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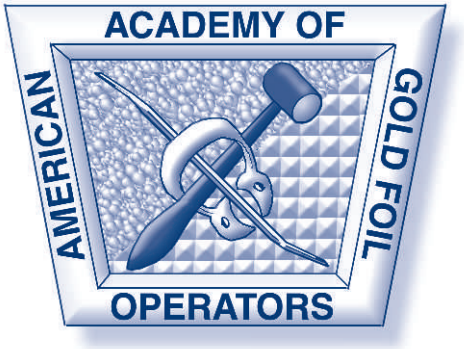

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Operative Dentistry publishes articles that advance the practice of operative dentistry. The scope of the journal includes conservation and restoration of teeth; the scientific foundation of operative dental therapy; dental materials; dental education; 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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Clinical Research

Two-Year Clinical Performance of Class V Resin-Modified Glass-Ionomer and Resin Composite Restorations

WW Brackett • A Dib • MG Brackett
AA Reyes • BE Estrada

Clinical Relevance

Although the incidence of failed restorations was higher for resin composite than for glass ionomer, no statistically significant difference was observed in overall clinical performance between the two materials. The appearance of resin composite restorations was judged to be better than the resin-modified glass ionomer.

SUMMARY

While a one-year report had been previously published, this study was undertaken to evaluate the clinical performance and appearance of a resin-modified glass ionomer and a resin composite over two years. Thirty-seven pairs of restorations of FujI II LC and Z 250/Single Bond were placed in caries-free cervical erosion/abfraction lesions without tooth preparation. Restorations were clinically evaluated at baseline, 6, 12, 18 and 24 months using modified Ryge/USPHS criteria. No statistically significant difference (p=0.13) was observed in the overall performance of the materials. Retention was 96% for the resin-modified glass ionomer and 81% for the resin composite, with no additional restorations of either material lost after one year. As previously reported, retention of the Z 250 restorations at six months was below the minimum specified in the ADA Acceptance Program for Dentin and Enamel Adhesives. The resin composite restorations generally had a better appearance, with a 100% alpha rating in color match, versus 85% for the resin-modified glass ionomer.

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INTRODUCTION

Resin-modified glass ionomer restorative materials are well proven in clinical trials as adhesive restorations in non-retentive cervical cavities (Boghosian, Ricker & McCoy, 1999; Brackett & others, 2001). Whether they are adequate in appearance in Class V restorations relative to resin composites is controversial, with some

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clinical investigators reporting a deterioration in appearance of glass ionomers over time (Duke & Trevino, 1998; Gladys & others, 1999). Others report nearly ideal color match for up to five years (Boghosian & others, 1999; Brackett & others, 2001), although none of the cited investigations included resin-modified glass ionomers and resin composites. Three studies have evaluated the appearance of both materials in Class V restorations over one-to-three years, of which one has reported resin composites to be superior, (Folwaczny & others, 2000) , while the other two report equal appearance (Neo & Chew, 1996; Neo & others, 1996).

Resin composites placed with recent dentin adhesives have also been demonstrated to be effective restorations for non-retentive cervical cavities. Most studies report very good appearance and clinical performance with resins when adhesives with separate primers and adhesives are used with the total-etch technique (Alhadainy & Abdalla, 1996; Van Meerbeek & others, 1996; Browning, Brackett & Gilpatrick, 2000). Although less is known about the adhesives most commonly supplied today, where the primer and adhesive are combined in a single bottle, available clinical data indicate that single-bottle adhesives perform well in unprepared Class V resin composite restorations for intervals of one-to-three years (Peters & others, 2001; Ripps & others, 2001; Swift & others, 2001a; Swift & others, 2001b).

Two clinical studies of similar protocol have appeared since this study began, one (Burgess & others, 2002) demonstrates the clinical performance of the resin-modified glass ionomer evaluated in this study as being superior to a hybrid resin composite but inferior to another in unprepared Class V restorations. A second (Folwaczny & others, 2001) demonstrated approximately equal overall performance for the same glass ionomer and another hybrid resin composite in prepared and unprepared Class V restorations. In both, resin restorations demonstrated a higher percentage of alpha ratings for most criteria.

This study evaluated two representative products, a resin-modified glass ionomer and a resin composite placed with its single-bottle adhesive, in appearance and clinical performance in non-retentive Class V restorations. This article is a follow-up of previously published one-year data (Brackett & others, 2002).

METHODS AND MATERIALS

The resin-modified glass ionomer restorative material chosen for this study was Fuji II LC (GC America, Inc, Alsip, IL, USA), while the resin composite chosen was Z 250 (3M ESPE, St Paul, MN, USA), which was placed using the same manufacturer's single-bottle adhesive, Single Bond. The study was conducted according to the protocol for clinical studies set forth in the 1994 American Dental Association acceptance program for dentin and enamel adhesive materials.

Thirty-seven pairs of equivalent-sized cervical erosion/abfraction lesions, primarily in premolar and anterior teeth, were identified in 24 healthy patients who presented for treatment at the student clinics of the Facultad de Estomatología, Benémerita Universidad Autónoma de Puebla. The study was conducted in accordance with all local regulations for the ethical treatment of human subjects. The median age of these patients was 47 years, while the patients ranged in age from 28 to 73 years. Each pair of cervical erosion/abfraction lesions received one restoration of each material, assigned randomly. No patient received more than two pairs of restorations. Included in the study were pairs of lesions of varied sizes and axial depth. The approximate size of each lesion and any sensitivity of the lesion to air from the dental unit were recorded prior to restoration (Table 1).

Table 1: *Distribution of Restorative Materials to Teeth Restored; Axial Depth and Preoperative Sensitivity of Erosion/Abfraction Lesions*

Z 250/Single Bond	**Incisors**						**Canines**						**Premolars**						**Molars**					
	n	*s*	*o*	*a*	*b*	*c*	*n*	*s*	*o*	*a*	*b*	*c*	*n*	*s*	*o*	*a*	*b*	*c*	*n*	*s*	*o*	*a*	*b*	*c*
Maxillary	3		3		3		7	2	5	1	5	1	9	4	5		9		2		2		2	
Mandibular	1		1	1			4	1	3		4		7	1	6	1	5	1	4	2	2		4	
Fuji II LC	**Incisors**						**Canines**						**Premolars**						**Molars**					
	n	*s*	*o*	*a*	*b*	*c*	*n*	*s*	*o*	*a*	*b*	*c*	*n*	*s*	*o*	*a*	*b*	*c*	*n*	*s*	*o*	*a*	*b*	*c*
Maxillary	3	1	2		3		1		1		1		9	3	6		9		3	2	1		3	
Mandibular	1		1		1		5		5		5		14	8	6		12	2	1		1		1	

s=sensitive to air
o=insensitive to air

a=axial depth <1 mm
b=axial depth 1-2 mm
c=axial depth >2 mm

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One investigator (MGB) placed all restorations. Isolation was accomplished using cotton rolls, with gingival retraction cord placed to expose any subgingival margins. All restorations were placed according to the manufacturers' instructions. Other than cleaning with plain pumice and water in a rubber prophylaxis cup, no mechanical preparation or abrasion of tooth surfaces was done. Surfaces restored with Fuji II LC received a 10-second application of GC Cavity Conditioner (GC America), while surfaces restored with Z 250 were etched for 15 seconds with the supplied 35% phosphoric acid, then coated twice with Single Bond. Each coat was lightly air dried, then two coats of adhesive were light cured for 10 seconds.

Each restoration was placed in a single increment and light-cured for 40 seconds. Light output of the XL3000 curing light (3M ESPE) employed was found to exceed 450 mW/cm^2 prior to and after the study and was verified during placement of the restorations with the unit's built-in radiometer. For both materials, the shade considered the closest match using a Vita shade guide (Vita-Zahnfabrik, Bad Säckingen, Germany) was selected. Restorations of both materials were shaped with a plastic instrument prior to light curing, contoured with ET finishing diamonds (Brasseler USA, Savannah, GA, USA) using air/water coolant and polished with wet Diacomp abrasive rubber points (Brasseler, USA).

At baseline, 6, 12, 18 and 24 months, the restorations were clinically evaluated by two other calibrated investigators using modified Ryge/USPHS criteria (Cvar & Ryge, 1971) listed in Table 2. The examiners were unaware which material had been used in a restoration, and any discrepancy between examiners was resolved before the patient was dismissed. A further recall is planned at 36 months.

For purposes of statistical analysis, restorations receiving a score of "charlie" in any category were classified as failed restorations. The incidence of failures

Table 2: *Modified USPHS Rating System*

Category	Score	Criteria
Retention	Alpha	No loss of restorative material
	Charlie	Any loss of restorative material
Color match	Alpha	Matches tooth
	Bravo	Acceptable mismatch
	Charlie	Unacceptable mismatch
Marginal Discoloration	Alpha	No discoloration
	Bravo	Discoloration without axial penetration
	Charlie	Discoloration with axial penetration
Secondary Caries	Alpha	No caries present
	Charlie	Caries present
Anatomic Form	Alpha	Continuous
	Bravo	Slight discontinuity, clinically acceptable
	Charlie	Discontinuous, failure
Marginal Adaptation	Alpha	Closely adapted, no detectable margin
	Bravo	Detectable margin, clinically acceptable
	Charlie	Marginal crevice, clinical failure
Surface Texture	Alpha	Enamel-like surface
	Bravo	Surface rougher than enamel, clinically acceptable
	Charlie	Surface unacceptably rough

Table 3: *Results of Clinical Evaluation for Resin-Modified Glass Ionomer and Resin Composite Restorations (%)*

Z 250/Single Bond

	Retention**			Color Match			Marginal Discoloration		Secondary Caries		Anatomical Form		Marginal Adaptation		Surface Texture	
	n*	alpha	charlie	n*	alpha	bravo	alpha	bravo	alpha	charlie	alpha	bravo	alpha	bravo	alpha	bravo
baseline	37	100	0	37	100	0	97	3	100	0	100	0	100	0	97	3
6 months	32	88	12	28	100	0	96	4	100	0	100	0	75	25	93	7
12 months	31	84	16	26	100	0	96	4	100	0	96	4	88	12	100	0
18 months	31	84	16	26	100	0	96	4	100	0	96	4	88	12	96	4
24 months	27	81	19	22	100	0	95	5	100	0	95	5	82	18	86	14

Fuji II LC

	Retention**			Color Match			Marginal Discoloration		Secondary Caries		Anatomical Form		Marginal Adaptation		Surface Texture	
	n*	alpha	charlie	n*	alpha	bravo	alpha	bravo	alpha	charlie	alpha	bravo	alpha	bravo	alpha	bravo
baseline	37	100	0	37	100	0	100	0	100	0	100	0	100	0	45	55
6 months	32	97	3	31	100	0	100	0	100	0	94	6	94	6	23	77
12 months	31	97	3	30	90	10	100	0	100	0	93	7	87	13	93	7
18 months	31	97	3	30	87	13	100	0	100	0	93	7	94	6	87	13
24 months	27	96	4	26	85	15	100	0	100	0	92	8	96	4	85	15

**sample size larger for retention than for other criteria because lost restorations unavailable for evaluation.*
***cumulative throughout the study.*

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was analyzed as a pairwise comparison using an exact binomial test at a confidence level of 5%.

RESULTS

At the end of two years, 27 pairs of restorations were available for evaluation—a recall rate of 73%. Five Z 250 restorations were lost, four prior to the six-month recall and one during the 6-12 month interval. One Fuji II LC restoration was lost prior to the six-month recall. No restorations of either material were lost during the 12-18 month interval. The percentage of lost restorations, which were the only scores of "charlie" assigned in the study, was 19% for Z 250 and 4% for Fuji II LC over two years. Restorations of Z 250 demonstrated a better color match with tooth structure than Fuji II LC, with 100% and 85% alpha scores, respectively, although the materials were approximately equal in the surface texture category. The percentage of restorations receiving alpha scores for all other criteria were relatively high and nearly equal for both materials after two years, except for Z 250 restorations, which received 82% alpha scores for marginal adaptation compared to 96% for Fuji II LC.

None of the teeth with retained restorations that exhibited sensitivity to air at the beginning of the study were sensitive at recall. No statistically significant difference in the incidence of failed restorations was found between the materials (exact binomial test; p=0.13). Complete results are presented in Table 3.

DISCUSSION

The authors were surprised at the relatively poor performance of the single-bottle resin adhesive in this study, given that its two-bottle predecessor had performed very well in a previous study of the same protocol (Browning & others, 2000) . Because four of the five lost resin composite restorations were lost from insensitive areas, it may be that a longer etch is indicated with this product for dentin known to be insensitive to ensure resin infiltration. As in most clinical studies of this type, lost restorations were displaced early in the study, which lends credence to the ADA acceptance program protocol and to reporting early clinical data. Although short-term results are probably representative of the performance of adhesives, even two-year studies may be too short for development of any secondary caries, which were not observed in this study despite the absence of water fluoridation in the community in which the study subjects reside. For this reason, a three-year recall is planned.

The use of diamond-impregnated rubber points, while common for final finishing of resin composites, is less commonly reported for resin-modified glass ionomers that, in previous studies, have usually been finished with abrasive disks. These points produced a relatively rough surface in the glass ionomers over the first six months of the study and are probably not the best choice of finishing method for restorations of this material.

The effect of repeated measures on confidence level precludes running a statistical test for each criterion in a study of this size. For this reason, only the overall clinical performance, in terms of failed restorations of the two materials, was compared statistically. Although not statistically compared, the color match of the resin composite restorations appears to be superior, while the surface texture of the two materials appears to be approximately equal. Despite the lack of a statistical difference, if the results of this study are representative, Single Bond would not qualify for acceptance as a dentin and enamel adhesive under the ADA acceptance program, while Fuji II LC would have qualified for full acceptance after the 18-month recall.

Of these two materials, the authors consider the resin-modified glass ionomer to be the better choice for Class V restorations because of retention and the potential for cariostasis but acknowledge that a veneer of resin composite would likely be eventually necessary in highly-visible teeth of esthetically-conscious patients.

CONCLUSIONS

Although a larger number of resin composite restorations was lost, no statistically significant difference in clinical performance in non-retentive Class V restorations was observed between the two restorative materials. Although not statistically compared, the resin composite restorations appear superior in color match to the resin-modified glass ionomer restorations.

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Effect of Resin Viscosity and Enamel Beveling on the Clinical Performance of Class V Composite Restorations: Three-Year Results

LN Baratieri • S Canabarro
GC Lopes • AV Ritter

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Clinical Relevance

Beveling enamel margins might not be necessary in non-carious Class V restorations placed with a total-etch, one-bottle adhesive.

SUMMARY

This study evaluated the effect of the elastic modulus and margin configuration on the clinical performance of resin-based composite restorations in Class V non-carious defects. One hundred and five cervical non-carious defects on buccal surfaces of canines and premolars were included in this study. Defects were randomly divided into three Groups and restored according to the following techniques: Group 1—no enamel bevel was placed and the defect was restored with a microfilled resin-based composite (Durafill VS); Group 2—the enamel margin was beveled and the defect restored as in Group 1; Group 3—the enamel margin was beveled and the defect was restored with a flowable resin-based composite (Natural Flow). Each group comprised 35 lesions. A total-etch, one-bottle adhesive (One-Step) was used in all groups. Retention rate, pre- and post-operative sensitivity, marginal discoloration and secondary caries were determined over a three-year period and the data were analyzed statistically.

At six months post-insertion, the restorations placed with beveled enamel margins resulted in 100% retention regardless of the composite used compared to a 66% retention of the non-beveled margins. At two and three years, no significant

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difference in retention rate was found among the three groups. Post-operative sensitivity, marginal discoloration and secondary caries were not affected by enamel beveling and restorative material.

Beveled enamel margins resulted in significantly better clinical retention in the first six months only. Enamel beveling and composite viscosity appeared to not significantly affect the clinical performance of Class V non-retentive composite restorations after three years.

INTRODUCTION

Cervical non-carious erosion/abrasion or abfraction defects are used as a clinical model to evaluate the efficacy of dentin bonding agents in non-retentive tooth preparations. This model is recommended by the American Dental Association (ADA) in its Acceptance Program for Adhesive Restorative Materials (ADA—Council of Scientific Affairs, 2001). When applied to non-retentive cervical defects, the clinical performance of resin-based composite restorations relies mostly on the bond generated by the adhesive, with retention being an important criterion to determine the appropriateness of the technique/material.

Even though emphasis is always placed on the adhesive system used to restore non-retentive cervical defects, the restorative material and the preparation margin configuration might also influence the clinical performance of the restoration. It has been proposed that microfilled composites perform better than hybrid composites in abfraction defects (Heymann & others, 1991; Levitch & others, 1994). When compared to hybrid composites, microfills have a lower elastic modulus. It is believed that this allows the material to flex with the tooth during function, reducing the chances for failure of the bonded interface and dislodgment of the restoration (Heymann & others, 1991). Based on this hypothesis, flowable composites that have an even lower elastic modulus than microfill composites might further minimize the development of stress during function.

Several simplified adhesive systems have been introduced during the past decade. "One-bottle" systems combine primer and adhesive functions in one component. Even though the *in vitro* performance of these systems has been extensively tested in bond strengths and microleakage studies, such materials have to be validated in controlled clinical trials. Currently, few long-term clinical evaluation studies have been reported on such materials. This *in vivo* study evaluated the clinical performance of a microfill and flowable composites bonded on beveled and non-beveled, non-carious cervical defects with a total-etch, one-bottle adhesive system.

METHODS AND MATERIALS

The protocol and consent form for this study were reviewed and approved by the Federal University of Santa Catarina Committee on Investigations Involving Human Subjects. Written informed consent was obtained from all participants. After screening several potential participants, 50 participants ranging in age between 28 and 55 years presenting with cervical abrasion/abfraction defects requiring restoration were accepted into the study. One hundred and five defects were included in the study. All defects had similar characteristics regarding geometry (saucer-shaped), size (approximately 2 mm X 2 mm), depth (approximately 1.5 mm) and degree of sclerosis. The degree of sclerosis was determined to be 2 according to the University of North Carolina dentin sclerosis scale (Heymann & Bayne, 1993).

All restorations were placed under rubber dam isolation. A Vita shade guide (Vita Zahnfabrik, Germany) was used to determine tooth shade prior to rubber dam isolation. Each part had three defects that were restored with a different material or margin configuration. All restorations were done by one operator (SC). A total of 35 defects were restored in each experimental group as follows:

Group 1 (n=35): The tooth to be restored was cleaned with pumice/water and the defect etched with 32% phosphoric acid for 15 seconds (Uni-Etch, BISCO, Schaumburg, IL, USA), rinsed and blot dried. A one-bottle adhesive (One-Step, BISCO) was applied and light-cured following the manufacturer's instructions. The defect was then restored with a microfill resin-based composite (Durafill VS, Heraeus Kulzer, Dormagen, Germany) in two increments.

Group 2 (n=35): The defects were treated and restored as in Group 1, but the enamel margins were beveled with a #860-014 flame-shaped diamond bur (Brasseler USA, Savannah, GA, USA) prior to the acid etching step.

Group 3 (n=35): The defects were treated as in Group 2 (bevel, acid-etched, bonding) and restored with a flowable resin-based composite (Natural Flow, Scientific Pharmaceuticals, Inc, Pomona, CA, USA).

Distribution of restorations per participant was done by blocked randomization, so that if a participant received more than one restoration, they were placed with a different technique. Eighteen participants received three restorations, 19 received two restorations and 13 received one restoration. The resin-based composites were applied into the defects in two increments, and each increment was polymerized for 40 seconds with a XL3000 curing light (3M ESPE, St Paul, MN, USA) with a power output maintained at 400mW/cm^2.

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The restorations were finished and polished one week post-insertion. Fine and extra-fine diamond burs (Brasseler USA) were used for finishing and contouring, and polishing was performed with silicon impregnated finishing points and polishing pastes (Enhance, Dentsply LD Caulk, Milford, DE, USA).

All restorations were evaluated using modified United States Public Health Service (USPHS) criteria (Cvar & Ryge, 1971) at baseline (one week post-insertion) and at 6, 12, 24 and 36 months after baseline. Two examiners evaluated the restorations independently and a consensus was reached if the evaluations were not the same. The criteria evaluated in this study were retention, marginal discoloration, pre- and post-operative sensitivity, secondary caries and white line formation at margins. The results expressed in percentage for each criterion among the three groups were subjected to statistical analysis using a proportion test at a confidence level of 95% using SPSS 10.0 for Windows (SPSS Inc, Chicago, IL, USA).

RESULTS

Results are summarized in Table 1. All participants were recalled at all evaluation times. Statistical analysis of the data collected in each recall interval revealed no significant differences in marginal discoloration, secondary caries and white line at margins among the groups and for all time periods (Table 1). Analysis of the data related to pre- and post-operative sensitivity indicates that the restorations resulted in a reduction of sensitivity and that post-operative sensitivity decreased from baseline to the six-month evaluation. Post-operative sensitivity was absent (100% alpha scores) at all evaluation periods after six months.

Retention results are plotted in Figure 1. Beveled margins resulted in 100% retention at six months, regardless of restorative material (Groups 2 and 3), compared to 66% retention of the non-beveled margins (Group 1). This difference was statistically significant ($p<0.05$). Microfill and flowable composites performed similarly in beveled margins at six months. At 12 months, statistical analysis revealed two equivalent subsets, Groups 1 and 2 and Groups 2 and 3. Group 3 had significantly more retained restorations (100%) than Group 1 (63%). At 24 and 36 months, there was no significant difference in retention rates among the three Groups.

DISCUSSION

Most of the adhesive systems currently available use three basic principles in bonding to dental tissues: demineralization, infiltration and polymerization. In general, adhesives include components that contemplate each of these principles. The adhesive used in this study (One-Step, BISCO) was the first "single-component" adhesive system introduced where the primer and the adhesive are combined in the same solution. One-Step is an acetone-based adhesive. The high concentration of solvent (70%) allows for part of the dentin humidity to be volatilized together with the acetone. In addition, the system contains 2-hydroxyethyl methacrylate (HEMA) and biphenyl dimethacrylate (BPDM), which had already been used on primer B of the All Bond II adhesive system.

Some bond strength studies with One-Step offer results above 20 MPa when applied to wet dentin (Kanca, 1996; 1997). However, other studies using different methodologies reveal results around 15 MPa (Perdigão & others, 1999). Swift and

Table 1: *Number of Restorations Presented and Evaluated (N) and the Results for USPHS Criteria (alpha %) per Group at Baseline and at Each Recall Interval*

	Baseline	6 months	12 months	24 months	36 months
• Group 1: USPHS alpha (%)					
N	**35**	**23**	**22**	**22**	**19**
Retention	100	66	63	63	54
Marginal discoloration	100	100	82	82	84
Pre-op sensitivity	20	NA	NA	NA	NA
Post-op sensitivity	57	100	100	100	100
Secondary caries	100	100	100	100	100
White line at margin	94	91	91	95	100
• Group 2: USPHS alpha (%)					
N	**35**	**35**	**27**	**23**	**18**
Retention	100	100	77	66	51
Marginal discoloration	100	100	92	69	83
Pre-op sensitivity	48	NA	NA	NA	NA
Post-op sensitivity	74	100	100	100	100
Secondary caries	100	100	100	100	100
White line at margin	100	100	100	100	100
• Group 3: USPHS alpha (%)					
N	**35**	**35**	**32**	**28**	**24**
Retention	100	100	91	80	69
Marginal discoloration	100	100	100	100	100
Pre-op sensitivity	66	NA	NA	NA	NA
Post-op sensitivity	91	100	100	100	100
Secondary caries	100	100	100	100	100
White line at margin	94	100	100	100	100

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others (1997) reported a mean shear bond strength of 7.3 MPa for One-Step. A recent study reported a mean dentin bond strength value of 6.4 MPa (Lopes & others, 2002) for One-Step. In addition, after simulated clinical use for three weeks, One-Step's bonding capacity to dentin decreased when compared to baseline values, possibly due to high acetone volatilization (Perdigão, Swift & Lopes, 1999).

According to Perdigão and others (1996), upon applying One-Step *in vivo*, a well-defined interdiffusion zone results, with areas of union alternating with areas of fissure formation between the hybrid and the adhesive layers. Single-component adhesives usually result in a thin adhesive layer. Manufacturers recommend multiple applications or coatings in an attempt to compensate for this thin layer, but an adhesive with a high quantity of solvent on the surface can result in a film that is rich in non-polymerized monomers (Van Meerbeek & others, 1994).

Research has shown that simplified adhesives do not always result in a more effective bond. In fact, studies reveal a reduction in bond strength due to the difficulty in completely removing the solvent (Swift & others, 1997). It should also be noted that, in the laboratory, the thickness of the adhesive layer can be more easily controlled than under clinical situations. That might explain why an adhesive system with high *in vitro* bond strengths (Kanca, 1996; 1997) and a non-significant index of marginal microleakage (Castelnuovo, Tjan & Liu, 1996) attained relatively poor retention performance in this study. Consensus seemingly exists that one-bottle adhesives are more sensitive to the technique than systems of two or more components (Kanca, 1996; Tyas, 1994; Finger & Fritz, 1996).

It is not always possible to compare the retention rates of the different studies due to the many variables that can influence the retention rate of Class V restorations *in vivo*. These variables include, but are not limited to, patient age, occlusion, etiology of the lesion, location, degree of dentin sclerosis, available enamel at the margins, geometry, depth and size of the lesion, margin configuration of the preparation, restorative technique used and restorative material used. Retention rates for Class V composite restorations placed in non-carious lesions as reported in the literature are variable. Van Meerbeek and others (1996) evaluated the clinical performance of Class V microfill composite (Silux Plus, 3M ESPE) restorations placed in non-carious lesions with or without enamel beveling. For the multi-bottle adhesive Scotchbond Multi-Purpose (3M ESPE), they reported a 98% and 96% retention rate for beveled and non-beveled enamel margins, respectively, after three-years. Sturdevant and others (1999) reported an 81% retention using Pro-Bond adhesive (Dentsply/Caulk) and Prisma APH (Dentsply/Caulk) composite and non-beveled enamel margins after three years. Clinical studies with the All Bond II adhesive system (BISCO) have also been reported, and the retention rates vary from 93% at two years with a microfill composite (Bis-Fil M, BISCO) in beveled enamel margins (Ianzano & Gwinnett, 1993), to 83% at six months with Bis-Fil M and non-beveled enamel margins (Snuggs & others, 1992), to 70% at 14 months with Silux (3M ESPE) in non-beveled enamel margins (Tyas, 1994).

The retention rates observed in this study are relatively low when compared to the above mentioned studies and those of others (Van Dijken, 2000). Our results for the beveled margins restored with flowable resin composite correlate with those of Schwartz and others (1998), who reported an 89% and 93% retention rate after 18 months using One-Step (BISCO) and a microfilled resin composite (AeliteFil, BISCO) or a "flexible" liner (BISCO), respectively, in diamond bur-abraded cervical lesions. Burrow and Tyas (1999) reported 100% and 95% retention rates using One-Step after six and 12 months, respectively, using a microfilled composite (Silux, 3M ESPE) and a flowable resin composite (AeliteFlo, BISCO) and non-beveled enamel margins. These authors reported no difference in retention between a hybrid flowable composite and a microfilled composite.

It has been reported that microfilled composites leak less at the interface when compared to hybrid composites (Fruits & others, 2002). However, Fitchie and others (1995) found no such differences. The use of flowable composites for Class V restorations has been suggested (Miller, 1997; Christensen, 1996). In theory,

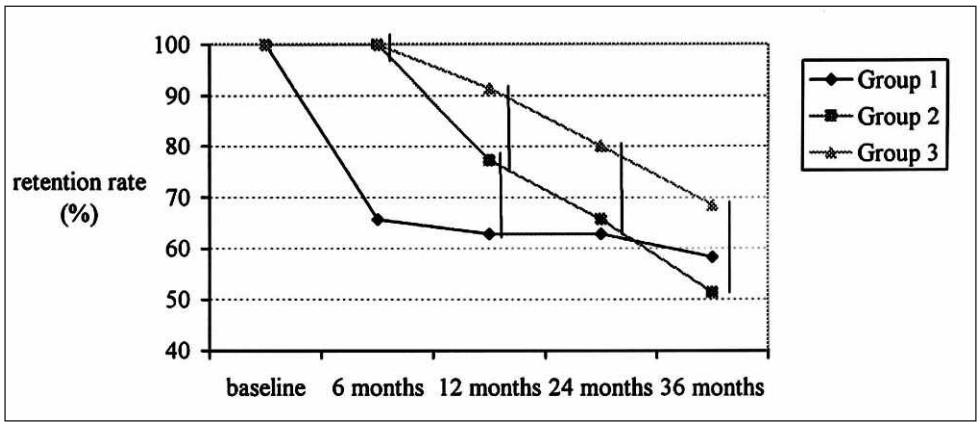

Figure 1. *Retention rates for the different groups over time. Vertical lines indicate statistically similar values at each time interval (p ≥ 0.05).*

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flowable composites flex more than hybrid and microfill composites upon and after curing, allowing for greater relaxation of tensions imposed to the tooth-resin composite interface by shrinkage during its polymerization, as well as thermal expansion/contraction and occlusal forces (Kemp-Scholte & Davidson, 1990). In this study, all of the above factors have apparently collaborated to enhance three-year clinical results of retention in the Group where a flowable resin composite (Natural Flow) was used (69%) when compared to the Group restored with microfilled resin (51%).

Regardless of the composite used, clinical effectiveness increased when the adhesive system was applied in combination with beveling of the enamel margin for six and 12 months. Such results may well indicate that initial adhesion to dentin using the One-Step system is poorer than enamel bond. However, after the second year of evaluation, the best results with the bevel are found only when flowable composite is used, which was not statistically significant.

Finally, it is important to emphasize that retention should not be considered the only parameter to assess performance of Class V restorations. However, this study confirmed what is frequently reported in a number of studies, that many other clinical parameters remain stable over the observation periods commonly reported. It can be concluded from these observations that longer evaluation times might be necessary to detect changes in marginal discoloration and secondary caries.

CONCLUSIONS

Based on the results of this study, beveled enamel margins resulted in better retention rates for Class V adhesive composite restorations only at six months. At 12, 24 and 36 months, beveled margins did not contribute to increased retention rates. The viscosity of the restorative material appeared not to affect the clinical performance of Class V adhesive composite restorations.

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A Clinical Evaluation of Two In-Office Bleaching Products

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Clinical Relevance

With increased patient demand for esthetic improvements, bleaching has become a popular treatment in dentistry, and new bleaching products are being introduced to the practice. In this study, two in-office products performed similarly. After applying the second in-office bleaching treatment, tooth lightness improved. Hence, a single in-office treatment is not the maximum whiteness that can be achieved for a patient.

SUMMARY

This half-mouth design, two-week treatment phase, combined with an 11-week evaluation double-blinded randomized clinical trial was conducted to compare two in-office bleaching products, StarBrite (35% hydrogen peroxide) with Opalescence Xtra Boost (38% hydrogen peroxide), for degree of color change of teeth, any relapse effect (darkening) associated with discontinued use and gingival irritation and tooth sensitivity associated with use. The degree of color change and relapse was evaluated by using a colorimeter, shade guide and color slide photographs. Participants self-evaluated their gingival irritation and tooth sensitivity. They recorded daily the level of gingival irritation and tooth sensitivity experienced during the first three weeks of the study.

The results of this study showed no statistical difference between products during active treatment periods and any follow-up visits using the three-color evaluation methods. Color relapse began after the bleaching treatments were finished and continued until the fifth week, after which no further significant changes appeared. Also, there was no statistical difference in gingival irritation and tooth sensitivity between the products.

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INTRODUCTION

Cosmetic dentistry has become a very important part of restorative dental practice. The appearance and color of teeth is important to many individuals seeking dental treatment. Dentistry has succeeded in preserving natural teeth, even in older patients, so that lighter-colored teeth have become attainable for most people. Vital bleaching has been accepted as a method for treating discolored teeth. Increased interest in treating tooth staining and discoloration is demonstrated by the

large number of tooth whitening agents appearing on the market. Today, the majority of practitioners perform vital tooth bleaching on patients with discolored teeth with a high rate of success (Christensen, 1998). Bleaching is the most conservative treatment for discolored teeth when compared to other treatment modalities, such as veneers, crowns or composite bonding (Barghi, 1998).

Although at-home bleaching has increased dramatically in popularity, in-office bleaching products are still in demand and strongly promoted by manufacturers (Blankenau, Goldstein & Haywood, 1999). There are still many indications for in-office bleaching. These include cases where a patient cannot wear bleaching trays, or when patients want to have their teeth bleached quickly by applying several in-office treatments and are unwilling to wait two weeks for at-home techniques. Moreover, in selected cases, in-office bleaching sometimes augments the whitening effects of at-home bleaching (Barghi, 1998). Results of a published survey of general practitioners showed that 33% of dentists in the United States use in-office bleaching (Clinical Research Associates, 2001).

This study compared the ability of two different in-office bleaching products to lighten teeth. It was also designed to evaluate any relapse effects associated with discontinued use, and tooth sensitivity and gingival irritation associated with the bleaching treatments.

METHODS AND MATERIALS

Twenty subjects volunteered to participate in this study and signed a consent form prior to participating. All 20 subjects met the inclusion/exclusion criteria (Table 1).

A half-mouth design was used, in which each patient's six maxillary anterior teeth were bleached, with the bleaching products assigned to either the right or left anterior teeth. Each patient served as his or her own control. The assignment was conducted randomly by flipping a coin.

Two commercially available bleaching agents were used; StarBrite (Interdent, Inc, Los Angeles, CA, USA) Bleaching Gel with 35% hydrogen peroxide (which has received the ADA Seal of Acceptance) and Opalescence Xtra Boost (Ultradent Products, Inc, South Jordan, UT, USA) Bleaching Gel with 38% hydrogen peroxide. Manufacturers' instructions for handling and application were followed for both products used in this study.

All subjects received a dental screening and prophylaxis prior to beginning the study, using a fluoridated paste (Nupro Supreme, Dentsply Int, York, PA, USA) to remove extrinsic stains. The prophylaxis was performed one week prior to the active treatment phase being initiated. The preoperative evaluation was performed on the maxillary anterior teeth and their surrounding tissues; however, the gingival index was performed on all teeth using the Loe-Silness Gingival Index (Loe & Silness, 1963).

For each subject, an alginate impression was taken of the maxillary arch using Jeltrate Plus (Caulk Division Dentsply International Inc, Milford, DE, USA), into which Silky-Rock stone (Whip Mix Corp, Louisville, KY, USA) was poured. The resulting cast was used to construct a positioning jig with palatal coverage to ensure consistent positioning of the colorimeter (Chroma Meter CR-321, Minolta, Ramsey, NJ, USA). The Eichhold Positioning System with Pindex dual-pin precision attachment (Coltene/Whaledent Inc, Mahwah, NJ, USA) was used in this study (Mokhlis & others, 2000). At each appointment, color evaluation was performed using the following three methods:

1. Photographs recorded with Ectachrome Elite 100, 35-mm film (Kodak, Rochester, NY, USA) at each appointment in the same area with color corrected lighting. At the end of the study, slide photographs were projected onto an image of 3.0 x 4.5 feet and compared for color differences between the right and left sides by two independent evaluators. The degree of color difference was ranked: 0=no change; 1=slight; 2=moderate and 3=significant difference.

2. Subjective shade guide matching by an independent, experienced evaluator using the Trubyte Bioform

Table 1: *Inclusion and Exclusion Criteria*

Inclusion Criteria	Exclusion Criteria
Candidates must have all six maxillary teeth	Candidates with a history of any medical disease that may interfere with the study or require special considerations
The maxillary anterior teeth should not have more than 1/6 of the labial surface restored, and the location of the restoration, if any, must not interfere with placement of the colorimeter	Candidates who have had professionally applied in-office or at-home bleaching procedures in the past three years
Candidates must be willing to sign a consent form	Candidates who have used tobacco products during the past 30 days
Candidates must be at least 18 years of age	Candidates who have gross pathology in the oral cavity
Candidates must be able to return periodic examinations	Candidates with a gingival index score for greater than 1.0
Candidates must be willing to refrain from using tobacco products during the study period	Candidates who are pregnant or lactating
The maxillary anterior teeth must be darker than B -54 and lighter than B-4 shade tabs on the Truebyte Bioform Color Ordered shade guide	Candidates with tetracycline-stained teeth

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Color Ordered Shade Guide (Dentsply Int, York, PA, USA).

3. Colorimeter measurements (Matis & others, 1998; Zekonis & others, 2003). During evaluations, each of the six anterior maxillary teeth was color measured three different times. The colorimeter was calibrated before each subject. The colorimeter measures the color of teeth based on the CIE L* a* b* color space system. This system was defined by the International Commission on Illumination in 1967 and is referred to as CIELAB. The L* represents the value (lightness or darkness); a* is the measurement along the red-green axis and b* is the measurement along the yellow–blue axis. A positive a* value indicates the red direction, while negative a* value indicates the green direction. Also, a positive b* value indicates the yellow direction, while a negative b* value indicates the blue direction. Total color differences or distances between the two colors (ΔE) were calculated using the formula:

$$\Delta E_{ab}^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

A rubber dam was used to isolate and protect the soft tissue. Also, OpalDam (Ultradent Products, Inc), a light-cure resin was used at the midline as a barrier to separate the two bleaching gels. It was extended interproximally between the central incisors into the incisal embrasure at a width of 2 mm facially and 6 mm-8 mm lingually, then cured for 10 seconds (Figure 1).

At the initial appointment, the subjects received a color evaluation of their maxillary anterior teeth using the three measurement methods to determine the initial color (baseline). At the same appointment, the sides of the arch on which the products would be used were determined on a random basis. The specified isolation techniques were performed, and both products were applied on their respective sides. No color evaluation was conducted immediately after the bleaching process because of the dehydration effect of the bleaching treatment. At week one, subjects returned for color measurement and the second bleaching procedure. Only color evaluations were accomplished on the second, fifth and eleventh week appointments. Table 2 shows the data collection design.

StarBrite and Opalescence Xtra Boost were mixed and handled according to manufacturers' instructions. For StarBrite, two hydrogen peroxide ampoules were added to one powder tub, then two energizer ampoules were added to the mix prior to applying the gel. For Opalescence Xtra Boost, activator and bleaching agent were mixed using the syringes provided by the manufacturer. After applying for five minutes, both whitening gels were stirred using a brush, while remaining on the teeth. The gels were allowed to remain on the teeth for a total of 10 minutes. The gels were then rinsed off and the teeth dried. Both procedures were repeated two more times at the same sitting, providing a total of 30 minutes of bleaching with both agents.

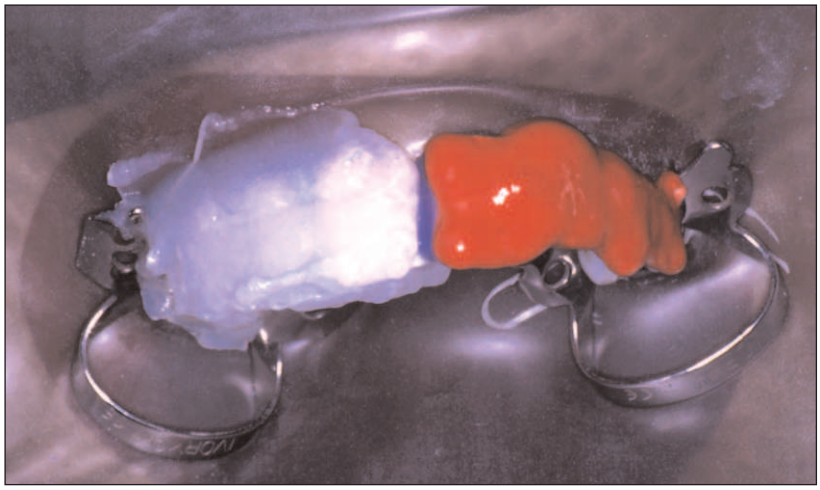

Figure 1. *Isolation technique and gel application.*

During the 11 weeks of the study, subjects were asked to brush their teeth with fluoridated, non-desensitizing, non-whitening toothpaste twice daily. They were given a form on which they recorded daily the level of tooth sensitivity and gingival irritation they experienced during the first three weeks of the study. Tooth sensitivity was defined as any sensitivity from cold temperature, while gingival irritation was defined as any sensitivity or discomfort from food and tooth brushing. Candidates recorded any gingival irritation or tooth sensitivity (indicating the side of the arch) into one of five categories: 1) none, 2) slight, 3) moderate, 4) considerable or 5) severe.

