights emit significantly less heat from their light guides than QTH lights. This makes sense, considering the fact that the filtered emission spectrum of a QTH lamp is broader than that of an LED and includes wavelengths of light that are capable of generating higher heat. Figure 5 illustrates this difference in spectrums. However, it is important to note that the first generation of LED curing lights had low power

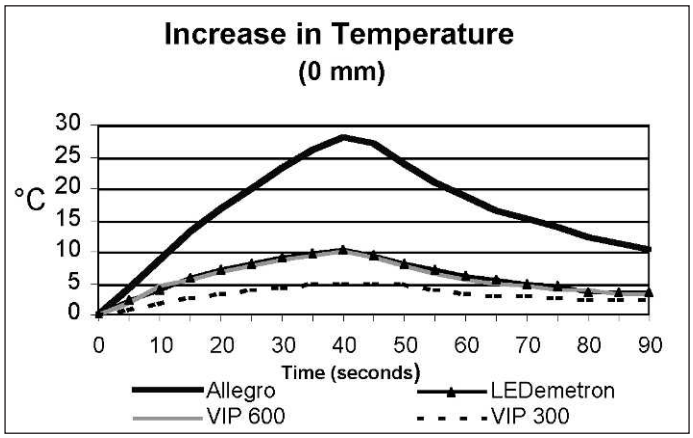

Figure 2. *Thermal emissions (°C) over time (seconds) recorded at 0 mm from tip of light guide. Lights activated for 40 seconds.*

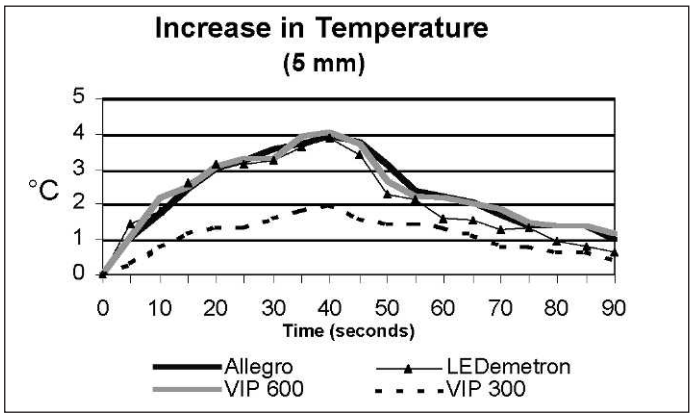

Figure 3. *Thermal emissions (°C) over time (seconds) recorded at a distance of 5 mm from tip of light guide. Lights activated for 40 seconds.*

Table 2: *Maximum Temperature Increase in Degrees Centigrade at O- and 5-mm Distances in Air and at the Pulpal Root*

Light	Power Density (mW/cm^2)	Energy Density (J/cm^2)	Mean Maximum Temperature Rise °C (std dev)		
			0 mm	5 mm	pulpal
Allegro	1437	57.5	28.2 (1.3) a	3.9 (0.3) a	3.1 (0.1) a
LEDemetron 1	602	24.1	10.3 (0.5) b	3.9 (0.4) a	2.9 (0.1) b
VIP	608	24.3	10.0 (0.4) b	4.0 (0.1) a	3.0 (0.2)a,b
VIP	312	12.5	12.5	4.0 (0.5) b	1.5 (0.1) c

Letters denote significant differences by column.

densities (Leonard & others, 2002). The lower power densities require longer curing times to deliver equivalent energy densities and depth of cure. Longer curing times may translate into higher heat development. The second generation of LED lights generally have much higher power densities and potentially much higher thermal emission and depth of cure (Price & others, 2003). This study, in fact, found that to be the case. Allegro, with its significantly higher power density, produced greater heat at 0 mm in air and higher KHN ratios at a curing distance of 1 mm.