Table 2: *Data Collection Design*

Base Line		Week 1 (1 Week After 1st Bleaching Treatment)		Week 2 (1 Week After 2nd Bleaching Treatment)	Week 5	Week 11
Color measurement	1st bleaching treatment	Color measurement	2nd bleaching treatment	Color measurement	Color measurement	Color measurement

STATISTICAL METHODS

Analysis of variance (ANOVA) was used for comparing baseline L*, a*, b*, shade guide rank, ΔL*, Δa*, Δb*

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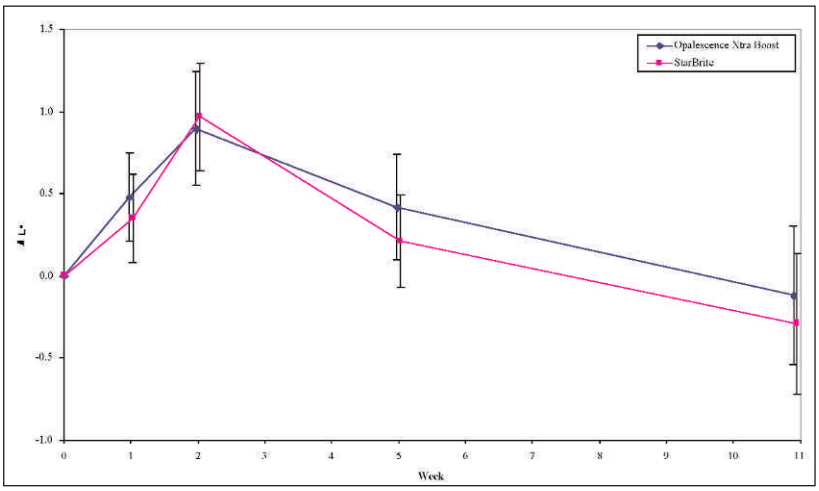

Figure 2. *ΔL* for Opalescence Xtra Boost and StarBrite showing error bars.*

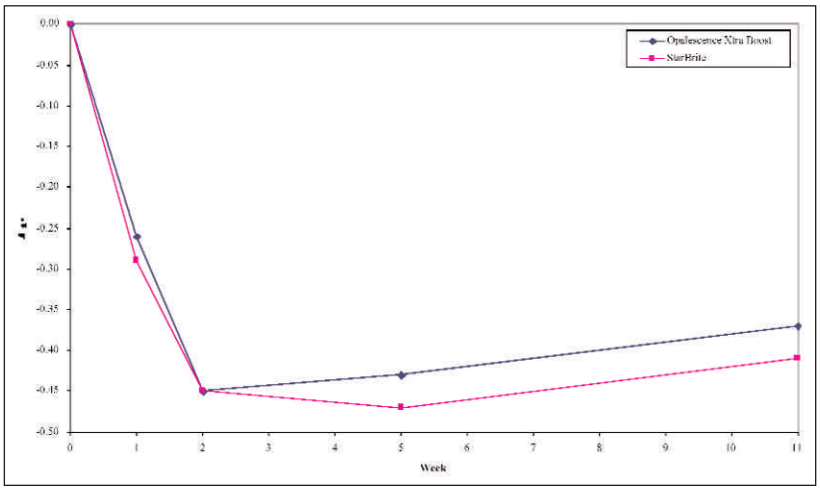

Figure 3. *Δa* for Opalescence Xtra Boost and StarBrite.*

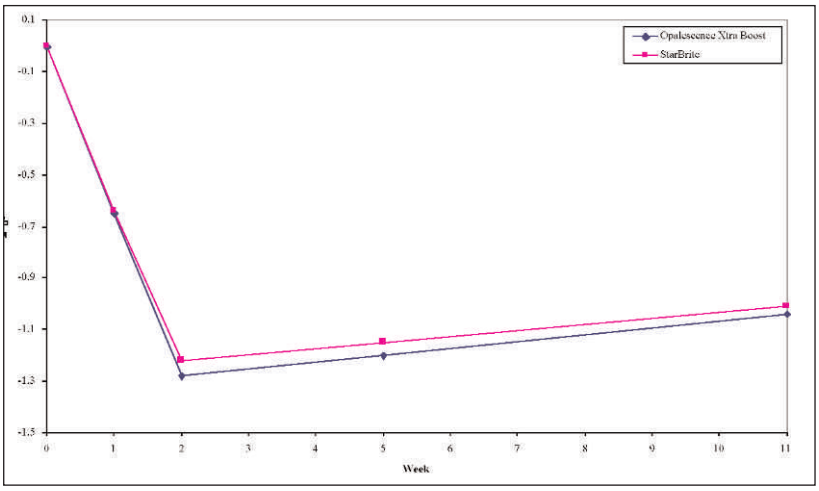

Figure 4. *Δb* for Opalescence Xtra Boost and StarBrite.*

ΔE and Δ shade guide. It was also used for comparing daily gingival irritation and tooth sensitivity. Wilcoxon Sign Rank tests were used to determine significant differences in tooth color by slide assessment.

RESULTS

Twenty subjects were enrolled and completed the study. Ten participants were female (50%) and 10 male (50%), with an age range from 30 to 71 years, with an average age of 55 years.

Chroma Meter Data

At baseline, the products did not have significantly different L* (p=0.38), a* (p=0.96) or b* (p=0.84) colorimeter measurements. At 1, 2, 5 and 11 weeks, the products were not significantly different in ΔL* (p=0.74) (Figure 2), Δa* (p=0.41) (Figure 3), Δb* (p=0.76) (Figure 4) and ΔE (p=0.36) (Figure 5). This was for overall examinations and any individual follow-up examination (ΔL* p=0.99, 0.94, 0.88 and 0.97), (Δa* p=0.89, 1.00, 0.77 and 0.78), (Δb* p=1.00, 0.98, 0.99 and 1.00) and (ΔE p=0.98, 0.91, 0.22 and 1.00) for all teeth and separately for centrals, laterals and cuspids, respectively.

Shade Guide Data

At baseline, the products did not have a significantly different shade guide (p=0.74). The products were not significantly different in the delta shade guide overall (p=0.65) or for any individual follow-up examination (p=1.00, 1.00, 1.00 and 0.97 for one week, two weeks, five weeks and 11 weeks, respectively) for all teeth and separately for centrals, laterals and cuspids (Figure 6).

Slide Assessment Data

At baseline, the products did not have significantly different slide assessments (p=1.00) or at any follow-up examination (p=0.25 at one week, p=1.00 at all other examinations) (Table 3). Figures 7-9 show clinical pictures at baseline and after one and two in-office bleaching applications.

Sensitivity Data

Opalescence Xtra Boost and StarBrite did not have significantly different gingival irritation (p=0.27) and tooth sensitivity (p=0.36) for all days pooled or for any of the individual days (gingival p>0.13) (tooth p>0.37) (Figures 10-11).

DISCUSSION

In this study, the authors did not measure the changes in tooth color immediately after

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bleaching treatment, because they did not want dehydration of the teeth to affect the measurements. It has been found that rubber dam isolation can cause the teeth to dehydrate, and the researchers needed to wait at least 30 minutes after isolation removal in order to rehydrate the teeth (Russell, Gulfraz & Moss, 2000). Color evaluation was postponed until one week after the bleaching treatment. This avoided any dehydration effect from being included erroneously in the color evaluation data. Changes were measured before the first and second bleaching treatment and at week two, five and 11 after baseline. The amount of change in CIE L*a*b* and shade guide that occurred immediately after the bleaching treatment and during the seven days following the bleaching treatment were not measured and, therefore, the maximum changes and rebound effects during that period are unknown.

Because of the half-mouth design used for this study, all comparisons were within-subject and the standard deviations shown in the tables were between-subject. This design helps to reduce patient variations.

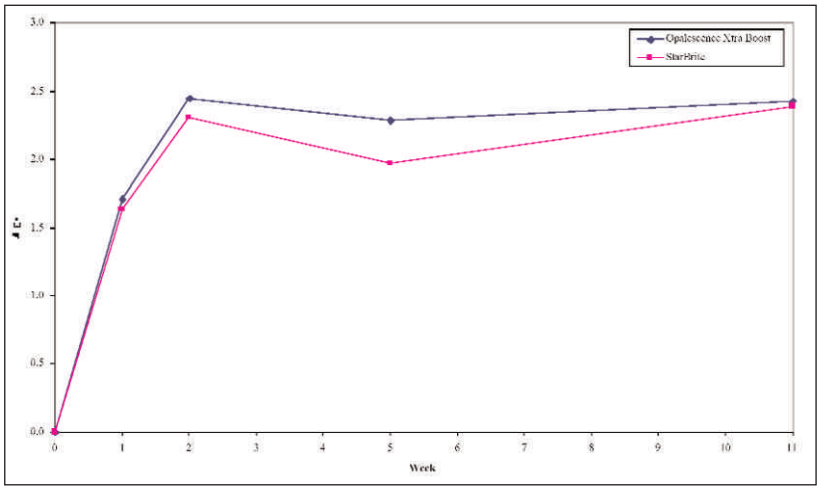

Figure 5. *ΔE for Opalescence Xtra Boost and StarBrite.*

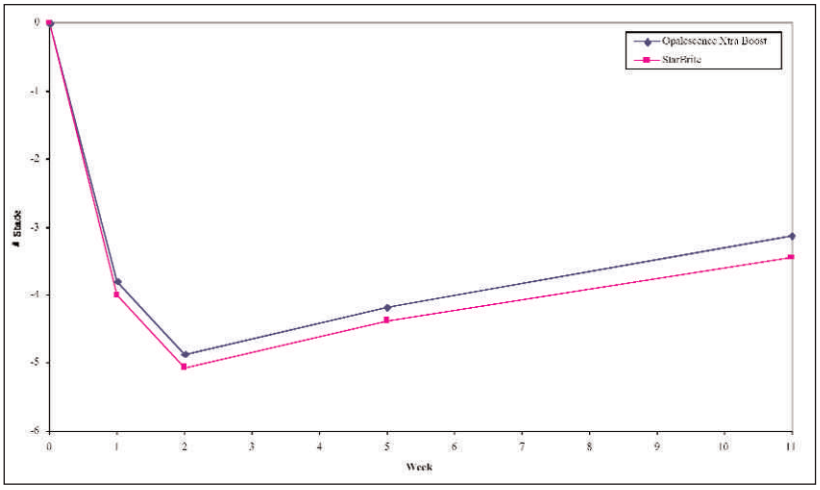

Figure 6. *Δ Shade for Opalescence Xtra Boost and StarBrite.*

Table 3: *Slide Assessment Data for Opalescence Xtra Boost and StarBrite*

	StarBrite Lighter		No Difference		Opalescence Xtra Boost Lighter	
Week	#	%	#	%	#	%
0	0	0%	20	100%	0	0%
1	0	0%	17	85%	3	15%
2	0	0%	20	100%	0	0%
5	0	0%	20	100%	0	0%
11	0	0%	19	95%	1	5%

One must be careful when comparing results from different bleaching studies. It is generally believed that peroxide concentration is related to efficacy. However, current evidence suggests that the technique for applying the product, including the number of applications and duration of each bleaching application, can be of equal importance (Lu, Margiotta & Nathoo, 2001).

At two weeks, mean ΔE (total color change) reached 2.45 for Opalescence Xtra Boost and 2.31 for StarBrite, which meant that subjects achieved the same results clinically. This disagrees with mean ΔE obtained by Zekonis and others (2003), who compared in-office and at-home bleaching procedures using a half-mouth design. Zekonis and others (2003) reached 4.33 for mean ΔE for the in-office treatment using StarBrite.

Two concepts might explain the difference in brightness attained after two bleaching treatments, which was a Δ 4.4 L* by Zekonis and others (2003) compared to Δ 1.0 L*, which was achieved in this study. The first concept is that the dehydration effect was included in the color evaluation data when the data was recorded 15 minutes after the bleaching treatment in the previously mentioned study. The second concept is that the latent peroxide from the at-home bleaching present in the patients' saliva may have augmented the bleaching effect from the in-office bleaching, because both bleaching treatments were carried out for the same period of time. Studies (Wattanapayungkul & others, 1999; Al-Qunaian, 2003) that investigated the degradation of carbamide peroxide during the first hour found that the total amount of

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Figure 7. *Clinical picture at baseline.*

Figure 8. *Clinical picture after the first in-office bleaching application.*

Figure 9. *Clinical picture after the second in-office bleaching application.*

hydrogen peroxide in patients' saliva was highest during the first hour. Wattanapayungkul and others (1999) measured the amount of hydrogen peroxide in patients' saliva when they used 10% carbamide peroxide. Wattanapayungkul and others (1999) found that there was a mean of 2.14 mg of carbamide peroxide in saliva collected up to one hour of wearing the bleaching tray. Dahl and Becher (1995) have shown that a safe amount of ingesting carbamide peroxide is 10 mg with a safety factor of 100.

The labeled hydrogen peroxide concentration for the products is 35% for StarBrite and 38% for Opalescence Xtra Boost. The authors determined by chemical analysis the actual concentration of the products they tested. Matis (2000, 2003) has shown that the labeled concentration is not always the actual concentration. Tests were accomplished in triplicate and showed that the mean hydrogen peroxide percent for StarBrite was 31.5% and 35.8% for Opalescence Xtra Boost.

In this study, color relapse (darkening) began after the bleaching treatments were finished and continued until the fifth week, after which there was no significant change in ΔL^*, Δa^*, Δb^* and ΔE for either product. This relapse pattern agrees with Zekonis and others (2003). This means that two weeks are needed for the color to stabilize after the bleaching treatments have been completed. Therefore, practitioners are recommended to wait at least two weeks post-bleaching when making a good color match, if they are planning to place a tooth-colored restorative material in the anterior teeth.

Subjective shade guide matching was performed using the Trubyte Bioform Color Ordered Shade Guide. At week two, Δ shade guide reached the peak of –4.87 for Opalescence Xtra Boost and -5.07 for StarBrite after two bleaching applications. It is interesting to note that the shade guide rank difference was –3.1 for Opalescence Xtra Boost and –3.4 for StarBrite at the end of the study, yet the ΔL^* values were slightly negative from baseline. This can be accounted for by the fact that there was a decrease in b* (about 1.00), which represented a decrease in yellowness. This decrease in yellowness was probably the reason for the lower shade guide values.

Using the shade guide, the color stabilized by the fifth week ($p>0.05$) at a level significantly different from the baseline for Opalescence Xtra Boost ($p=0.0001$) and StarBrite ($p=0.0001$). It is difficult to compare between studies in subjective data, because disagreement between dentists in shade matching the same tooth has been documented by Culpepper (1970), but the same relapse pattern and color stabilization was also found by Zekonis and others (2003).

Two independent evaluators performed the subjective slide evaluation. The Kappa (k) inter-evaluator reliability was determined to be 0.66, with 98% agreement between the two evaluators. A trend of Opalescence Xtra Boost's side being lighter was found using the subjective slide evaluation, which diminished after the bleaching treatments were finished. Three participants (15%) were assessed having the Opalescence Xtra Boost's side lighter at week one, and one participant (5%) was assessed having the Opalescence Xtra Boost's side lighter at week 11, but it was not statistically sig-

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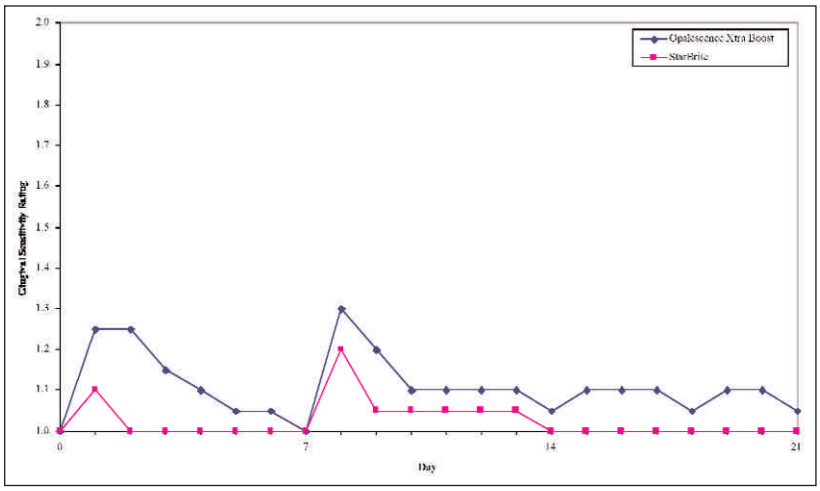

Figure 10. *Gingival irritation for Opalescence Xtra Boost and StarBrite.*

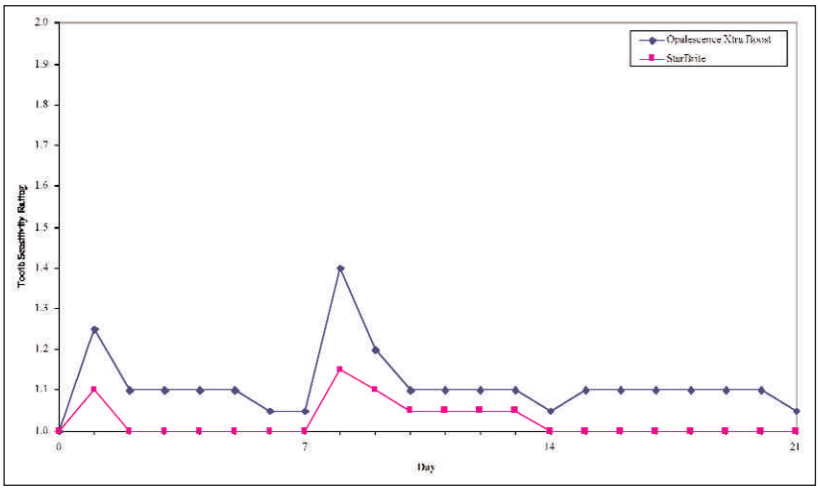

Figure 11. *Tooth sensitivity for Opalescence Xtra Boost and StarBrite.*

nificant. Opalescence Xtra Boost and StarBrite did not have significantly different patient-assessed lightness at any follow-up examination (p=0.25 at one week; p=1.00 at all other examinations).

All participants started with no gingival irritation or tooth sensitivity before any bleaching treatment. Some participants reported slight gingival irritation and tooth sensitivity by both products. However, it took only two days for the gingival irritation and tooth sensitivity to return to the pretreatment level. This pattern could be explained by the acute exposure to high concentration of hydrogen peroxide followed by a week of no bleaching treatment, which allowed the sensitivity to abate.

CONCLUSIONS

The results of this study showed no statistical difference between the two in-office bleaching products during active treatment periods or at any of the follow-up visits according to all three-color evaluation methods. Color relapse began after the bleaching treatments were finished and continued until the fifth week, after which there were no significant changes. Also, there was no statistical difference between products regarding gingival irritation and tooth sensitivity. Within the bounds of this study, the peroxide composition is the most important component of the bleaching materials. Other formula components are much less significant.

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The Clinical Effect of Amorphous Calcium Phosphate (ACP) on Root Surface Hypersensitivity

S Geiger • S Matalon • J Blasbalg
MS Tung • FC Eichmiller

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Clinical Relevance

Sequential applications of solutions that form amorphous calcium phosphates are effective for rapidly decreasing dentin hypersensitivity.

SUMMARY

Dentin hypersensitivity is a transient condition that often resolves with the natural sclerotic obturation of dentin tubules. A method of rapidly forming calcium phosphate compounds within these tubules can mimic sclerosis and lead to rapid reduction in hypersensitivity. Amorphous calcium phosphates (ACP) can be formed *in situ* by the sequential application of calcium and phosphate solutions. In this clinical study, 30 patients with reported dentin hypersensitivity were randomly assigned to parallel treatment or placebo groups. In the experimental treatment group, ACP was formed by topical application of a 1.5 mol/L aqueous solution of $CaCl_2$ followed by topical application of 1.0 mol/L aqueous K_3PO_4. The placebo group was treated with a topical application of 1.0 mol/L aqueous solution of KCl followed by topical application of distilled water. Treatments were repeated at the 7-day and 28-day recall appointments. Response to air and tactile stimuli were measured immediately before treatment using a visual analog scale initially on day 1, then on days 7, 28 and 180. The results showed that both the experimental and placebo treatments resulted in a reduction in hypersensitivity at 180 days. However, the ACP treatment

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group showed a much more rapid reduction in hypersensitivity over time. The change in sensitivity was much more apparent using the air stimulus than the tactile stimulus. These results show that topical placement of ACP can rapidly reduce dentin hypersensitivity.

INTRODUCTION

The clinical condition of dentin hypersensitivity is a significant problem in the adult population. Gingival recessions, root surface debridement or periodontal surgical procedures often cause the exposure of orifices of the dentin tubules at the root surface. The origin of dentin sensitivity has been described (Brännström, Linden & Aström, 1967; Pashley, 1990) as being due to mechanical, osmotic or thermal stimuli that can communicate with pulpal sensory nerves through these open tubules. The current approach to treating this condition is to obturate the dentinal tubules using a variety of agents, such as fluoride varnishes (Lukomsky, 1941; Lutins, Greco & McFall, 1984), Oxalate salts (Pashley & Galloway, 1985), and resinous agents (Pashley, & others, 1988; Trowbridge & Silver, 1990). The treatments mentioned above generally result in only partial or short-term relief of the dentin hypersensitivity (Morris, Davis & Richardson, 1999). Many treatments are not compatible with ongoing dental treatment, such as periodontal flap surgery.

Calcium phosphate minerals are the main inorganic constituents of dentin, and mineral rich deposits obstruct tubule orifices in the physiological process of dentin sclerosis (Frank, 1968). A method of mimicking this natural process may provide an effective clinical treatment for dentin hypersensitivity. Of the available calcium phosphate compounds, amorphous calcium phosphate (ACP) has the highest rate of formation and dissolution under physiologic oral conditions. This compound is also capable of rapid conversion into hydroxyapatite crystals under these same conditions, which can precipitate in the lumen of open dentinal tubules (Tung & others, 1992; Tung & others, 1993; Tung, O'Farrell & Liu, 1993). Previous research by Tung and others (1997) established the concentrations and mode of application for clinically applying ACP as a topical desensitizer, and preliminary clinical investigations indicated that this might provide an efficient treatment for dentin hypersensitivity. This study clinically assessed the effect of topical application of ACP forming calcium phosphate solutions on dentin hypersensitivity.

METHODS AND MATERIALS

Thirty adult patients exhibiting clinical symptoms of dentin hypersensitivity participated in this study. The clinical history of all the candidates was documented, and those suffering any illness or allergies or those receiving any kind of medication were excluded. Subjects participating in the study presented a minimum of two sensitive teeth in a minimum of two different quadrants. All subjects exhibited good dental care and had their last oral hygiene treatment within the last six months. Patients were randomly assigned to a treatment or a control group in a parallel design. This design was chosen to prevent crossover from the experimental treatment to the control teeth. No hazards or deleterious side effects were observed in previous studies of this material and none were anticipated in this study. The Helsinki Committee of Tel-Aviv University approved the study. Each subject signed a consent form after a full explanation of the treatment procedure and the known risks and effects.

Materials

Amorphous Calcium Phosphate is precipitated under oral physiological conditions by the sequential application of calcium chloride solution followed by a potassium phosphate solution (Tung & others, 1993). For the experimental group patients, two solutions were sequentially applied to the root surface: Solution A containing an aqueous solution of 1.5 mol/L calcium chloride, and Solution B containing an aqueous solution of 1.0 mol/L tripotassium phosphate. In the control group, a placebo material composed of two solutions was also sequentially applied: Solution PA containing 1.5 mol/L KCL, and Solution PB containing distilled water. For both the experimental and control treatments, the two solutions were applied successively over the root surfaces of each quadrant using a cotton pellet saturated with the solution and rubbing the material on the sensitive tooth surface for five seconds. Cotton rolls were used to isolate the treatment area. After rubbing, the solutions stayed undisturbed on the tooth surface for 60 seconds. The patients were then asked not to rinse, eat or drink for 30 minutes after treatment. The treatment was repeated at 7- and 28-day recalls, for a total of three treatments. Both the ACP and placebo solutions were coded and were unknown to the patient or the examiner during the experiment.

Sensitivity Test

The level of sensitivity was evaluated at the initial visit and at recalls after one week (seven days), four weeks (28 days) and six months (180 days). At the 7- and 28-day recalls, sensitivity measurements were made prior to re-treatment. No treatment was delivered at the 180-day recall. The patient was asked to define the degree of sensitivity using a visual analog scale by placing a vertical mark on a 100 mm line where 0 was "no pain" and 100 was "severe pain." The location of the mark was measured in mm from the line of origin and the results were coded and recorded for further statistical analysis. Tactile sensitivity was evaluated by measuring the response to the contact of an explorer over the cervical area of each tooth. Air sensitivity was exam-

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ined using the dental unit triple syringe and blowing a short blast of room temperature air over each root surface. Each tooth was isolated during measurement by placing cotton rolls over the adjacent teeth.

Statistical Analysis

The results were analyzed using two-way Analysis of Variance (ANOVA) with repeated measures of time and treatment as primary variables. The significance level used was ≤ 0.05 one tailed. Standard Deviations (SD) were calculated for all analyses. The statistical analysis was performed using the SPSS program.

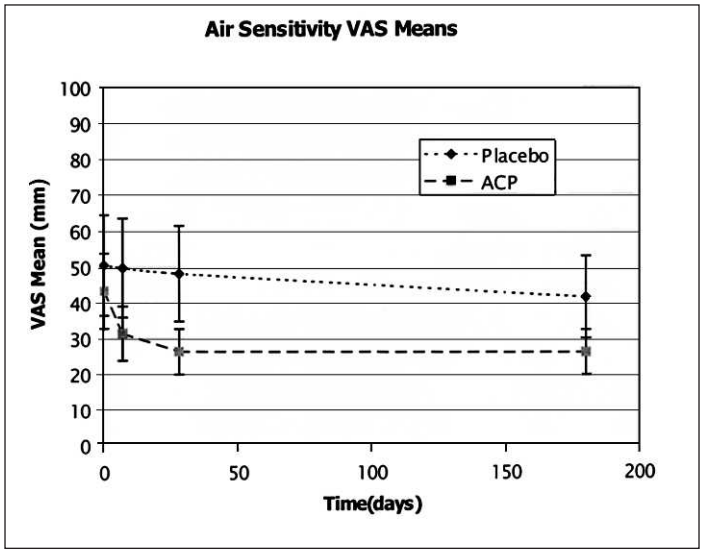

Figure 1. *The visual analog means and standard deviations for air sensitivity measurements made at 1, 7, 28 and 180 days. The standard deviation is given as the estimated standard uncertainty of the mean for these measurements.*

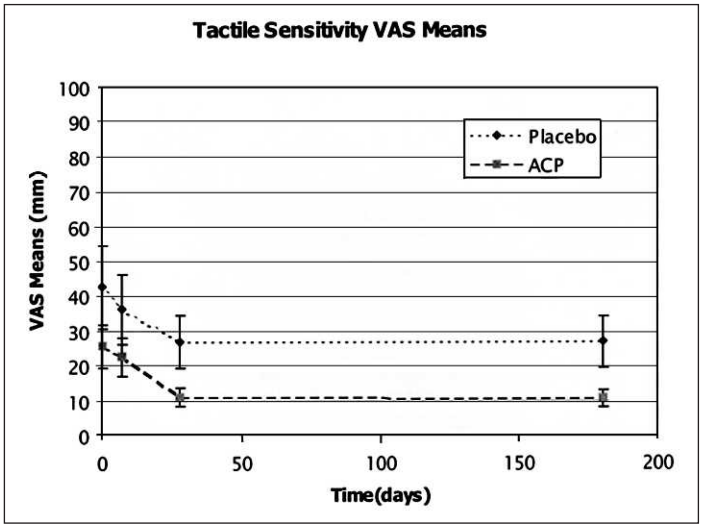

Figure 2. *The visual analog means and standard deviations for tactile sensitivity measurements made at 1, 7, 28 and 180 days. The standard deviation is given as the estimated standard uncertainty of the mean for these measurements.*

RESULTS

Chi-square analyses were conducted to determine whether significant correlations could be found between treatments and such factors as gender; periodontal condition, rated as healthy, mild inflammation or severe inflammation; quality of oral hygiene rated as good, fair or poor; toothbrush type rated as soft, medium and hard; whether a mouthwash was used; whether previous periodontal surgery was done; whether a six month recall had been completed; and whether they were smokers. No significant correlations were found, indicating that the patients were fairly balanced over the study groups.

Applying ACP to the root surfaces resulted in a rapid decrease in air sensitivity. Within seven days after treatment, air sensitivity was reduced from the initial mean value of 43.2 to a mean value of 31.4. Examination of these same patients three weeks later revealed a further decrease in sensitivity, reaching values of 26.3 and remaining at about the same level at the fourth examination after 180 days (Figure 1). The control treatment, where no significant change was noted from 50.4 to 49.7 after seven days, to 48.1 after 28 days and to 41.3 after 180 days (Figure 1), was also noted. The Analysis of Variance on the transformed data for air response showed that time was a significant factor ($p<0.004$) with sensitivity score dropping over time. Treatment was also significant ($p=0.041$), but no significant interaction was present between time and treatment ($p=0.230$).

Examination of tactile sensitivity (Figure 2) revealed a slower reduction in sensitivity after applying ACP from 25.7 to 22.6 after seven days, to 10.9 after 28 days, with no additional reduction (10.4) after 180 days. The control group showed a nearly parallel reduction, but with a higher sensitivity starting value at the initial measurement (Figure 2). The Analysis of Variance for tactile sensitivity showed, again, that time was a significant factor ($p<0.001$), with sensitivity being reduced with time. Treatment method was also significant ($p=0.041$), and there was no significant interaction between time and treatment ($p=0.984$).

DISCUSSION

Dentin hypersensitivity is a clinical situation characterized by a short, sharp pain initiated by many types of stimulation, such as tactile, thermal, osmotic or chemical (Pashley, 1990). According to the hydrodynamic theory of Brännström, Linden and Aström (1967), these stimulations, applied at the exposed dentin surface, cause an inward pressure on the tubular fluid, exciting the nerve endings within the pulp. Most current strategies for treating dentin hypersensitivity are by occlusion of tubular orifices at the tooth surface. In order to create an efficient, long lasting seal,

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the material used to occlude this orifice should penetrate the tubule, be chemically stable and adhere to the intratubular dentin. Combe and Douglas (1998) proposed that developing a biomimetic material that is structurally similar to the dental hard tissue could possibly provide a solution for treating dentin hypersensitivity. Tung and others (1997) has postulated that since calcium phosphate is the main inorganic component of the tooth, calcium phosphate minerals are ideal candidates for the obstruction of dentinal tubules. Tung and others (1992) have also shown that calcium phosphate solutions at high concentrations and at pH=9.5 rapidly precipitate amorphous calcium phosphate that obstructs the dentinal tubules and decreases dentin permeability by 85% or more.

In this study, the effectiveness of ACP was evaluated clinically as a possible therapeutic agent for the treatment of dentin hypersensitivity. The results demonstrated an immediate relief of sensitivity tactile and air stimulation after topical application of ACP. The alleviation was noted after 7 and 28 days, with further reduction in sensitivity after each repeated treatment. Beyond 28 days, the effect leveled off, and at 180 days, the difference between ACP and the placebo treatment was less notable. A possible explanation for this phenomenon might be that a spontaneous reduction in sensitivity occurs, probably due to minerals from salivary origin obstructing the tubular orifices. Alleviating sensitivity to air stimulation was more striking than alleviating sensitivity to tactile stimulation. A possible hypothesis that might explain this phenomenon is that mineral plugs of calcium phosphate that obstruct the tubular orifices at the dentin surface are not stable at the initial phase. Immediate contact of the explorer at the surface causes a pressure that is exerted on the tubular fluid, stimulating nerve endings at the pulp and causing the pain sensation. It is also difficult to precisely stimulate the same area of the tooth from one examination to another, leading to greater variation in results and the inability to discern differences in treatments. Tung and others (1997) also reported greater variability in tactile response in a prior clinical study.

The results of this study indicate that ACP may be used clinically for the fast relief of dentin hypersensitivity. The mode of application was found to be easy, and may be feasible even for auxiliary staff. No prior surface conditioning was used other than normal brushing and flossing. The solutions were easily placed using cotton tip applicators and isolation with cotton rolls was maintained during treatment. The precipitation of calcium phosphates provides a very natural and biocompatible treatment that does not require surface conditioning or the application of resins or polymers. This may make the treatment much more compatible with periodontal procedures such as deep scaling and root planing or surgical flap procedures.

CONCLUSIONS

The results of this clinical study showed that sequential application of calcium and phosphate-containing solutions that can form amorphous calcium phosphate *in situ* will more rapidly and more effectively reduce dentin hypersensitivity than a placebo treatment.

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Certain commercial materials and equipment are identified in this paper to specify the experimental procedure. In no instance does such identification imply recognition or endorsement by the National Institute of Standards and Technology, the Hebrew University School of Dental Medicine or the ADA Health Foundation or that the material or equipment identified is necessarily the best available for the purpose.

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Laboratory Research

Fracture Resistance of Teeth with Class II Bonded Amalgam and New Tooth-Colored Restorations

J Görücü • G Özgünaltay

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Clinical Relevance

Fracture resistance of teeth weakened with MOD preparations was only obtained with hybrid composite, packable composite and ormocer restorations, and it was determined to be similar to intact, unprepared teeth when compared to bonded amalgam restorations.

SUMMARY

This study compared the cuspal fracture resistance of posterior teeth restored with four different adhesive restorations. Fifty sound, maxillary human premolars were randomly divided into a control group and four experimental groups with 10 teeth in each. Specimens in the first group were intact teeth that were tested as unprepared. The remaining four groups received mesio-oclusodistal cavity preparations and were restored with a hybrid composite (Filtek Z250), a packable composite (Filtek P60), an ormocer (Definite) and an amalgam (SDI Permite) with an amalgam bonding agent (Amalgam Bond Plus). All groups were stored in water at 37°C for 15 days and thermocycled 1000 times between 5°-55°C. The specimens were preloaded five times in compression to 10 kg using two metal rods that contacted only the teeth on the cuspal inclines. The teeth were then loaded occlusally in an Instron Universal Testing Machine until fracture occurred. The means of force required to fracture the teeth in each of the five groups was analyzed using one-way ANOVA and Tukey Test.

The difference between the mean cuspal fracture resistance of the unprepared control teeth and those restored with amalgam groups was found to be statistically significant ($p<0.05$). No significant differences in resistance to cuspal fracture were found among the restoration groups, the unprepared control group and those teeth restored with hybrid composite, packable composite and ormocer groups ($p>0.05$).

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INTRODUCTION

Cusp fracture often occurs in teeth with restorations that have more than one-third of the intercuspal distance (Mondelli & others, 1980; Cavel, Kelsey & Blankenau, 1985; Larson, Douglas & Geistfeld, 1981). Amalgam used as an intracoronal restoration that lacks adhesion to tooth structures provides no significant change in the fracture resistance of the cusps when compared to prepared, unrestored premolars (McCullock & Smith, 1986; Boyer & Roth, 1994). The use of adhesive resins with amalgam was first reported in 1983 (Stevenson, 1983; Zardiackas & Stoner, 1983). Eakle, Staninec and Lacy (1992) reported increased fracture resistance of teeth restored with bonded amalgam compared with teeth restored with non-bonded

amalgam. However, other studies reported conflicting results (Santos & Meiers, 1994; Bonilla & White, 1996; Dias De Souza & others 2001).

The use of adhesive materials to reinforce weakened teeth was first proposed by Denehy and Torney in 1976. Using dentin bonding agents in conjuction with acid etching of enamel further enhanced the strengthening effects of these restorations (Jagadish & Yogesh, 1990). However, the use of traditional direct composites as a substitute for amalgam in restorating posterior teeth has been problematic (Leinfelder, 1988). Stress-bearing posterior areas have poor wear resistance when compared with amalgam (Leinfelder, Barkmeier & Goldberg, 1983) polymerization shrinkage that leads to poor marginal integrity (Roulet, 1988; Yap & others, 2000) and difficulty in handling (Rukmo & Wilson, 1985). Traditional composites offer no resistance to placement forces in their unpolymerized state and they tend to be "sticky," resulting in a tendency to pull away from the cavity wall when a placement instrument is withdrawn.

Recently, several manufacturers have introduced "packable" composites to the marketplace as alternatives to amalgam (Leinfelder, Radz & Nash, 1998). They are mainly characterized as having less stickiness or higher viscosity compared to traditional composites. Therefore, "packable" composites are appropriate for stress-bearing posterior restorations with improved handling properties. An application technique similar to the manipulation of amalgam can be used for the placement. These materials were expected to exhibit superior physical and mechanical properties in addition to showing improvements in handling.

Ormocer, another tooth-colored restorative material, has arisen from the progressive development of materials. Multi-functional urethane and thioether (meth) acrylate alkoxysilanes as sol-gel precursors have been developed to synthesize inorganic-organic co-polymer ormocer composites as the dental restorative materials (Hickel & others, 1998; Manhart & others, 1999). The alkoxysilyl groups of the silane allow for the formation of an inorganic Si-O-Si network by hydrolysis and polycondensation reactions and the (meth) acrylate groups are available for photochemically induced organic polymerization (Manhart & others, 1999). After incorporating the filler particles, dentists prefer a hybrid composite that can manipulate the ormocer. Ormocers are characterized as having novel, inorganic-organic copolymers in their formulation that allow for a wide range of modification of mechanical parameters.

Currently, there is no report in the literature that compares the effects of using hybrid composite, packable composite, ormocer and bonded amalgam restorations to reinforce weakened teeth.

This study determined whether using a hybrid composite resin, a packable composite resin, an ormocer and a bonded amalgam restoration could increase the fracture resistance of posterior teeth with MOD cavity preparations.

METHODS AND MATERIALS

Intact, non-carious, recently extracted human maxillary first premolars were cleaned and stored in 10% formalin solution (pH=7.0) at 20°C. All teeth were inspected under light microscopy and transilluminating fiber-optic light to detect the presence of cracks. Those with apparent cracks were excluded from the study. Teeth of similar crown size were selected for the study. Fifty sound, maxillary premolars were selected and included.

At no stage in the investigation were the teeth allowed to dehydrate. The roots of the teeth were embedded in acrylic resin to 2 mm below the cementum enamel junction (approximately the level of the alveolar bone in a healthy tooth), with the cusp tips aligned in the same plane to ensure a more equal distribution of the load during testing. The teeth were randomly divided into five groups. One group served as the intact control (Group 1) and the remaining four groups were prepared and restored in the following manner.

Tooth Preparation

Standardized MOD cavity preparations were made in each tooth using a diamond bur (835-010-4 ML, Diatech Dental AG CH 9435 Heerbrugg, Switzerland) in a high-speed handpiece with air/water spray. A new bur was employed for each five teeth. The depth of the occlusal isthmus was 2/3 the mean height of the cusps above the cementoenamel junction and the cavity width was 1/3 the intercuspal distance. The buccolingual width of the

Table 1: *Products Used in This Study*

Group	Product		Manufacturer
2	Hybrid Composite Bonding Agent	Filtek Z 250 Single Bond	3M Dental Products St Paul, MN, USA
3	Packable Composite Bonding Agent	Filtek P 60 Single Bond	3M Dental Products St Paul, MN, USA
4	Ormocer Bonding Agent	Definite Etch&Prime 3.0	Degussa AG Geschäftsbereich Dental, D, 63403 Hanau, Germany
5	Amalgam	SDI Permite	Southern Dental Industries Limited Bayswater Victoria, 3153 Australia
	Bonding Agent	Amalgam Bond Plus	Parkell, Farmingdale, NY, USA

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approximal boxes was 1/3 of the total buccolingual width of the tooth. The floor of the approximal boxes was maintained at 1 mm above the cementoenamel junction and the gingival seat width was 1.5 mm wide. All internal line angles were rounded, and no additional mechanical retention was cut.

Tooth Restorations

Table 1 lists the restorative materials tested. The exact techniques utilized for each group are:

Group I: Unprepared, intact teeth (Control).

Group II: After cavity preparation, enamel and dentin were etched with 37% phosphoric acid gel for 15 seconds. The preparation was rinsed with water spray, followed by slightly drying the cavities. According to the manufacturers' instructions, Single Bond was applied with a brush to the enamel and dentin walls and left to diffuse through the conditioned substrate for 20 seconds. After 20 seconds, the teeth were dried and a second coat of single component was applied and immediatelly dried, then light cured for 20 seconds. Each tooth was restored immediatelly after following adhesive application with Filtek Z250. A metal matrix band was placed in a Tofflemire matrix retainer (Union Brocach Corp, Long Island City, NY, USA) and positioned on the tooth. The composites were then placed incrementally. The proximal boxes were filled in three layers and the occlusal boxes in two. Each layer was polymerized for 20 seconds using a visible light-curing unit (Hilux Expert, Benlioglu Dental, Ankara, Turkey) with a light intensity of 500 W/cm^2. Care was taken to keep the depth of the increments to no more than 1.5 mm to ensure adequate curing. Then, the matrix band was removed. To ensure that the deepest parts of the interproximal box had been cured adequately, each restoration was cured for an additional 40 seconds from the buccal aspect and 40 seconds from the lingual aspect of the box. Then, the restoration surface was finished and polished sequentially with a finishing diamond, gray politip finisher silicon rubber and green politip-polisher silicon rubber (Vivadent-Liechtenstein) in a low-speed handpiece.

Group III: The same procedures were followed as the hybrid composite (Filtek Z 250), except that Filtek P 60 was packed into the cavity according to the manufacturer's recommendations.

Group IV: Etch and Prime 3.0 was used. One drop of universal and one drop of catalyst were mixed and applied with a brush to the enamel and dentin walls for 30 seconds. The teeth were dried and light cured for 10 seconds. The same procedure was repeated twice more. Definite was placed and cured in the same way as the hybrid composite (Filtek Z 250), except that each layer of Definite was polymerized for 40 seconds.

Table 2: *Mean Force Required to Fracture Teeth in Each Group (n=10)*

GROUP	Mean (Kg)	SD	Range (Kg)
Intact Teeth (Unprepared)	158.4[a*]	30.6	115.9-198.5
Restored with hybrid composite	127.1[ab*]	30.5	89.4-175.3
Restored with packable composite	130.6[ab*]	28.2	95.5-173.2
Restored with ormocer	124.7[ab*]	27.5	82.7-166.1
Restored with amalgam with amalgam bond	110.3[b*]	23.9	69.8-135.4

*Means with same letter (a or b) are not statistically different at $p<0.05$.