Early studies suggested that LED curing lights have greater curing efficiency over QTH lights (Mills & others, 1999; Fujibayashi & others, 1998). These initial investigations used prototype, laboratory LED lights with a relatively large number of LEDs or they reduced the output of the QTH light to coincide with the lower output of the LED light. Stahl and others (2000) developed a model to explain the LED's curing effectiveness over QTH lights, stressing the importance of looking at the higher "integrated relative curing potential" of the LED with its narrow, more efficient emission spectrum. Leonard and others (2002) found that LED lights have a higher percentage of their output in the absorption spectrum of camphoroquinone compared with QTH curing lights. A recent study by Halvorson and others (2004) found that, despite a 31% greater relative efficiency, scrapeback lengths from composite polymerized using the LED light were only 6% greater than those polymerized with a QTH light at similar energy densities. Recently published abstracts comparing commercially available LED and QTH lights have found no statistical difference in curing efficiency at equal energy densities (Vandewalle & others, 2002; Ramp & others 2003). LED curing lights are optimized for materials containing the photoinitiator camphoroquinone. Recently, Uhl, Mills and Jandt (2003) found that some resin composites (Solitare 2, Heraeus Kulzer, Armonk, NY, USA) containing other photoinitiators, in addition to camphoroquinone, may not develop hardness values equivalent to those attainable with the broader spectrum QTH curing lights. The resin composites used in this study only contain the photoinitiator camphoroquinone.

This study attempted to control as many confounding variables as possible. Multiple combinations of commercially available LED and QTH curing lights were evaluated to find the correct combination that would produce similar power, power density and energy density using an identical light guide. However, the results of this study found no significant differences in maximum surface and pulpal temperatures and KHN ratios between the VIP at maximum output and the LEDemetron 1. Apparently, the narrow emission spectrum of the tested commercially available LED curing lights may not play a statistically significant role in reducing thermal emissions from the tip of the light guides or increasing the curing efficiency of the composites used in this study. Hardness testing is a popular method of assessing the effectiveness of polymerization because of its simplicity and reasonable correla-

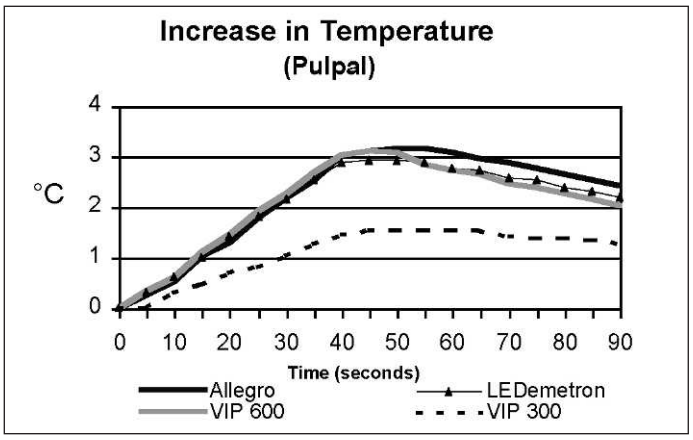

Figure 4. *Thermal emissions (°C) over time (seconds) recorded at the central pulpal root of third molar. Lights activated for 40 seconds.*

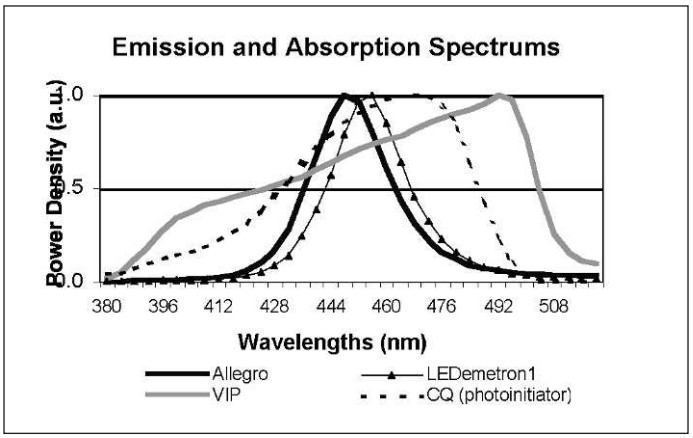

Figure 5. *Spectral emissions of the curing lights and absorption spectrum of camphoroquinone.*

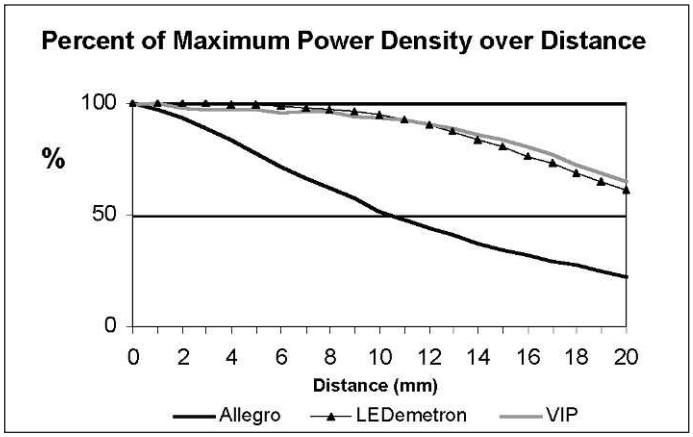

Figure 6. *The percentage of maximum power density over a 20-mm distance from tip of light guides.*

tion with other methods (DeWald & Ferracane, 1987). A good correlation can be found between hardness and the relative degree of conversion for a specific composite (Ferracane, 1985). It was not surprising to find that the microfilled resin composite demonstrated a decreased KHN ratio compared with the hybrid resin composite. Kawaguchi, Fukushima and Miyazaki (1994) showed that microfilled composites have a lower transmission coefficient and depth of cure than hybrid and small particle composites. Microfilled resin composites may be more difficult to cure, because the natural agglomeration of their small filler particles may cause light to scatter, decreasing the effectiveness of the curing light (Ruyter & Oysaed, 1982; McCabe & Carrick, 1989).

Different curing light guides can have a dramatic influence on the focusing effect of the emitted light (Price & others, 2000). Figure 6 shows the effects of distance on power density. The solid acrylic light guide of the Allegro curing light produced a dramatic reduction in power density over distance. The Allegro light guide has a stronger focusing effect of light due to the larger entrance and smaller exit surface areas of its turbo light guide. This greater focusing effect increases the power density near the tip of the light guide but, at greater distances, the light diffuses at a faster rate as shown in Figures 6 and 7. This agrees with Price and others (2000), who found that as the distance from the tip of a light guide increased, the power density decreased, but the rate of decrease was greater for turbo light guides than for standard light guides. The authors speculate that in this study, a higher rate of power density decrease would have also occurred with the LEDemetron 1 or VIP with the substitution of a turbo light guide. However, Allegro's uncoated acrylic light guide herniated light laterally, which may have additionally contributed to the poor collimation of light over distance compared to a typical fiber optic light guide.

As noted earlier, Allegro produced greater heat than the other units at 0 mm in air. However, due to the relatively poor collimation of the emitted light, Allegro dramatically lost heat over distance and was similar in maximum heat output to the other curing lights at greater distances. A similar effect was noted with the KHN ratios. Allegro showed a reduction in composite hardness at a 5-mm distance and was similar to the LEDemetron 1 and VIP at maximum output. The VIP light was also tested at one half of its maximum output to represent the lower end of acceptability in recommended power density (Rueggeberg & others, 1994). The maximum surface and pulpal temperature increases at this reduced power density were one-half the recorded values for the same light at maximum output. The KHN ratios were notably reduced, as well.

It was not the primary purpose of this study to investigate the effects of thermal emissions on pulpal tissue. However, studies have shown that temperature increases from light curing units can cause pulpal damage (Hussey & others, 1995; Hannig & Bott, 1999). Previous studies have shown temperature increases from 1.5°C to 4°C in the pulp chamber of extracted teeth during light curing of resin composite restorations (Thompson, Gomez & Puckett, 1997; Bennett & others, 1984). Zach and Cohen (1965) determined that the critical temperature increase for irreversible pulpal damage is 5.5°C. The maximum pulpal temperature increase in this study, 3°C, produced by all three curing lights at maximum output, suggests that the tested