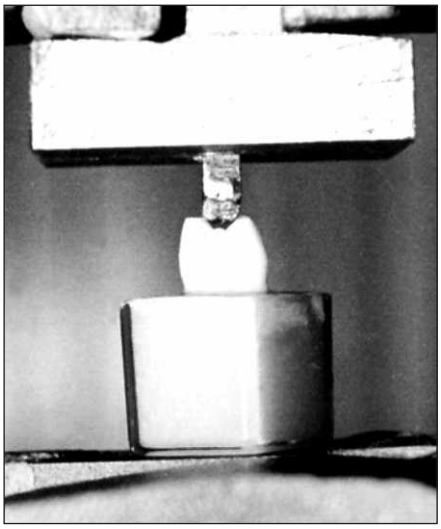

Figure 1. *Compressive loading using steel rods, testing head contacted inclines of cusps but no contact restoration.*

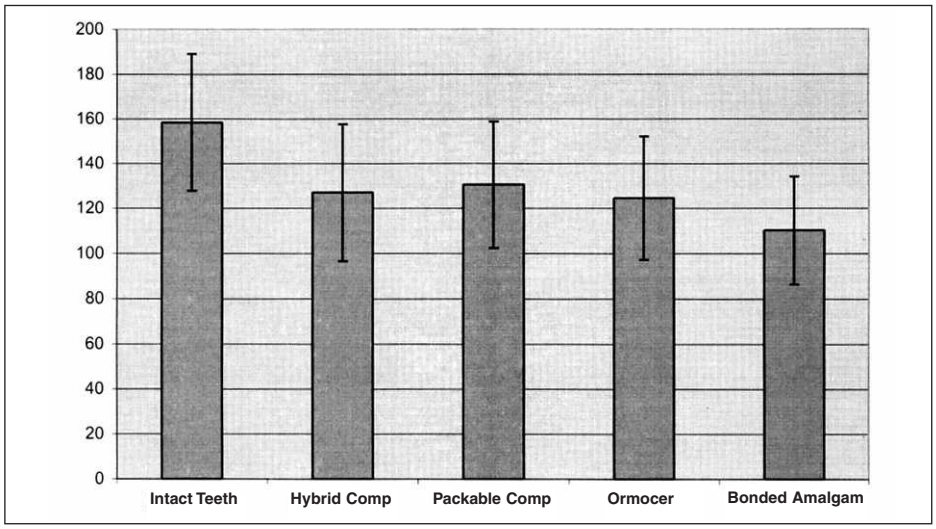

Figure 2. *Illustrative graphic of mean and standard deviations for the fracture resistance test.*

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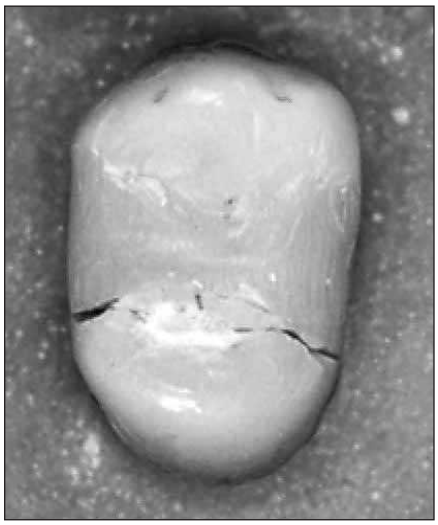

Figure 3. *Fracture at the interfacial area between the tooth and composite restoration. Occsionally, a small fragment of resin composite was found attached to the tooth.*

Figure 4. *Fracture at the interfacial area between the tooth and amalgam restoration.*

Figure 5. *Bulk fracture of restorative materials.*

Figure 6. *Fracture involving only tooth structure.*

Figure 7. *Fracture involving the tooth structure and restoration.*

Group V: Dentin activator was dispensed into a mixing well, applied to the enamel and dentin walls for 30 seconds and rinsed and air dried. Three drops of base, one drop of catalyst and one scoop of HPA (High Performance Additive) were dispensed into a mixing well and gently stirred. The mixture was applied in a thin layer to the cavity walls. Each tooth was restored immediatelly following adhesive application with a spherical lathe-cut amalgam A metal matrix band was placed in the same way as the other restorations. The amalgam was triturated. The plastic amalgam was hand condensed into the cavity while the adhesive liner was still wet. The matrix band was removed and the restoration surface was carved and finished to the orginal margins and anatomy.

The same investigator performed all restorative procedures throughout the study.

Testing

The restored teeth were stored in water at 37°C for 15 days prior to testing. During storage, the teeth were subjected to 1,000 thermocycles between 5° and 55°C with a dwell time of 30 seconds.

All specimens were tested on the Instron Universal Testing Machine (Model 4301, Instron Corp, Attleboro, MA, USA) for resistance to fracture. The instrument developed by Blaser and others (1983) and Joynt and others (1987) appears to provide precision in loading the specimen and, with modification, it was used as a model for this investigation. Two metal rods, each approximately 2 mm in diameter and having contact with the specimens parallel to the occlusal surface were used. A bidirectional joint was also added to the shaft between the crosshead and the metal rods to allow for appropriate adjustment to the various occlusal surfaces. The use of two rods instead of one assured contact with tooth structure alone without loading the restorative material during the test procedure (Figure 1). The specimens were placed on a foam cushion on the Instron platen, which permitted even seating of the load. The teeth were preloaded to a maximum force of 10 kgf at a speed of 5-mm/minute five times before testing to failure. The preloading procedure was introduced to simulate intraoral forces and improve uniform loading of the teeth (Oliveira, Cochran & Moore, 1996).

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The teeth were then tested to failure at a speed of 5 mm/minute and the force recorded. The data were analyzed using a one-way analysis of variance (ANOVA) and a Tukey test. Fractured tooth and restoration surfaces were viewed under stereomicroscopy.

RESULTS

The mean force required to produce fracture of the teeth in each group and standard deviation are presented in Table 2 and Figure 2. The one-way analysis of variance indicated a significant difference among the means of five groups (f=3.840, p=0.009). The Tukey multiple comparison tests showed that there were no statistically significant differences among the means of the unprepared group and the group restored with hybrid composite, packable composite and ormocer. Also, no statistically significant differences were found among the restoration groups (p>0.05). However, statistically significant differences were noted between the restored group with amalgam and the unrestored control group (p<0.05) (Table 2).

Fractures of restored teeth with hybrid and packable composites often occurred at the interfacial area between the tooth and restoration (Figure 3), not in the bulk of the resin composite. Occasionally, small fragments of resin composite were found attached to the tooth (Figure 3). Fractures at the interfacial area between the tooth and the restorations were found in a small number of ormocer and amalgam specimens (Figure 4) compared to hybrid and packable composite specimens. The amalgam and ormocer groups' demonstrated cohesive failure in restoration materials associated with tooth crack (Figure 5), although in the other groups, fractures did not occur through the bulk of the materials. Fewer fractures were observed in all restored groups that involved only the tooth structure (Figure 6) and also involved tooth structure and restoration (Figure 7).

DISCUSSION

Conventional amalgam restorations do not provide cusp support (McCullock & Smith, 1986; Boyer & Roth, 1994). Static loading of the amalgam restoration, irreversible deformation and crack propagation risk on the remaining cusps has been shown (Assif, Marshak & Pilo, 1990). Several *in vitro* studies have reported that teeth restored with amalgam bonded to etched enamel with resin cement have a significantly greater fracture resistance than teeth conventionally restored with amalgam (Eakle & others, 1992; Staninec & Holt, 1988; Eakle & others, 1994; Dias De Souza & others, 2001). In this study, intact teeth had a significantly greater fracture resistance than teeth restored with amalgam using Amalgam Bond Plus. These results are similar to those of Oliveria, Cochran and Moore (1996), who evaluated the effects of bonded amalgam on the fracture strength of teeth using five adhesive systems. In their study, Amalgam Bond Plus, All-Bond 2 Primer/Liner F and varnish control resulted in strengths significantly lower than the unprepared group.

Most studies have demonstrated a significant decrease in resistance to cuspal fracture of teeth prepared involving more than one-third of the intercuspal distance (Joynt & others, 1987; Çötert, Sen & Balkan, 2001; Mondelli & others, 1980).

The cuspal reinforcing effect of bonded resin composite restorations has generally been accepted. This suggests that in teeth that are etched, bonded and restored with resin composite, the cusps are mechanically splinted together. The reaction of these teeth to a load was similar to the reaction of unprepared teeth. In this study, the means of the fracture resistance for teeth restored with hybrid composite, packable composite and ormocer materials were not statistically significant compared to the unprepared control group. The results of this study confirm the results of previous studies that showed an increase in the fracture resistance of teeth restored with bonded composite resins (Eakle, 1986a; McCullock & Smith, 1986).

In this study, the Tukey multiple comparison tests revealed no significant differences between the restoration groups (p>0.05). The amalgam group showed lower cuspal fracture resistance compared to all the other groups. However, this difference was not statistically significant. Also, the unprepared group showed higher cuspal fracture resistance than the teeth restored with composites (hybrid, packable) and ormocer, but the differences showed no significance (p>0.05).

In this study, the teeth weakened with MOD preparation were restored with hybrid composite, packable composite, ormocer and bonded amalgam. The data showed that the fracture resistance of teeth restored with etched and bonded composites (hybrid, packable) and ormocer restorations were found to be similar to intact, unprepared teeth when compared to amalgam. Composites have a lower elastic modulus than amalgam; therefore, more load is absorbed within the composite than the amalgam. Composites, therefore, may transmit less of the applied load to the underlying tooth structure.

Wieczkowski and others (1988) evaluated structurally weakened teeth restored with resin composite and placed with a bulk and an incremental technique that may reduce the effects of polymerization shrinkage. They reported that posterior resin placed with an incremental technique produced greater resistance to cuspal fracture than posterior resin placed with a bulk technique. Since polymerization contraction stresses of the packable composite and ormocer materials were higher than that of hybrid composites (Chen & others, 2001), the hybrid composite restorations were expected to provide greater resistance to fracture of the remaining

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tooth structure. According to the results of this study, however, no significant differences were found among the hybrid composite, packable composite and ormocer materials.

Previous studies had determined that the strength of restored teeth was lower than that found in the current study (Joynt & others, 1987; Wieczkowski & others, 1988; Real & Mitchell, 1989). As bonding agents have progressed over the years, the strength values found in this study were higher than previous studies (Harada & others, 2000; Ogata & others, 2001).

Most fractures of teeth restored with hybrid and packable composites were shown to be at the interfacial area between the tooth and the restoration, not in the bulk of the resin composite. These findings of failure were similar to the fracture modes found in other studies (Eakle, 1986a,b); however, in this study, ormocer and amalgam were shown to fracture in cohesive.

Related to the results of this study, the cohesive failure of amalgam was similar to the results of a study by Oliveria, Cochran and Moore (1996). For ormocer and amalgam, the number of fractures seen in the tooth and the restoration were higher than the hybrid and packable composite restorations.

In view of the reinforcing ability of all the tooth-colored restorative materials (hybrid, packable, ormocer) studied, it is suggested that where cavity cusp width is one-third of the intercuspal distance, reinforcement of the weakened cusp should be considered a possible alternative for posterior amalgam and cast restorations to protect the remaining tooth. However, durability tests and long-term clinical trials are still required.

CONCLUSIONS

Cuspal fracture resistance of posterior teeth restored with various adhesive restorations were observed in this study. According to the results, the following conclusions were drawn:

1. A statistically significant difference was found in the resistance to cuspal fracture between the unprepared control groups and the group restored with bonded amalgam.
2. No significant differences were found in resistance to cuspal fracture among the restoration groups.
3. Differences among the unprepared teeth and those restored with hybrid composite, packable composite and ormocer were not statistically significant.

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Radiopacity of Direct Esthetic Restorative Materials

MD Turgut • N Attar • A Önen

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Clinical Relevance

Significant variations in radiopacity values were found among esthetic restorative materials when compared to enamel. All materials except for the microfilled resin composite Filtek A 110 had radiopacity values greater than dentin.

SUMMARY

This study determined the radiopacity of 21 commercially available direct esthetic restorative materials with reference to an aluminum step wedge and an equivalent thickness of enamel and dentin. A total of 168 samples measuring 6 mm in diameter and 1 mm in thickness, with eight samples of each material, were prepared from restorative materials. Enamel and dentin samples 1-mm thick were also prepared by longitudinally sectioning eight extracted human permanent molars using a microslicing machine. The optical densities of each restorative material, along with one tooth section and an aluminum step wedge were measured from radiographic images using a transmission photodensitometer. The optical density values of the specimens were used to determine the aluminum thickness equivalent values. The data were analyzed with one-way analysis of variance (ANOVA) and Duncan's multiple range tests. The results showed statistically significant differences among materials. Tetric Ceram had the greatest radiopacity value and was higher than enamel. All materials except for the microfilled resin composite Filtek A 110 had radiopacity values greater than dentin and possessed sufficient radiopacity to meet ISO 4049 standard. Significant differences were found among materials of the same composition when compared to enamel.

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INTRODUCTION

Tooth-colored restorative materials have increasingly been used as alternatives to amalgam. Up-to-date resin composites and glass ionomer cements may be the most refined materials, as new versions are routinely being developed. Since conventional glass ionomer cements have low mechanical properties and moisture sensitivity, hybrid versions, resin-modified glass ionomer cements and polyacid-modified resin composites have been developed to overcome these shortcomings (Toledano & others, 1999).

Resin composites have been widely used as esthetic materials because they are mercury-free, thermally non-conductive and resist corrosion (Boksman & others, 1986), with the progression of composites from macrofills to microfills and from hybrid to microhy-

brids and the development of new materials, flowable, condensable and ion-releasing composites have been introduced to the dental market (Estafan & others, 2000; Bayne & others 1998; Manhart, Chen & Hickel, 2001; Vivadent, 1998).

Research that focuses on increasing the mechanical properties of tooth-colored restorative materials has been conducted in order to improve clinical performance. Of these, radiopacity is a valuable property that allows the clinician to assess the adequacy of restorations, distinguish secondary caries and evaluate marginal adaptation, voids and interfacial gaps. In addition, adequate radiopacity permits detection of interproximal contours, contacts and overhangs and serves an important role in detecting aspirated or dislocated restorations (Cook, 1981; Abou-Tabl, Tidy & Combe, 1979; Chandler & others, 1970; Akerboom & others, 1993).

According to the International Standards Organization (ISO), the radiopacity of material should be equal to or greater than the same thickness of aluminum and shall be no less than 0.5 mm of any value claimed by the manufacturer (ISO Standard 4049, 2000).

In many studies, radiopacity has been evaluated by comparing specific sample thicknesses to aluminum step wedges under typical radiographic conditions. Radiopacity is usually expressed in terms of aluminum thickness (Cook, 1981; Abou-Tabl & others, 1979; Omer, Wilson & Watts, 1986).

Although many new esthetic restorative materials have been introduced to the dental market, there have been only a few recent studies regarding their radiopacity (Bouschlicher, Cobb & Boyer, 1999; Hara & others, 2001). This study, therefore, determined the radiopacity values of esthetic restorative materials and compared them to enamel and dentin.

METHODS AND MATERIALS

Table 1 lists the 21 esthetic restorative materials used in this study. Round, disc-shaped samples, eight samples per material, measuring 6 mm in diameter and 1 mm (+/- .01 mm) in thickness were prepared by using Teflon molds that were placed between two glass plates, clamped under pressure and light cured from

Table 1: *Restorative Materials Used in the Study*

Type	Commercial Name	Manufacturer	Batch #
Packable composite	SureFil	Dentsply-De Trey, GmbH D 78467 Konstanz, Germany	606.05.750
Packable composite	P-60 Filtek	3M, Dental Products, St Paul, MN, USA	70-2010-2553-6
Packable composite	Pyramid Dentin	BISCO Inc, 110 W Irving Park Rd, Schaumburg, IL, USA	9800001488
Packable composite	Pyramid Enamel	BISCO Inc, 110 W Irving Park Rd, Schaumburg, IL, USA	9800001491
Packable composite	Solitaire 2	Heraeus Kulzer D-41538 Dormagen, Germany	64715227
Flowable composite	Heliomolar Flow	Vivadent Ivoclar AG, FL-9494, Schaan, Liechtenstein	557032 BN
Flowable composite	Composan LCM Flow	Promedica, D-24537 Neumünster, Germany	5126781195
Hybrid composite	Tetric Ceram	Vivadent Ivoclar AG, FL-9494, Schaan, Liechtenstein	546307 AN
Hybrid composite	TPH Spectrum	Dentsply-De Trey, GmbH D 78467 Konstanz, Germany	0108293
Hybrid composite	Filtek Z-250	3M, Dental Products, St Paul, MN, USA	70-2010-2244-2
Hybrid composite	Composan LCM	Promedica, D-24537 Neumünster, Germany	CE 0482
Hybrid composite	Glacier	Southern Dental Industries Limited, Bayswater, Victoria 3153, Australia	010324
Microfilled composite	Heliomolar Radiopaque	Vivadent Ivoclar AG, FL-9494, Schaan, Liechtenstein	541503 AN
Microfilled composite	Filtek A-110	3M, Dental Products, St Paul, MN, USA	70-2010-3035-3
Ion-releasing composite	Ariston pHc	Vivadent Ivoclar AG, FL-9494, Schaan, Liechtenstein	A09636
Glass-ionomer cement	Chem Flex Syringeable	Dentsply-De Trey, GmbH D 78467 Konstanz, Germany	pow: 606.07.002 liq: 606.07.030
Glass-ionomer cement	Chem Flex Condensable	Dentsply-De Trey, GmbH D 78467 Konstanz, Germany	pow: 606.07.002 liq: 606.07.030
Resin-modified GIC	Vitremer	3M Dental Products, St Paul, MN, USA	Pow: 318 liq:33031
Polyacid-modified resin composite	Dyract AP	Dentsply-De Trey, GmbH D 78467 Konstanz, Germany	0005000764
Polyacid-modified resin composite	Compoglass F	Vivadent Ivoclar AG, FL-9494, Schaan, Liechtenstein	546975
Polyacid-modified resin composite	F2000	3M, Dental Products, St Paul, MN, USA	0930 AR

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both sides for 30 seconds with a light source (Hilux Dental Curing, Benlioglu Dental Inc, Ankara, Turkey) at 500 mW/cm². Chem Flex Syringeable and Condensable were chemically set and prepared according to the manufacturers' instructions using the scoops provided. All samples were polished using 180, 220, 320 and 400 grit sandpaper, then measured with a digital micrometer to verify that the thickness remained at the critical tolerance of 1± .01 mm. Enamel and dentin samples 1-mm thick were also prepared by longitudinally sectioning eight extracted permanent molars using a micro-slicing machine (Accutom, Struers Co, Copenhagen, Denmark). The samples and tooth sections were randomly divided into eight groups, each containing one sample of each material and one tooth section. Then, each group was positioned on an occlusal x-ray film, (Kodak Ultra-speed DF 50, Eastman Kodak Company, Rochester, NY, USA) with an aluminum step wedge (99.5% purity), which increases 1 mm in thickness per step and has a thickness range of 0.5 to 13.5 mm. This step wedge was used to enable accurate correlation of the optical density of the images of the specimens to that of the aluminum step wedge. Films were exposed for 0.37 seconds using a dental x-ray machine (Prostyle Intra Planmeca OY, SF-00810, Helsinki, Finland) at 70 kV and 10 mA with a focus object distance of 40 cm. The film was processed in a standard automatic processor (Dent X 9000 Film Processors, Elmsford, NY, USA) using fresh Super Defiks developer and fixer.

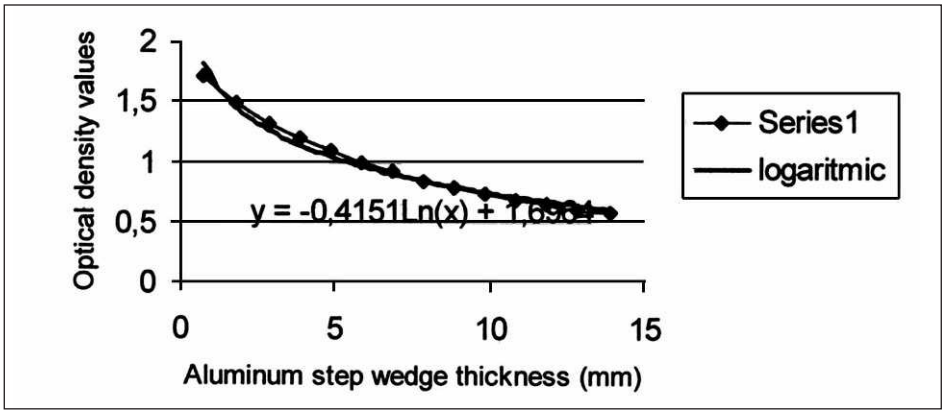

Figure 1. *Optical densities of aluminum step wedge.*

Table 2: *Radiopacity Test Results*

Material	Mean OD (Std Deviation)		Equivalent Al Thickness (mm) (Std Deviation)	
Tetric Ceram	1.212 (0.017)		3.390 (0.127)	
Dyract AP	1.266 (0.009)		2.994 (0.06)	
Chem Flex Condensable	1.329 (0.008)	a	2.596 (0.045)	
Compoglass F	1.343 (0.01)	ab	2.510 (0.054)	j
Ariston pHc	1.351 (0.013)	bc	2.469 (0.07)	jk
Chem Flex Syringeable	1.358 (0.015)	bcd	2.427 (0.083)	kl
TPH Spectrum	1.363 (0.008)	cde	2.403 (0.043)	klm
Filtek Z-250	1.371 (0.007)	de	2.360 (0.034)	lmn
F2000	1.375 (0.011)	e	2.337 (0.059)	mn
SureFil	1.379 (0.012)	e	2.316 (0.062)	n
P-60 Filtek	1.419 (0.019)	f	2.115 (0.09)	o
Heliomolar Flow	1.422 (0.015)	f	2.101 (0.072)	o
Heliomolar RO	1.437 (0.013)	g	2.023 (0.062)	p
Enamel	1.439 (0.031)	g	2.022 (0.143)	p
Vitremer	1.565 (0.017)	h	1.519 (0.059)	q
Composan LCM	1.566 (0.008)	h	1.515 (0.029)	qr
Glacier	1.569 (0.015)	h	1.504 (0.053)	qr
Pyramid Dentin	1.586 (0.009)	i	1.447 (0.031)	rs
Solitaire 2	1.590 (0.008)	i	1.433 (0.026)	s
Composan LCM Flow	1.633 (0.015)		1.299 (0.043)	
Pyramid Enamel	1.664 (0.004)		1.210 (0.01)	
Dentin	1.693 (0.021)		1.135 (0.055)	
Filtek A 110	1.834 (0.032)		0.824 (0.06)	

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

The optical density of the materials' images on the x-ray films was measured by means of a transmission densitometer (DT 1105, Ryparry Limited, England). A minimum of four readings was obtained from each radiograph for each material for each tooth. A graph was plotted between the entire step wedge thickness and its optical density values [y=-0.4151Ln(x) + 1.6964] (Figure 1). From this graph, the optical density values of the specimens were used to determine the aluminum thickness equivalent values. According to ISO standards, the value of the aluminum thickness equivalent or the radiopacity of the restorative resins was expected to be higher than the same thickness of aluminum (ISO Standard 4049, 2000).

One-way analysis of variance (ANOVA) and Duncan's multiple range tests were conducted to statistically analyze the differences in the optical density values and the aluminum thickness equivalent values of the materials.

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RESULTS

Figure 2 shows a radiograph of all tested materials, along with a tooth section and the aluminum step wedge. Table 2 records the optical density values and aluminum thickness equivalent values of all tested materials. One-way analysis of variance (ANOVA) indicated significant differences both for the optical density values and aluminum thickness equivalent values among the materials (p=0.0001).

Duncan's multiple range test showed Tetric Ceram to have a radiopacity value (3.390 mm Al) significantly greater than all other materials, whereas Filtek A110 had a radiopacity value (0.824 mm Al) significantly lower than the other materials (p<0.05).

Heliomolar Flow, P-60 Filtek, SureFil, F-2000, Filtek Z-250, TPH Spectrum, Chem Flex Syringeable, Ariston pHc, Compoglass F, Chem Flex Condensable, Dyract AP and Tetric Ceram were found to have radiopacity values (2.101, 2.115, 2.316, 2.337, 2.360, 2.404, 2.427, 2.469, 2.511, 2.596, 2.994, 3.390 mm Al, respectively) that exceeded enamel (2.022 mm Al).

Heliomolar RO had a radiopacity value (2.029 mm Al) that was not significantly different from enamel (p>0.05).

Vitremer, Composan LCM, Glacier, Pyramid Dentin, Solitaire 2, Composan LCM Flow, Pyramid Enamel and Filtek A110 had radiopacity values (1.519, 1.515, 1.504, 1.447, 1.443, 1.299, 1.210 and 0.824 mm Al, respectively) lower than enamel.

All materials except Filtek A110 (0.824 mm Al) had radiopacity values higher than dentin (1.135 mm Al).

Figure 3 shows the bar chart representation of optical densities and Figure 4 shows the aluminum thickness equivalents of all tested materials with enamel and dentin.

DISCUSSION

The radiopacity of a restorative material serves as a valuable diagnostic tool, especially when evaluating the long-term success of restorations. Adequate radiopacity has been considered a prerequisite for diagnosis of secondary caries and marginal defects since it facilitates evaluating the radiographic view of restorations (Tveit & Espelid, 1986; Prevost & others, 1990).

Secondary caries is reported to be one of the major problems with replacing esthetic restorations (Phillips & others, 1973). Marginal defects and secondary caries are usually located on the gingival part of Class II restorations. Therefore, the first increment must be sufficiently radiopaque to clearly evaluate the tooth-restoration interface (Bouschlicher & others, 1999). Restorative materials with less radiopacity than

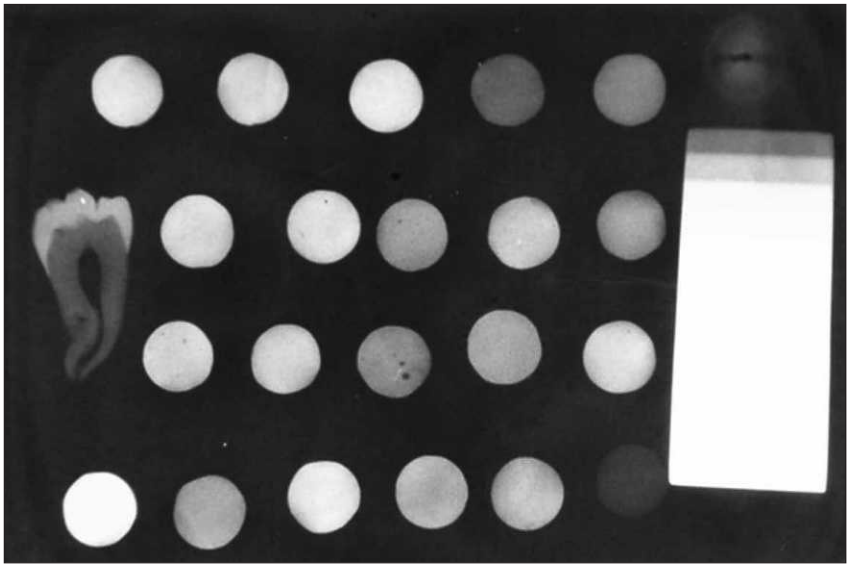

Figure 2. *A radiograph of all the tested materials along with a tooth section and the aluminum step wedge.*

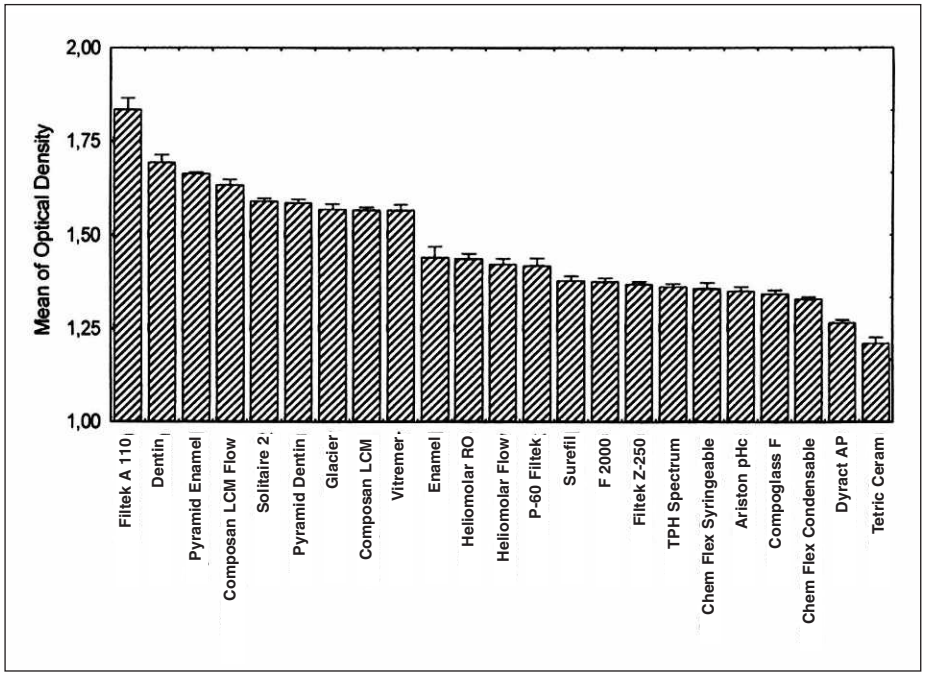

Figure 3. *Bar chart representation of optical density values of all materials.*

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dental hard tissues are inconvenient for this purpose (Tveit & Espelid, 1986). Whether a restorative material with a radiopacity greater than dentin complies with ISO standards, it may not be possible to evaluate small defects and the extent of the restorations (Bouschlicher & others, 1999). It has been recommended that the radiopacity of resin composites should be equal to or greater than enamel (Van Dijken, Wing & Ruyter, 1989; Murchison, Charlton & Moore, 1999). Materials with a radiopacity less than enamel were reported not to be suitable for use, especially as an initial increment in areas prone to secondary caries (Bouschlicher & others, 1999).

In this study, there was considerable variation in the radiopacity values of esthetic posterior restorative materials. Three of the five hybrid resin composites, Tetric Ceram, TPH Spectrum and Filtek Z-250, had radiopacity values greater than enamel but Composan LCM and Glacier had radiopacity values less than enamel.

The popular materials for posterior esthetic restorations, the packable resin composites, also showed different radiopacity values. Of the five packable composites used in this study, only SureFil and P-60 Filtek had radiopacity values higher than enamel, whereas Pyramid Dentin, Pyramid Enamel and Solitaire 2 had lower values than enamel.

Flowable composites have been recommended for use especially in areas that are difficult to access. The radiopacity of a flowable composite is important since it is indicated as gingival increments in Class II restorations to reduce microleakage (Bayne & others, 1998; Leevailoj & others, 2001; Prager, 1997; Small, 1997). Heliomolar Flow, the flowable composite used in the study, was higher in radiopacity compared to enamel, but in contrast, Composan LCM Flow was lower.

Resin composites that contain radiopaque glasses have atoms with high atomic numbers such as barium, strontium and zirconium (Craig & Ward, 1997). The radiopacity of a resin depends on selection of the polymer matrix, chemical nature of the filler particles, their size, density and addition level (Chandler & others, 1970; Bowen & Cleek, 1972). This resulted in considerable variations in radiopacity values of the restorative materials used in the study.

Williams & Billington (1990) suggested that radiopacity is not a requirement for anterior restorations but is essential for posterior restorative materials. However, in the opinion of the authors, all restoratives, including anterior, posterior and lining materials, must be sufficiently radiopaque to be detected against a background of enamel and dentin since the contrast facilitates the evaluation of restorations in every region of the mouth. For this reason, the anterior restorative materials were also included in the study. Of the microfilled resin composites, Heliomolar RO had a radiopacity greater than enamel. Conversely, Filtek A 110 had a radiopacity lower than enamel and dentin and it failed to meet even ISO standard 4049 (2000).

In addition to being used in permanent dentition, glass ionomer cements and hybrid versions have become popular, especially for the anterior and posterior restorations of primary teeth (García-Godoy, 2000). The conventional glass ionomer cements used in this study, Chem Flex Condensable and Chem Flex Syringeable, and the polyacid-modified resin composites, Dyract AP, Compoglass F and F2000, had a radiopacity higher than enamel but the resin-modified glass ionomer cement Vitremer had a lower radiopacity. The fluoride containing resin composite Ariston pHc also showed a radiopacity value greater than enamel.

It was suggested that in order to fulfill clinical requirements, the radiopacity of restorative materials should permit the diagnosis of secondary caries and marginal defects. However, it was reported that the higher radiopacity of amalgam restorations may lead to under- and over-scoring of secondary caries and marginal defects compared to composite restorations.

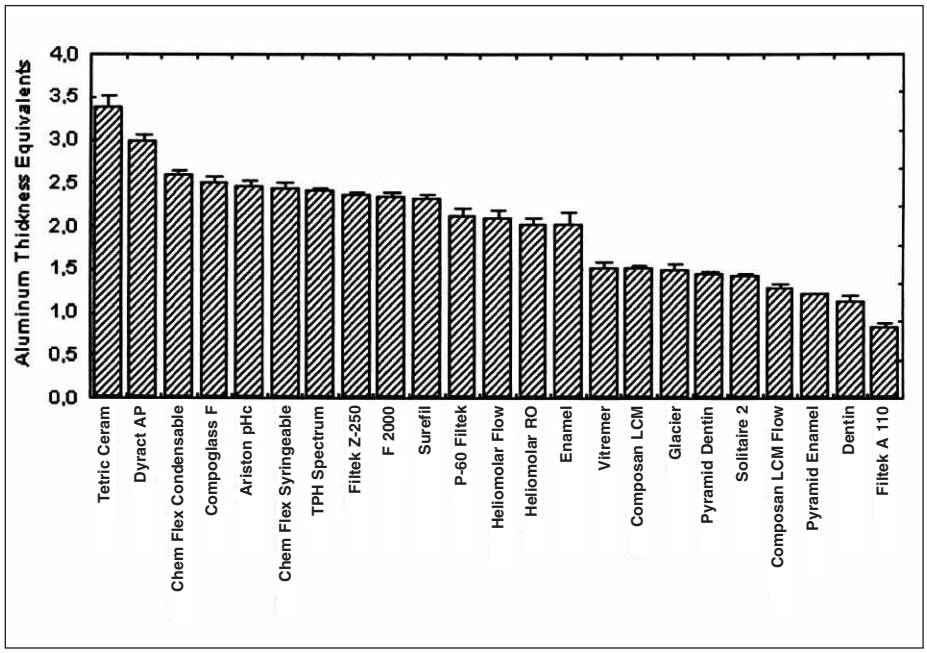

Figure 4. *Bar chart representation of aluminum thickness equivalents of all materials.*

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It was concluded that caries lesions and marginal defects may be over-diagnosed with high radiopaque restorations. A caries lesion near the buccal or lingual margin of a Class II restoration may be masked by a highly radiopaque restorative material. Moderate radiopacity might be more favorable and will make caries detection easier (Tveit & Espelid, 1986). This phenomenon might depend on the angulation of the x-ray beam superimposing high radiopaque restorations over carious tooth structure (Goshima & Goshima, 1990). Radiographically, distinguishing the restoration from tooth structures was reported to be more visible in areas primarily composed of dentin due to the lower radiopacity of dentin compared to resin composites. The remaining enamel and dentin adjacent to and superimposed on the restoration along with the cavity configuration influence the radiographic evaluation. High radiopacity of restorative materials were reported to decrease this influence (Stanford & others, 1987).

In many studies, an aluminum equivalent was used to compare the radiopacities of different restorative materials (Akerboom & others, 1993; Curtis, Von Fraunhofer & Farman, 1990; Bouschlicher & others, 1999; Omer & others, 1986). Although Akerboom and others (1993) suggested using human teeth as a standard, they reported that variations in radiopacity of samples may cause a disruption between studies. The radiopacity values of enamel and dentin obtained from this study (2.02 mm Al/1 mm enamel and 1.13 mm Al/1 mm dentin) were determined to be comparable to those reported by el-Mowafy, Brown and McComb (1991) (1.84 mm Al/1 mm enamel and 1.16 mm Al/1 mm dentin), Williams and Billington (1990) (2.2 mm Al/1 mm enamel and 1 mm Al/1 mm dentin) and Stanford and others (1987) (2.22 mm Al/1 mm enamel and 0.79 mm Al/1 mm dentin).

It was reported that variability in radiopacity values of the same restorative materials among different studies depends on a number of factors including speed of the x-ray film, exposure time, voltage used and the age of the developing and fixing solutions (el-Mowafy & others, 1991; el-Mowafy & Benmergui, 1994). In this study, x-ray films were processed with fresh developing and fixing solutions. It was reported that optical density can vary with the age of the developing and fixing solutions. A change in the voltage from 65 to 70 kV has an effect on radiopacity (el-Mowafy & others, 1991; el-Mowafy & Benmergui, 1994).

Since dental materials are constantly being refined, clinicians seek better mechanical properties for restorative materials. Adequate radiopacity of restorative materials assists in the radiological diagnosis of caries and the overall condition of existing restorations. Adequate radiopacity must, therefore, be accepted as one of the major factors when evaluating the clinical success of restorations.

CONCLUSIONS

In this study, all materials except for the microfilled resin composite Filtek A 110 were found to have radiopacity values greater than dentin and possessed sufficient radiopacity standards of ISO 4049 (2000). Considerable variations in radiopacity values were found among materials when compared to enamel.

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Fiber Post Adhesion to Resin Luting Cements in the Restoration of Endodontically-Treated Teeth

D Prisco • R De Santis • F Mollica
L Ambrosio • S Rengo • L Nicolais

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Clinical Relevance

With respect to the adhesion properties of carbon fiber posts and glass fiber posts used in the restoration of endodontically-treated teeth, they perform equally well if used in combination with chemically cured luting cements or with light activated ones.

SUMMARY

Fiber posts are widely used in the restoration of endodontically treated teeth. Scientific evidence demonstrates that the mechanical performance of teeth restored with fiber posts in combination with resin luting cements is improved with respect to metallic post restorations. The post is cemented inside the root canal using low modulus elastic polymer resins. In this study, the mechanical resistance of four different post-cement systems was assessed by means of a micro-mechanical pull-out test assisted by a simulation using the finite element methodology. This *in vitro* test is specifically designed to accurately characterize the post-cement interface. The results show no significant difference among the adhesion properties of the various types of post-cement systems used.

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INTRODUCTION

Post and core restoration is used to restore functionality when the loss of tooth structure has been significant. The post is cemented inside the root canal and the core is retained by an apical extension of the post. The cast restoration, replacing the coronal portion of the tooth, is subsequently cemented on the restored core (Fredriksson & others, 1998; Torbjorner, Karlsson & Odman, 1995). The use of post and core restoration will continue to increase because of the current trend to preserve natural tooth structure in the mature years of life (Christensen, 1998).

In the 1990s, fiber posts, usually made of stretched, aligned fibers embedded in various epoxy-resin matri-

ces (Purton & Love, 1996; Torbjorner & others, 1996), were introduced in order to obtain improved mechanical strength, retention and corrosion properties with respect to metallic posts that are prone to fatigue failure and corrosion (Milot & Stein, 1992).

Fiber posts are generally used with low modulus resin cements in combination with dentin adhesive systems. This restorative technique has been shown to have less microleakage than other bonding cements (zinc phosphate and glass ionomer) (Mannocci, Ferrari & Watson, 2001a; Bachicha & others, 1998; Viguie & others, 1994).

The increased popularity of fiber posts in restorative dentistry is also due to its capability to better distribute chewing loads along the radicular walls and, therefore, prevent dangerous stress accumulations that can lead to untreatable vertical tooth fracture. In fact, from a mechanical viewpoint, the post acts as a bypass, concentrating the chewing stresses at the root tip. This can lead to fracture of the surrounding dentinal walls weakened by the root canal preparation (Purton & Love, 1996; Torbjorner & others, 1996; Sidoli, King & Setchell, 1997; Huysmans & van der Varst, 1995; Assif & others, 1993). This is especially true when the post diameter increases. Root fracture of teeth restored with prefabricated posts or cast gold posts is related to their mechanical stiffness (Dean, Jeansonne & Sarkar, 1998), which is higher than the surrounding tissue, thus, leading to stress concentration at the tip (Martinez-Insua & others, 1998).

A longitudinal clinical follow-up study after three years has indicated that fiber post restoration is a viable alternative in some cases to traditional cast metal post and core or metal prefabricated post restoration (Fredriksson & others, 1998).

Carbon fiber posts were initially used in combination with low modulus chemically cured luting cements. Biomaterials research introduced new materials for post manufacturing, for example, E-glass or quartz fiber embedded in transparent epoxy-resin matrix, to be used with newly developed light cured luting cements.

From a clinical viewpoint, evaluating the adhesion properties at the post-cement interface is an aspect of particular importance since the post has primarily a retention function (Fan, Nicholls & Kois 1995; Assif & others, 1993).

The mechanical properties of fiber posts and data on adhesive retention strength of cemented fiber posts in root canals are widely reported in the scientific literature. However, little information is available regarding the interface between the fiber post and the resin luting cement. In fact, the direct measurement of the bond strength between the fiber posts and cement has been carried out only in the case of carbon fiber posts (De Santis & others, 2001).

In this work, the fracture resistance of the post-cement interface of four post-cement systems was investigated using an *in vitro* mechanical pull-out test. The stress distribution in the cement layer was studied using the finite element method (FEM).

METHODS AND MATERIALS

Forty selected, sound third molars stored in an aqueous solution of 1% chloramines-T, for less than 30 days after extraction were used to obtain 40 middle coronal dentin disks (thickness=2 mm). A post-hole (diameter D=2.4 mm) was drilled in the center of each dentin slice and four different posts (diameter d=2 mm) were cemented into the post-hole according to the manufacturers' instructions. Four different combinations of fiber post–luting cement were used: carbon fiber posts [CFP] (Composipost RTD, France) + a chemically cured cement [C&B] (C&B Cement, BISCO Dental Products, Schaumburg, IL, USA), hybrid posts, that is, carbon fiber posts covered by a mantle of glass fiber [WAP] (White Aestetic plus RTD, France) + C&B, glass fiber posts [AEP] (Aestetic post RTD, France) + C&B, glass fiber posts in a translucent epoxy resin matrix [LP] (Light post RTD, France) + a dual cured cement [DL] (Duo-link cement, BISCO). The rationale behind the choice of cement in the last system lies in the fact that since Light Post allows for the transmission of light, a dual cured cement is recommended in clinical practice. Samples were light cured using a 600 mW/cm² output

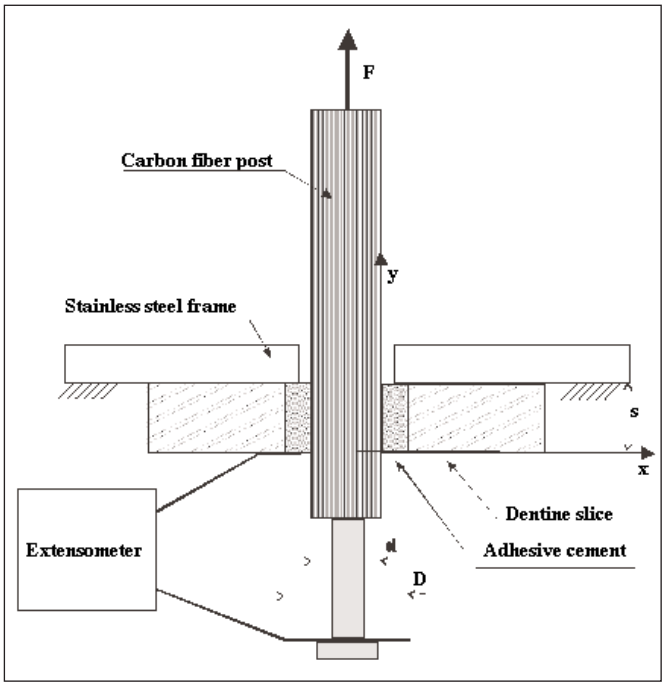

Figure 1. *Mechanical pull-out test set-up.*

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halogen lamp (Optilux, Demetron Research Corporation, Danbury, CT, USA) for 40 seconds. This was done in order to investigate whether the adhesion properties were improved by photopolymerization.