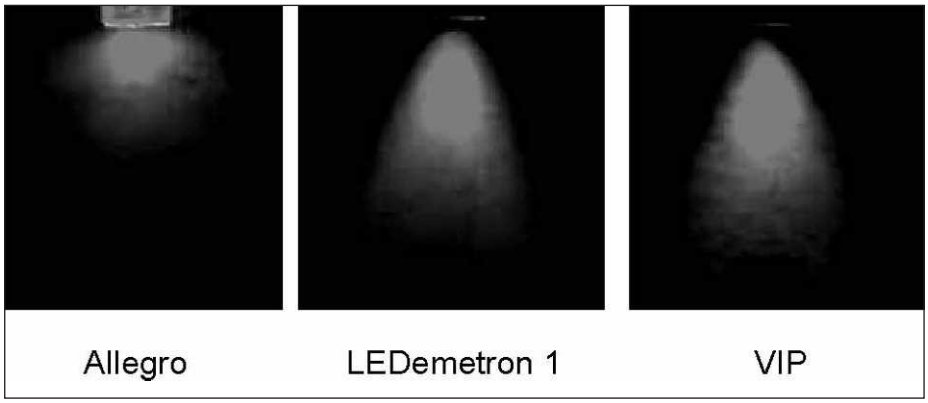

Figure 7. *Light diffusion from the three curing lights.*

Table 3: *Mean KHN ratios (% bottom/max) of resin composites at 1- and 5-mm distances. Maximum KHN for Z100 = 45.8 kg/mm² and A110 = 20.8 kg/mm²*

Light	Power Density	Energy Density	Mean Knoop Hardness Ratios (% bottom/max) (std dev)			
			Z100		A110	
	(mW/cm²)	(J/cm²)	1 mm	5 mm	1 mm	5 mm
Allegro	1404	28.1	94.8 (1.0) a	85.2 (2.0) a	78.5 (3.6) a	61.3 (2.0) b
LEDemetron 1	598	12.0	85.4 (1.5) b	86.3 (1.9) a	63.8 (1.9) b	67.9 (3.2) a
VIP	605	12.1	86.0 (1.9) b	84.7 (2.2) a	64.8 (2.3)b	63.4 (3.1) ab
VIP	304	6.1	69.5 (1.9) c	69.1 (1.3) b	44.2 (2.8) c	40.4 (1.8) c

Letters denote significant differences by column.

lights have less potential to cause pulpal damage. It is very difficult to predict the temperature rise in any particular tooth due to multiple variables such as dentin thickness, preparation depth, output intensity and exposure time (Yap & Soh, 2003). Although the actual critical temperature necessary to cause pulpal damage is controversial, pulpal temperature changes should be kept as small as possible. According to Lloyd, Joshi and McGlynn (1986), the most significant factor for increasing pulpal temperature while light curing is the energy absorbed during irradiation. The exothermic composite polymerization process provides secondary increases.

CONCLUSIONS

Based on the limitations of this study, there was no statistically significant difference between the maximum surface and pulpal temperatures between LEDemetron 1 (LED) and VIP (QTH) at maximum output and equivalent energy densities. The latest generation of LED curing lights, exemplified by Allegro, have significantly higher power densities and are capable of producing high thermal emissions. Also, there was no statistically significant difference between the mean KHN ratios of the resin composites cured in this study with LEDemetron 1 (LED) and VIP (QTH) at maximum output and equivalent energy densities.

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Disclosure

The views expressed in this article are those of the authors and do not reflect the official policy of the Department of Defense or other departments of the United States government.

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The Effect of Whitening Agents on Caries Susceptibility of Human Enamel

T Al-Qunaian

Clinical Relevance

The results of this study provide support for the concept that vital tooth whitening does not produce caries susceptibility in human enamel.

SUMMARY

This *in vitro* study evaluated whether the treatment of human enamel with whitening agents containing different concentrations of carbamide or hydrogen peroxide changes the susceptibility of enamel to caries. Twenty-four sound human incisors were selected for this study. For each tooth, the crown was sectioned into two halves in the cervical-incisal direction. One half of the sectioned tooth was treated and the other half was used as a control specimen. Each half was randomly divided into three treatment groups (eight two-halves/group). The whitening agents were 10% carbamide peroxide, 20% carbamide peroxide with fluoride and 35% hydrogen peroxide. Following pretreatment, the specimens were demineralized for four days in an *in vitro* microbial caries model and then analyzed using a confocal laser scanning microscope (CLSM). Results showed that there were no significant differences between the treated and controlled specimens for teeth treated with 10% carbamide peroxide or 35% hydrogen peroxide. However, specimens treated with 20% carbamide peroxide with FP (0.11% fluoride and potassium nitrate) were less susceptible to caries than their controls at $p \leq 0.05$. In conclusion, application of bleaching agents does not increase the caries susceptibility of human enamel.

INTRODUCTION

At-home and in-office bleaching procedures have been recognized as successfully treating discolored teeth in esthetic dentistry. When vital teeth are bleached, direct contact is established between the bleaching agent and the outer enamel surface (Ernst, Marroquin & Willershausen-Zonnchen, 1996). Clinical observations have shown no important clinically adverse effects as a result of tooth whitening with moderate concentrations of hydrogen peroxide or carbamide peroxide (Haywood, 1994; Goldstein, 1995). Most laboratory studies have confirmed the clinical observation that whitening gels containing carbamide or hydrogen peroxide have not demonstrated a significant effect on hard tissue morphology (Ernst & others, 1996; Oltu & Gurgan, 2000; Gultz & others, 1999; McCracken & Haywood, 1996).

Clinically, however, it is not unusual to see a white spot develop during the whitening process, which disappears after discontinuing the procedure. The concern has been raised whether or not these white spots are precarious lesions. Therefore, this *in vitro* study was conducted to evaluate whether treating human enamel with whitening agents containing different concentrations of carbamide or hydrogen peroxide changes the susceptibility of enamel to caries.

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METHODS AND MATERIALS

Three groups of extracted teeth were used in this study. Two groups of teeth were pretreated for eight hours with 10% or 20% carbamide peroxide (Opalescence). The 20% carbamide peroxide product also contained 0.11% fluoride and potassium nitrate (FP). The 10% whitening agent is accepted by the American Dental Association (ADA) as a "safe" and "effective" product to be used at home, and the 20% agent was also recommended for home use. The third group was pretreated with 35% hydrogen peroxide (Opalescence Xtra). This agent is approved for professional in-office use and was applied three different times with three 10-minute applications, for a total of nine applications.

Twenty-four sound human incisors were selected for this study. For each tooth, the root was removed and the crown sectioned into two halves in the cervical-incisal direction. One half of the sectioned tooth was treated and other half was used as a control specimen. Each half was attached with cyanoacrylate adhesive to one end of a plexiglass rod. The cut side of the specimen was sealed with nail varnish to prevent bacterial penetration into opened tubules when the specimens were placed in the demineralizing model. Each half was randomly divided into three treatment groups (eight groups of two-halves).

The control halves were placed in deionized water. The eight tooth halves in Group 1 were treated with 10% carbamide peroxide (Opalescence 10%, Ultradent Products, Inc, South Jordan, UT, USA) for eight hours on 14 consecutive days by placing the specimen upright in a holder and applying the whitening agent to the labial surface. The halves in Group 2 were similarly treated with 20% carbamide peroxide with FP (Opalescence 20% PF, Ultradent Products, Inc). This whitening agent contains 0.11% sodium fluoride and 3% potassium nitrate. The third group had 35% hydrogen peroxide placed on the labial surfaces. A curing light was then placed approximately 0.25 inches from the specimen surfaces, with the specimens being exposed for 30 seconds and the gel left in place for a total of 10 minutes. Hydrogen peroxide was then rinsed from the surface of each tooth. This procedure was repeated two more times during each treatment session. A total of three treatment sessions, each involving three individual treatments, separated by a minimum of three days, were applied to the specimens in Group 3 during a 14-day treatment period. During non-treatment times, all teeth were placed in a highly humid environment at 4°C.

Following pretreatment, the specimens were demineralized for four days an *in vitro* microbial caries model (Fontana & others, 2000). The eight specimens in each treatment group and their controls were mounted onto acrylic plates that fit tightly on the stirring magnet of four caries-forming vessels. All the specimens were gas sterilized, then placed in the microbial demineralizing model to generate carious lesions.

All the groups were inoculated once at the beginning of the experiment with a mid-log phase culture of *S mutans* TH16. Following inoculation, the specimens were incubated at 37°C for two hours to allow the bacteria to adhere to tooth surfaces before beginning treatment. The specimens were then exposed at 37°C to circulating trypticase soy broth supplemented with 5% sucrose (TSBS) for 30 minutes three times a day and to a mineral washing solution (MW) for total of 22.5 hours per day. The circulating fluids were delivered to and removed from the treatment vessels by time-regulated peristaltic pumps. The TSBS solution reproduced nutrient intake three times/day and the MW was used as an artificial saliva buffering solution. Each treatment group had its own TSBS bottle. The specimens were connected to two MW bottles. On days one and three, the drainage containers for all three groups were changed and the drainage fluid was monitored for pH, bacterial viability and the absence of contamination. After four days of demineralizing treatment in the model, the tooth specimens were removed and monitored for the development of primary caries in all specimens, including halves treated and non-treated with whiteners.