All posts were cemented using the coronal portion, where the diameter is constant (d=2 mm). During the preparation, all samples were stored for no more than 24 hours in an isotonic 0.5 wt% sodium chloride solution at 37°C to prevent dentin dehydration. Coupled Teflon molds were used to keep each fiber post coaxial with the dentin hole during the curing of the cement, leading to a uniform cement mantle thickness of (D-d)/2=0.2 mm (Figure 1).

In a preliminary FEM analysis, two different loading geometries were simulated in order to select the one with a preferential stress accumulation at the post-cement interface. In the first case, the upper surface of the dentin was constrained, while the cement was free to move vertically during the application of the load. In the second case, about 3/4 of the free surface of the cement was constrained with the dentin. The axial-symmetry of the geometry led to the development of a two-dimensional model that was solved using the package ANSYS 5.6 (Swanson Corp). The FEM analysis demonstrated that in the second case the post-cement interface was preferentially loaded, while the dentin-cement interface was basically stress-free. Consequently, a custom-made stainless steel frame was used to constrain the samples following indications obtained from this preliminary FEM analysis.

The pull-out tests were performed with an INSTRON 4204 testing machine and the pulling load was increased at a cross-head speed of 2.0 mm/minute in the axial direction of the post. The axial displacement of the post was monitored using an INSTRON extensometer 2620-601 positioned between the dentin slice and the post (Figure 1). The load and the extensometer displacement were acquired at a sampling rate of 10 points per second.

The average shear strength (τ) of the post-cement interface was computed by dividing the average maximum load (F) by the bonding area cross-section:

$$\tau = \frac{F}{\pi \bullet d \bullet s} \qquad (1)$$

where s is the dentin slice thickness (s=2 mm) and d is the post diameter (d=2 mm).

All the posts were assumed to be transversely isotropic, while the luting cements were assumed to be isotropic elastic—perfectly plastic materials. The manufacturers provided all the relevant data concerning the posts and luting cements.

RESULTS

Table 1 shows the average recorded maximum load and the average bond shear strength for each post-cement system. Plotting the pull-out load against the displacement for all the post-cement combinations, one can see that the load increases quasi-linearly, then levels up, reaching the ultimate load F before drastically dropping as the post-cement interface fails (Figure 2). One-way ANOVA statistical analysis suggests that there are no significant differences among samples at a probability level of 0.5.

FEM was used to investigate the stress distribution in the luting cement. The analysis, performed on all the post-cement systems, has shown that the stress components σ_x, σ_y, σ_z, and τ_{xy} are relevant in the cement, particularly in the region near the upper part of the cement layer (the part in contact with the steel frame, see Figure 1). In Figure 3, the σ_x, σ_y, σ_z, and τ_{xy} distributions in the cement layer for the case of CFP + C&B loaded at 40% of the ultimate load are depicted as an example, the other systems being very similar. Since the state of stress in the relevant parts of the cement is generally three-dimensional, the FEM analysis produced graphs of a suitable stress measure, namely, the von Mises equivalent stress (Khan & Huang, 1995):

$$\sigma_e = \sqrt{1/2 \bullet \left[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2\right]} \qquad (2)$$

where σ_1 σ_2 and σ_3 denote the principal stresses. The distributions of the von Mises equivalent stresses in the cement layer for each of the four post-cement systems were used to compare the different combinations and to check for the locations where critical stress values were reached and are shown in Figure 4. As can be seen, the post-cement interface (the left side of each distribution) is more loaded by about a factor of 2 than the dentin-cement interface (the right side of each distribution). This was desired, since the purpose of the test was to evaluate the mechanical properties of the post-cement interface rather than the cement-dentin interface.

Figure 4 also shows that the stress distribution along the post-cement interface is not uniform, reaching its maximum value near the upper portion of the cement layer and slowly decreasing towards the bottom. This maximum value is well above the average shear stress

Table 1: *Average Recorded Maximum Load and Average Bond Shear Strength for Each Post Cement System Tested*

	CFP	WAP	AEP	LP
Ultimate strength N (St dev)	315 (78)	316 (70)	347 (84)	341 (68)
Ultimate shear stress MPa (St dev)	25.1 (6.2)	25.2 (5.6)	27.6 (6.7)	27.1 (5.4)

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calculated using equation (1) for all the post-cement systems that were considered.

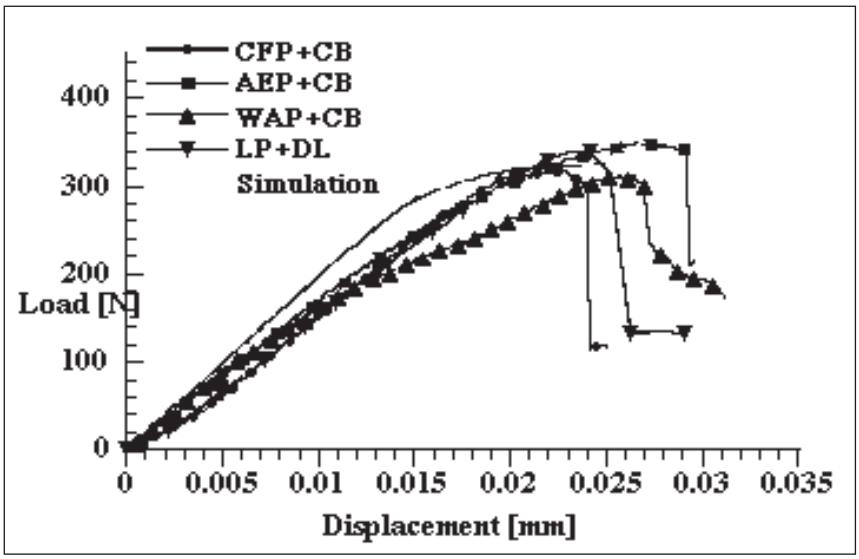

Figure 2. *Pulling load against displacement for the different post-cement systems and FEM simulation for the CFP+C&B system.*

Another result of the FEM analysis is that the stress distribution in the luting cement for the four post-cement systems is substantially similar, suggesting that it has no direct relationship with the type of fiber reinforcement of the post.

A complete simulation of the pull-out test was performed for the CFP + C&B system. The axial load-axial displacement graph obtained from the FEM analysis is shown in Figure 2, where it can be compared with the experimental data.

DISCUSSION

A cemented post does not equally distribute the load along the root length. The contribution of intraradicular posts to fracture resistance of restored teeth is negligible because the post occupies the canal region, which is a neutral region as regards the bending loads acting on the coronal portion (Assif & others, 1993; Assif & Gorfil, 1994). Thus, regarding the mechanical integrity of the restored tooth,

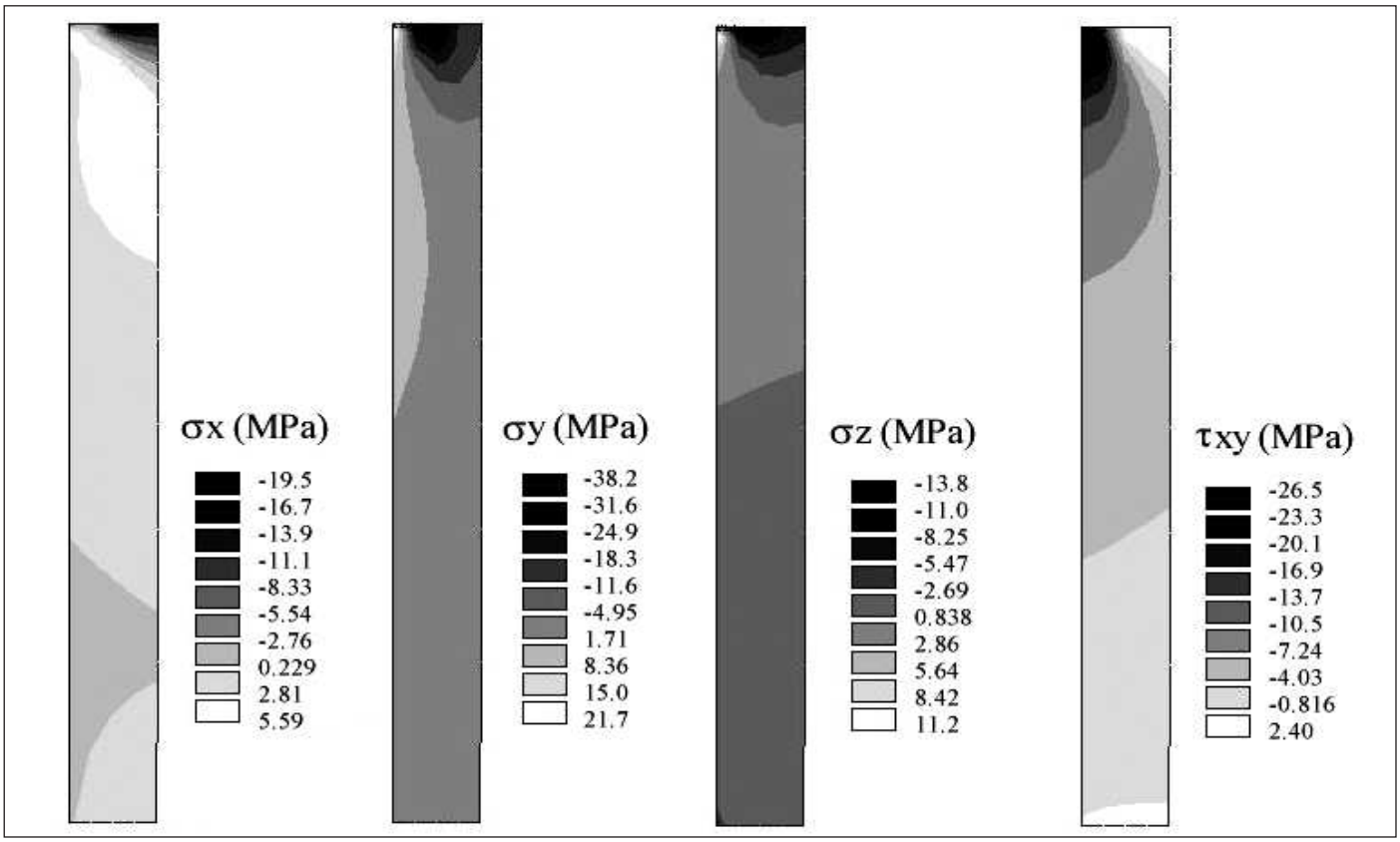

Figure 3. *FEM-distributions of the stress components σ_x, σ_y, σ_z, and τ_{xy} in the cement layer of the CFP + C&B post-cement system loaded at 40% of the ultimate load. The left and right side of each distribution represent the post-cement and the cement-dentin interfaces, respectively. The upper part of each distribution is the region of the cement that is in contact with the stainless steel frame, as represented in Figure 1.*

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the main requirements of the post should be retention and passivity rather than strength.

As retention properties are essential from a clinical perspective, it is important to study the properties of the interfaces involved in the restoration.

In a fiber post restoration, there are at least two main interfaces: one between the post and the luting cement and another between the cement and the dentin substrate.

The interface between the luting cement and the dentin has already been studied by Mannocci and others (2001b); thus, it is interesting to consider the post-cement interface, as well. Moreover, as it is well known, the coupling of materials with different mechanical properties usually generates severe stress concentrations at the interface when the system is loaded, and these concentrations become more dangerous when the differences between the two materials are higher. In the case of fiber post restorations, the highest differences are between the post and the luting cement; thus, it is particularly important to investigate the properties of the post-cement interface. This can be achieved using the authors' experimental set-up that loads the post-cement interface (shown in Figures 3 and 4).

The mechanical tests demonstrated that all the various systems withstood loads up to 347 N, suggesting that chemical and micro-mechanical bonding takes place at the post-cement interface independent of the reinforcing fiber used in the post. The differences among the various post-cement combinations were not statistically significant, which may be due to the fact that the matrix substantially had the same chemical composition.

The FEM analysis was necessary to determine the stress distribution within the luting cement layer. According to the FEM simulation, the stress distribution at the post-cement interface (Figure 4) has its maximum value in the upper part of the bonded length, while the minimum is located at the lower edge of the interface. The shear stress calculated using equation (1) is an average value obtained, assuming that the shear stress distribution within the cement layer is uniform. In the case of CFP + C&B, the maximum shear stress (τ_{xy}) resulting from FEM depicted in Figure 3 is 26.5 MPa. This value is basically equal to the average shear stress calculated using equation 1

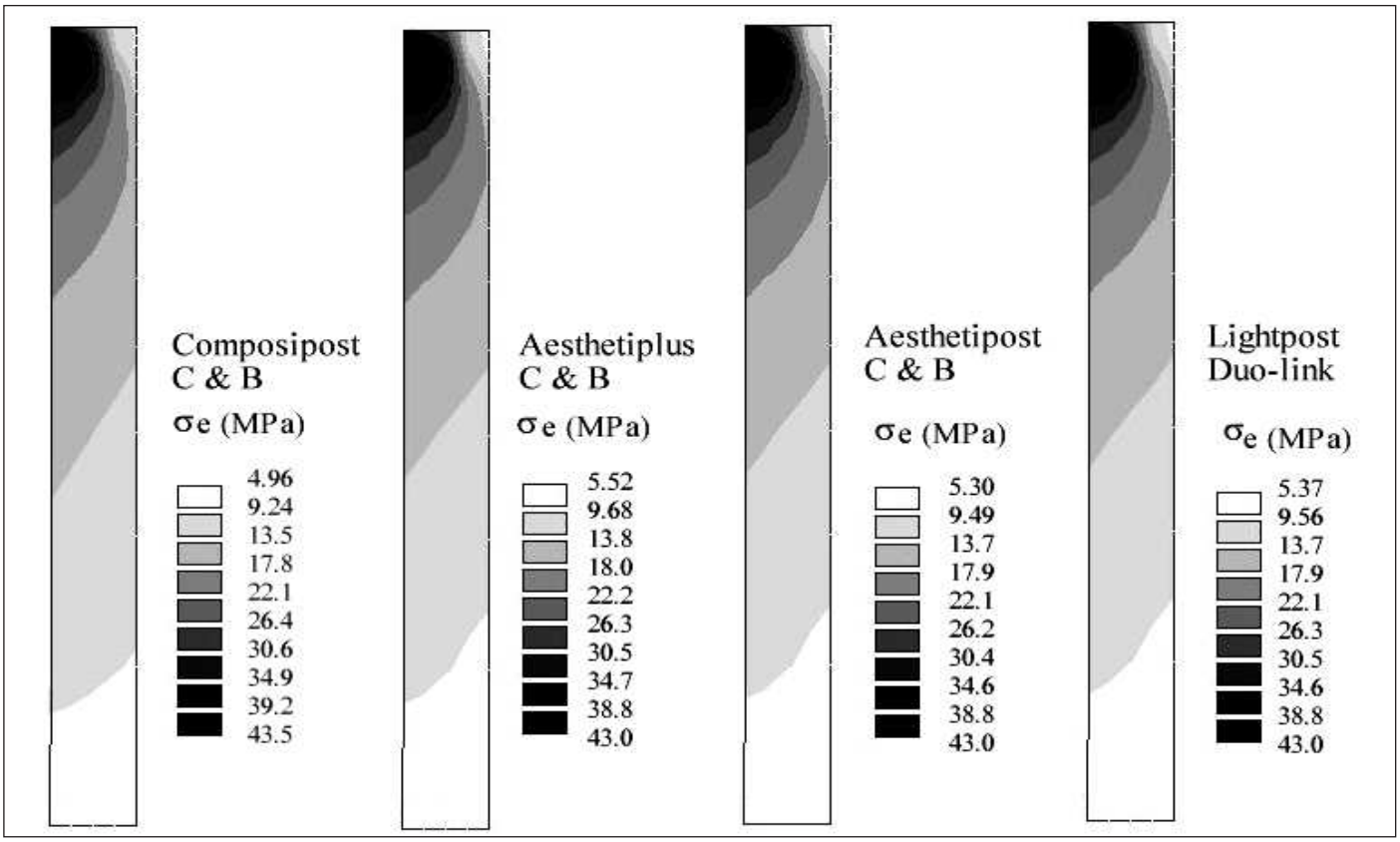

Figure 4. *FEM-distributions of the von Mises equivalent stresses in the cement layer for each post-cement system loaded at 40% of the ultimate load. The left side and the right side of each distribution represent the post-cement and the cement-dentin interfaces respectively. The upper part of each distribution is the region of the cement that is in contact with the stainless steel frame, as represented in Figure 1.*

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(τ = 25.1 MPa) for the same system, but one has to take into account that the FEM picture was obtained for a loading that is 40% of the loading used in equation 1. Thus, it can be concluded that using equation 1 leads to a gross underestimation of the maximum shear stress within the cement layer, hence, underlining the mechanical reliability of this interface in the fiber post restorations.

The non-uniform stress distribution obtained with FEM suggests that the post-luting cement system likely fails when a crack starts on the upper part of the cement layer (Figure 1) and propagates downward along the post-cement interface.

By comparing the FEM analysis of the various post-cement systems that were tested, it can be seen that the stress distribution in the cement changes very slightly, especially for the first three systems in which the same cement was used. FEM analysis thus confirms that the type of post does not play a significant role in the entity of the maximum stress in the cement nor in its distribution.

The FEM simulation of the pull-out test, shown in Figure 2, closely reproduces the experimental pull-out tests.

CONCLUSIONS

The FEM-assisted pull-out test assesses correct information about the actual bond strength values between fiber posts and resin luting cements. The difference between the relative rigidity of the cement and the post is higher than between the cement and dentinal substrate; thus, the luting cement will most probably fracture at the post-cement interface. This is consistent with the clinical observation of post debonding from root canals.

The ultimate strength values measured with the pull-out test showed no significant difference among the various post-cement systems, thus, as far as the post retention properties are concerned, the kind of fiber reinforcement of the epoxy resin of the post is not an influent factor.

Observing a remarkably non-uniform stress distribution within the cement layer, irrespective of the minimal thickness of the dentin slice used in our pull-out test, suggests that the stress concentrations may be reduced by modifying the local stiffness of the materials involved in the design; thus, improving the load transfer pattern and the retention properties. Further studies should focus on the influence of these factors on the strength of the fiber post cement interface.

The experimental and theoretical characterization of the post-cement interface obtained with this pilot study is essential for simulation of the mechanical behavior of an *in vivo* post-cement system, where additional parameters, such as environmental effects and aging, have to be considered.

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Effect of Bur Cutting Patterns and Dentin Bonding Agents on Dentin Permeability in a Fluid Flow Model

T Vaysman • N Rajan • VP Thompson

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Clinical Relevance

Dentin surface topography created by various bur designs may influence the surface sealing quality of resin bonding agents and, therefore, affect post-operative sensitivity and microleakage associated with composite bonding procedures. Dentin bonding agents do not appear to routinely seal the pulpal wall (before application of resin composite and creation of polymerization shrinkage stress).

SUMMARY

This study investigated the effects of bur cutting surface roughness and bonding systems on dentin permeability. A conventional straight edged bur, cross-cut serrated bur and an extensively serrated bur were utilized with two different bonding systems. Null hypothesis was that increased surface roughness does not decrease the permeability of dentin sealing after application of bonding agents. This study incorporated a fluid flow model for measuring dentin permeability. Seventy caries-free extracted molars were used and sectioned 2 mm apical to the cemento-enamel junction (CEJ). The pulp tissue was removed and the chamber cleaned using 37% phosphoric acid followed by 1:1 sodium hypochlorite solution. The specimens were mounted to plexiglass plates using cyanoacrylate cement. A flow system was established through the pulp chamber and gravity pressurized phosphate buffered saline (PBS) was forced into the chamber and the "closed system" permeability measured. Burs were used to prepare flat surfaces in dentin, and after etching with phosphoric acid, the "open tubule" permeability was determined. After applying the dentin bonding agents, fluid flow from the pulp chamber into dentin was again measured. The percentage of reduction in permeability following bonding agent application was then calculated. A two-way ANOVA found a significant ($p<0.05$) decrease in bonded dentin permeability in specimens prepared using the cross-cut serrated and extensively serrated bur as compared to the conventional straight-edged bur. No significant differences were detected between bonding systems and no combination of surface topography or bonding agent completely sealed the surface.

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INTRODUCTION

The use of resin composite restorations has been associated with postoperative sensitivity. This post-operative sensitivity has been shown to affect up to 56% of patients with Class I occlusal restorations subjected to loading (Opdam & others, 1998; Browning, Johnson &

Gregory, 1997; Borgmeijer & others, 1991). Causes of post-operative sensitivity after bonding procedures include incomplete caries removal, desiccation of tooth structure by over-drying, over-etching the dentin, bacterial contamination, improper restorative placement, incomplete polymerization and occlusal interferences (Hornbrook, 1996). An additional cause of sensitivity has been associated with the movement of dentinal fluid in the tubules. This fluid is a filtrate of plasma and its movement within the tubules is thought to cause post-operative sensitivity and occasional pulpal inflammation (Pashley, Nelson & Pashley, 1981).

Dentin bonding agents are intended to adhere to the dentin substrate and seal these tubules. While there is much literature regarding the production of an intimate bond between bonding resins and tooth structure, fewer studies have investigated the effects of surface topography on dentin bonding. Eick and others (1972) reported that one of the main factors in surface wetting of bonding resins is the surface topography of the prepared dentin and that a roughened surface creates a greater surface area. Others have considered surface rugosity and irregularities to promote the wettability of resins by producing increased surface area for the adhesive bond (Mowery, Parker & Davis, 1987; Al-Omari, Mitchell & Cunningham, 2001).

This study compared the effects of dentin surface roughness on dentin permeability using a fluid flow model (Pashley & others, 1985). The fluid flow system utilized was not meant to measure actual bond strengths, but rather to measure the existing dentin bonding system's ability to seal etched dentin. As mentioned above, incomplete sealing of dentin tubules by the bonding system has been implicated in the etiology of post-operative sensitivity after composite bonding procedures (Pashley & others, 1981).

METHODS AND MATERIALS

Seventy caries-free extracted human molars were first placed in a 1:1 solution of sodium hypochlorite for 24 hours. They were then stored in a phosphate buffered saline solution (PBS) until used for sample preparation. The PBS solution was prepared using protocol from Quality Biological, Inc (Gaithersburg, MD, USA) and consisted of Na_2HPO_4 (anhydrous) 726 mg/ml, KH_2PO_4 210 mg/ml and NaCl 9000 mg/ml. The specimens were sectioned 2 mm apical to the CEJ using a high-speed handpiece with copious water. The apical crown segment was milled flat using a polisher/grinder unit (Beuhler, Economet 4, Lake Bluff, IL, USA). The pulpal tissues were removed and the chamber cleaned with a 30-second application of 37% phosphoric acid followed by a 30 second-treatment with 1:1 sodium hypochlorite solution. The specimens were mounted with cyanoacrylate cement to 50 x 50 mm plexiglass plates. The cynaoacrylate cement was applied to the milled apical portion of the cut crown segment, taking care not to introduce any cement into the root canal chamber. A flow system was established using two 18-gauge hollow stainless steel cylinders connected through the pulp chamber. The specimens were coated with a thin layer of cyanoacrylate (ZAP Pacer Tech, Rancho Cucamonga, CA, USA) cement to seal the coronal portions.

Figure 1 shows the apparatus for the fluid flow model for measuring dentin permeability. The fluid reservoir containing sterile phosphate buffered saline (PBS) solution (pH=7.4) was placed at a height of 58 cm to provide intrapulpal pressure to tooth specimen. The *in-vitro* fluid pressure (here 58 cm H_20) was set higher than *in vivo* intrapulpal pressure (15 cm H_20) to facilitate flow through the dentinal tubules (Gerzina & Hume, 1995). This was within the range of similar fluid flow studies (Pashley & others, 1981). Other investigators have reported that intrapulpal pressure is known to vary in magnitude, is significantly higher in inflammatory states and is positively directed toward the dentinal walls (Gerzina & Hume, 1995). Accordingly, some authors have used intrapulpal pressures up to 100 cm H_20 in similar studies (Bouillaguet & others, 2000). The left side of the circuit consisted of the fluid reservoir joined to the specimen with 18-gauge polyurethane tubing. A 1-cc syringe connected to the left side of the circuit allowed for the introduction of an air bubble to visualize and measure the volume of the PBS solution that moved from the pulp chamber to the prepared dentin surface. A millimeter ruler approximated to the polyurethane tubing allowed for visual measurements of air bubble travel. Another 1-cc syringe connected to the right side of the circuit

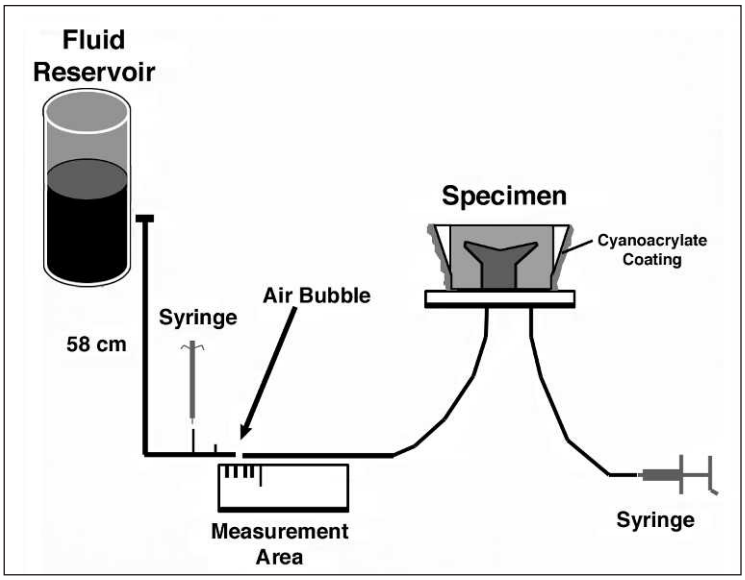

Figure 1. *Schematic diagram of fluid flow apparatus.*

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allowed for air bubble "zeroing" at the beginning of each test. The connections were examined to ensure a sealed fluid system and minimize visible leaks.

The specimens were connected to the fluid flow apparatus and equilibrated for 45 minutes to allow for any initial fluid transfers to occur. After the equilibration period, flow was measured over a 15-minute interval to establish a baseline *sealed* measurement. This allowed each tooth to act as its own control. A flat surface was then prepared on the dentin at a depth of 1.5 mm (one-half the cutting length of a 556 carbide bur) from the central fossa using a diamond bur on a high-speed handpiece. The bur was held perpendicular to the long axis of the tooth, leaving an exposed, unetched dentin surface. The thin layer of cyanoacrylate cement applied initially remained only on the external axial walls of the crown segment and was not in contact with the exposed dentin used in testing dentin bonding agents. The prepared surface was then etched with 37% phosphoric acid for 30 seconds. The flow through the etched surface was recorded over 15 minutes to establish initial dentin permeability of the crown segment. Based on this initial dentin permeability, specimens were assigned to each of the six experimental groups so that each group received a variety of initial dentin permeabilities.

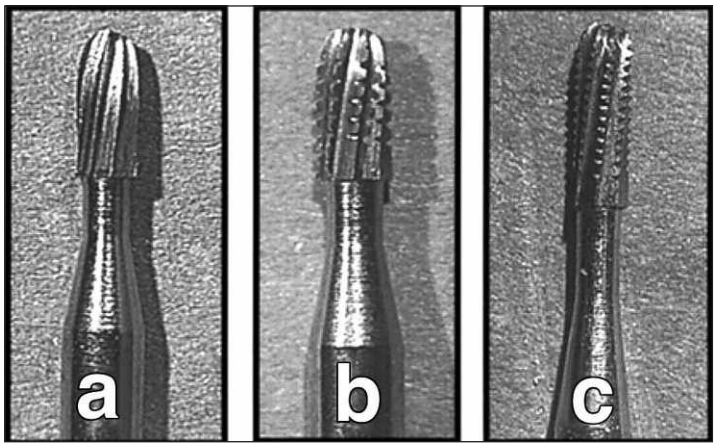

Figure 2. *Photographs of burs used in the study. 2a: Fissure Bur (FB); 2b, Cross-Cut (CC); and 2c Extensively Serrated (ES).*

Table 1: *Materials Utilized and the Manufacturer's Information*

Material	Manufacturer
Fissure Bur (FG 1158 17709) Cross-Cut Serrated Bur (FG 15924 1558) Extensively Serrated Bur (Prototype A)	SS White Co 1145 Towbin Ave Lakewood, NJ, USA
Clearfil SE Bond (Lot #61184)	Kuraray Co, Ltd 1-12-39, Umeda Kita-ku, Osaka, 530-8611, Japan
Optibond Solo Plus (Lot #101159)	Kerr Corporation 1717 West Collins Ave Orange, CA, USA

Table 2: *Assignment of 62 Samples in the Six Experimental Groups. FB Indicates Fissure Bur, CC Indicates Cross Cut Bur, ES Indicates Extensively Serrated Bur, SE Refers to Clearfil SE Bond; OB refers to Optibond Solo Plus*

GROUPS	N	Bur	Bonding Agent
Group 1	8	FB	SE
Group 2	16	FB	OB
Group 3	10	CC	SE
Group 4	9	CC	OB
Group 5	7	ES	SE
Group 6	12	ES	OB

Once the specimens were equally distributed, the dentin surface was re-prepared using a straight fissure bur (FB), cross-cut serrated bur (CC) or extensively serrated bur (ES) (SS White Co, Lakewood NJ, USA) (Figure 2 a-c). The surfaces were re-etched using 37% phosphoric acid. The flow was measured over 15 minutes and recorded as the *etched* system measurement. This flow rate was considered to represent 100% dentin permeability for the specimen being tested. After establishing the *etched* system permeability, the pressure in the fluid reservoir was reduced to zero to avoid moisture contamination during the bonding procedure. The bonding agent was applied to the flat occlusal dentin surface using either Clearfil SE Bond (Kuraray, Osaka, Japan) or Optibond Solo Plus (non-self-etching variant) (Kerr Corporation, Orange, CA, USA) (Table 1). The bonding agents were applied to the etched dentin surface and light cured with a regularly calibrated light source (The MAX, Dentsply Caulk, Milford, DE, USA) according to the manufacturer's specifications; 10 seconds for Clearfil SE Bond and 10 seconds for Kerr Optibond Solo Plus. The established testing procedure necessitated applying Clearfil SE bond to an already etched surface since an etched 100% permeability value needed to be recorded prior to bonding.

The fluid permeability was again measured immediately after applying the bonding system to establish a *bonded* system measurement. All samples were stored in an air-tight, moist environment before and after the testing procedure. The decrease in dentin permeability was expressed as the Percent Permeability Decrease (PPD). PPD was calculated by comparing the *etched* and *bonded* system permeability measurements, each of which was normalized by subtracting the *sealed* system permeability. The formulas are as follows:

PPD = [(etched-sealed) – (bonded-sealed)]/(etched-sealed) x 100

Eight specimens were discarded in the study for producing initial *etched* permeability of less than 2.2 x 10^-17 nL/min and greater than 4.4 x 10^-18

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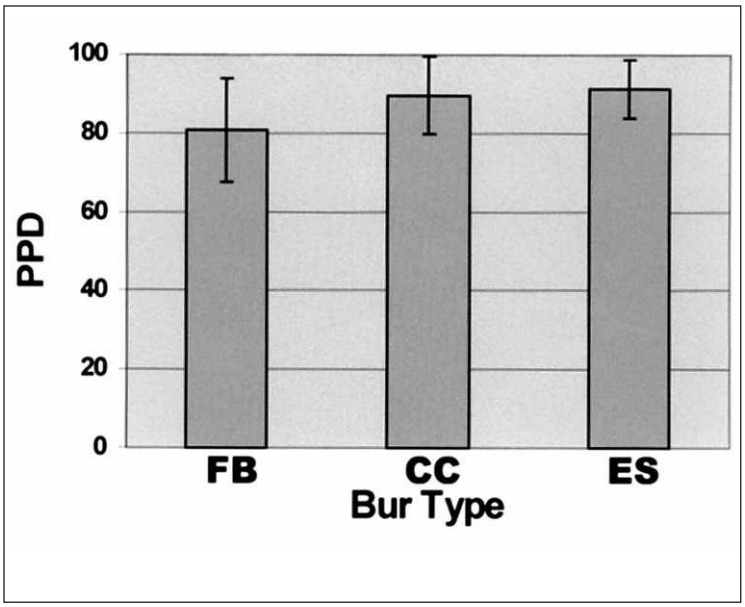

Figure 3. *Percent Permeability Decrease (PPD) with the Fissure Bur (FB), Cross-Cut (CC) and Extensively Serrated (ES).*

nL/min over a 15-minute interval. Removing these samples necessitated having unequal samples groups. The remaining 62-specimen distribution is shown in Table 2. Mean differences in PPD as a function of bur, bonding agent and their interaction were compared in a two-way ANOVA for independent groups. Post-hoc *t*-tests were used to compare groups where a significant omnibus *f*-test was found. Experiment-wise error rates were controlled in these post-hoc tests by means of Tukey HSD procedure (Kirk, 1995). Statistical significance was set as $p<0.05$ in all tests.

For scanning electron microscopy (SEM), the specimens were removed from their plexiglass plates and the undersurfaces were further milled to facilitate placement in the SEM chamber. The samples were mounted on stainless steel studs and vacuum desiccated for approximately four days, then carbon coated. Samples were inspected in the SEM at 50 kV acceleration voltage and at magnifications between 35-1500X for surface topography and coating characteristics.

RESULTS

Measuring dentin permeabilites in this extracted tooth model yielded significant variability. Typical permeabilities of etched (open) tubules ranged from 0.27 ml/min to 0.68 ml/min. Once the etched dentin was bonded with DBA, typical permeabilities ranged between zero and 0.26 ml/min. Using the formula described above, mean PPD values ranged from a low of 80.1% (± 12.9%) to a high of 94.5% (± 6.6%) (Figure 3). Increased serrations of the bur cutting patterns yielded a statistically significant ($p<0.05$) increase in sealing ability when the data were averaged over the bonding systems. Post-hoc *t*-tests showed that the average PPD for both serrated type burs was significantly more effective than the straight fissure bur, therefore, indicating that samples prepared with the extensively serrated carbide bur provided the highest

Figure 4: *Scanning Electron Micrographs (SEM) of prepared surfaces.*

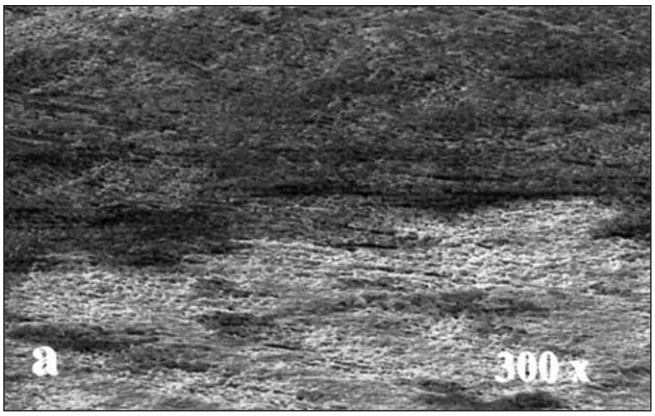

Figure 4a. *Fissure bur (FB).*

Figure 4b. *Cross-cut bur (CC).*

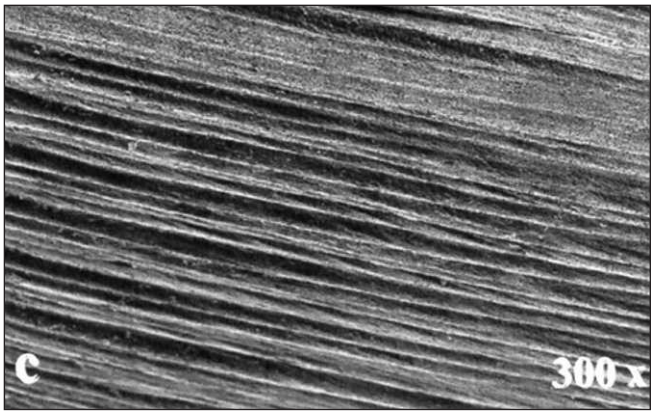

Figure 4c. *Extensively serrated bur (ES).*

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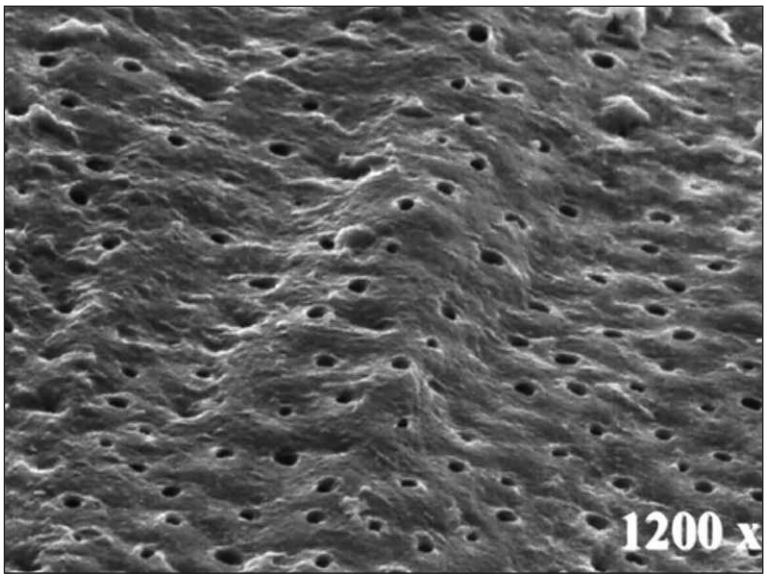

Figure 5. *Scanning Electron Micrograph (SEM) of dentin cut with Extensivley Serrated (ES) bur and etched with 37% phosphoric acid.*

reduction in dentin permeability. Although the trend to higher values with increased serration was evident, the cross-cut fissure bur was not different from the extensively serrated bur. The effect of the two bonding systems, when averaged over the three burs used, demonstrated no statistical difference in sealing ability (PPD). No interaction between bur and bonding system was detected. In addition, no group exhibited 100% reduction in dentin permeability of all specimens in a group after application of the dentin-bonding agent. SEM observation revealed a relatively smooth surface with limited surface roughness when a fissure bur was employed as depicted in Figure 4a. Cross-cut serrated burs created serrations and grooves that appeared uniform and widely separated (Figure 4b). Extensively serrated burs created numerous grooves that were closer together compared to cross-cut serrated burs (Figure 4c). A higher magnification SEM examination of the extensively serrated surface revealed orientation of the grooves, peaks and valleys in the dentin, and patent dentin tubules (Figure 5).

Examination of the dentin prepared with cross-cut serrated burs followed by application of Clearfil SE Bond revealed that the bonding agent covered the peaks and valleys created by the bur. The surface appeared uniform with some irregularities (Figure 6a). Higher SEM examination of all surfaces revealed limited areas where the bonding agent had separated from the underlying dentin, especially in the region of irregularities. This resulted in separation of polymerized resin tags and exposed dentin tubules (Figure 6b). However, desiccation of the specimens for SEM observation resulted in dentin cracking and the dentin bonding agent separation observed may be an artifact.

DISCUSSION

Bonded samples with PPD measurements that approached 100% were considered to have sealed the majority of the exposed dentin tubules, thereby preventing the outward flow of the pressurized PBS solution supplied by the fluid reservoir. Bonded samples with lower PPD measurements were considered to have areas of unsealed etched dentin, causing localized fluid flow of the PBS solution.

Specimens with initial *etched* permeability less than 2.2 x 10^-17 nL/min and greater than 4.4 x 10^-18 nL/min over a 15-minute interval were eliminated from the study. Very high initial flow volumes were thought to result from microcracks or from limited dentin thickness in specimens prepared too close to the pulp, giving an abnormally high permeability. Specimens producing very low etched permeability, baseline flow 2.2 x 10^-17 nL/min, were discarded. Since shallow restorations exhibit little post-operative sensitivity, this elimination was thought to be inconsequential to the outcome of the study.

As stated in Methods and Materials, the *in vitro* fluid pressure was introduced at a pressure significantly higher than what exists in a healthy tooth. However, since intrapulpal pressure has been reported to be markedly increased in times of inflammation (as in a post-operatively sensitive tooth), the authors felt that creating an increased pressure environment may more closely duplicate biological situations. It has been shown that polymerization shrinkage of resin composites during direct placement causes dimensional strains within the cavity preparation (Ciucci & others, 1997). These dimensional strains have been shown to appear as gaps at the tooth-restorative material interface due to the separation of the resin-bonding agent from the underlying dentin, especially along the pulpal wall. The volume of these internal gaps has been reported to be as large as 0.7 µl in Class I restorations (Ciucci & others, 1997). Carvalho and others (1996) reported these polymerization stresses (and resulting strain) are expected in Class I preparations where there is a high ratio of bonded to unbonded surfaces (high C-factor) (Yoshikawa & others, 1999). In addition, bonds to the pulpal floor are often lower because deep dentin is involved (Carvalho & others, 1996; Kinomoto & Torii, 1998). The presence of these gaps, regardless of the bonding agent polymerization method or whether flowable or heavily filled resin composite formulations were employed, was recently demonstrated in Class I dentin restorations (Cho & others, 2002). Gaps beneath resin composite restoration materials may allow for the accu-

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Figure 6: *SEM image of dentin surface prepared with CC and bonded with Clearfil SE Bond.*

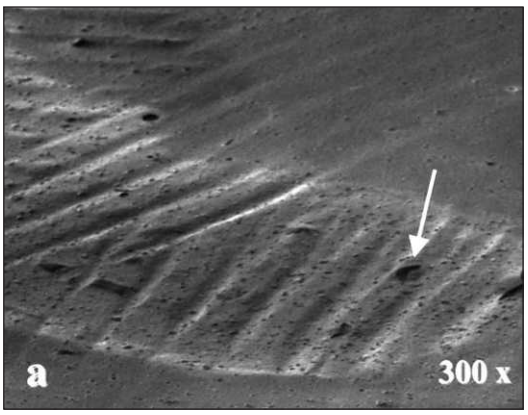

Figure 6a. *Arrow points to micro-cracks in bonded resin layer.*

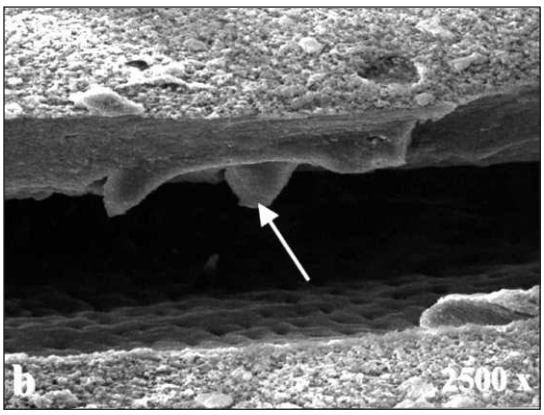

Figure 6b. *SEM image of a micro-crack. Arrow points to a de-bonded polymerized resin tag.*

mulation and movement of dentinal fluid during mastication.

The Brännström "hydrodynamic theory of dentin hypersensitivity" proposes that fluid transfer across dentin in either direction produces pain (reviewed by Pashley & others, 1981). Therefore, maintenance of a continuous low permeability interface between the prepared tooth surface and the resin-bonding agent is essential to limit fluid build-up and/or flow. Resin bonding agents have a dual role when used under resin composites. First, they provide micro-retention, primarily through the peritubular dentin, for bonding to the composite restorative. Second, they infiltrate and seal intratubular dentin with resin tags (Pashley & Carvalho, 1997). These polymerized resin tags form a plug to seal the etched dentin tubules and prevent any movement of dentinal fluid under the resin composite restorations (Bouillaguet & others, 2000). Therefore, an ideal resin bonding agent should provide effective peritubular bonding to facilitate retention of the composite and complete sealing of the dentinal tubules to prevent permeability of dentinal fluid that may cause post-operative sensitivity.

In clinical practice, amalgam restorations are seldom associated with post-operative sensitivity. While this study did not include an amalgam sample group due to flat preparation design, previous studies have compared dentin permeabilities in Class I cavities restored with varnish lined amalgams to composite restorations using a similar fluid flow system. These studies concluded that at 24 hours after insertion, varnish lined amalgam exhibited up to a 98% decrease in apparent dentin permeability, while composite restortations produced only a 62% apparent permeability decrease (Derksen, Pashley & Derksen, 1986).

The results of this study indicated that samples prepared with the extensively serrated carbide bur provided the highest reduction in etched and bonded dentin permeability. One possible explanation is that increased surface area on the dentin allows for a better infiltration of the bonding resins. In addition, the bur-created surface topography, if extended to the pulpal wall preparation, may help to withstand the polymerization shrinkage stress of resin composite in high C-factor cavity preparations (Carvalho & others, 1996; de Gee, Feilzer & Davidson, 1993; Feilzer, de Gee & Davidson, 1987). No test group consistently sealed the dentin to a 100% level of permeability decrease, irrespective of the bur and bonding agent used. These findings are consistent with other studies testing resin bonding agent properties (Del-Nero, Escribano & de la Macorra, 2000; Bouillaguet & others, 2000).

The results of this study indicate that if a uniform roughness could be created on the pulpal floor of cavity preparations, the initial sealing capability of existing bonding resins may be improved. In clinical practice, creating serrated bur topography on pulpal walls may be difficult but is the focus of continuing research in the authors' laboratory. Although no bur exists today that will create this sort of variegated surface on the pulpal floor, the results of this study elude to the possibility of pulpal floor topography playing a role in improving dentin bonding agent sealing ability. While many procedures and techniques have been proposed to improve bonding procedures, cavity surface topography is generally not considered. As a result of this study, future work in the area of surface topography may be warranted.

CONCLUSIONS

Within the limitations of this study, bur created surface roughness has a significant role in reducing dentin permeability after bonding.

The bonding systems that used (Clearfil SE Bond and Kerr Optibond Solo Plus) did not differ significantly in their ability to reduce permeability of etched dentin surfaces.

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Effect of Food-Simulating Liquids on the Shear Punch Strength of Composite and Polyacid-Modified Composite Restoratives

AUJ Yap • MK Lee • SM Chung
KT Tsai • CT Lim

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Clinical Relevance

The effect of food-simulating liquids on the shear strength of resin-based filling materials was product dependent. Composites based on BisEMA may be more resistant to degradation by alcohol.

SUMMARY

This study evaluated the effects of food-simulating liquids on the shear punch strength of two composites (Tetric Ceram [TC], Vivadent; Esthet X [EX], Dentsply), a conventional (Compoglass [CG], Vivadent) and a posterior polyacid-modified (Dyract Posterior [DP], Dentsply) composite. Thirty-two specimens (10-mm in diameter and 1.6-mm thick) of each material were made, randomly divided into four groups of eight and conditioned for one week as follows—Group 1 (control): air at 37°C; Group 2: distilled water at 37°C; Group 3: 75% ethanol-water solution at 37°C and Group 4: heptane at 37°C. After conditioning, the specimens were restrained within the test apparatus and subjected to shear punch strength testing using a 3.2 mm diameter punch at a crosshead speed of 0.5 mm/minute. The shear punch strength of the specimens was computed and the data was subjected to ANOVA/Scheffe's tests at significance level 0.05. The effect of food-simulating liquids on shear strength was found to be material dependent. All materials with the exception of EX were significantly weakened by ethanol solution. For DP, a significant increase in strength was observed after conditioning in water. EX was significantly stronger than TC and DY after conditioning in air, water and ethanol solution. The shear punch strength of EX and CG was significantly higher than DY after conditioning in heptane.

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INTRODUCTION

The clinical use of composite and polyacid-acid modified composite restoratives has increased substantially over the last few years due to improvements in formulation, simplification of bonding techniques, increased aesthetic

demands by patients and decline in the popularity of amalgam. Composites may be defined as three-dimensional combinations of at least two chemically different materials with a distinct interface (Phillips, 1981). Dental composites consist of a resin matrix (organic phase), inorganic filler particles (dispersed phase), filler-matrix coupling agent (interface) and minor additions including polymerization initiators, stabilizers and coloring pigments. Polyacid-modified composites or compomers are resin composites that contain either or both of the essential components of glass ionomer cements but at a level insufficient to promote acid-base cure reaction in the dark (McLean, Nicholson & Wilson, 1994). Composites are recommended for restoring all cavity classes in anterior and posterior teeth, while polyacid-modified composites are indicated in non-stress bearing areas like Class III and V cavities. Polyacid-modified composite materials have, however, been recently re-formulated for use in posterior teeth.

Composites, conventional and resin-modified glass ionomer cements are all susceptible to various modes of chemical degradation *in vitro*. Reduction in hardness, wear, fracture toughness and flexural strength had been reported after exposure to various food-simulating liquids (McKinney, Antonucci & Rupp, 1987; Kao, 1989; Ferracane & Marker, 1992; Mante & others, 1999; Yap, Low & Ong, 2000a; Yap & others, 2000b; Yap & others, 2002b). In an *in vivo* situation, it can be assumed that saliva, food components and beverages can degrade and age dental restorations. Wu and others (1984) reported that clinically damaged composite restorations had altered layers on both stress bearing and non-stress bearing occlusal surfaces. It was hypothesized that chemical or thermal environment contributed to the *in vivo* degradation of these materials. Thus, interactions among many substances in the oral cavity at 37°C may have a negative impact on the long-term durability of dental restorations. Although the effects of food-simulating liquids on composites had been widely investigated, only a few studies had been conducted on posterior polyacid-modified resin composite (Yap & others, 2000a,b).

Direct dental restorative materials are subjected to evaluation using different ISO (International Standards Organization) testing. Dental amalgams (ISO 1995) and water-based cement (ISO 1991) are evaluated using a compression test, while resin-based filling materials (ISO 1988) and light-activated water-based cements (ISO 1999) are evaluated using a flexural test. A single standard test for all dental restorative materials is desirable. The shear punch test has previously been used in standards testing and has been suggested as a suitable test for evaluating dental cements (Roydhouse, 1970; Smith & Cooper, 1971). Results of a recent study suggest that this simple test is suitable for standardized testing of different types of direct restorative materials (Nomoto, Carrick & McCabe, 2001). Composite and amalgam gave the highest shear punch strength values, while glass ionomers and polycarboxylate provided the lowest. Polyacid-modified composite and resin-modified glass ionomer materials gave intermediate values of shear punch strength. The rank order obtained with the shear punch test was consistent with what is known about the clinical performance of the different material types (Nomoto & others, 2001). This study investigated the effects of food-simulating liquids on the shear-punch strength of composites and polyacid-modified composites, including a recently introduced posterior polyacid-modified composite material. Inter-material comparison after conditioning in the various mediums was also performed.

METHODS AND MATERIALS

Two resin composites (Tetric Ceram [TC], Vivadent, Schaan, Liechtenstein; Esthet X [EX], Dentsply, Konstanz, Germany), a conventional (Compoglass [CG], Vivadent) and a posterior polyacid-modified (Dyract Posterior [DP], Dentsply) composite were selected for the study. Table 1 shows the technical profiles of the materials and their manufacturers. All materials were of the A2 shade. Shear punch specimens were made by placing the restorative materials in stainless steel washers (18-mm outer diameter, 10-mm inner diameter and 1.6-mm thickness) supported by glass slides. A second glass slide was placed on top of the washers and gentle pressure was applied to extrude excess material. The top and bottom surfaces of the specimens were then cured using a Max polymerization unit (Dentsply/Caulk, Milford DE, USA) with a light exit window of 13 mm according to manufacturers' cure times. The mean intensity of the light source (410 ± 4 mW/cm^2) was determined with a commercial radiometer (CureRite, EFOS Inc, Ontario Canada) prior to starting the experiment. Thirty-two specimens of each material were made and randomly divided into four groups of eight. The specimens, together with their washers, were conditioned for one week as follows—Group 1 (control): air at 37°C; Group 2: distilled water at 37°C; Group 3: 75% ethanol-water solution at 37°C and Group 4: heptane at 37°C.

At the end of the conditioning period, the shear punch strength of the materials was assessed. A special shear punch apparatus was designed and fabricated in collaboration with Instron, Singapore. The apparatus (Figure 1) allowed for restraining the specimens during testing, self-location of the specimens and testing in a thermally-controlled liquid environment. Shear punch strength testing was conducted using an Instron Uniaxial testing machine (Model 4302, Instron Corp, Canton, MA, USA) at a crosshead speed of 0.5 mm/minute under water at room temperature. Prior to testing, the shear punch apparatus was aligned to the loading axis of the Instron machine to ensure minimal

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frictional force compared to the value of force required to fracture the test specimens. The thickness of each specimen was measured with a digital vernier calliper prior to placement in the shear punch apparatus. The specimens were positioned in the apparatus by means of a self-locating recess that provided a snug-fit, with the washers holding the specimens. The specimens were subsequently restrained by tightening the screw clamp on top of the specimens. An M2 tool steel punch with a flat end (3.2-mm diameter) was used to create shear force by sliding through a punch hole and having minimal clearance. A small disc from the center of each specimen was punched out and the maximum load recorded. Shear strength was then computed using the following formula:

$$\text{Shear strength (MPa)} = \frac{\text{Force (N)}}{\pi \times \text{Punch diameter (mm)} \times \text{Thickness of specimen (mm)}}$$

All statistical analysis was carried out at significance level 0.05. The interaction between materials and conditioning mediums was evaluated using two-way ANOVA. One-way ANOVA and Scheffe's post-hoc tests were used to determine inter-medium and inter-material differences.

RESULTS

The mean shear strength of the materials after conditioning in the various mediums is shown in Table 2 and Figure 2. Results of the statistical analyses are shown in Tables 3 and 4.

Two-way ANOVA revealed significant interaction between materials and conditioning mediums. The effect of conditioning mediums on shear strength was therefore material dependent. The shear strength of TC and CG were significantly reduced after conditioning in ethanol solution. For DP, a significant increase in shear strength was observed after conditioning in water. Conditioning in ethanol solution resulted in significantly lower shear punch strength values compared to conditioning in water. No significant difference in shear strength was observed between Group 1 (air) and Group 3 (ethanol solution) specimens for DP. For EX, no significant difference in shear strength was observed among the four treatment groups.

The shear strengths of EX, TC and CG were significantly greater than DP after conditioning in air (Group 1). In addition, EX was significantly stronger than TC. When conditioned in water (Group 2) and ethanol solution (Group 3), the shear strength of EX was signifi-

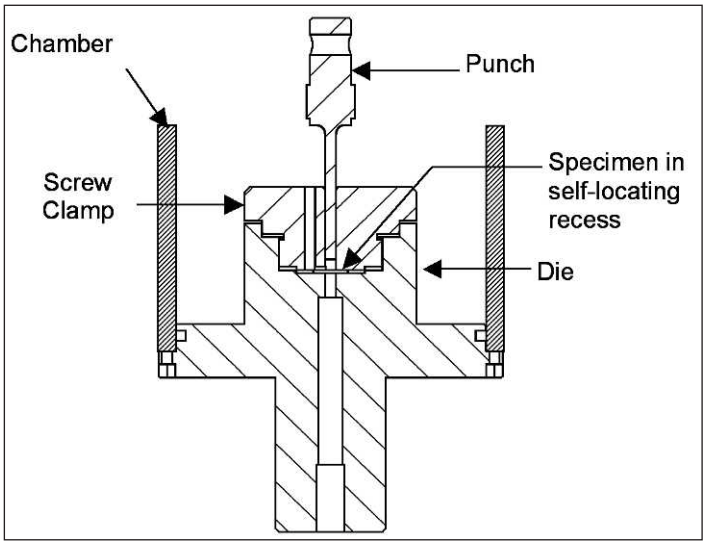

Figure 1. *Diagrammatic representation of the shear punch apparatus.*

Table 1: *Technical Profiles and Manufacturers of the Materials Evaluated*

Material (Lot #)	Cure Time	Resin Matrix	Filler Type	Filler Size (μm)	Filler Content (% Vol)
Tetric Ceram (TC) (D54267)	40 seconds	BisGMA, UDMA, TEGDMA	Barium glass, Ytterbium trifluoride, Ba-Al-fluorosilicate, Silicon dioxide	0.04—1.0	60
Esthet X (EX) (0108102)	20 seconds	BisGMA, BisEMA, TEGDMA	Ba-Al-fluorosilicate, Silicon dioxide	0.04—1.0	60
Compoglass (CG) (D51370)	40 seconds	UDMA, PEGDMA, DCDMA	Ytterbium trifluoride, Ba-Al-fluorosilicate	1.0 (mean)	55
Dyract Posterior (DP) (0107001376)	40 seconds	UDMA, TCB	Strontium-fluoro-silicate, strontium fluoride	0.8 (mean)	47

BisEMA=Ethoxylated bisphenol-A-glycidyl methacrylate
BisGMA=Bisphenol-A-dimethacrylate
DCDMA=Cycloaliphatic dicarbonic acid dimethacrylate
PEGDMA=Polyethylene glycoldimethacrylate
TEDGMA=Triethylene glycol dimethacrylate
TCB=Reaction product butane tetracarboxylic acid and HEMA
UMDA=Urethane dimethacrylate

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cantly greater than TC and DP. The shear strength of EX was also significantly greater than CG after conditioning in ethanol solution. EX and CG were significantly stronger than DP after conditioning in heptane.

DISCUSSION

The food-simulating liquids used for conditioning the materials were among those recommended in the FDA guidelines as food stimulants (Food and Drug Administration, 1976). Heptane simulates butter, fatty meats and vegetable oils, while the ethanol solution simulates certain beverages, including alcohol, vegetables, fruits, candy and syrup. Distilled water was included to simulate the wet oral environment provided by saliva and water. The solubility parameters for these organic food simulating liquids were 1.5×10^{-4}, 3.1×10^{-4} and 4.8×10^{-4} $J^{1/2}m^{-3/2}$ for heptane, 75% ethanol solution and water, respectively. A 75% ethanol solution was selected, as this concentration of ethanol has been shown to cause maximum softening of BisGMA-based composite materials (Kao, 1989; McKinney & Wu, 1985). In addition, 75% ethanol solution is commonly used to study the effects of solvent and environmental degradation on material properties in dental research (Ferracane & Marker, 1992; Lee & others, 1994; Lim & others, 2001; Yap & others, 2002b). As occlusal and incisal forces during mastication and parafunction induce shear stresses in teeth and restorations, the shear punch test reflects qualities of clinical significance (Roydhouse, 1970).

One of the main advantages of shear punch testing is the simplicity of specimen preparation. Results of flexural, diametral and compression testing are highly dependent on the production of high quality specimens. In these tests, the quality of the surfaces and edges of the specimens are critical (Nomoto & others, 2001). The quality of the edges of the disc around the circumference, however, has no direct influence on testing outcome for shear punch testing. The only requirement is that the two main faces of the disc are flat and parallel to allow uniform stress distribution around the punch circumference. Specimens were constrained during shear punch testing, as Nomoto and others (2001) reported significantly lower strength values for specimens not restrained with a screw clamp. They hypothesized that unrestrained specimens are able to bend on application of the punch, creating localized stress concentration that leads to premature failure. Although a 15-cm kg increase in screw clamp torque resulted in an increase in shear strength, the increase was not statistically significant. The punch diameter (3.2 mm) employed in the current study was identical to that used by Nomoto and others (2001). Specimen thickness was, however, increased to a clinically more relevant dimension (1.6 mm). The shear punch apparatus used in this experiment gave highly reproducible results, and the standard deviations observed were generally low (under 10%) despite solvent effects. The aforementioned corroborates a theory by Nomoto and others (2001) that the shear punch test is suitable for standardized testing of different restorative materials.

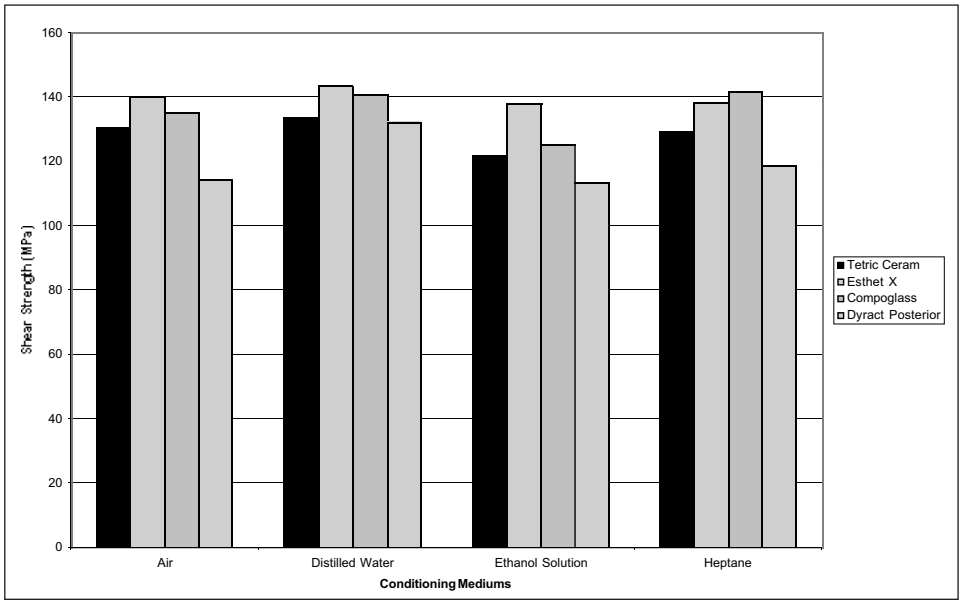

Figure 2. *Mean shear strength of the materials.*

Table 2: *Mean Shear Strength (MPa) of the Various Materials After Conditioning in the Various Mediums*

Materials	Group 1 Air	Group 2 Distilled Water	Group 3 Ethanol Solution	Group 4 Heptane
Tetric Ceram	130.54 (3.57)	133.58 (3.93)	121.84 (6.97)	128.94 (6.83)
Esthet X	139.95 (3.14)	143.41 (5.70)	138.01 (3.867)	138.22 (3.33)
Compoglass	135.12 (1.43)	140.81 (2.58)	125.38 (9.21)	141.61 (5.48)
Dyract Posterior	114.55 (8.14)	132.39 (8.78)	113.52 (11.15)	118.67 (14.43)

Standard deviations indicated in parentheses.

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The effect of food-simulating liquids on shear strength was found to be material dependent. TC and CG were significantly weakened after conditioning in ethanol solution. The significant decrease in shear strength values of TC may be attributed to the fact that 75% ethanol solution has a solubility parameter value approximating that of BisGMA used in TC (McKinney & Wu, 1985). Maximum softening occurred (Kao; 1989; McKinney & Wu, 1985), resulting in the lower shear strength observed. The significant decrease in shear strength of CG could also be attributed to the softening effect of 75% ethanol solution on UDMA (Kao, 1989). For EX and DP, no significant difference in shear strength was observed between conditioning in air (control) and ethanol solution. EX is both BisGMA and BisEMA-based. BisEMA, which is an ethoxylated version of BisGMA, is highly hydrophobic and may make EX more resistant to the softening effect of ethanol-water solution. This finding was consistent with that of Yap and others (2000a), who found that BisEMA-based composites were highly resistant to the degradation effect of food-simulating liquids including ethanol. BisEMA is hydrophobic, as it does not contain any unreacted hydroxyl groups on the main polymer chain (Ruyter & Nilsen, 1993). Although no significance in strength was observed between DP specimens conditioned in air and ethanol, significant differences were observed between DP specimens conditioned in water and ethanol. The strength of DP was significantly increased by water storage. This increase in strength is probably due to the acid-base reaction between the hydrated acidic functional groups and fluoroalumino silicate glasses present in DP. The acid-base reaction, however, occurs only at the surface layers (uppermost 100 µm layers) of the material and reaches a saturation point only after four weeks (Eliades, Kakaboura & Palaghias, 1998). In view of the latter, a further increase in shear strength is anticipated with the extension of water storage time. The shear strength of all materials was not significantly affected by heptane.

Table 3: *Results of Statistical Analysis Based on Materials*

Materials	Differences
Tetric Ceram	Air, Water > Ethanol solution
Esthet X	NS
Compoglass	Air, Water, Heptane > Ethanol solution
Dyract Posterior	Water > Air, Ethanol solution

Results of one-way ANOVA and Scheffe's test (p<0.05); > indicates statistical significance while NS indicates no statistical significance

Table 4: *Results of Statistical Analysis Based on Conditioning Mediums*

Mediums	Differences
Group 1 Air	EX, CG, TC > DP; EX > TC
Group 2 Distilled Water	EX > TC & DP
Group 3 Ethanol solution	EX > CG, TC & DP
Group 4 Heptane	EX, CG > DP

Results on one-way ANOVA and Scheffe's test (p<0.05); > indicates statistical significance while NS indicates no statistical significance

For the control group, significant differences in shear strength between materials could be explained in part by filler volume. Studies have reported a positive correlation between the mechanical properties and volume fraction of fillers (Chung & Greener, 1990; Kim & others, 1994; Yap & others, 2002a). Materials with higher filler volumes like EX, TC and CG are therefore expected to have higher shear strength than DP, which had a lower filler volume. Although EX and TC had identical filler volumes, EX was significantly stronger than TC. This is probably due to the differences in resin matrix formulation. The shear strength of EX remained significantly higher than DP after conditioning in water, ethanol and heptane. Despite its high filler volume, TC was not significantly stronger than DP after conditioning in the aforementioned food-simulating liquids. This may be due in part to the increase in strength of DP after conditioning in water and a slight weakening of TC after conditioning in ethanol and heptane. EX was most resistant to the degradation effect of alcohol. As EX was significantly stronger than all materials after conditioning in ethanol, it may be the composite of choice for the restoration of posterior teeth in patients who consume alcohol frequently and in large quantities. Differences in shear strength between materials after conditioning in heptane were similar to that of the control group with the exception of TC. The latter was due to a slight decrease in strength of TC after conditioning in heptane and a slight increase in strength of DP. Since the posterior polyacid-modified composite (DP) was no stronger than the conventional material (CG), its use for the restoration of posterior teeth should be handled with caution. The clinical success of materials cannot be determined by shear strength alone. Other factors, such as wear, water sorption and bonding to tooth structure are equally important. The shear punch test, however, offers a simple, effective and reliable means of screening resin-based filling materials and evaluating material-environment interactions.

CONCLUSIONS

Under the conditions of this *in vitro* study:

1. The effect of food-simulating liquids on shear strength of composites and polyacid-modified composites was material dependent.
2. With the exception of Esthet X, the shear strength of all materials was significantly reduced after exposure to ethanol solution.

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3. The shear strength of Dyract Posterior was significantly increased after storage in water.
4. The shear punch test is a simple, reliable method for testing resin-based filling materials.

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Mechanical Properties of Luting Cements After Water Storage

A Piwowarczyk • H-C Lauer

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Clinical Relevance

Luting cements differ considerably with respect to their mechanical properties. These differences should be taken into consideration when selecting a material for clinical application.

SUMMARY

This study determined the effect of water storage on flexural strength (FS) and compressive strength (CS) of 12 luting cements from different material classes. In addition, the influence of the curing method on the mechanical properties was investigated. The materials examined were two zinc phosphate cements (Harvard cement and Fleck's zinc cement), two glass ionomer cements (Fuji I and Ketac-Cem), three resin-modified glass ionomer cements (Fuji Plus, Fuji Cem and RelyX Luting), four resin cements (RelyX ARC, Panavia F, Variolink II and Compolute) and one self-adhesive universal resin cement (RelyX Unicem). The samples were prepared and tested according to ISO specifications. Specimens for FS and CS were loaded to fracture at a constant crosshead speed of 1 mm/minute. The mechanical properties were measured after the materials were stored in distilled water at a temperature of 37.0 ± 1.0°C for 24 hours and 150 days after mixing. In a one-way ANOVA, multiple mean value comparisons using Duncan's multiple comparison tests were performed. Resin cements had the highest flexural and compressive strengths, followed by self-adhesive universal resin cement. These materials were statistically stronger than resin-modified glass ionomer cements, glass ionomer cements and zinc phosphate cements.

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INTRODUCTION

Dental luting cements form the link between a fixed restoration and the supporting tooth structure. Numerous types of commercial luting cements are currently available for long-term cementation. Traditionally, zinc phosphate cement has been the most popular dental cement and has been in use for more than 90 years (Ames, 1892). Zinc phosphate cements are not adhesive to tooth substance or to restorative materials. The mixing technique is critical in developing the optimal cement, because the strength is almost linearly dependent on the powder:liquid ratio (Diaz-Arnold, Vargas & Haselton, 1999). Glass ionomer restorative materials have been available since the early 1970s (Wilson & Kent, 1971) and were derived from silicate cements and polycarboxylate cements. The solubility of these materials can be adversely affected by early moisture contamination (Curtis, Richards & Meiers, 1993). So, it is essential that cement margins be covered with a layer of protective

varnish immediately after seating the restoration. Glass ionomer cements (GIC) have been gaining in popularity because of fluoride release that may prevent recurrent secondary caries (Muzynski & others, 1988). In addition, the clinical success of glass ionomer cement as a luting agent has been well-documented (Metz & Brackett, 1994). Resin-modified glass ionomer cements (RMGICs) harden by an acid-base reaction between fluoroaluminosilicate glass powder and an aqueous solution of polyalkenoic acids (Diaz-Arnold & others, 1999). These materials have greater compressive and diametral tensile strengths compared to zinc phosphate and some glass ionomers (White & Yu, 1993a; Mathis & Ferracane, 1989). RMGICs have been shown to exhibit high water absorption, which has been related to the presence of polyHEMA (Poly-Hydroxy-ethyl-methacrylat) and unconverted monomer in the set cement (Kanchanavasita, Anstice & Pearson, 1997). Resin cements are variations of filled Bis-GMA and other methacrylates. They exhibit high mechanical properties and have excellent esthetic shade-matching potential (Li & White, 1999). On the other hand, they require skillful usage and multiple steps with respect to their handling characteristics, especially time-consuming bonding procedures and excess removal. The self-adhesive universal resin cement represents a new type of luting cement. It is claimed that this cement combines the advantages of glass ionomer cements and resin cements. It shows self-adhesion to tooth structure without a conditioning and/or bonding step and exhibits high mechanical values (Piwowarczyk & others, 2002) and excellent esthetic properties.

Clinically, the tooth-cement interface can withstand masticatory and parafunctional stresses for many years in a warm, wet oral environment. Stress may cause recoverable elastic or permanent plastic deformation, marginal fracture or discoloration of the cement and secondary caries, all of which impairs the longevity of the restoration (Smales & Webster, 1993). When exposed to water or saliva, hydrolytic degradation is likely to occur and could be one of the reasons for submargination. Söderholm (1981) reported that water enhances the surface deterioration of polymer resin materials because of water-matrix interactions and/or filler debonding.

As the prognosis for prosthetic restorations is largely a function of maintenance of the luting cement and the adhesive bond (Milleding, Örtengren & Karlsson, 1995), and as moisture and water interact with both, it seems relevant to investigate the effect of water on the mechanical properties of luting cements. This study analyzed the influence that water has on three-point flexural strength (FS) and compressive strength (CS) after 24 hours and 150 days. It also investigated the effect of the curing method on mechanical properties.

METHODS AND MATERIALS

Twelve luting cements from various material classes were investigated: two zinc phosphate cements (Harvard cement and Fleck's zinc cement), two glass ionomer cements (Fuji I and Ketac-Cem), three resin-modified glass ionomer cements (Fuji Plus, Fuji Cem and RelyX Luting), four dual-cure resin cements (RelyX ARC, Panavia F, Variolink II and Compolute) and one dual-cure self-adhesive universal resin cement (RelyX Unicem) (Table 1). Handling of the materials was carried out according to the manufacturers' directions at a temperature of 23.0 ± 1.0°C and a relative humidity of 50 ± 5%. Four materials (Ketac-Cem, Fuji Plus, Compolute and RelyX Unicem) were supplied in predosed capsules. Upon activation, these materials were mechanically triturated with a rotational mixing machine (RotoMix; 3M ESPE, Seefeld, Germany) for the time recommended by the manufacturers (8 or 10 seconds). Fuji Cem and RelyX ARC were supplied in pre-measured delivery systems. Panavia F and Variolink II were mixed with a hard plastic spatula in a ratio of 1:1 base to catalyst paste on a mixing block for approximately 20 and 10 seconds, respectively. The powder:liquid ratio of Harvard cement and Fleck's zinc cement was weighed according to the manufacturer's instructions using an analytical balance (± 1 mg). The mixing technique was completed on a cool slab, over a wide area, to incorporate small increments of powder into the liquid for approximately 90 seconds (Harvard cement) and 120 seconds (Fleck's zinc cement).

To evaluate the influence of the curing method, the resin cements and the self-adhesive universal resin cement were polymerized using a dental light-curing unit (Elipar Trilight; 3M ESPE, Seefeld, Germany). Output of this curing light was checked to ensure adequate curing with a measuring device (Cure Rite, LD Caulk, Milford, DE, USA) prior to curing each group of cements.

Measurements were performed at 24 hours and 150 days following the first mixing. These measurements were performed on 10 samples at each occasion.

Three-point Flexural Strength (FS)

The test bars were prepared in a split stainless steel precision mold (length = 25 mm, height = 2 mm, width 2 = mm) according to ISO 4049 (International Standards Organization, 2000a). To prevent adhesion of the cement, molds were lubricated prior to filling, using 3% Luwax solved in Hexan. The mold was placed on a glass slab and freshly mixed cement was applied until the mold was slightly overfilled. Then, a transparent matrix band was placed on top of the mold and a second glass slab was pressed on it. Specimens of

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light-activated materials (resin cements and the self-adhesive universal resin cement) were cured through the glass slab from the top and the bottom of the specimen following International Organization for Standardization (ISO), 2000a, #4049. Each half of the test bar was polymerized on both sides with a curing light (Elipar Trilight) with overlapping irradiation. In accordance with the manufacturers' instructions, RelyX ARC, Variolink II and Compolute were light cured for 40 seconds and Panavia F and RelyX Unicem for 20 seconds. After the mold and the glass slabs were put in the screw clamp and tightened, all specimens were placed into an incubator (Memmert model 200, Schwabach, Germany) at 37° ± 1°C and 95% to 100% relative humidity for three minutes after first mixing. After 60 minutes the molds were removed and separated. Specimens were checked visually for air entrapments or chipped edges. Defective specimens were discarded. Appropriate test bars were stored in distilled water (37° ± 1°C). No surface protection was used on any of the materials.

Three-point flexural strength tests were carried out on the test bar at a span of 20 mm and a constant crosshead speed of 1 mm/minute using a universal testing machine (Zwick 1435, Ulm, Germany) and a 50 N force-measuring device (Type: KAP-S; Angewandte System-Technik, Dresden, Germany). The dimensions of the samples were measured with an electronic digital caliper accurate to ± 10 µm. FS was calculated according to the following formula in Megapascal (MPa) (ISO, 2000b, #4049): $FS = 3\ F\ L / (2\ b\ d^2)$, where F is the maximum force (N), L is the distance between the layers (mm), b is the width of the specimen (mm) and h is the height of the specimen (mm).

Compressive Strength (CS)

The specimens were prepared in split cylindrical rigid plastic molds 6 mm in height and 4 mm in diameter, which corresponds to ISO 9917 (ISO, 2000b). CS was determined in a manner similar to the three-point flexural strength test above. Light-curing the cements was carried out on each side of the mold with a curing light according to the manufacturers' instructions. The samples were stored in distilled water at 37° ± 1°C until testing, which occurred 24 hours and 150 days after mixing. The wet specimens were placed with the flat ends vertically between the platens of a universal testing machine and the failure load was recorded. The determination of CS was measured with a universal testing machine at a constant crosshead speed of 1 mm/minute using a 5 kN force-measuring device (Type: U2A, Hottinger Baldwin Messtechnik GmbH,

Table 1: *Luting Cements and Their Characteristics*

Material	Manufacturer	Classification	Mixing Process (seconds)	Powder/Liquid Ratio (g)	Batch #
Harvard Cement	Richter & Hoffmann, Berlin, Germany	zinc phosphate cement	manual (90 seconds)	1.5:1.0	Powder 2112400005 Liquid 21111000006
Fleck's Zinc Cement	Mizzy Inc, Cherry Hill, NJ, USA	zinc phosphate cement	manual (120 seconds)	0.4:0.26	Powder P67 Liquid L49
Fuji I	GC Corp, Tokyo, Japan	glass ionomer cement	manual (20 seconds)	1.8:1.0	0001261
Ketac-Cem	3M ESPE, Seefeld, Germany	glass ionomer cement	mechanical (8 seconds)*	Capsules	106291
Fuji Plus	GC Corp, Tokyo, Japan	resin-modified glass ionomer cement	mechanical (10 seconds)*	Capsules	0012042
Fuji Cem	GC Corp, Tokyo, Japan	resin-modified glass ionomer cement	manual (10 seconds)	Cartridge system	0012012
RelyX Luting	3M ESPE, Seefeld, Germany	resin-modified glass ionomer cement	manual (30 seconds)	1.6:1.0	20000627
RelyX ARC	3M ESPE, Seefeld, Germany	dual-cure resin cement	manual (10 seconds)	Pre-dosed delivery system	20010627
Panavia F	Kuraray, Osaka, Japan	dual-cure resin cement	manual (20 seconds)	1.0:1.0	Base 038AA Catalyst 015AA
Variolink II	IvoclarVivadent, Schaan, Liechtenstein	dual-cure resin cement	manual (10 seconds)	1.0:1.0	Base D51333 Catalyst D51333
Compolute	3M ESPE, Seefeld, Germany	dual-cure resin cement	Mechanical (8 seconds)*	Capsules	106595
RelyX Unicem	3M ESPE, Seefeld, Germany	dual-cure self-adhesive universal resin cement	mechanical (10 seconds)*	Capsules	CM611

**Rotational mixing machine (RotoMix; 3M ESPE, Seefeld, Germany).*

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Darmstadt, Germany). The CS values were computed as follows: $CS = 4F/\pi d^2$, where F is the maximum force (N), d is the specimen diameter (mm) and L is the specimen length (mm).

The statistical analysis of the data was performed after checking measured values for normal distribution with the Kolmogorov-Smirnov tests and calculating the mean value and the corresponding standard deviation for each material. In a one-way ANOVA, comparisons of multiple means were made using Duncan's multiple comparison tests. The level of significance for the differences was determined to be $p \leq 0.05$. As the data were metrically scaled and could be shown to be normally distributed, the prerequisites for a *t*-test for dependent samples existed.

RESULTS

The arithmetic mean values and standard deviations of flexural strength data were reported in Table 2 and Figure 1 and compressive strength in Table 3 and Figure 2. The statistical analysis demonstrated significant differences between luting cements with regard to measuring time.

Harvard cement, Fleck's zinc cement, Fuji I, Ketac-Cem and Fuji Cem exhibited the lowest and Variolink II self-cured and light-cured the highest three-point flexural strength after 24 hours and 150 days (Figure 1). When the 24-hour and 150-day results were compared, only Panavia F and RelyX Unicem light-cured had significant ($p \leq 0.05$) decreases in flexural strength. Fuji I, Ketac-Cem and RelyX Luting increased significantly ($p \geq 0.05$). Among the other materials, no statistical significant differences ($p \leq 0.05$) were established using the paired *t*-Test with respect to the two times of measurement (Table 2).

For compressive strength, the one-way ANOVA showed that after 24 hours and 150 days, Fleck's zinc cement and Ketac-Cem were the weakest cements, followed by RelyX Luting, Harvard cement and Fuji Cem. Variolink II (self-cured and light-cured) reached the highest measured values of all materials examined (Figure 2). Comparing the 24-hour and 150=day results, Fuji Cem, RelyX Luting, RelyX ARC self-cured and Variolink II (self-cured and light-cured) had a significant ($p \geq 0.05$) increase in strength, only RelyX Unicem light-cured decreased significantly (Table 3).

By comparing the polymerization mechanism of the dual-cure cements, it became apparent that light curing could not generally enhance the mechanical properties significantly after 150 days of water storage.

Table 2: *Three-Point Flexural Strength [Mean (MPa) and Standard Deviation]*

Material	24 hours	150 days
Harvard cement	14.9 ± 1.8 (a)	15.2 ± 2.3 (a)
Fleck's zinc cement	10.4 ± 2.5 (a)	8.4 ± 1.7 (a)
Fuji I	10.5 ± 4.3 (a)	13.3 ± 4.8 (b)
Ketac-Cem	6.3 ± 2.4 (a)	11.6 ± 4.8 (b)
Fuji Plus	27.6 ± 5.4 (a)	33.0 ± 9.0 (a)
Fuji Cem	13.6 ± 2.5 (a)	15.5 ± 3.5 (a)
RelyX Luting	22.1 ±4.3 (a)	30.2 ± 9.4 (b)
RelyX ARC	86.7 ± 8.8 (a)	81.2 ± 10.0 (a)
RelyX ARC light-cured	102.7 ± 15.7 (a)	90.6 ± 15.4 (a)
Panavia F	91.3 ± 9.7 (a)	76.4 ± 14.7 (b)
Panvavia F light-cured	82.2 ± 13.0 (a)	77.9 ± 8.8 (a)
Variolink II	100.9 ± 10.8 (a)	101.2 ± 12.3 (a)
Variolink II light-cured	105.5 ± 17.9 (a)	103.3 ± 11.2 (a)
Compolute	91.9 ± 9.3 (a)	86.1 ± 11.6 (a)
Compolute light-cured	100.1 ± 11.8 (a)	95.4 ± 10.2 (a)
RelyX Unicem	49.6 ± 7.8 (a)	53.6 ± 7.8 8 (a)
RelyX Unicem light-cured	63.0 ± 7.2 (a)	56.3 ± 8.1 (b)

**Means followed by distinct letters are significantly different (p≤ 0.05) groups, representing time differences (horizontal comparisons).*

Table 3: *Compressive Strength [Mean (Mpa) and Standard Deviation*

Material	24 hours	150 days
Harvard cement	103.1 ± 21.5 (a)	107.2 ± 26.6 (a)
Fleck's zinc cement	56.6 ± 14.4 (a)	64.5 ± 19.9 (a)
Fuji I	129.1 ± 11.8 (a)	123.0 ± 20.8 (a)
Ketac-Cem	78.9 ± 12.9 (a)	87.4 ± 16.0 (a)
Fuji Plus	129.7 ± 12.0 (a)	141.9 ± 13.3 (a)
Fuji Cem	95.9 ± 10.5 (a)	115.4 ± 13.8 (b)
RelyX Luting	90.3 ± 4.2 (a)	106.6 ± 15.2 (b)
RelyX ARC	263.8 ± 29.0 (a)	286.5 ± 18.7 (b)
RelyX ARC light-cured	284.5 ± 17.7 (a)	288.9 ± 16.9 (a)
Panavia F	250.7 ± 35.9 (a)	259.3 ± 10.4 (a)
Panvavia F light-cured	244.2 ± 25.5 (a)	257.8 ± 28.1 (a)
Variolink II	284.0 ± 22.7 (a)	318.3 ± 19.2 (b)
Variolink II light-cured	303.5 ± 32.1 (a)	325.8 ± 11.5 (b)
Compolute	266.3 ± 13.6 (a)	269.3 ± 19.1 (a)
Compolute light-cured	257.8 ± 20.1 (a)	249.1 ± 22.0 (a)
RelyX Unicem	198.3 ± 16.5 (a)	201.8 ± 13.8 (a)
RelyX Unicem light-cured	240.6 ± 9.4 (a)	194.5 ± 23.3 (b)

**Means followed by distinct letters are significantly different (p≤ 0.05) groups, representing time differences (horizontal comparisons).*

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However, only light curing of RelyX ARC and Compolute showed a significant increase in flexural strength (Figures 1 and 2).

DISCUSSION

Considerable differences in flexural and compressive strengths were recorded among the tested luting cements (Figures 1 and 2). Results of the mechanical tests only apply to the specific conditions in this study. Such conditions included the experimental variables of specimen fabrication procedures, specimen geometries, storage times, storage environments, test configurations and temperatures (Oilo & Orstavik, 1985). All specimens were treated identically throughout the study, which was based on ISO 4049 and 9917 (ISO 2000a,b).