Confocal Laser Scanning Microscope (CLSM) Analysis

These analyses were done blindly, without any knowledge of the specimen groups' assignment or pretreatment. The specimens were analyzed in numerical order, thereby randomizing the treatment groups. Each specimen half was cut in half, perpendicular to the cervical incisal axis, using a Silverstone Taylor hard tissue microtome. One-half of each cut was stained overnight with an 0.1 mM solution of Rhodamine B fluorescent stain. The cut stained surface was then allowed to dry before being analyzed with a CLSM to determine the presence and extent of lesions. The samples were examined with a CLSM using Metamorph software (Universal Image Corp, West Chester, PA, USA). After being brought into focus using a 10x Nikon objective, NA 0.25, the specimens were illuminated with the argon laser using a 488 nm excitation wavelength. Confocal slits were set at 25 µm 100%. For collection of the images, the samples were frame-averaged using 100 frames per image. Lesion measurements were made in both the whitener-pretreated half and the non-pretreated half, and the following parameters were measured: area of the fluorescent lesion, fluorescence (total gray) of the lesion and depth of the lesion. The differences in lesion parameters between the pretreated and non-treated halves of the tooth were calculated.

Mean CLSM parameter data (± SD) was determined for the pretreated and non-pretreated lesions in each treatment group and analyzed for significant intra-group differences using a paired *t*-test. For each CLSM measured parameter, differences between the lesion in whitener-pretreated and non-pretreated enamel were calculated for each specimen and mean differences (± SD) were determined for each group. Multiple inter-group comparisons were then conducted using one-way analysis of variance (ANOVA). Multiple comparisons of the three groups were done using Tukey's procedure to determine significant differences among the group means when significant F-values were found. The level of significance accepted for all parameters measured in this study was $p \leq 0.05$.

Table 1: *Paired* t-*test Group 1 (10% CP)*

Parameters	Specimen	N	Mean	SD+/-	Sig
Total area (μm)²	Treated	8	8603.4	1727.5	
	Controlled	8	7152.5	2604.3	0.301
Total gray value	Treated	8	109.25	51.36	
	Controlled	8	74.67	30.76	0.178
Lesion depth (μm)	Treated	8	37.1	5.58	
	Controlled	8	39.97	7.56	0.451

Table 2: *Paired* t-*test Group 3 (35% HP)*

Parameters	Specimen	N	Mean	SD+/-	Sig
Total area (μm)²	Treated	8	8295.4	2438.82	
	Controlled	8	10667.0	3018.16	0.590
Total gray value	Treated	8	78.08	19.68	
	Controlled	8	120.42	62.89	0.200
Lesion depth (μm)	Treated	8	40.44	5.41	
	Controlled	8	48.34	10.2	0.087

Table 3: *Paired* t-*test Group 2 (20% CP with FP)*

Parameters	Specimen	N	Mean	SD+/-	Sig
Total area (μm)²	Treated	8	6060.8	2151.7	
	Controlled	8	8887.4	2302.5	0.027
Total gray value	Treated	8	69.7	36.37	
	Controlled	8	108.6	50.11	0.027
Lesion depth (μm)	Treated	8	30.13	9.2	
	Controlled	8	42.18	9.26	0.034

RESULTS

The paired *t*-test showed that there were no significant differences between the treated and controlled specimens for teeth treated with 10% carbamide peroxide or 35% hydrogen peroxide (Tables 1 and 2). However, specimens treated with 20% carbamide peroxide with FP were less susceptible to caries than their controls at $p \leq 0.05$ (Table 3). Figures 1, 2 and 3 illustrate the CLSM images from representative specimens in the study. ANOVA and Tukey's tests revealed that the total lesion area of the first group was significantly higher than the third group at $p \leq 0.05$.