The results of the three-point flexural strength tests demonstrated a significant variation (6.3 MPa to 105.5 MPa) of the 12 materials. Generally, the luting cements followed a trend, with zinc phosphate cements, glass ionomer cements and resin-modified glass ionomer cements having the lowest flexural strength, followed by the self-adhesive universal resin cement and the resin cements. These findings are in accordance with previous studies that ranked resin cements stronger than resin-modified glass ionomer cements or glass ionomer cements (Knobloch & others, 2000; Li & White, 1999; Rosenstiel, Land & Crispin, 1998; Li & others, 1996; Miyazaki, Moore & Onose, 1996).

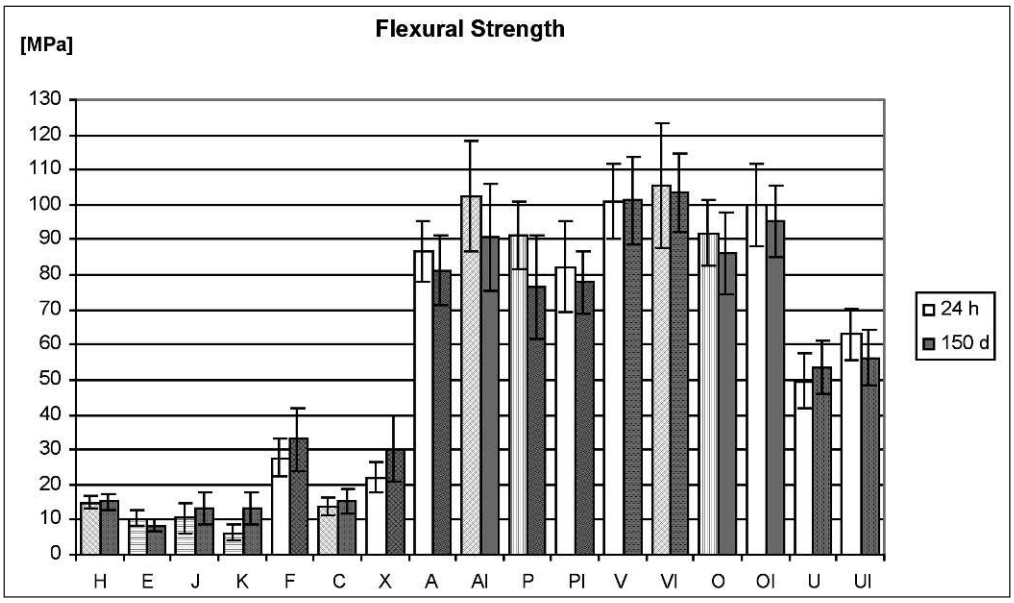

Figure 1. *Three-point flexural strength (ISO 4049) of various luting cements (24 hours and 150 days after first mixing; n ≥10 measurements for each material and time of measurement). H = Harvard cement, E = Fleck's zinc cement, J = Fuji I, K = Ketac-Cem, F = Fuji Plus, C = Fuji Cem, X = RelyX Luting, A = RelyX ARC, P = Panavia F, V = Variolink II, O = Compolute, U = RelyX Unicem; l = light-cured.*
Identical patterns within the bar columns indicate groups with no significant differences (p≤0.05) between the tested materials at a given time of measurement.

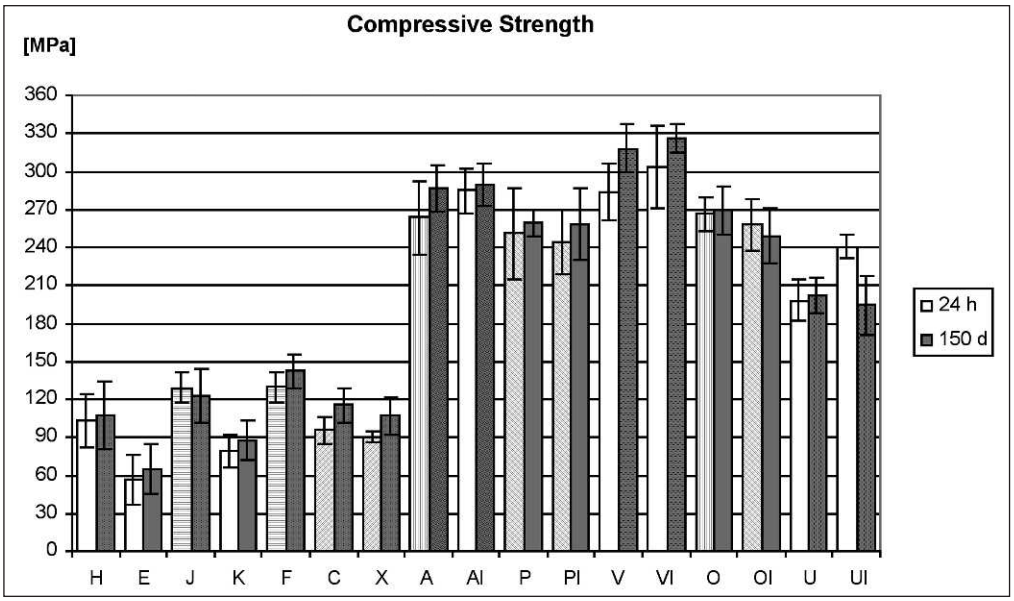

Figure 2. *Compressive strength of various luting cements (24 hours and 150 days after first mixing; n≥ 10 measurements for each material and time of measurement). H = Harvard cement, E = Fleck's zinc cement, J = Fuji I, K = Ketac-Cem, F = Fuji Plus, C = Fuji Cem, X = RelyX Luting, A = RelyX ARC, P = Panavia F, V = Variolink II, O = Compolute, U = RelyX Unicem; l = light-cured.*
Identical patterns within the bar columns designate groups with no significant differences (p≤ 0.05) between the tested materials at a given time of measurement.

Resin cements were up to 10x stronger in flexural strength than zinc phosphate cements, glass ionomer cements and one resin-modified glass ionomer cement (Fuji Cem). The resin-modified glass ionomer cements Fuji Plus and RelyX Luting revealed two times higher results compared to the latter group with low flexural strength values. The high mechanical properties of resin cements are primarily influenced by type and

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composition of the resin matrix, filler type and filler load. The filler particles incorporated into the matrix provide much better mechanical properties than the matrix itself. The study by White and Yu (1993b) suggested that filler content is related to compressive strength, diametral tensile strength and resistance to indentation. The goal of developing the self-adhesive universal resin cement (RelyX Unicem) was to combine the easy handling and non-pre-treatment steps of glass ionomer cements with the high mechanical properties of resin cements. This study found that the flexural strength of this cement achieved lower results (up to 0.6x) compared to resin cements. On the other hand, the self-adhesive universal resin cement was up to 4.5 times stronger than zinc phosphate cements, glass ionomer cements and one resin-modified glass ionomer cement (Fuji Cem).

The material ranking for the compressive strength testing was similar to the flexural strength rankings. Overall conclusions can be drawn regarding the cements on the basis of their material class. In general, compressive strength was lowest for phosphate cements, glass ionomer cements and resin-modified glass ionomer cements, higher for self-adhesive universal resin cement and highest for resin cements.

Compressive strength has been used as a predictor of clinical performance (Yettram, Wright & Pickard, 1976) because a high compressive strength is necessary to resist masticatory forces, although the exact value is unknown. The American National Standards Institute/American Dental Association (ANSI/ADA) specification No 96 (ISO, 2000b) for dental water-based cements requires a minimum compressive strength of 70 MPa at 24 hours. At the first measuring time after 24 hours, only Fleck's zinc cement with a result of 56.6 MPa did not reach this standard. The other luting cements tested were over the required limit of 70 MPa.

The compressive strength of glass ionomer cements and resin-modified glass ionomer cements, in particular, continues to increase over several weeks. This continued increase is thought to be due to reconstruction of the silicate network (Matsuya, Maeda & Ohta, 1996). By comparing the results of the measurements in this study after 24 hours and 150 days, a slight increase in compressive strength was found for four of five glass ionomer and resin-modified glass ionomer cements. After 150 days water storage, these cements achieved results ranging from 87.4 MPa for Ketac-Cem and 141.9 MPa for Fuji Plus, respectively. The compressive strength of the self-adhesive universal resin cement ranged between 198.3 MPa and 240.6 MPa, lower than the results of the resin cements (244.2 MPa to 325.8 MPa). It is unclear as to how valuable compressive strength data are in selecting a clinical material, especially when choosing among different types of luting cements. However, a correlation between high strength and low margin wear of resin cements has been demonstrated *in vitro* (Peutzfeldt, 1995).

Comparing the dual-cure cements when they were cured under manufacturer's instructions with the appropriate visible light source, and under conditions where the curing light was excluded and curing was reliant on the chemical reaction only, revealed mainly no significant differences. The flexural strength did not improve more than 16 MPa for RelyX ARC after 24 hours and 9.4 MPa for RelyX ARC after 150 days between all dual and chemically cured specimens. The highest enhancement after light-curing the dual-cure cements with regard to the compressive strength was after 24 hours 19.5 MPa (Variolink II) and after 150 days 6.7 MPa (Variolink II). These small differences after 150 days of water storage could be explained by the light-curing process of dual-cure cements having a marked effect in the initial 30 minutes, but after this period, samples provided by photo initiation or chemical reaction advanced at very similar rates (Darr & Jacobsen, 1995).

Conflicting reports have been published on the effects of water storage on mechanical properties. The results of a study by Ortengren and others (2000) indicated that water has an important effect on flexural properties of a resin composite cement (Variolink; IvoclarVivadent, Schaan, Liechtenstein) after 60 days storage time. Cattani-Lorente and others (1999) observed a decrease in flexural strength of five resin-modified glass ionomer cements immersed in water. A correlation was established between the decrease in their physical properties and the water uptake. In contrast, McCabe (1998) found that water storage produced a small initial decrease in strength but had little long-term effects. Similarly, this study observed that water storage did not influence the results of the compressive strength of nearly all tested materials. Looking at the three-point flexural strength test, a slight decrease in the measurements of the resin cements and the self-adhesive universal resin cement usually resulted after 150 days water storage. Water uptake in resin composite materials has been shown to be a slow process at 37°C, and several months must pass before water reaches the central parts of the sample (Øysæd & Ruyter, 1986). A factor that probably influences mechanical properties, at least in the long run, is stress corrosion of the filler particles, which takes place in water-stored composite materials (Øysæd & Ruyter, 1986; Söderholm, 1983). On the other hand, the current study revealed a small increase in flexural strength in the glass ionomer and resin-modified glass ionomer cements after 150 days. However, as pointed out by Anstice and Nicholson (1992), changes that were observed to occur in distilled water would not necessarily take place in an oral environment.

Other authors have examined some materials in artificial saliva as a storage medium (de Gee & others,

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1996; Söderholm, Mukherjee & Longmate, 1996). Kanchanavasita, Anstice and Pearson (1998) investigated the effects of long-term storage of up to one year for resin-modified glass ionomer cements in two storage media. This study showed that specimens aged in distilled water and artificial saliva had similar flexural strength values.

When discussing the clinical implications of the results of this study, one must bear in mind that the testing procedure differs in several ways from *in vivo* conditions. However, the great diversity among the material classes of luting cements indicates that mechanical properties need to be proved before selecting a material for clinical use. Flexural and compressive strength are only two criteria for the selection of a luting cement choice, but are crucial. Stronger cements provide more even stress distributions, less probability of failure and greater probability for clinical success. In addition to the mechanical properties, other parameters, such as biocompatibility, post-treatment sensitivity, clinical performance, esthetics and working properties should be taken into consideration when choosing an ideal material for cementation of fixed restorations.

CONCLUSIONS

1. The three-point flexural and compressive strengths of luting cements varied widely.
2. In general, the highest measured values in flexural and compressive strengths were achieved by resin cements. Next came the self-adhesive universal resin cement, followed by significantly lower measured values recorded for resin-modified glass ionomer, glass ionomer and zinc phosphate cements.
3. The resin cement Variolink II demonstrated the highest measured data in flexural and compressive strengths after 24 hours and 150 days.
4. Light curing of the dual-cure cements could not provide an increase in mechanical strengths after 150 days water storage.

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Bonding of Photo and Dual-Cure Adhesives to Root Canal Dentin

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J Tagami • H Miura

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Clinical Relevance

Light exposure of photo and dual-cure adhesives and dual-cure resin composites was necessary for optimum bonding to root canal dentin.

SUMMARY

This study evaluated the regional tensile bond strength of a dual-cure resin composite core material to root canal dentin using photo and dual-cure adhesives and different modes of polymerization. Nineteen extracted premolars were decoronated and their root canals prepared to a depth of 8 mm and a width of 1.4 mm using Para Post drills. For the microtensile bond strength (µTBS) test, 15 roots were randomly divided into five groups and their canal walls treated with a dual-cure self-etching primer (Clearfil Liner Bond 2V Primer, Kuraray Medical Co, Japan). Adhesive resin (Clearfil Liner Bond 2V Bond A) was applied to two of the groups and light cured for 20 seconds. A dual-cure adhesive resin (Clearfil Liner Bond 2V Bond A+B, Kuraray Medical Co, Japan) was applied to the remaining three groups, one of which was light cured. The post spaces of all the groups were filled with a dual-cure resin composite (DC Core) and three were light cured for 60 seconds from a coronal direction. Chemical-cure resin composite was placed on the outer surfaces of the roots, which were then stored in water for 24 hours. They were serially sliced perpendicular to the bonded interface into eight 0.6 mm-thick slabs, then transversely sectioned into beams approximately 8 x 0.6 x 0.6 mm for the µTBS test. All of the failure modes were observed under SEM and analyzed using the Kruskal-Wallis Rank test. For Knoop hardness testing, four specimens were prepared in a similar manner, two were light-cured and the other two chemically-cured. The specimens were longitudinally sectioned into two pieces, and three indentations were made at 100 µm intervals from a coronal to an apical direction in the eight halves. Data were divided into two groups (coronal/apical halves of post space) and analyzed using two-way ANOVA and Scheffe's test ($p<0.05$). For each curing strategy, there were no significant differences in µTBS and Knoop hardness between the coronal and apical regions ($p>0.05$). Light exposure of both the adhesive resin and resin composite resulted in significantly higher µTBS than chemical-cure alone ($p<0.05$). Light exposure also significantly increased Knoop hard-

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ness at both the coronal and apical regions ($p<0.05$). When the bonding resin and dual-cure resin composite were chemically-cured, failures occurred cohesively within the resin. Photo-initiated polymerization of the adhesive resin and dual-cure resin composite was necessary to achieve good bonding to root canal dentin, which was not dependent upon region.

INTRODUCTION

Recently, a new technique has been proposed for the restoration of endodontically-treated teeth, the so-called frictionless bonded post technique (Ferrari & others, 2000a). Fibre posts in combination with adhesive resin cements are used to form the dowel (Ferrari & others, 2000a). Fibre posts are prefabricated and require the post space to be prepared using custom preshaping and finishing drills before being luted with a resin cement (Ferrari, Vichi & García-Godoy, 2000b). Factors, such as extended caries, trauma to an immature tooth, pulpal pathology or iatrogenic causes may result in a root canal that is flared with thin dentinal walls (Lui, 1999). If a fibre post is cemented into a flared canal, it might be difficult to maintain an even thickness of resin cement between the canal wall and the post. The thickness of the cement could be excessive in the coronal region of the post-space and may not possess the necessary mechanical properties to withstand occlusal loading.

Previous research has demonstrated the suitability of chemical-cure resin composite as a dowel and core material (Plasmans, Welle & Vrijhoef, 1988); however, because of its limited working time, controlled placement within the canal is difficult (Lui, 1994). To overcome this problem, Lui (1994) proposed using light-cured resin composite in combination with a light-transmitting post to reinforce the flared canal prior to inserting a matching post. More recently, reinforced resin composite cements have been shown to compensate for the retention of parallel-sided and tapered dowels with a reduced length (Nissan, Dmitry & Assif, 2001). Currently, newer dual-cure resin composite core materials are being developed and may offer a longer working time and an ability to polymerize in regions distant from the light source.

Published research on the bonding of resin composite to root canal dentin is extremely limited (Ishizuka & others, 2001). Development of the microtensile bond strength test has enabled the measurement of resin/dentin bond strengths at several regions within the same tooth (Sano & others, 1994). Subsequently, modifications in specimen preparation have enabled the regional measurement of resin/dentin bond strengths within root canal dentin (Foxton & others, 2003; Shono & others, 1999). A recent study by Gaston and others (2001) reported that when post-spaces were prepared in root canals and filled with chemically polymerized resin cement, there were no significant differences in microtensile bond strength between the coronal and middle-third of the post space. Authors had noted that these regions should be considered to be clinically relevant regions for bonding, since posts are commonly cemented into post spaces prepared in the cervical and middle-third of the root canal (Gaston & others, 2001). However, to date, there is no published information on the bonding of dual-cure resin composite at different regions of root canal dentin.

Therefore, this study evaluated the bonding of a recently developed dual-cure resin composite core material to different regions of root canal dentin using photo and dual-cure adhesives and different modes of polymerization. The null hypotheses tested were that the mode of polymerization does not affect the bonding of dual-cure resin composite to root canal dentin, nor are there any regional differences in bond strengths

METHODS AND MATERIALS

Nineteen single-rooted human premolars, recently extracted from adolescents for orthodontic reasons and stored frozen, were used in this study. Their crowns were removed at the cementoenamel junction using a low-speed diamond saw (Isomet, Buehler, Ltd, Evanston, IL, USA) with post spaces prepared in the root canals using Gates drills (Matsutani Seisakusho Co, Ltd, Takanezawa, Japan) and Para Post drills (Whaledent International, New York, USA) in a low-speed handpiece under copious water irrigation to a depth of 8 mm and a width of 1.4 mm. The roots were then randomly divided into two groups, a group of 15 for the microtensile bond strength test, and a group of four for microhardness testing. A self-etching/dual-cure adhesive system (Clearfil Liner Bond 2V, Kuraray Medical Co, Tokyo, Japan) and a dual-cure resin composite (Clearfil DC Core, Kuraray Medical Co, Tokyo, Japan) were used in this study. Table 1 shows the chemical composition of these materials.

Microtensile Bond Strength Test

The 15 roots were randomly divided into five groups of three and their walls treated with self-etching primer using a microtip applicator (GC Corporation, Tokyo, Japan) according to the manufacturer's instructions. The five groups were then treated with adhesive resin and filled with dual-cure resin composite as follows (Table 2): (1) light-cured adhesive resin (Bond A) was applied to the canal walls using a microtip applicator and light cured for 10 seconds from a coronal direction using a conventional light source (Optilux 500, Demetron, Danbury, CT, USA). Prior to each bonding procedure, the power density of the light source was checked with a digital radiometer (Jetlite light tester, J Morita USA Inc, Irvine, CA, USA) to ensure that the power density of the light source was between 550 and

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570 mW/cm^2. The dual-cure resin composite was then injected into the canal using a centrix syringe (Centrix Inc, Shelton, CT, USA) and light cured for 60 seconds (Bd A Lt/DC Lt). (2) Bond A was light cured for 10 seconds, then dual-cure resin composite was chemically-cured by placing in darkness for 30 minutes (Bd A Lt/DC No). (3) Bond A+B was applied and light cured for 10 seconds, then the dual-cure resin composite was light cured for 60 seconds (Bond AB Lt/DC Lt). (4) Bond A+B was applied with no light exposure, then, dual-cure resin composite was light cured for 60 seconds (Bond AB No/DC Lt). (5) Bond A+B was applied with no light exposure and the dual-cure resin composite was chemically-cured (Bond AB No/DC No).

Chemical-cure resin composite (Clearfil FII, Kuraray Medical Co, Tokyo, Japan) was placed on the outer cementum surfaces of all the roots to make grips for testing and stored in water for 24 hours at 37°C. Each bonded specimen was then attached to the arm of a low-speed diamond saw (Isomet, Buehler Ltd) and vertical cuts were made perpendicular to the bonded interface under water-cooling to harvest eight slabs approximately 0.6-mm thick as depicted in Figure 1. The coronal four slabs were considered to represent the coronal portion of the post space (Cor) corresponding to the coronal third of the root canal, and the apical four slabs were considered to represent the apical region of the space (Api) corresponding to the middle third of the root canal. Each slab was

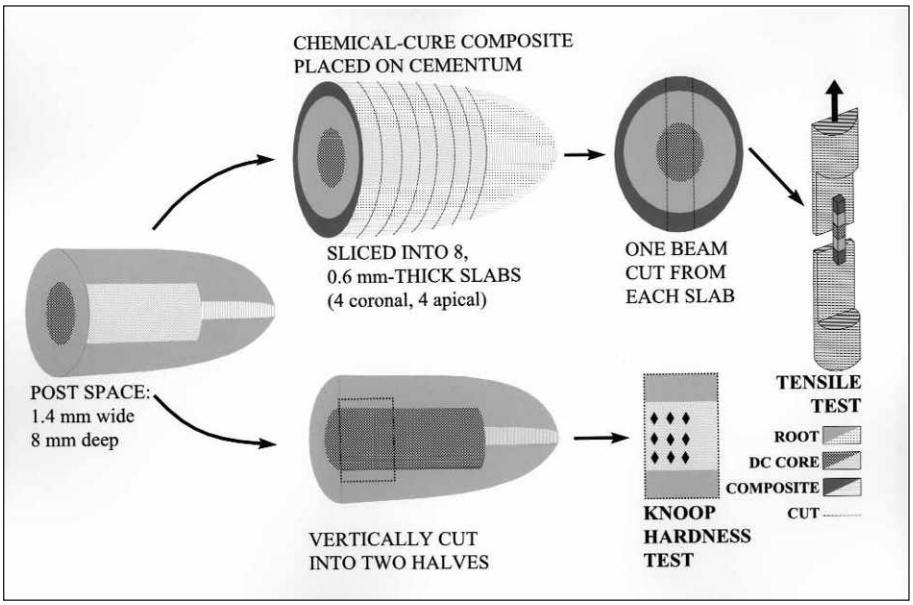

Figure 1. *Schematic illustration of the methodology of specimen preparation for the microtensile bond strength and Knoop hardness tests.*

Table 1: *Materials Tested in This Study*

Material	Batch #	Composition (according to manufacturers)
Clearfil Liner Bond 2V		
Primer A,B	A: 00082A	MDP, HEMA, H_20, photoinitiators
	B: 00080B	HEMA, H_20, chemical initiators
Bond A,B	A: 01139A	MDP, dimethacrylates, photoinitiator, microfillers
	B: 00022A	HEMA, dimethacrylates, chemical initiator, microfillers
Clearfil DC Core Paste		
Catalyst	0169	Silanated inorganic filler, microfillers, dimethacrylates photo/chemical initiator
Universal	0162	Silanated inorganic filler, microfillers, dimethacrylates photo/chemical initiator

Abbreviations: MDP = 10-methacryloyloxydecyl dihydrogen phosphate; HEMA = 2-hydroxyethyl-methacrylate

Table 2: *Experimental Groups and Bonding Procedures*

Group	Bonding Resin	Light Cure	Dual-Cure Resin Composite	Light Cure	Procedures*
Bd A Lt/ DC Lt	Bond A	10 seconds	DC Core	60 seconds	a; b; d; f; g
Bd A Lt/ DC No	Bond A	10 seconds	DC Core	No	a; b; d; f; h
Bd AB Lt/ DC Lt	Bond A+B	10 seconds	DC Core	60 seconds	a; c; d; f; g
Bd AB No/ DC Lt	Bond A+B	No	DC Core	60 seconds	a; c; e; f; g
Bd AB No/ DC No	Bond A+B	No	DC Core	No	a; c; e; f; h

**Procedures: a = mix one drop of primer A + B, apply for 30 seconds, then gently air dry; b = apply Bond A; c = mix 1 drop of Bond A and B and apply; d = light-cure for 10 seconds; e = no-light cure; f = mix equal amounts of catalyst and universal paste, then apply; g = light-cure for 60 seconds; h = no-light cure.*

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bonded separately using cyanoacrylate glue (Zapit, DVA, Anaheim, CA, USA) to a glass microscope slide (Microslide glass, Matsunami Glass Ind Ltd, Tokyo, Japan) to prevent any movement as it was being sliced into beams. A custom-molded acrylic base-plate was slotted into the outer casing of the diamond saw. Attached longitudinally to the base-plate was another glass slide positioned 0.6 mm from the diamond saw blade. The edge of the glass side acted as a guide for the transverse slicing of the slabs into beams approximately 8 mm in length with a mean cross-sectional area of 0.35 ± 0.06 mm^2 measured using digital calipers (Mitutoyo CD15, Mitutoyo Co, Kawasaki, Japan). For each slab, only the central beam was designated for tensile testing as shown in Figure 1. The remaining beams were discarded.

Using cyanoacrylate glue, each beam was carefully bonded onto a testing device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) mounted in a tabletop material testing machine (EZ Test, Shimadzu, Kyoto, Japan) and subjected to a tensile force at a crosshead speed of 1 mm/minute. The load at failure divided by the cross-sectional area of the beam was used to calculate the microtensile bond strength (μTBS) in units of stress (MPa).

The tested beams were glued to brass tablets, gold sputter coated and their failure modes determined under a scanning electron microscope (JSM-5310, JEOL, Tokyo, Japan). In the case of the beams that failed during slicing (pre-test failure), failure modes were determined visually. Failure modes of the tested

Table 3: *Microtensile Bond Strengths (MPa) Including Zero Bond Strengths*

	Bd A Lt/ DC Lt	Bd A Lt/ DC No	Bd AB Lt/ DC Lt	Bd AB No/ DC Lt	Bd AB No/ DC No
Coronal Half	49.1 ± 11.2[ab]	29.9 ± 19.6[ab]	33.9 ± 15.6[ab]	28.6 ± 16.5[ab]	17.1 ± 18.3[b]
Apical Half	37.7 ± 16.0[ab]	19.8 ± 10.5[b]	23.5 ± 10.1[ab]	33.2 ± 16.8[ab]	16.6 ± 12.0[b]

All values are mean ± SD.
Number of specimens in each group = 12
Groups with the same case superscript letters are not significantly different (p>0.05).

Table 4: *Microtensile Bond Strengths (MPa) Excluding Zero Bond Strengths*

	Bd A Lt/ DC Lt	Bd A Lt/ DC No	Bd AB Lt/ DC Lt	Bd AB No/ DC Lt	Bd AB No/ DC No
Coronal Half	49.1 ± 11.2[a] (12)	35.9 ± 15.2[ab] (10)	33.9 ± 15.6 (12)[ab]	34.2 ± 10.7[ab] (10)	25.6 ± 16.6[ab] (8)
Apical Half	41.1 ± 11.3[ab] (11)	21.5 ± 8.9[b] (11)	23.5 ± 10.1 (12)[b]	33.2 ± 16.8[ab] (12)	19.9 ± 10.1[b] (10)

All values are mean ± SD (Number of specimens).
Groups with the same case superscript letters are not significantly different (p>0.05).

Table 5: *Knoop Hardness of DC Core at Different Regions of the Post Space*

	Curing Strategy	
	Light (60 seconds)	No Light
Coronal half	91.9 ± 6.8[a]	72.3 ± 3.9[b]
Apical half	88.2 ± 7.3[a]	76.0 ± 3.3[b]

All values are mean ± SD
Groups identified with the same superscript letter are not significantly different (p>0.05)

Table 6: *Failure Modes After Microtensile Test and Kruskal-Wallis Mean Rank*

	# of Specimens for Each Failure Mode (failure mode/score)				
Subgroup	A/1	B/2	C/3	D/4	Kruskal-Wallis Rank
Bd A Lt/DC Lt Cor	2	3	2	5	101.9
Bd AB Lt/DC Lt Cor	8	2	2	0	68.4
Bd A Lt/DC Lt Api	8	4	0	0	66.8
Bd A Lt/DC No Cor	9	2	1	0	63.0
Bd AB Lt/DC Lt Api	10	2	0	0	57.7
Bd A Lt/DC No Api	11	1	0	0	53.1
Bd AB No/DC Lt Cor	12	0	0	0	48.5
Bd AB No/DC Lt Api	12	0	0	0	48.5
Bd AB No/DC No Cor	12	0	0	0	48.5
Bd AB No/DC No Api	12	0	0	0	48.5

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specimens were classified as A: Cohesive failure within resin; B: Mixed failure in resin and the hybrid layer; C: Mixed failure in the hybrid layer and dentin and D: Cohesive failure within dentin.

Microhardness Measurement

The remaining four roots were treated with the self-etching primer, and the adhesive resin Bd A, which was light cured. After injecting the dual-cure resin composite into the post spaces, two roots were light cured for 60 seconds from the coronal direction and two were chemically-cured as described earlier. After 24 hours at 37°C, the four roots were sectioned parallel to the long axis of the root through the filled post space to provide eight halves, four dual-cured and four chemically cured. These halves were placed into acrylic rings that were attached to adhesive tape and embedded in epoxy resin. After the epoxy resin had set, the acrylic resin blocks were polished using ascending grades of abrasive SiC papers under running water, then polished using diamond pastes (DP-paste P, Struers A/S, Denmark) down to 1 µm. Each block was positioned on the traveling micrometer stage of a micro-indentation tester (Akashi MVK-E hardness tester, Omron Takeisi Electronics Co, Tokyo, Japan) fitted with a diamond Knoop indenter. A load of 50 g was applied for a dwell time of 15 seconds. Indentations were made vertically, starting from the coronal end in the upper left, mid- and right-third regions of the dual-cure resin composite at 100 µm intervals. Using 400x magnification, the length of the long diagonal (µm) of each indentation was measured and the Knoop Hardness Number (KHN) calculated. The data were equally divided into two, the coronal and apical regions of the post space.

Statistics

The microtensile bond strength data and microhardness data were analyzed by two-way ANOVA factorial analysis (chemical-vs dual-curing of DC Core and region) and Scheffe's post-hoc test. Statistical significance was considered to be $p<0.05$. Statistical analysis of the failure modes was performed using the Kruskal-Wallis rank test. For this, each failure mode was given a score from 1 to 4 prior to statistical analysis. A score of 1 was given for cohesive failure in resin, a score of 2 for mixed failure in resin and the hybrid layer, a 3 score for mixed failure in the hybrid layer and dentin and a score of 4 for cohesive failure within dentin. The higher the score, the stronger the bond (Matsumura, Kato & Atsuta, 1997; Foxton & others, 2002).

RESULTS

The microtensile bond strength (µTBS) test data including the pre-test failures are presented in Table 3, and the data not including the pre-test failures is shown in Table 4. The microhardness data are presented in Table 5, with the failure modes of the beams shown in Table 6.

Two-way ANOVA revealed that the bond strengths were influenced by mode of polymerization of the dual-cure resin composite ($p<0.001$) but not by the region of the post space ($p=0.0679$). The microhardness of the dual-cure resin composite was influenced by the mode of polymerization of the dual-cure resin composite ($p<0.001$) but not by the region of the post space ($p=0.9836$). In addition, in the case of microhardness, there was a significant interaction between the mode of polymerization and region ($p=0.0259$).

For each of the five groups, there was no significant difference in µTBS between the coronal and apical regions of the post space ($p>0.05$). The highest µTBS occurred when the adhesive resin was light cured and dual-cure resin composite was dual-cured, and the lowest µTBS occurred when both the dual-cure adhesive resin and dual-cure resin composite were chemically cured. When the adhesive resin was light-cured, µTBS was higher when the resin composite was dual-cured than when the resin composite was chemically cured at both the coronal and apical regions.

Regarding microhardness, KHN of the dual-cure resin composite was significantly higher when the resin composite was dual-cured than when curing was chemical only at both regions of the post space ($p<0.05$). For each curing strategy, there was no significant difference between the KHN at the coronal and apical region of the post space ($p>0.05$).

Regarding failure modes of the tested specimens, when the dual-cure adhesive resin was chemically cured, the beams failed cohesively within the bonding or resin composite. Representative SEM views of the upper and lower surfaces of a failed beam from the Bd AB No/DC No group, which was ranked the lowest, are shown in Figures 2a-d. On the other hand, when the bonding resin was light or dual-cured, some beams were found to fail cohesively within the hybrid layer or dentin. Figures 3a-d show the SEM views of the upper and lower surfaces of a failed beam from the Bd A Lt/DC No group. Moreover, when the bonding resin was light cured and the resin composite dual cured, some beams in the coronal region exhibited cohesive failure in dentin. Figures 4a-d provide SEM views of the upper and lower surfaces of a failed beam from the Bd A Lt/DC Lt group that was ranked the highest. This group also recorded the highest µTBS.

Out of a maximum total of 120 beams, 12 failed during preparation for bond strength testing, one from Bd A Lt/DC Lt, three from Bd A Lt/DC No, two from Bd AB No/DC Lt and six from Bd AB No/DC No. These are included in the results as zero bond strengths. These beams failed cohesively within resin.

DISCUSSION

Presently, very limited information is available on the bonding of dual-cure resin composite at different regions

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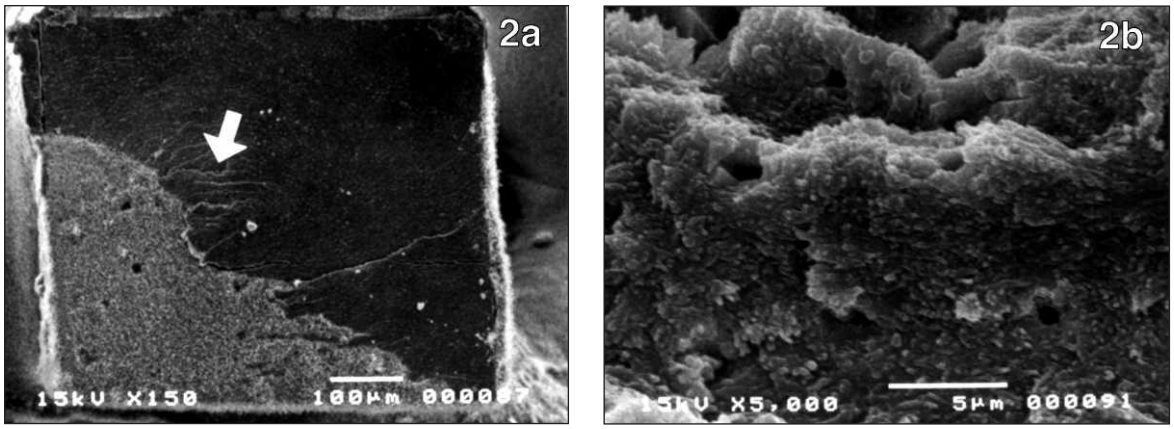
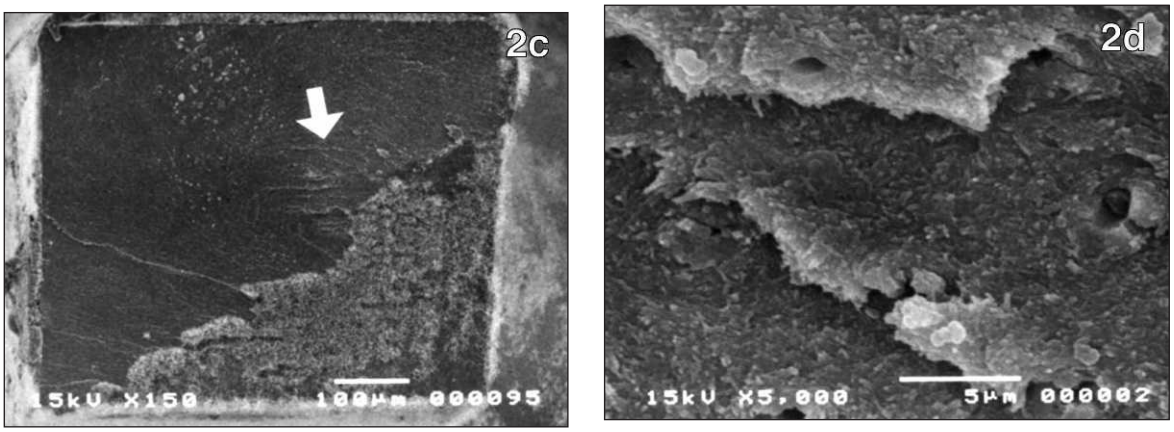
Figure 2



Figures 2 a and b: *SEM photographs of one side of a fractured beam from the Bd AB No/DC No. Group showing cohesive failure within the bonding resin* and composite resin. The area marked by an arrow is shown at higher magnification. (a) 150x (b) 5000x.



Figures 2c and d*: SEM photographs of the opposing side of the fractured beam shown in 2a. The area marked by an arrow is shown at higher magnification. (c) 150x (d) 5000x.*

of root canal dentin. In this study, when dual-cure resin composite was bonded to root canal dentin using a dual-cure self-etching primer/adhesive resin, no significant differences were found between the µTBS at the coronal and apical regions of the post space in each of the five groups. These results agree with those from a study by Gaston and others (2001), that evaluated the microtensile bond strength of two chemically-cured resin cements to root canal dentin in teeth that had not been endodontically-treated. The authors of that study found that for both materials, there were no significant differences between the µTBS at the coronal and middle thirds of root canal dentin and concluded that bond strength is related more to the area of solid dentin than to tubule density (Gaston & others, 2001). Therefore, the null hypothesis of the current study has to be partially rejected.

Giannini and others (2001) examined the influence of tubule density and the area of solid dentin on resin/dentin bond strengths of two adhesive systems, a self-etching system (Clearfil Liner Bond 2V) and an acetone-based system in conjunction with phosphoric acid etching (Prime & Bond 2.1) at superficial, middle and deep regions of coronal dentin. The authors of that research found that the self-etching system was less sensitive to dentin depth and tubular density than the acetone-based system (Giannini & others, 2001). While no significant differences in bond strengths of the self-etching system were found at the superficial, middle and deep regions of coronal dentin, significant differences in tubule density were observed (Giannini & others, 2001).

Regarding tubule density in root dentin, Ferrari and others (2000a) reported that tubule density was highest in the cervical region and significantly reduced in the middle and apical thirds. This study found that there were no significant differences in the µTBS of the dual-cure resin composite at the coronal and apical regions of the post space irrespective of whether polymerization was light or chemically activated, which agrees with the findings of Giannini and others (2001) using coronal dentin. Therefore, the authors speculate that in this study, resin/dentin bond strengths may not have been dependent on the density of the dentinal tubules. This may have been due to the self-etching primer used in this study having a pH of around 2.8, which is sufficient to infiltrate the smear layer and etch the underlying intact dentin (Okada & othes, 1998; Tay & others, 2000). After application and polymerization of the bonding resin, a thin hybrid layer is created, which is made up of a resin impregnated smear layer and resin impregnated intertubular dentin and hybridized smear plugs in the dentinal tubules (Tay & others, 2000).

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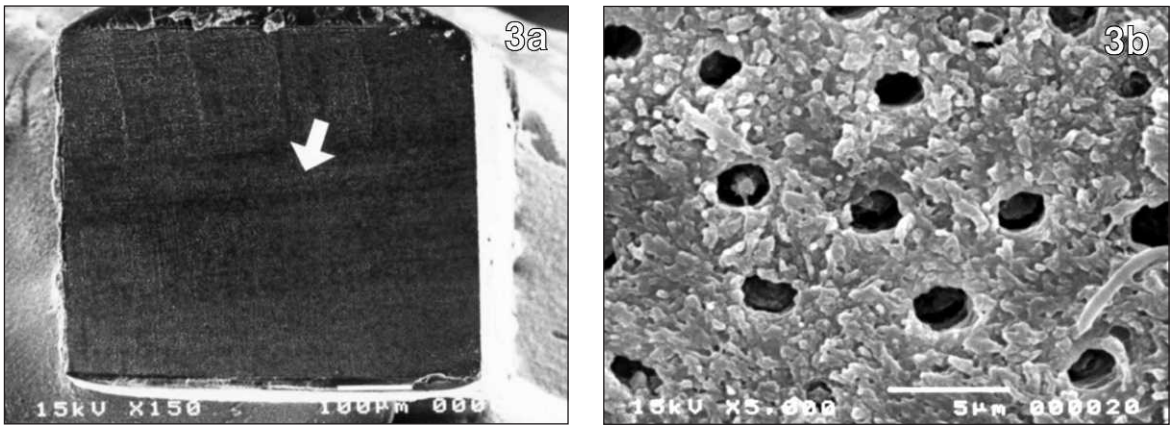
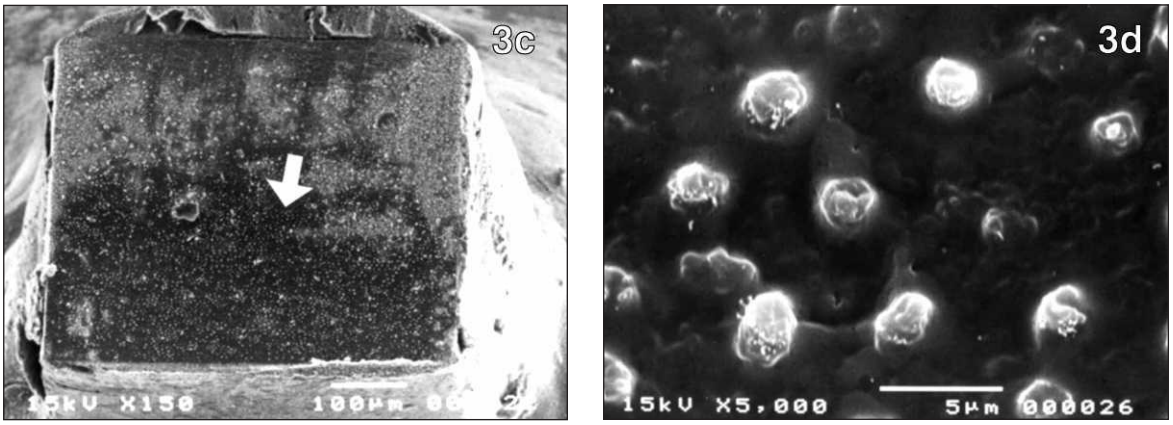
The group Bd A Lt/DC Lt, in which the adhesive resin was light cured, exhibited higher bond strengths than Bd AB Lt/DC Lt, in which the adhesive resin was dual cured at both the coronal and apical regions of the post space. Moreover, SEM observation of the tested beams revealed that cohesive failures in dentin had occurred in the Bd A Lt/DC Lt group but not in the Bd AB Lt/DC Lt group. In addition, when the failure modes were statistically analyzed, both the Bd A Lt/DC Lt sub-groups were ranked the highest. These findings indicate that the mechanical properties of the adhesive resin Bd A, when photo-cured, may have been greater than those of the adhesive resin Bd A+B when dual cured. The authors speculate that this was due to differences in the chemical compositions of Bond A and mixed Bond A + Bond B. Bond A contains photoinitiators and the acidic phosphate monomer MDP, whereas Bond B contains chemical initiators and no MDP (Table 1). When Bond A and Bond B were mixed, a reduction in the concentration of photoinitators and MDP occurred. The authors speculate that although mixed Bond A + B is able to polymerize through the dual action of the chemical and photoinitiators, for this particular adhesive resin, photoinitated polmerization is more important. The lower concentration of photoinitiators and MDP in Bond A+B compared to Bond A probably resulted in a reduction in the degree of polymerization and bonding ability. In contrast, the group with the lowest µTBS was Bd AB No/DC No, in which both the adhesive resin and resin composite were chemically cured. In addition, the Knoop Hardness Number of DC Core was significantly lower when polymerization was chemically initiated as opposed to photo-initiated. Moreover, all the beams in the Bd AB No/DC No groups failed cohesively within resin and these groups were ranked among the lowest (Table 6). Together, these results indicate that for this particular dual-cure resin composite, resin/dentin bond strengths may also have been dependent upon the mechanical properties of both the bonding resin and resin composite.

Figure 3



Figures 3 a and b: *SEM photographs of the dentin side of a fractured beam from the Bd A Lt/DC No group showing cohesive failure within the hybrid layer. The area marked by an arrow is shown at higher magnification. (a) 150x (b) 5000x.*



Figures 3c and d*: SEM photographs of the composite side of the fractured beam shown in 3a. The area marked by an arrow is shown at higher magnification (c) 150x (d) 5000x.*

Previous research has shown that variations in the microhardness of photo-initiated resin composites are related to the degree of cure or the degree of conversion (Cook, 1980; Ruyter & Øysæd, 1982). In this experiment, a significant reduction in microhardness occurred when polymerization of the dual-cure resin composite was chemically initiated. This indicates that the degree of conversion in the dual-cure resin composite was lower when polymerization was chemically initiated compared to photo-initiated. Therefore, the lower bond

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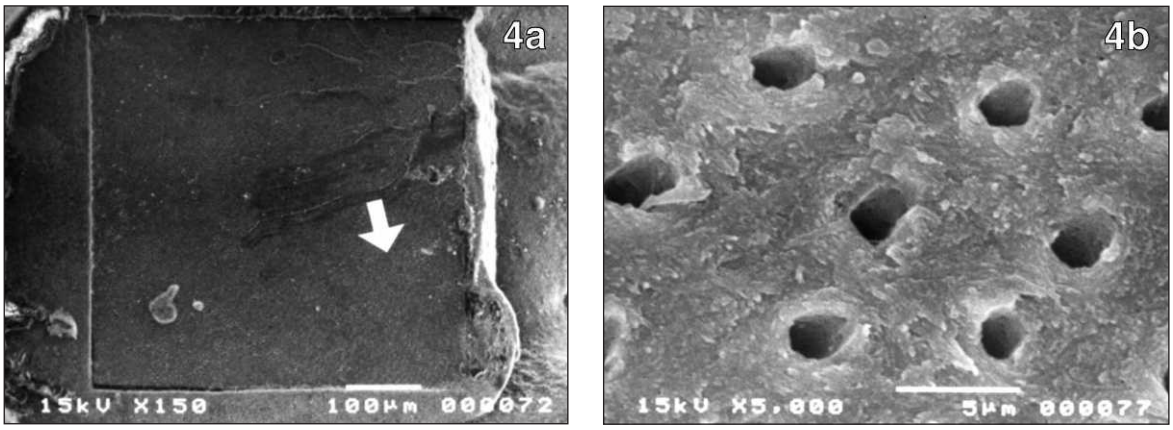
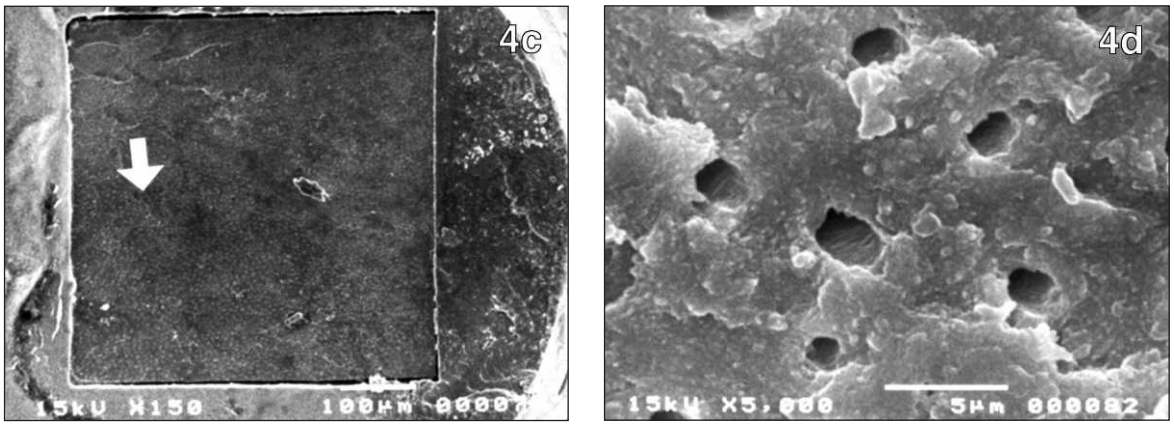
Figure 4



Figures 4 a and b: *SEM photographs of the dentin side of a fractured beam from the Bd A Lt/DC Lt group showing cohesive failure within dentin. The area marked by an arrow is shown at higher magnification. (a) 150x (b) 5000x.*



Figures 4c and d*: SEM photographs of the opposing side of the fractured beam shown in 4a. The area marked by an arrow is shown at higher magnification. (c) 150x (d) 5000x.*

strength of the groups in which DC Core was chemically-cured (Bd A Lt/DC No and Bd AB No/DC No) compared to those groups in which DC Core was dual-cured (Bd A Lt/DC Lt, Bd AB Lt/DC Lt and Bd AB No/DC Lt) may have been the result of a lower degree of conversion occurring in the dual-cure resin composite. On the other hand, there was no influence of region on microhardness and no significant differences in KHN between the coronal and apical regions of the post space when polymerization was photo-initiated, which indicates that the degree of conversion may have been similar at both the coronal and apical regions even though the energy of light irradiation may have been at the apical region. This suggests that for this particular dual-cure resin composite, the amount of light necessary for adequate photo-initiated polymerization may be small, whereas, chemically-initiated polymerization is not sufficient to obtain the optimum mechanical properties for bonding.

Recent research by Sanares and others (2001) has suggested that adverse surface reactions might occur between one-bottle light-cured adhesives and chemically-cured composites, resulting in significantly lower bond strengths. Moreover, Tay and others (2001) reported that when a hybrid composite was bonded to dentin using one of two single-step bonding systems and light-curing was delayed for up to 20 minutes, microtensile bond strengths decreased and adverse interactions occurred along the adhesive-composite interface. However, when an adhesive resin was placed after applying and curing the single-step adhesive, no adverse interactions were found (Tay & others, 2001). They postulated that this might be due to an acid-base reaction occurring between the tertiary amine, which is necessary for activation of the photo-initiator, and the acidic resin monomer, resulting in inactivation of the photoaccelerator-photoinitiator system (Tay & others, 2001). This study used a two-step self-etching primer/bonding system and found that there were no significant differences in µTBS among the groups in which the adhesive resin was dual-cured (Bond AB Lt/DC Lt) and chemically cured (Bond AB No/DC Lt) and the resin composite dual-cured. In addition, when the adhesive resin was light-cured and the resin composite was chemically cured, SEM examination of the fractured surfaces of the de-bonded beams showed no blistering or void formation in the adhesive layer (Figures 3a-d). This may be due to the insensitivity of the self-etching primer to the intrinsic wetness of dentin (Giannini & others, 2001; Pereira & others, 1999). In addition, the adhesive resin Bond A+B contains photo and chemical initiators and, thus, was able to polymerize without exposure to light. In this study, the post

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spaces were prepared in root canals that had not been endodontically-treated or treated with NaOCl so as not to alter the biomechanical behavior of the root dentin (Ishizuka & others, 2001; Morris & others, 2001; Nikaido & others, 1999).

CONCLUSIONS

Within the limitations of this study, it can be concluded that photo-initiated polymerization of the tested dual-cure adhesive resin and dual-cure resin composite was necessary for optimum bonding of root canal dentin. A reduction in bond strength occurred when polymerization of the dual-cure resin composite was chemically initiated, which was associated with a significant reduction in the degree of conversion of the resin composite. No regional differences were found in resin/dentin bond strengths and microhardness between the coronal and apical regions of the post space irrespective of whether polymerization of the adhesive resin and dual-cure resin composite were chemically or photo-initiated. Further research on other similar materials and their suitability with recently developed fibre posts for the restoration of endodontically-treated teeth is therefore warranted.

(Received 22 August 2002)

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Effect of Delayed Polishing Periods on Interfacial Gap Formation of Class V Restorations

M Irie • R Tjandrawinata • K Suzuki

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Clinical Relevance

Delaying polishing for one day resulted in improved gap formation for Class V restorations of conventional and resin-modified glass ionomers and a microfilled composite. Delaying polishing was not necessary for a compomer.

SUMMARY

This *in vitro* study evaluated the effect of the initial polishing period through 30 minutes, 3 hours, 12 hours, 24 hours and 1 week after setting on the gap formation around Class V restorations. Three resin-modified glass ionomers, one compomer, one conventional glass ionomer and one microfilled composite were used as controls.

When specimens of the two types of glass ionomers and a microfilled composite were polished immediately after the setting procedure, this study showed 100-140 gaps around the X-section of the restorations. In contrast, only 10-40 gaps around the Class V restoration were observed when the specimens were polished after 12 hours of storage. Significant differences were observed between polishing immediately and polishing after 12 hours of storage in the two glass ionomer restorative materials. The compomer did not show this pattern. No significant differences were observed among the six polishing periods of the sum number of gaps at the cavity-restoration interfaces. The tendency of Silux Plus was similar to the two types of glass ionomer materials; namely, when the specimen was polished and inspected after storage in water for 24 hours or one week, the authors observed almost 30 gaps around the restorative cavities.

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INTRODUCTION

Class V restorations are an indication of a conventional glass ionomer cement (CGIC), a resin-modified glass ionomer cement (RMGIC) and a polyacid-modified resin composite (compomer) (Ferrari & others, 1998; Gladys & others, 1998; Tyas, 2000; Folwaczny & others, 2000a,b; Di Lenarda & others, 2000; Brackett & others, 2001; Brackett & others, 2002). CGIC has several beneficial properties, such as physicochemical bonding to the tooth substrate, fluoride release and uptake and tooth color. However, it also demonstrates brittle fracture, erosion and wear in the oral environment (Wilson & McLean, 1988). To improve these deficiencies, two

types of combination materials, RMGIC and compomer, have been developed. RMGIC is unlike a light-cured resin composite or a CGIC; it has a dual setting reaction consisting of an acid-base reaction and a photochemical polymerization process. The final set materials have been described as having a complex structure in which glass particles are sheathed in a matrix consisting of two networks—one derived from the glass ionomer, the other from the resin (Wilson, 1990; Mitra, 1991). In these dual-setting systems, the resin reinforcement provides higher mechanical strength (Uno, Finger & Fritz, 1996; Irie & Suzuki, 1999; Irie & Suzuki, 2000; Yap & others, 2001) and higher bond strength to tooth surfaces compared with CGICs. RMGIC claimed to have an improved marginal seal and gap formation of restoration by hygroscopic expansion (Sidhu, Sherriff & Watson, 1997; Irie & Suzuki, 2000) and improved bonding ability after storage in water (Fritz, Finger & Uno, 1996a,b; Irie & Suzuki, 1999; Irie & Suzuki, 2000; Irie & Suzuki, 2002).

A compomer may be a single- or two-component material containing one or both of the essential components of a glass ionomer. However, the components do not react as part of the setting process (McLean, Nicholson & Wilson, 1994). It consists of a resin matrix and a fluoroaluminosilicate glass filler. However, it undergoes no dimensional change by hygroscopic expansion (Irie & Suzuki, 2000). Therefore, it may not improve the marginal seal and gap formation of a restoration (Irie & Suzuki, 2002). The monomer in resin matrix ionizes following water uptake during storage after light activation. The released hydrogen ions then react with the glass filler to initiate an acid-base reaction. Ionic cross-linking also occurs and fluoride is released (Hammesfahr, 1994). Therefore, it may improve the marginal seal and gap formation of restorations by enhancing the bond ability.

The polishing periods serve as other factors that may influence the seal ability around a cervical restoration. Polishing after storage in water for one day resulted in improved gap formation for three positioned cervical restorations of one RMGIC and one CGIC. However, delayed polishing did not improve gap formation for a compomer (Irie & Suzuki, 2002). Due to the structure of glass ionomers and their hydrophilic nature, water sorption and subsequent swelling may lead to partial compensation of the shrinkage. The preservation of sealing around a restoration would benefit most if water sorption and setting shrinkage could occur simultaneously. However, water sorption occurs only at a later stage compared with setting shrinkage (Feilzer & others, 1995). Currently, no information is available regarding the gap formation behavior around Class V of a CGIC, RMGIC and a compomer.

In this study, the effects of the polishing period on the gap formation around Class V restorations using RMGICs and a compomer were evaluated *in vitro*. The hypothesis tested was that the difference in polishing times would result in gap formation for the tested materials.

METHODS AND MATERIALS

Human premolars, extracted for orthodontic reasons, were used for the experiment. After extraction, each tooth was immediately stored in cold distilled water at 4°C for one to two months before testing. The basic properties of three RMGICs, one compomer, one CGIC and one microfilled composite are summarized in Tables 1 and 2. All processing of the materials was carried out according to the manufacturer's instructions. Capsules of Photac-Fil were triturated using a high-

Table 1: *Restorative Materials Investigated*

Material	Manufacturer	Batch #	Type (Powder/Liquid)/Components
Fuji II LC	GC Corp Tokyo, Japan	P: 030921 L: 250721	Resin-modified GIC (3.0g/1.0g) P: fluoroaluminosilicate glass L: copolymer of acrylic and maleic acid, HEMA, water
Vitremer	3M Dental Products St Paul, MN, USA	19930203	Resin-modified GIC (2.5g/1.0g) P: fluoroaluminosilicate glass L: polyalkenoate copolymer, HEMA, water
Photac-Fil	3MESPE GmbH Seefeld, Germany	X033	Resin-modified GIC (Precapsulated) P: fluoroaluminosilicate glass L: copolymer of maleic acid and acrylic acids monomers oligomers, water
Dyract	DeTrey/Dentsply Konstanz, Germany	B 930776	Compomer fluoroaluminosilicate, polyacrylic acid, UDMA
Fuji II	GC Corp Tokyo, Japan	P: 300802 L: 120901	Conventional GIC (2.7g/1.0g) P: fluoroaluminosilicate glass L: copolymer of acrylic and maleic acids, polybase carboxylic acid, water
Silux Plus	3M Dental Products St Paul, MN, USA	1DW1	Microfilled resin composite colloidal silica (38 vol%), Bis-GMA, TEGDMA

Key: GIC, Glass ionomer cement; HEMA, 2-Hydroxtethyl methacrylate; UDMA, urethane dimethacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate.

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speed mixer (Silamat, Vivadent, Schaan, Liechtenstein) for 15 seconds. For light activation of the priming or sealing agents and of the restorative materials, a curing unit (New Light VL-II, GC, Tokyo, Japan; irradiated diameter: 8 mm) was used. The curing distance from tip to material surfaces was as near as possible. The light intensity was checked immediately before each application of the adhesive resin and restorative material using a radiometer (Demetron/Kerr, Danbury, CT, USA). During the experiment, the light intensity was maintained at 450 mW/cm^2. For each material and storage period, 10 specimens were made. All procedures, except for cavity preparation, were performed in a thermo-hygrostatic room kept at 23±0.5C° and 50±2% relative humidity.

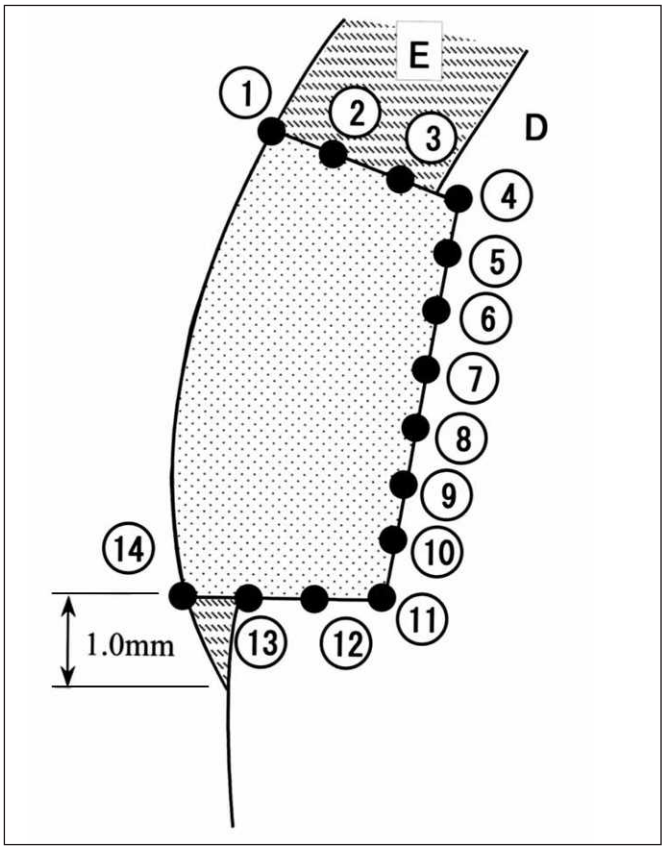

Figure 1. *Class V restoration and each measured point in the Class V restorations. E: Enamel substrate, D: Dentin substrate.*

Class V Restoration

Cavity preparations were made in the premolars on the facial surface. A cylindrical cavity was prepared with a tungsten carbide bur (200,000-rpm) and a fissure bur (8,000-rpm) under wet conditions to a depth of 1.5 mm with a diameter of 3.5 mm. A cavity preparation was placed parallel to the cementoenamel junction (CEJ) with the preparation extended 1.0 mm above the CEJ (Figure 1). Cavosurface walls were finished to a butt joint. One cavity was prepared in each tooth. In total, cavities were prepared in 360 teeth (6 materials X 6 polishing times X 10 repeats).

The prepared cavity surface was treated with the conditioner/primer according to each manufacturer's instructions. Dentin Conditioner and Ketac Conditioner were applied for 20 and 10 seconds, respectively, and rinsed with water. Vitremer Primer was applied for 30 seconds, air dried and light cured for 20 seconds. An ample amount of Dyract-PSA was applied and left undisturbed for 30 seconds. Excess solvent was removed by compressed air. The light curing time was 10 seconds. A second layer of Dyract-PSA was applied with a brush. Etchant was applied for 15 seconds, rinsed and air dried. Primer was applied and air dried following Adhesive that was photocured for 10 seconds.

The cavity was filled with mixed materials using a syringe tip (Centrix C-R Syringe System, Shelton, CT, USA) and covered with a plastic strip and

Table 2: *Conditioner/Primer Agents Investigated*

Material	Manufacturer	Batch #	Components and Surface Treatment
Dentin Conditioner	GC Corp Tokyo, Japan	151021	Polyacrylic acid, water Apply with brush 20 seconds → rinse 15 seconds → gently dry five seconds
Vitremer Primer	3M Dental Products St Paul, MN, USA	35	HEMA, maleic acid in aqueous solution ethyl alcohol Apply with brush 30 seconds → gently dry five seconds → light cure 20 seconds
Ketac-Conditioner	3MESPE GmbH Seefeld, Germany	0002	Polyacrylic acid, water Apply with brush 10 seconds → rinse 15 seconds → gently dry five seconds
Dyract-PSA	DeTrey/Dentsply Konstanz, Germany	931020	PENTA, TEGDMA, acetone Apply 30 seconds → gently dry five seconds → light cure 10 seconds
Scotchbond Multi-Purpose	3M Dental Products St Paul, MN, USA	Etchant(2AC) Primer(2AC) Adhesive(2AB)	10% maleic acid, water HEMA, polyalkenoic acid, coplymer, water Etch 15 seconds → rinse 15 seconds → gently dry five seconds→ Apply with brush (Primer) gently dry five seconds → Apply with brush (Adhesive) → light cure 20 seconds

Key: HEMA, 2-hydroxtethyl methacrylate; PENTA, dipentaerythritolpenta-acrylatemonophosphate; TEGDMA, tri-ethylene-glycol dimethacrylate; Bis-GMA, bisphenol A-glycidyl methacrylate

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hardened. Fuji II LC, Vitremer, Photac-Fil, Dyract and Silux Plus were exposed to a visible light source with irradiation times of 20 seconds, 40 seconds, 20 seconds, 40 seconds and 40 seconds, respectively. Fuji II was stored in an incubator at 37°C and 100% relative humidity for four minutes after mixing to allow for chemical-curing to occur and was coated with a varnish (Fuji Varnish, Tokyo, Japan).

Inspection Procedure

Immediately after light curing, setting or after storage in distilled water at 37°C, and at 30 minutes, 3 hours, 12 hours, 24 hours and 1 week, the restorations were polished with abrasive points (Silicone Mide, Shofu, Kyoto, Japan) in wet condition to avoid desiccation and breakdown. Each tooth was sectioned in a buccolingual direction through the center of the restoration with a low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA). Thus, the presence or absence of marginal gaps was measured at 14 points (each 0.5-mm apart) along the cavity restoration interface (n=10; total points measured=140). This was performed using a traveling microscope (x1,000, Measurescope, MM-11, Nikon, Tokyo, Japan) positioned parallel with the cavity wall and bottom on each half of the sample (Figure 1). The number of gaps in each sample was totaled and expressed as the sum of each sample (Irie & Suzuki, 2002).

The results were analyzed statistically using Tukey Test (Non-parametric) (Conover & Iman, 1981; Irie & Suzuki, 2002).

RESULTS

Table 3 summarizes the findings for the gap formation of RMGIC (Fuji II LC) observed in the Class V restorations with various storage periods. With immediate inspection conducted after setting, the authors observed 101 gaps at the restoration interfaces. The severest points, 1 and 14, showed all gaps in this condition. At the cervical corner area, 10-11, most also showed gaps. The axial regions, 5-9, showed more than half the number of gaps in the same condition. However, when the specimen was cut and inspected after storing in water for only 30 minutes, 34 gaps were observed around the restorative cavities. Significant differences were observed between the two conditions. No significant differences were found among the sum of gaps for five different storage period restorations (30 minutes—1 week). However, polished points, 1 and 14 (enamel substrate), showed half the number of gaps in the restorative cavities. The cervical corner, 11, also showed many gaps.

Table 4 summarizes the findings for the gap formation of RMGIC (Vitremer) observed in the Class V restorations with various storage periods. With the three storage periods (immediately, after 30 minutes and after three hours), the authors observed 128, 119 and 95 gaps, respectively, around the restorative cavities. At the severest points, 1 and 14, all showed gaps in the three conditions. The coronal and cervical areas also mostly showed gaps. The axial regions showed more than half the number of gaps in the same condition. However, when the specimen was cut and inspected after storing in water for 12 hours, the authors observed 37 gaps around the restorative cavities. Significant differences were observed among the three storage periods (immediately, after 30 minutes and after three hours) and after the 12 hour-storage period. No significant differences were observed among the sum of gaps of the three different storage period restorations. The axial

Table 3: *Effect of Delayed Polishing Period on Interfacial Gap Formation Around Class V Restoration: RMGIC (Fuji II LC)*

	Number of Specimens Showing Gaps														
Delayed Polishing Period	**Coronal**					**Axial**						**Cervical**			**Sum**
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
Immediately	10	7	6	6	7	7	6	8	9	10	10	0	5	10	101 (A)
30 minutes	6	3	1	2	2	2	1	0	0	1	3	2	1	10	34 (B)
3 hours	3	0	0	0	1	2	1	2	0	0	2	0	0	6	17 (B)
12 hours	3	0	0	1	0	1	0	0	1	0	3	0	0	4	14 (B)
24 hours	1	0	0	1	1	2	0	0	2	1	4	0	0	5	17 (B)
1 week	5	0	1	0	0	1	1	0	1	0	0	0	1	7	17 (B)

N=10 (total measuring points, 1~14=140)
Values with the same letters were not significantly different by Tukey Test (Conover & Iman, 1981) ($p<0.05$).

Table 4: *Effect of Delayed Polishing Period on Interfacial Gap Formation Around Class V Restoration: RMGIC (Vitremer)*

	Number of Specimens Showing Gaps														
Delayed Polishing Period	**Coronal**					**Axial**						**Cervical**			**Sum**
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
Immediately	10	10	10	10	9	10	8	8	8	7	10	8	10	10	128 (A)
30 minutes	10	10	10	10	6	7	7	8	8	8	8	8	9	10	119 (A)
3 hours	10	9	8	5	5	4	2	5	4	7	9	9	8	10	95 (A)
12 hours	10	2	0	1	1	2	2	1	1	1	2	2	2	10	37 (B)
24 hours	9	1	0	2	0	0	0	1	2	1	3	0	0	10	29 (B)
1 week	10	1	0	0	0	0	0	0	3	1	5	3	0	10	33 (B)

N=10 (total measuring points, 1~14=140)
Values with the same letters were not significantly different by Tukey Test (Conover & Iman, 1981) ($p<0.05$).

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Table 5: *Effect of Delayed Polishing Period on Interfacial Gap Formation Around Class V Restoration: RMGIC (Photac-Fil)*

	Number of Specimens Showing Gaps														
Delayed Polishing Period	**Coronal**						**Axial**					**Cervical**			**Sum**
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
Immediately	10	10	9	10	10	10	10	10	10	10	10	9	10	10	138 (A)
30 minutes	10	9	8	10	10	10	10	10	10	10	10	10	10	10	137 (A)
3 hours	9	7	5	1	0	2	6	6	6	4	10	7	9	10	82 (B)
12 hours	6	2	0	0	0	0	2	2	4	3	6	3	0	4	32 (C)
24 hours	7	0	0	0	1	1	1	0	0	4	8	0	0	10	32 (C)
1 week	9	0	0	1	1	1	0	0	0	1	8	3	2	9	35 (C)

N=10 (total measuring points, 1~14=140)
Values with the same letters were not significantly different by Tukey Test (Conover & Iman, 1981) (p<0.05).

Table 6: *Effect of Delayed Polishing Period on Interfacial Gap Formation Around Class V Restoration: Compomer (Dyract)*

	Number of Specimens Showing Gaps														
Delayed Polishing Period	**Coronal**						**Axial**					**Cervical**			**Sum**
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
Immediately	9	6	3	1	2	1	0	0	0	0	1	0	6	9	38 (A)
30 minutes	10	10	4	2	0	0	0	0	0	0	4	1	1	10	42 (A)
3 hours	10	10	5	4	0	0	0	0	0	0	0	2	2	10	43 (A)
12 hours	10	5	1	4	2	1	0	0	0	0	4	0	2	10	39 (A)
24 hours	9	4	3	2	3	2	0	0	0	0	0	0	3	9	35 (A)
1 week	9	7	6	2	1	1	0	0	0	0	2	0	0	7	35 (A)

N=10 (total measuring points, 1~14=140)
Values with the same letters were not significantly different by Tukey Test (Conover & Iman, 1981) (p<0.05).

regions showed a few gaps in the same conditions. However, at polished points, 1 and 14 (enamel substrate), all showed gaps in the restorative cavities.

Table 5 summarizes the findings for gap formation of RMGIC (Photac-Fil) observed in Class V restorations with various storage periods. With the two storage periods (immediately and after 30 minutes), the authors observed 138 and 137 gaps, respectively, around the restorative cavities. At the severest points, 1 and 14, all showed gaps in the two conditions. The coronal, axial and cervical areas also mostly showed gaps. However, when the specimen was cut and inspected after storing in water for three hours, 82 gaps were observed around the restorative cavities. Significant differences were observed between the two storage periods (immediately and after 30 minutes) and after the three-hours storage time. Next, when the specimen was cut and inspected after storing in water for 12 hours, the authors observed 32 gaps around the restorative cavities. Significant differences were observed between the 3- and 12-hour storage times. No significant differences were observed among the sum of gaps for the three different storage period restorations (12 hours, 24 hours and 1 week). The axial regions showed a few gaps in the same conditions. However, polished points 1 and 14 (enamel substrate) mostly showed gaps in the restorative cavities.

Table 6 summarizes the findings for gap formation of the Compomer (Dyract) observed in the Class V restorations with various storage periods. When the six storage period procedures were compared, the immediate procedure was 38, the 30-minute period was 42, the 3-hour period was 43, the 12-hour period was 35, the 24-hour period was 35 and the 1-week period was 35, respectively. No significant differences were observed among the six-period sum number of gaps in the cavity-restoration interfaces. For Dyract, there was no effect in delaying the polishing for 12 hours, 24 hours or 1 week. When the restoration surface was polished, 1 and 14 (enamel substrate) showed the most gaps after the polishing procedures for all six conditions. The authors observed several gaps at a closer point, 2 (enamel substrate). Namely, there was no significant difference among the storage period procedures. However, the dentin margin showed no or few gaps in the six conditions.

Table 7 summarizes the findings of the gap formation of CGIC (Fuji II) observed in the Class V restorations with various storage periods. With immediate inspection after setting, the authors observed 117 gaps around the restorative cavities. At the severest points, 1 and 14, most showed gaps in this condition. At the cervical corner area, 9-11, most also showed gaps. At the axial regions, most showed gaps in the same condition. However, when the specimen was polished and inspected after storing in water for only 30 minutes, 26 gaps were observed around the restorative cavities. Significant differences were observed between the two conditions. No significant differences were observed among the sum of gaps for the five different storage period restorations. When the specimens were polished and inspected after storing in water for more than three hours, only a few gaps were observed around the restorative cavity. However, at polished points 1 and 14 (enamel substrate), a few gaps appeared in the restorative cavities. The cervical corner, 11, also showed many gaps. The presence of gaps was almost zero at all

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inspected points in the axial region, 4-11.

Table 8 summarizes the findings of the gap formation for the Composite (Silux Plus) observed in the Class V restorations with various storage periods. When the six storage time procedures were compared, the immediate procedure was 64, the 30-minute was 48, the 3-hour was 53 and the 12-hour was 51. No significant differences were observed among the four sum numbers of gaps in the cavity-restoration interfaces. At the severest points, 1 and 14, half showed a gap in this condition. However, when the specimen was polished and inspected after storing in water for 24 hours or 1 week, 28 and 25 gaps were observed around the restorative cavities, respectively. Significant differences were observed between the 12-hour and 24-hour condition. However, not only the dentin margin but also the enamel margin, namely, the entire margin, showed several gaps in the six conditions.

Table 7: *Effect of Delayed Polishing Period on Interfacial Gap Formation Around Class V Restoration: GIC (Fuji II)*

	Number of Specimens Showing Gaps														
Delayed Polishing Period	**Coronal**					**Axial**					**Cervical**				**Sum**
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
Immediately	10	5	7	6	8	8	9	8	10	9	10	8	9	10	117 (A)
30 minutes	10	0	0	1	0	0	0	0	0	2	4	0	0	9	26 (B)
3 hours	2	0	0	0	0		0	0	0	0	4	0	0	3	9 (B)
12 hours	1	0	0	0	0	0	0	0	0	0	2	0	0	3	6 (B)
24 hours	0	0	0	1	1	0	0	0	0	1	3	0	0	1	7 (B)
1 week	2	0	0	0	0	0	0	0	0	0	2	0	0	1	5 (B)

N=10 (total measuring points, 1~14=140)
Values with the same letters were not significantly different by Tukey Test (Conover & Iman, 1981) (p<0.05).

Table 8: *Effect of Delayed Polishing Period on Interfacial Gap Formation Around Class V Restoration: Composite (Silux Plus)*

	Number of Specimens Showing Gaps														
Delayed Polishing Period	**Coronal**					**Axial**					**Cervical**				**Sum**
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
Immediately	8	4	3	8	6	4	4	3	2	4	8	2	3	5	64(A)
30 minutes	5	2	0	1	4	5	4	3	2	3	6	1	4	8	48 (A, B)
3 hours	5	1	1	3	7	6	5	4	6	4	6	0	1	4	53 (A, B)
12 hours	4	1	0	0	3	5	6	7	6	4	7	2	2	4	51 (A, B)
24 hours	3	0	0	0	2	4	5	3	2	3	4	0	1	1	28 (B)
1 week	5	0	0	5	1	2	3	4	3	1	0	0	0	1	25 (B)

N=10 (total measuring points, 1~14=140)
Values with the same letters were not significantly different by Tukey Test (Conover & Iman, 1981) (p<0.05).

DISCUSSION

This study clearly demonstrated that polishing three RMGICs and one CGIC (Fuji II LC, Vitremer, Photac-Fil and Fuji II) should not be performed immediately after the filling and setting procedure. Polishing should be delayed between 3 and 12 hours to prevent gap formation at the material-tooth cavity interface. In contrast to the presence of approximately 100-140 of 140 (total measurement points) gaps at the material-tooth cavity interface in the Class V cavity of specimens polished immediately after setting, the gap was near zero or only a few when the specimen was polished after storage in water for 3 or 12 hours. RMGIC or CGIC shrinks during the setting reaction. Gaps form as the adhesion between the tooth cavity and glass ionomer does not resist the stress formed by cement shrinkage (Feilzer, de Gee & Davidson, 1988; Sidhu, 1994; Feilzer & others, 1995). One reason for the gap being dependent on the storage period may be the hygroscopic expansion of the glass ionomer. As soon as adhesion of a restoration is disturbed by shrinkage stress during setting, subsequent swelling by water sorption will never lead to closure. A mismatch between the surface of the cavity wall and the opposing surface of the restoration, due to dimensional changes of the various restorations, almost prevent this. After 12 hours of water storage, curing contraction stresses of the materials are effectively compensated for or even converted into expansion stress due to water uptake and swelling (Feilzer & others, 1995). This effect is reported for the uptake of water by the matrix of resin-modified glass ionomers forming a poly-HEMA complex (Wilson, 1990). In addition, CGIC forms a hydrogel of calcium and aluminum polyacrylates by its uptake of water (Wilson & McLean, 1988). Water absorption of RMGICs and CGICs reportedly affects cavity adaptation and reduces microleakage (Fritz & others, 1996b; Irie & Suzuki, 2000). Although hygroscopic expansion may not be enough to compensate for setting shrinkage, it plays an important role in reducing shrinkage caused by the cement setting reaction and thus improves the marginal seal (Sidhu & others, 1997; Irie & Suzuki, 2000).

The cement is expected to show higher bond and mechanical strengths when fully set rather than during setting reaction. It is suggested that the bonding ability to tooth substrate increases with the development of glass ionomer/tooth substrate interaction during stor-

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age in water, and the cohesive strength of the cement itself improves with the setting process (Irie & Suzuki, 2000). The pH, an index of the degree of hardening reaction of set glass ionomer, is reported to be lower at the initial stage (until 30 minutes) regardless of the type of cement, whether CGIC or RMGIC. Although there was no significant difference among 3-, 12- and 24-hour conditions, all of which prevent gap formation at the material-tooth cavity interface, the pH value of the set cement gradually increases for 24 hours (Tosaki & Hirota, 1994; Anusavice, 1996). Therefore, it can be presumed that completing the setting reaction of an RMGIC or CGIC requires 24 hours. Thus, RMGIC or CGIC requires 24 hours for adequate mechanical strength, which has a close relationship with bond strength (Irie & Suzuki, 2000). RMGIC has a dual setting reaction: one is the light-initiated cross-linking of methacrylate groups similar to the setting of light-cured resin composites; the other is an acid-base reaction similar to that of a CGIC (Wilson, 1990; Mitra, 1991).

The cervical corner of Class V cavity restorations showed more gaps than the coronal corner in RMGICs and CGICs. This was expected because bond strength to coronal dentin is usually higher than bond strength to cervical dentin, because cervical dentin is a less favorable bonding substrate (Heymann & Bayne, 1993; Irie & Suzuki, 2002).

Not one of the samples prevented gaps at the two polished points for Vitremer (1 and 14; enamel substrate). The reason is that Vitremer shows a low bond value to enamel after one day. A failure site after debonding showed a mixed failure of Vitremer bonded to enamel (Fritz & others, 1996a; Irie & Suzuki, 2000).

Photac-Fil showed no effect in preventing gaps around Class V restorations during the initial stage. Fracturing of the debonded area in the material-tooth cavity interface showed very weak bonding during the early phase and probably produced very low bond value to dentin (Irie & Suzuki, 2000). It is possible that exposure of Photac-Fil surface to water immediately after light activation may have a similar adverse effect on setting, as commonly found with CGICs after early water exposure. Other investigations have also reported the poor bonding capacity of Photac-Fil with Ketac Conditioner (Sidhu & Watson, 1995; Fritz & others, 1996a,b).

This study demonstrated that delaying polishing of a compomer for one day does not prevent gap formation in the material-tooth cavity interface. Therefore, it did not prevent bonding to the tooth structure and the hygroscopic expansion of compomer, itself (Irie & Suzuki, 2000). The marginal gaps observed even after the specimen was stored in water for one day indicated that hygroscopic expansion does not fully compensate for shrinkage caused by setting reaction. The degree of hygroscopic expansion of the compomer was smaller than RMGICs, because the composition of the compomer is similar to the resin composite system (Hammesfahr, 1994; Irie & Suzuki, 2000). The values of the bond strengths to enamel and dentin measured after one-day storage were not significantly higher than those measured immediately for the compomer (Irie & Suzuki, 2000). Since no micro-mechanical interlocking with conditioned enamel was available when Dyract-PSA was used, a low shear bond strength to enamel was shown even after storage for one day (Fritz & others, 1996a).

This study demonstrated that the polishing period of the compomer showed no effect in preventing gaps around Class V restorations, especially at the enamel margin. The gap width of the enamel margin was found to result from polishing procedures because the enamel margin had no gaps without the polishing procedure (Irie & Suzuki, 2002). The manufacturer suggests that tooth etching with Dyract is unnecessary for most restorative procedures. Although the recommended primer is considered adequate to condition tooth-hard tissues, it has been demonstrated that compomer restorations lack marginal adaptation (Ferrari & others, 1998; Tyas, 2000; Di Lenarda & others, 2000; Brackett & others, 2001). A modified formulation of Dyract is now available (Dyract AP, Dentsply/DeTrey, Konstanz, Germany). However, the manufacturer still considers etching unnecessary even for this material. To improve the enamel/compomer interface, the manufacturer's protocol should be modified, introducing enamel etching to achieve the compomer restorations' clinical success.

Silux Plus demonstrated that preventing gap formation to enamel substrate was poor immediately following setting, and for dentin substrate, it was not significantly better compared with RMGICs and CGICs in all conditions. It is suggested that bonding at the material-tooth cavity interface was insufficient. A previous study showed that not only the value of the bond strength to enamel substrate of Silux Plus immediately following setting was low (only 6.83 MPa), but also that the value of the bond strength to dentin substrate after one-day storage of Silux Plus was significantly lower (immediately: 5.97 MPa; after one-day storage: 8.63 MPa) than RMGICs and CGICs (Irie & Suzuki, 2000). It may be suggested that the enamel cavosurface margin is not a bevel but a butt joint.

The restorative materials tested in this study should not be polished at the placement appointment except for the compomer, based on gap formation at the material-tooth cavity interface and completion of the setting reaction. Instead, the prepared cavity should be filled at the placement appointment and polished at a subsequent appointment (Irie & Suzuki, 2000; 2002).

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CONCLUSIONS

All materials except compomer showed a significant reduction in interfacial gap formation by 24 hours after insertion. Although compomer did not exhibit a change in gap formation, the number of gaps at any time interval was similar to the other materials at their lowest gap conditions. Of the materials undergoing changes in gap formation, CGICs showed the most improvement and microfilled composite showed the least improvement.

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Effect of Surface Finishing and Storage Media on Bi-axial Flexure Strength and Microhardness of Resin-Based Composite

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Clinical Relevance

Resin-based composites left with a Mylar finishing must receive additional finishing to remove the resin-rich layer. This layer may be responsible for reduced values on flexural strength and microhardness.

SUMMARY

This *in vitro* study tested the following null hypotheses: (1) surface finishing treatments do not significantly affect the biaxial flexure strength and microhardness of resin-based composites (RBC) and (2) storage media do not significantly affect these physical properties. Discs (81 RBC and 81 UR; 3M/ESPE) were prepared using a circular polyethylene mold (2.4-mm thick x 16.7 mm in diameter) that was polymerized through a Mylar strip and divided into three surface finishing treatment groups: 1 µm aluminum oxide slurry; 15 µm diamond and a Mylar strip. Randomly selected controls for each finishing group were stored at room temperature in individual vials. Test specimens were immersed in water, stored at 37°C for two days and transferred for an additional seven days to one of three aqueous storage media at 37°C: coffee (pH 5.1), cola (pH 2.4) or red wine (pH 3.7). Post immersion (nine days total), the specimens were tested for biaxial flexure strength (BFS) and Vicker's microhardness (VHN). ANOVA and Tukey's HSD test were used for statistical analysis. ANOVA results indicated that surface finishing treatments had a significant effect on the biaxial flexure strength and microhardness of the RBC and the UR specimens. BFS results for RBC specimens were AL>DD>ML ($p<0.0001$) and VHN results were AL, DD>ML ($p<0.0001$). Storage in wine medium reduced the VHN of UR speci-

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mens significantly. Both alternative hypotheses were accepted. In addition, the Mylar finishing group, because of the resin-rich surface layer, yielded the lowest mean values of BFS and VHN.

INTRODUCTION

The intrinsic properties of resin-based composite (RBC) materials, such as hardness and strength, are important mechanical characteristics that contribute to the clinical success of the restorative material. Hardness, the resistance to permanent surface indentation, is related to ease in finishing the surface of the material and the resistance to surface scratches (Craig, 1997). Furthermore, hardness can indicate the depth of cure of the material. The biaxial flexure test can be used to determine the fracture characteristics of dental restorative materials. In addition, the physical characteristics of resin composites (McCabe & Kagi, 1991), particularly their hardness (Eliades, Vougiouklakis & Caputo, 1987; Wendt, 1987), have proven to be good predictors of the conversion rate of resins (Rueggeberg & Craig, 1988).