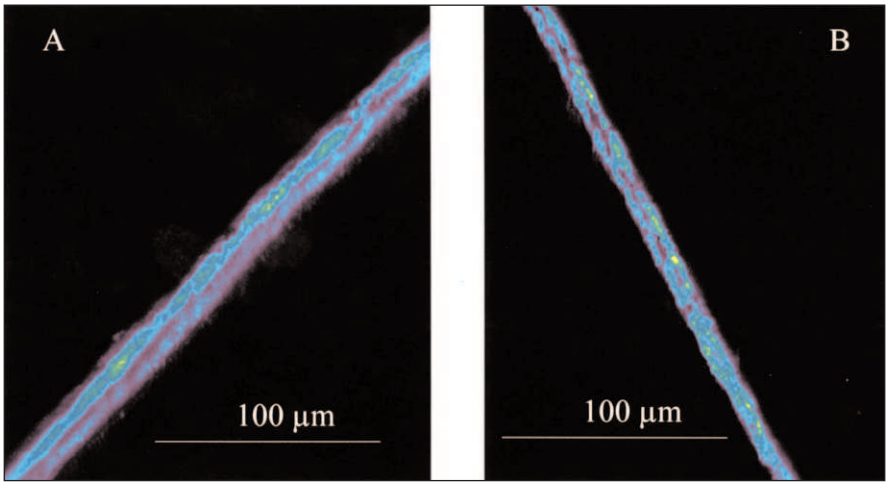

Figure 1. *(A) image of specimen treated with 10% CP, (B) control specimen of image (A). The pseudocolor shows the caries lesion in enamel.*

DISCUSSION

The CLSM images permit non-destructive examination of surface and subsurface hard tissue ultrastructural changes and provide evidence of structural changes within tissues (Duschner, Sonju-Clason & Ogaard, 1996).

In CLSM, laser light from out of focus planes is eliminated by the confocal pinhole technique, hence, only light remitted from the exact focus plane is recorded. The analog output of the photomultiplier detector is coordinated three-dimensionally with the scan position of the laser and the position of the object table. With computer assistance, this data is transferred into a three-dimensional matrix of digital values of light

intensity. For visualization, this information is transformed into an arbitrary scale of pseudocolors, then presented as three-dimensional images (White & others, 2000).

Hydrogen peroxide has a low molecular weight. Therefore, it can readily penetrate into enamel. Thus, inner oxidative effects are likely to occur in the subsurface enamel where more organic material is present and where oxidation is capable of altering the outer enamel and surface (Hegedus & others, 1999).

Haywood, Houck and Heymann (1991) studied, *in vitro,* the effect of three commercially available 10% carbamide peroxide solutions and a 1.5% hydrogen peroxide solution on enamel surface and color. No significant differences in enamel surface texture were detected between the treated and control enamel surfaces of the teeth in all groups. Leonard and others (1999) compared maxillary teeth after being bleached every night for six months with mandibular teeth that had not been bleached. They reported that there was "no discernable difference in surface texture between the maxillary and mandibular teeth as viewed under the Scanning Electron Microscope (SEM) at 200 or 2000 times magnification." Covington and others (1990) also looked at the effect of carbamide peroxide gel on the structure of human enamel in a SEM study. They reported the development of focal areas of very shallow erosion rather than pitting. Analysis of the outer 100 nm indicated a distinct loss of organic component from treated surfaces. He concluded that there was "… a controlled oxidation process in which the organic phase of the enamel is mobilized, without producing grossly unacceptable enamel surface topography."

Bitter (1998) reported that a 14-day exposure to whitening agents caused an alteration of the enamel surface, exposing the enamel prismatic layer and possibly dentin. This effect was seen up to 90 days post-treatment; however, no control was reported in this study for comparison. Also, the patients practiced poor oral hygiene with the teeth that were whitened, because the teeth were scheduled for extraction. Duschner and others (2000) used confocal laser scanning microscopy to examine the effects of bleaching on enamel and dentin.

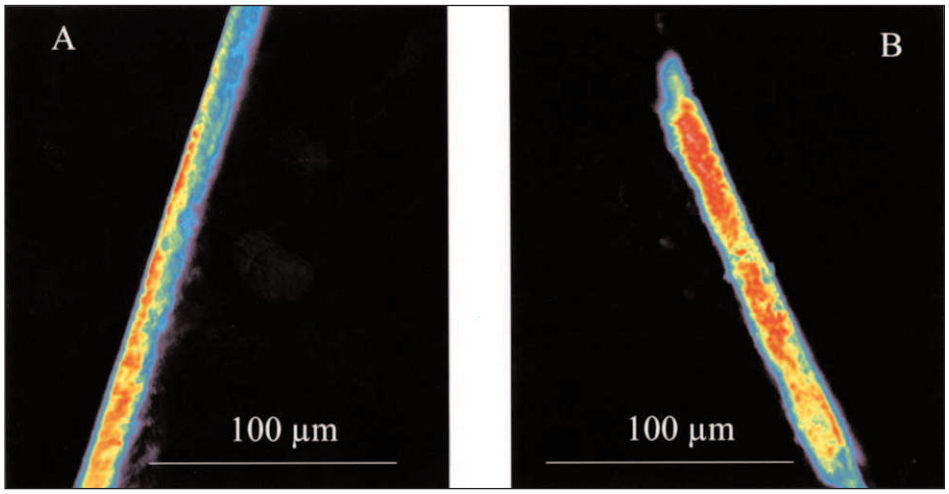

Figure 2. *(A) image of specimen treated with 20% CP, (B) control specimen of image (A). The pseudocolor shows the caries lesion in enamel.*

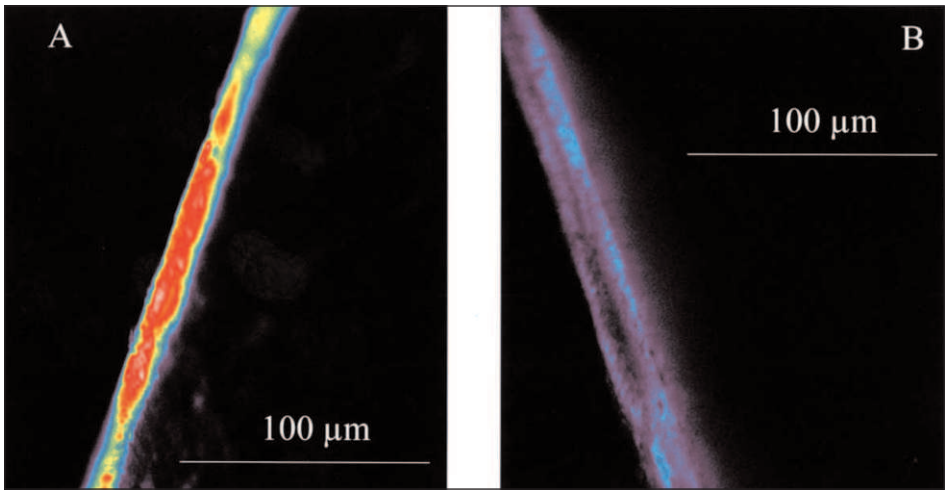

Figure 3. *(A) image of specimen treated with 35% HP, (B) control specimen of image (A). The pseudocolor shows the caries lesion in enamel.*

The results showed that no significant micromorphological changes were found in subsurface enamel. Potocnik, Kosec and Gaspersic (2000) studied the effect of 10% carbamide peroxide on enamel microhardness, microstructure and mineral content. They found local microstructural and chemical changes that were not clinically significant. Araujo and others (2003), in an *in situ* study, evaluated bleaching with 10% CP for one hour vs seven hours per day compared with a control and reported no difference in microhardness among the different regimens. White and others (2000) studied the effect of tooth-whitening gels on enamel ultrastructure by using confocal laser scanning microscopy. They found no significant micromorphological changes associated with the whitening process in subsurface enamel. Recently, a SEM study of dental enamel surfaces exposed to 35% hydrogen peroxide and 10% carbamide peroxide considered these gels safe for enamel

(Spalding, Taveira & de Assis, 2003). Basting, Rodrigues and Serra (2003) reported on the effects of seven carbamide peroxide bleaching agents on enamel hardness over time. His study showed a decrease in microhardness only after the first eight hours of a 45-day bleaching study.

In this study, no significant differences in caries susceptibility were observed between the untreated control specimens and those specimens treated with 10% carbamide peroxide or 35% hydrogen peroxide. On the other hand, specimens treated with gel containing 20% carbamide peroxide were significantly reduced in caries susceptibility when compared with their untreated controls. This is probably related to fluoride incorporation in 20% FP carbamide peroxide gels. This is in agreement with laboratory studies with fluoride enhanced enamel remineralization. A CLSM study by González-Cabezas and others (1998) revealed that remineralization is significantly greater in specimens treated with fluoride-containing dentifrices. Attin and others (1997) observed that remineralization of bleached enamel was improved by the application of fluoride. Future studies comparing the effect of 10% carbamide peroxide-containing fluoride and 20% carbamide peroxide-containing fluoride are recommended.

CONCLUSIONS

Application of bleaching agents does not increase caries susceptibility of human enamel. A bleaching agent-containing fluoride reduced caries susceptibility.

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