Finishing procedures affect various aspects of the final restoration, including surface staining, plaque accumulation and wear resistance (Weitman & Eames, 1975; Igarashi & others, 1976; Jefferies, 1998), indicating that surface finishing is an integral part of the material's integrity. However, different finishing treatments can also affect properties of the composite (Yap, Lye & Sau, 1997). Materials with low surface hardness are more susceptible to scratching. Surface scratches can compromise fatigue strength and lead to premature failure of the restoration (Craig, 1997). In addition, aggressive finishing techniques or overheating can damage the surface of RBC materials, resulting in accelerated wear (Wu & McKinney, 1982; Wu & others, 1984; Leinfelder, Wilder & Teixeira, 1986).

The resin matrix has been shown to be the critical component influencing the mechanical properties of RBC materials (Dietschi & others, 1994). This resin matrix is affected by different pH solutions (Ortengren, 2000) and alcohol-containing solutions (Deepa & Krishnan, 2000). Previous studies have reported that alcohol plasticizes the resin matrix, making it soft and prone to degradation (Ferracane & Marker, 1992; Ferracane & Berge, 1995).

This research investigated the influence of three finishing treatments and three aqueous storage media on the microhardness and biaxial flexure strength of resin-based composite material. The primary null hypothesis tested that finishing treatments do not significantly affect the microhardness and flexural strength of RBC materials. A second null hypothesis was that storage in liquid media would not affect the mechanical properties of the RBC.

METHODS AND MATERIALS

Eighty-one discs were prepared with a small particle resin-based composite (Filtek Z-250, shade A-1, Batch #6020A1, 3M/ESPE Dental Products, St Paul, MN, USA), and another eighty-one discs were prepared using unfilled resin (UR) from the same batch number (Batch #6020A1, 3M/ESPE Dental Products).

A circular polyethylene mold (16.7-mm in diameter x 2.4-mm thick) was used to fabricate each of the specimens individually. The discs were built incrementally and polymerized for 20 seconds on the first layer and 40 seconds on the second layer with a light-curing unit (Demetron, Division of Kerr Corporation, Danbury, CT, USA). The output of the light unit was measured daily with a curing radiometer (Demetron) to ensure a constant value of at least 470 mW/cm^2 (Rueggeberg, Caughman & Curtis, 1994). A transparent, 0.051-mm thick Mylar strip (DuPont Mylar, Henry Schein Inc, Port Washington, NY, USA) was placed against the bottom and top layers. The specimens of both material groups (RBC and UR) were randomly assigned to two test categories: biaxial flexure strength (BFS, n=72) and Vicker's microhardness test (VHN, n=9) as shown on Figure 1.

Each test group was further divided into three finishing treatment groups (n=24 for BFS; n=3 for VHN) with a final surface finishing of 1-µm aluminum oxide, 15-µm diamond and Mylar. Specimens in the aluminum oxide group were wet polished through a series of silicon carbide abrasives (400, 600, 1200 and 2000 grits; 3M). The final surface was polished with 1-µm aluminum oxide slurry. The diamond groups were initially wet polished with 400 grit SiC paper, then with 30- and 15-µm diamond plates (MB Diamond Plates, Mark V Laboratory, East Granby, CT, USA). The third group, Mylar, remained as cured through the Mylar strip.

All specimens, after surface treatment had been designated, were placed in individual vials with 20 mls of distilled water at 37°C for two days. Storage in water was important to simulate what occurs in a clinical situation after the restoration has been completed. Following water immersion, the BFS specimens of each surface treatment group were evenly divided into one control group and three storage subgroups (n=6): coffee (Jackson Creek Coffee, Mobile, AL, USA); red wine, (Concha Y Toro Frontera, Chile) and cola (Coca-Cola Classic, The Coca-Cola Company, Atlanta, GA, USA). The pH measurements of the storage media were taken prior to sample immersion. Post two-day water immersion, the specimens were immersed individually in 20 mls of the designated storage medium at 37°C for seven days. The control groups remained stored in individual vials and not subjected to further immersion. The biaxial flexure strength specimens, including controls, were tested on a universal testing machine (Instron

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Universal Testing Machine 1125, Instron Corp, Canton, MA, USA) using a pin-on-three-ball fixture with a 0.5mm/minute crosshead speed. The biaxial flexural strength in MPa was calculated using equations developed by Marshall (1980) and Shetty and others (1980). The following variables were applied in the formula for calculation of the failure stress: applied load at failure, Poisson's ratio, radius of the support circle, radius of the pin used on the loading surface and radius and thickness of the specimen.

Following two-day immersion in water, the VHN specimens were evenly divided into three storage media (n=1): coffee, red wine and cola. Prior to the seven-day immersion as described for BFS specimens, baseline microhardness values in MPa were determined using a Vicker's diamond indenter under a 200 gram load for a dwell time of 18 seconds on a Tukon Microhardness Tester (Wilson Instrument Division, Bridgeport, CT, USA). A total of 20 indentations were made per specimen. Following immersion, second

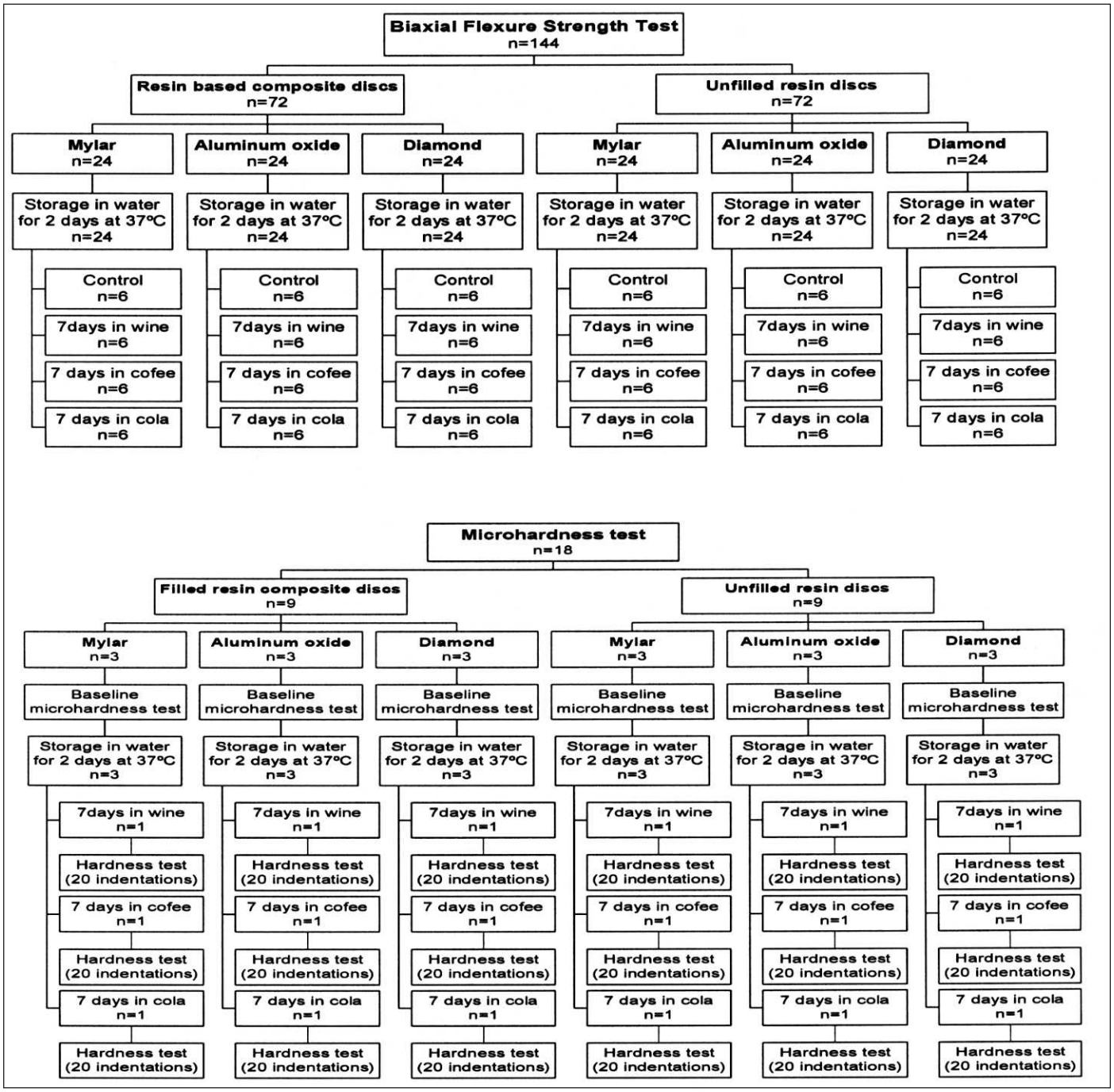

Figure 1. *Schematic diagrams illustrating the study design.*

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hardness measurements were taken on the same specimens but in different locations.

For both biaxial flexure strength and microhardness tests, the values were reported in MPa.

ANOVA and Tukey's HSD test (α=0.05) were used for statistical analysis.

RESULTS

This experimental design involved the following variables: filler particles, present (RBC) and not present (UR); three surface finishing treatments and three storage media, resulting in 18 experimental groups.

A three-way ANOVA showed that all three variables had statistically significant effects on the BFS values and there was interaction between finishing and medium (p=0.0016). Tukey's HSD test showed that RBC exhibited higher BFS values than UR specimens (α=0.05). Two-way ANOVA tests on the influence of finishing and storage media for RBC and UR were carried out. For RBC, both finishing and storage media exhibited significant influence on the BFS, and there was no interaction. Tukey's test showed that aluminum oxide and diamond finishing groups yielded higher BFS than Mylar finishing, and the control groups had higher BFS, while there was no difference among the three storage groups [coffee (pH 5.1), cola (pH 2.4), red wine (pH 3.7)]. Table 1 shows the BFS values of all test groups of RBC with respective grouping of finishing by Tukey's tests for each storage medium.

For UR specimens, the analysis showed the same results as in RBC, except that there was interaction between the finishing and the medium. A series of one-way ANOVA tests were performed to determine the effects of finishing for each storage condition. The tests showed that finishing exhibited significant influence in each storage medium. Table 2 shows the BFS values of UR and the respective grouping of finishing by Tukey's tests for each storage medium. In general, Mylar finishing was always in the lower BFS value groups, while aluminum oxide and diamond finishing exhibited higher BFS values or no difference to the Mylar group. Figure 2 shows the effect of storage media on the BFS values of UR for each finishing group. The control groups consistently showed the highest numbers, while the wine group showed the lowest values.

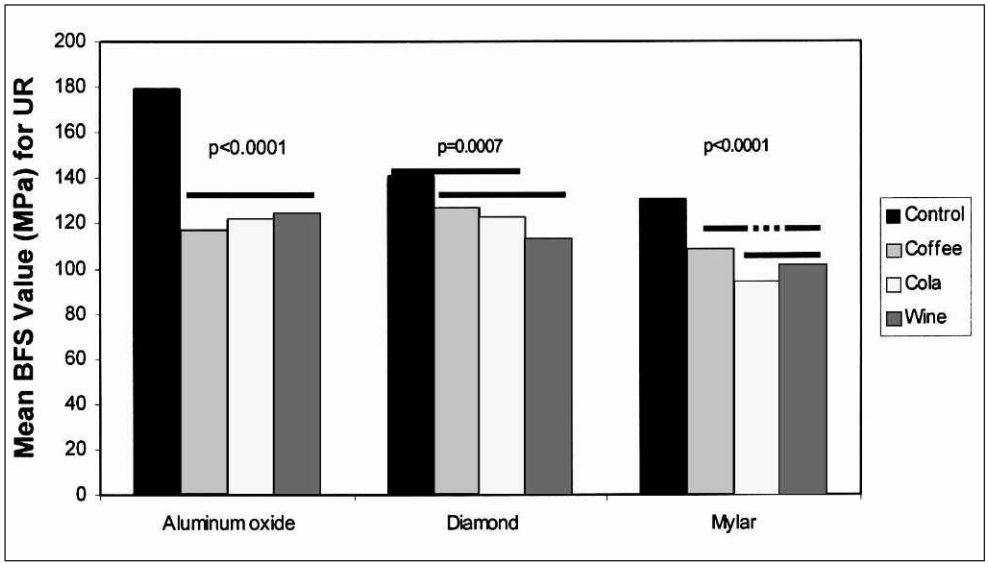

Figure 2. *Bar graph illustrating the influence of storage medium on the BFS values of each surface finishing group; bars under the same solid lines are not statistically different by Tukey's test (α=0.05).*

Table 1: *RBC Mean Biaxial Flexure Strength Values (MPa) for Untreated Controls and Post Nine Days Immersion*

Biaxial Flexure Strength (MPa) for RBC								
Finishing Treatment	**Controls**		**Coffee**		**Cola**		**Wine**	
	Mean(SD)	**THSD**	**Mean(SD)**	**THSD**	**Mean(SD)**	**THSD**	**Mean(SD)**	**THSD**
Aluminum oxide (1 µm)	218 (23)	A	165 (19)	A	172 (33)	A	179 (21)	A
Diamond (15 µm)	200 (20)	AB	179 (22)	A	147 (36)	A	137 (37)	B
Mylar	157 (41)	B	140 (26)	A	134 (20)	A	137 (16)	B

Standard deviation (SD) and Tukey's HSD grouping (THSD) are presented.

Table 2: *Unfilled Resin (UR) Mean Biaxial Flexure Strength Values (MPa) for Untreated Controls and Post-nine Days Immersion*

Biaxial Flexure Strength (MPa) for UR								
Finishing Treatment	**Controls**		**Coffee**		**Cola**		**Wine**	
	Mean(SD)	**THSD**	**Mean(SD)**	**THSD**	**Mean(SD)**	**THSD**	**Mean(SD)**	**THSD**
Aluminum oxide (1 µm)	179 (17)	A	117 (14)	A	122 (9)	A	124 (4)	A
Diamond (15 µm)	141 (15)	B	127 (5)	A	123 (13)	A	113 (14)	AB
Mylar	131 (6)	B	108 (9)	B	94 (10)	B	101 (8)	B

Standard deviation (SD) and Tukey's HSD grouping (THSD) are presented.

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Two-way ANOVA on the effect of finishing and media on the pre-immersion hardness values of RBC and UR showed that surface finishing exhibited significant influence but not the medium designation. In addition, there were no interactions between the two variables. This result was expected since the specimens had been assigned for immersion in certain medium but were not yet immersed. Therefore, all pre-immersion hardness values for each finishing group were considered as the baseline for the immersion groups, which was comparable to the control in the analysis of BFS values.

A three-way ANOVA was used to determine the effect of the filler presence, finishing and storage media (including baseline). The results showed that all three variables had significant influence on the hardness values, and there were interactions among variables. Since the microhardness values for RBC were significantly higher than UR, two-way ANOVA tests on the influence of finishing and storage media for RBC and UR were performed. For RBC specimens, both finishing and media storage had a significant influence on hardness values and there was interaction between the two variables. A series of one-way ANOVA tests were performed to examine the influence of finishing and storage media. Table 3 shows the results of grouping with respective finishing groups by Tukey HSD for each medium. Figure 3 shows the effect of storage medium on the hardness values of RBC for each finishing group. Baseline groups were consistently in the highest groups, while the wine group was always in the lowest groups.

For UR specimens, both finishing and storage media had significant influence on the hardness values and there was no interaction between the two variables. Tukey HSD test showed that the grouping of hardness values by finishing was diamond > aluminum oxide and Mylar finishing group, and the grouping by medium was baseline > coffee, cola > wine. Table 4 shows the hardness values of all test groups of UR with respective grouping of finishing by Tukey HSD for each medium.

DISCUSSION

The surface finishing of resin-based composite (RBC) restorations is a critical procedure pertaining to the maintenance of this restorative material. It is equally important to understand the effects of daily consumption of dietary drinks and foods. Several studies have shown that absorption of various solvents may cause adverse effects on RBC materials (Vogel, 1975; Ferracane & Marker, 1992; Nathoo & Gaffar, 1994). While limited uptake of a solvent may enhance the mechanical properties of a brittle resin through plasticization, further uptake may cause degradation of the resin matrix, facilitating crack propagation. It is also possible that the sorption of solvent might cause localized crazing and a degradation of the filler/matrix interface (Wu, 1982; Asmussen, 1984; McKinney & Wu, 1985; Ferracane, Berge & Condon, 1998).

The finishing procedure, as performed in a clinical setting, can also affect the physical properties of RBC materials (Wu & others, 1984; Leinfelder & others, 1986). Examination of removed composite restorations suggested that physico-chemical stresses had resulted in the formation of microcracks, microvoids or interfacial gaps at the interface between filler and matrix (Mair, 1989; 1991). It is important to note that these observations were not made on the effects of finishing procedure on surfaces with Mylar finishing, but on the composite surfaces with the resin-rich layer removed.

In this study, the mean flexure strength values and microhardness values of the Mylar finishing groups

Table 3: *Filled Resin Composite (RBC) Mean Microhardness Values (MPa) for Untreated Specimens Pre-immersion and Post-nine Days Immersion*

Vicker's Hardness Values (MPa) for RBC								
Finishing Treatment	**Baselines**		**Coffee**		**Cola**		**Wine**	
	Mean(SD)	**THSD**	**Mean(SD)**	**THSD**	**Mean(SD)**	**THSD**	**Mean(SD)**	**THSD**
Aluminum oxide (1 μm)	1147 (232)	A	1084 (84)	A	1056 (117)	A	1034 (108)	A
Diamond (15 μm)	1039 (142)	B	1044 (142)	A	1090 (124)	A	949 (76)	B
Mylar	1001 (165)	B	884 (74)	B	888 (103)	B	981 (120)	AB

Standard deviation (SD) and Tukey's HSD grouping (THSD) are presented.

Table 4: *Unfilled Resin (UR) Mean Microhardness Values (MPa) for Untreated Specimens Pre-immersion and Post-nine Days Immersion*

Vicker's Hardness Values (MPa) for UR								
Finishing Treatment	**Controls**		**Coffee**		**Cola**		**Wine**	
	Mean(SD)	**THSD**	**Mean(SD)**	**THSD**	**Mean(SD)**	**THSD**	**Mean(SD)**	**THSD**
Diamond (15 μm)	233 (46)	A	221 (43)	A	213 (50)	A	179 (22)	A
Aluminum oxide (1 μm)	189 (16)	B	167 (11)	B	170 (18)	B	140 (11)	B
Mylar	203 (40)	B	175 (9)	B	169 (9)	B	142 (17)	B

Standard deviation (SD) and Tukey's HSD grouping (THSD) are presented.

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were either the lowest of the three finishing groups or not statistically different from the lowest ones (Tables 1-4). In the Mylar finishing group, the resin was polymerized through a transparent 0.051-mm thick Mylar strip. Several studies have reported that the smoothest possible surface is obtained when RBC material is polymerized against a clear polyester strip (Pratten & Johnson, 1988; Stoddard & Johnson, 1991; Dietschi & others, 1994; Yap & others, 1997). When the surface of RBC has been cured under a certain pressure against a smooth surface, a resin rich surface layer is formed (Von Fraunhofer, 1971; Hachiya & others, 1984; Okazaki & Douglas, 1984; Stanford & others, 1985).

There is a possibility that the degree of polymerization of this resin-rich surface layer is lower than the bulk of the material because of oxygen inhibition. Previous studies have reported that incomplete polymerization due to oxygen at the restoration surface may affect aspects of resin-based composite materials (Gross & Moser, 1977; Rueggeberg & Margeson, 1990). The depth of this resin rich layer is approximately 5 µm by the SEM micrographs (Ozakai & Douglas, 1984). This magnitude of thickness would probably be removed during any finishing procedure on the other experimental groups used in the study. Since the resin-rich layer is a much weaker phase than the bulk of the cured material, existence of the layer would likely yield a strength value or hardness value lower than discs with the resin-rich layer removed. Apparently, the benefit of removing the resin-rich surface layer outweighed the potential ill effects that the finishing procedures could generate.

In general, there was a reduction in flexure strength and microhardness after nine days storage, which included two days in water and seven days in respective storage medium. The only exception was the hardness of RBC with diamond finishing immersed in cola, but the change was not statistically significant (Figure 3). By the results of Tukey's HSD tests, the mean BFS values of the group finished with 1-µm aluminum oxide usually ranked higher than the other two finishing methods, and occasionally, there was no statistical difference among the three methods (Tables 1 and 2). Although the mean BFS values of the group finished with 15-µm diamond and immersed in coffee exhibited the highest values, they were not statistically different from those finished with 1-µm aluminum oxide. The analysis also showed that finishing with 1-µm aluminum oxide and 15-µm diamond exerted similar influence on the mean microhardness values as for the mean BFS values. Since the resin matrix is susceptible to thermal changes (Wu & others, 1984; Leinfelder & others, 1986), it is reasonable to speculate that finishing procedures that require grinding through a series of abrasive papers of different grits and polishing with fine particles can generate enough thermal energy to cause further polymerization of the resin matrix. If the additional polymerization had occurred in this study, it would have augmented the strength and hardness of the discs in addition to those by the removal of resin-rich surface. This conjecture, however, could not be validated by this experiment, as the amount of water used in polishing kept the surface temperature low and minimized the potential for heating.

The mean BFS and microhardness values of the discs immersed in wine usually ranked in the group of lower values by Tukey's HSD tests. The resin matrix has been reported to be a critical component that influences the mechanical properties of the RBC (Li & others, 1985; Drummond & Savers, 1993; Ferracane & Berge, 1995; Ortengren, 2000), because it is responsible for the sorption of the RBC systems (Deepa & Krishnan, 2000). The wine used in this study had an alcohol content close to 12% by volume. Several studies have reported that alcohol weakens the resin matrix, changing the physical properties of RBC materials (Ferracane & Marker, 1992; Ferracane & Berge, 1995; Deepa & Krishnan, 2000). The weakened resin matrix has been shown to produce a reduction in the fracture resistance of BisGMA-based composites (Ferracane & Berge, 1995; Ferracane & others, 1998). Most of the studies

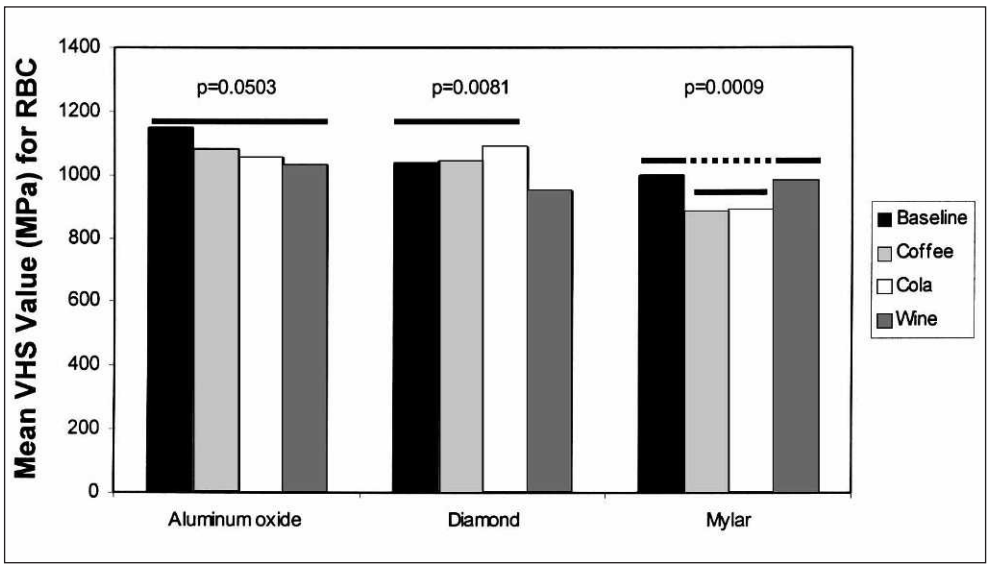

Figure 3. *Bar graph illustrating the influence of storage medium on the hardness values of each surface finishing group; bars under the same solid lines are not statistically different by Tukey's test (α=0.05).*

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reported storage time in alcohol for more than a month. In the current study, the specimens were stored in wine for only seven days, which was time enough to affect the unfilled resin group, suggesting that the matrix is the least resistant component of RBC materials changing upon uptake of different solvents.

CONCLUSIONS

This *in vitro* study showed that Mylar finishing produced the lowest mean biaxial flexure strength and Vicker's microhardness values for both resin based composite materials and unfilled resin.

Surface finishing treatments by polishing with abrasive paper and finishing with aluminum oxide or diamond under water essentially removed the resin-rich layer of Mylar finishing and significantly increased the biaxial flexure strength and the Vicker's microhardness of most of the groups tested. The use of 1-µm aluminum oxide often yielded greater improvement in strength and hardness than 15-µm diamond. The first alternative hypothesis was accepted.

Wine as a storage medium resulted in a significant reduction in mean microhardness and flexure strength values for the unfilled resin. The second alternative hypothesis was also accepted.

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Effect of Application Technique and Dentin Bonding Agent Interaction on Shear Bond Strength

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M Yashar • AA Caputo

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Clinical Relevance

Bond strength of resin composite to dentin can be improved by utilizing a one-second compressed air technique to form a thin, uniform adhesive layer that remains on the dentin surface.

SUMMARY

This study evaluated the interaction of five clinical application techniques and the shear bond strength of four DBAs (OptiBond FL, Clearfil SE Bond, PQ1 and Prime & Bond NT). A hybrid resin composite (Herculite XRV restorative resin) was attached to human dentin surfaces using five application techniques: Group A—adhesive spread with a 3M brush for 30 seconds, followed by compressed air 0.5 cm from the surface for one second to remove the excess adhesive. Group B—adhesive spread with a 3M brush for 30 seconds, followed by compressed air 0.5 cm from the surface for three seconds to remove the excess adhesive. Group C—adhesive spread with 3M brush for 30 seconds, excess adhesive removed with a clean brush, two strokes side by side, no compressed air. Group D—adhesive spread with a Micro-applicator brush for 30 seconds followed by compressed air 0.5 cm from the surface for one second to remove the excess adhesive. Group E—adhesive spread with a Micro-applicator brush for 30 seconds, the excess adhesive removed with a clean brush, two strokes side by side and no compressed air. The specimens were stored in distilled water at 37°C for 24 hours, followed by thermocycling between 5°C and 55°C for 1,000 cycles. The shear bond strengths were determined on a universal testing machine operating with a crosshead speed of 5 mm/minute. The fracture sites were examined by 20x stereo microscope to determine the type of failure that occurred during the debonding procedure. Bond strength data were compared with analysis of variance at a significance level of $p<0.05$. Post hoc comparisons of means were performed with *t*-tests with *p*-values adjusted for multiple comparisons. This *in vitro* study concluded that there was an interaction between the application technique and bonding agent tested. All DBAs utilized the one-second compressed air technique, which yielded the highest bond strengths.

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INTRODUCTION

Dentin adhesion has become fundamental in restorative dentistry (Swift, Perdigão & Heymann, 1995; Pashley & Carvalho, 1997). Traditional mechanical methods of retaining direct and indirect restorations are increasingly being replaced by tooth-conserving adhesive methods (Christensen, 1998; Shellard & Duke, 1999; Manhart & others, 2000; Belvedere, 2001). In order to overcome the challenge of bonding to dentin, several generations of dentin bonding agents (DBAs) have been developed to increase bond strength of restorative dental materials and reduce microleakage (Van Meerbeek & others, 1992). Lately, significant progress in the clinical performance of DBAs has been made due to improved formulations and modified application techniques (Nakabayashi, Ashizawa & Nakamura, 1992; Perdigão & others, 1999). It is now possible to develop a hybridized zone at the resin-dentin interface that produces bond strengths of 18 to 25 MPa (Nakabayashi & others 1982; Burrow, Satoh & Tagami, 1996). These values approximate the highly predictable bond strength of resin composite to enamel (Swift & others, 1995; Gallo, Burgess & Xu, 2001).

Most conventional DBAs use three steps (conditioner, primer, adhesive) to achieve formation of a resin reinforced dentin zone. The effects of different dentin treatments, including variations in application technique and surface wetness, have been evaluated for conventional DBAs. It has been recommended that the dentin surface should be maintained in a moist state prior to bonding (Kanca, 1992; Gallo & others, 2000).

In the past five years, two new types of DBA have been introduced in an effort to simplify the bonding procedure and improve the shear bond strength of resin composites to dentin. One type combines the primer resin with the adhesive resin into a single component. An etching conditioner is applied separately. The second type is a self-etching DBA that combines a weak phosphoric acid and a primer in one bottle and an adhesive in a second bottle.

Regardless of the bonding agent type, specific directions for application are usually prescribed to achieve a successful clinical outcome. For various reasons, clinicians may deviate from these recommendations, which may lead to a reduction in bond strength. However, there is little information on the interaction of the application technique on the resulting bond strength of DBAs. This *in vitro* study evaluated the interaction of five clinical application techniques and the resulting shear bond strength of four DBAs.

METHODS AND MATERIALS

Fifty caries-free human premolars were stored in distilled water for up to seven weeks following extraction. The teeth were mounted in 25-mm square blocks of cold-cure acrylic resin (Co-Oral-Ite, Dental Mfg Co, Diamond Springs, CA, USA). A modified surveyor table (The JM Ney Company, Hartford, CT, USA) was used to hold the blocks so that each tooth surface would be parallel to the high speed bur mounted in a metallic jig.

Four flat surfaces (facial, lingual and proximal) were cut on each tooth under a water-cooled high-speed turbine (Dentsply Midwest, Des Plaines, IL, USA). A medium grit (100 µm) diamond (Number 847-018, Brasseler USA, Savannah, GA, USA) was used to expose dentin, followed by a tungsten carbide fissure bur (560 XL, Brasseler USA) to cut approximately 0.8 mm into dentin. Flat dentin surfaces approximately 3-mm square were produced. A total of 200 tooth surfaces were prepared on the teeth that were stored in distilled water at room temperature until ready for use.

The DBAs included in this study were OptiBond FL (Kerr Corp, Orange, CA, USA), Clearfil SE Bond (Kuraray Medical Inc, Kurashiki, Okayama, Japan), PQ1 (Ultradent Products Inc, South Jordan, UT, USA) and Prime & Bond NT (Dentsply/Caulk, Milford, DE, USA) (Table 1). These DBAs were applied to the prepared dentin surface utilizing each of the following techniques: Group A—adhesive spread with 3M brush (3M Dental Products, St Paul, MN, USA) (Figure 1A) for 30 seconds, followed by compressed air 0.5 cm from the surface for one second to remove the excess adhesive. Group B—adhesive spread with 3M brush for 30

Table 1: *Dentin Bonding Agents Evaluated*

Bonding Agent	Etchant	Composition
OptiBond FL (Kerr)	37.5% H_3PO_4	Primer: HEMA, GPDM, PAMM camphorquinone (CQ), ethanol, water Adhesive: Bis-GMA, HEMA, GDMA Ba-Al-borosilicate, fumed silica, CQ
PQ1 (Ultradent)	35% H_3PO_4	Primer and adhesive together HEMA, CQ, Ba-borosilicates, natural resins, ethanol, fluoride
Prime & Bond NT (Dentsply/Caulk)	34% H_3PO_4	Primer and adhesive together Di and trimethacrylate resins, PENTA, acetone, CQ, nanofillers (amorphous silicon dioxide)
Clearfil SE Bond (Kuraray)	Self-etching	Primer: MDP, HEMA, CQ Hydrophilic DMA, water N, N-Di ethanol p-toluidine Adhesive: MDP, Bis-GMA, HEMA, Hydrophobic DMA, CQ, N, N-diethanol p-toluidine, silanated colloidal silica

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Figure 1: *Bonding agent application brushes.*

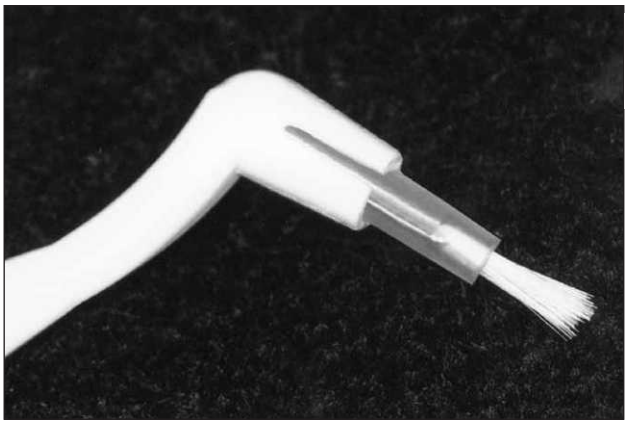

Figure 1A*: 3-M brush.*

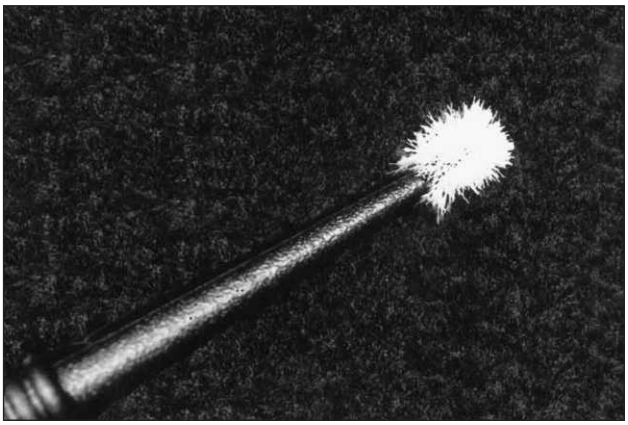

Figure 1B: Kerr Micro-applicator brush.

Table 2: *Summary of Shear Bond Strengths (Mean ± SD) MPa*

Group	Clearfil SE	Optibond	Prime&Bond	PQ1
A	29.5 ± 0.7	29.6 ± 5.9	28.0 ± 1.0	25.9 ± 4.8
B	12.3 ± 1.6	18.4 ± 7.2	16.6 ± 1.8	14.5 ± 2.8
C	23.3 ± 2.3	20.2 ± 4.0	19.2 ± 3.0	14.8 ± 2.9
D	28.5 ± 1.5	18.3 ± 8.6	24.2 ± 1.9	23.3 ± 6.3
E	18.1 ± 3.3	17.4 ± 2.3	11.9 ± 4.6	11.5 ± 3.6

seconds, followed by compressed air 0.5 cm from the surface for three seconds to remove the excess adhesive. Group C—adhesive spread with 3M brush for 30 seconds, excess adhesive removed with a clean brush, two strokes side by side, no compressed air. Group D—adhesive spread with a Micro-applicator brush (Kerr Corp) (Figure 1B) for 30 seconds followed by compressed air 0.5 cm from the surface for one second to remove the excess adhesive. Group E—adhesive spread with a Micro-applicator brush for 30 seconds, the excess adhesive removed with a clean brush, two strokes side by side, no compressed air. Prior to applying the DBAs, the cut dentin surfaces were etched according to the manufacturers' recommendations for those materials requiring etching. Since Clearfil SE Bond is a self-etching system, the cut dentin was not conditioned prior to DBA application.

A hybrid resin composite (Herculite XRV, Kerr Corp) was bonded to the treated dentin surfaces. The resin composite was packed into a plastic cylinder with dimensions 2-mm in diameter and 2-mm in height placed in contact with the dentin surface. The excess composite at the cylinder-tooth interface was removed with an explorer. The cylinder of resin composite was then subjected to a total 90 second exposure (two 45 second exposures around the circumference of the cylinder) with a Halogen curing light (Spectrum 800, Dentsply/Caulk). The intensity of the curing light was 650 mW/cm^2 and was checked with the radiometer incorporated into the unit. Each combination of DBA and application technique was replicated 10 times. The DBAs were randomly assigned to the cut buccal, lingual, mesial and distal tooth surfaces.

The specimens were stored in distilled water at 37°C for 24 hours, followed by thermocycling between 5°C and 55°C for 1,000 cycles. The shear bond strengths were determined on a universal testing machine (Model 1123, Instron Corporation, Canton, MA, USA) operating with a crosshead speed of 5 mm/minute (Figure 2). The fracture sites were examined with a 20x stereo microscope (Model L81-274, Edmund Industrial Optics, Barrington, NJ, USA) to determine the type of failure that occurred during the debonding procedure. Bond strength data were compared with analysis of variance at a significance level of $p<0.05$. Post hoc comparisons of means were performed with t-tests with p-values adjusted for multiple comparisons (Bonferroni method, Altman, 1991).

RESULTS

The mean shear bond strength values of the DBAs ranged from 11.5±3.6 MPa (PQ1—Group E) to 29.5±0.7 MPa (Clearfil SE Bond—Group A). The data of the mean shear bond strength and standard deviations are summarized in Table 2.

The highest bond strengths with Clearfil SE Bond were obtained with one-second compressed air exposure, Groups A and D. While these groups were not statistically different, they were significantly different

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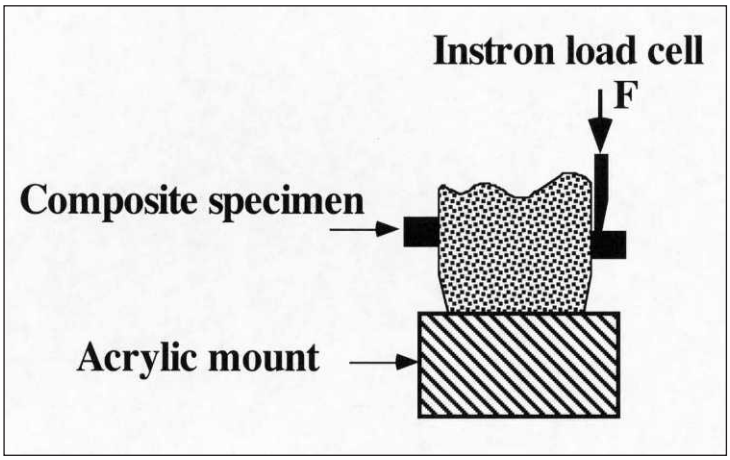

Figure 2. *Diagrammatic representation of shear bond strength test set-up.*

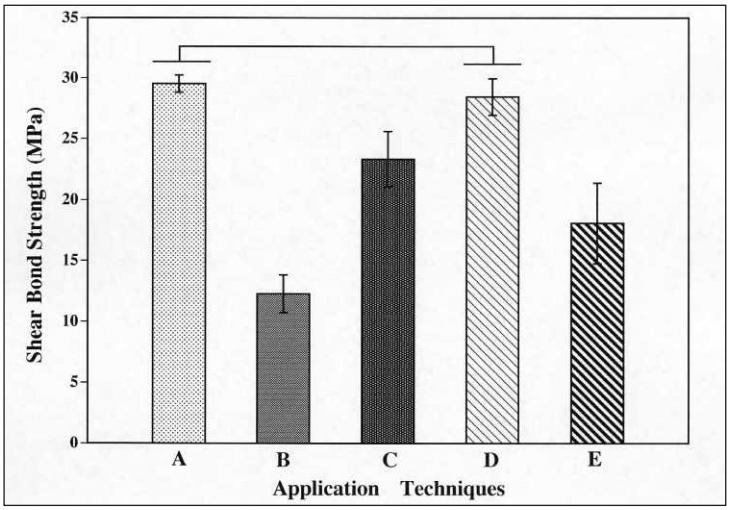

Figure 3. *Summary of shear bond strengths with Clearfil SE. Horizontal line connects groups that are not statistically different.*

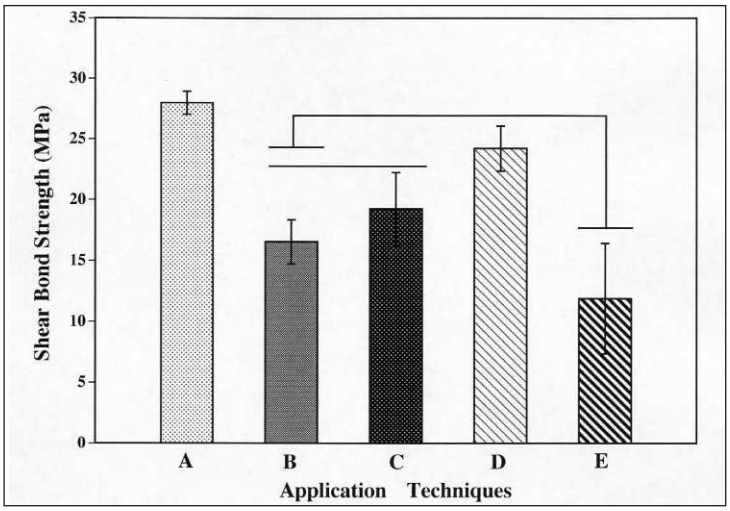

Figure 4. *Summary of shear bond strengths with Prime & Bond NT. Horizontal line connects groups that are not statistically different.*

from the remaining groups (Figure 3). The lowest bond strength was obtained after a three-second exposure to compressed air.

As was observed with Clearfil SE Bond, the highest bond strengths with Prime & Bond NT were obtained with one-second compressed air exposure, Groups A and D (Figure 4). However, these two groups were statistically different. A three-second exposure to compressed air produced significantly lower bond strength than with a one-second exposure. The lowest bond strength was noted after removing the excess adhesive with a clean brush (Group E) ($p<0.05$).

Again with PQ1, the highest bond strengths were obtained with one-second exposure to compressed air, Groups A and D, with no statistical difference between them. There were no statistical differences among the remaining Groups, which were all significantly lower (Figure 5).

In general, Optibond FL demonstrated greater variations than were observed with the other bonding agents. The highest bond strength was obtained with a one-second exposure to compressed air, and the lowest was noted after removing the excess adhesive with a clean brush (Group E) (Figure 6). These two groups were statistically different. There were no significant differences among the other groups.

Microscopic examination of the fracture surfaces after debonding revealed the following:

a) Cohesive failures in the dentin occurred with all specimens treated with Clearfil SE Bond in Groups A, C and D. Adhesive failure between the composite and dentin was seen with Groups B and E.

b) Cohesive failures in the dentin occurred with all specimens treated with OptiBond FL in Group A. Failure with all the other groups occurred at the composite-dentin interface.

c) Cohesive failures in the dentin occurred with all specimens treated with Prime & Bond NT and PQ1 in Groups A and D. Failure with the remaining groups was at the composite-dentin interface.

DISCUSSION

The application of DBAs to dentin to achieve strong bond strength with resin composite is dependent on many factors (Gwinnett & Kanca, 1992; Hashimoto & others, 2001). It has been shown that subtle differences in application techniques can affect bond strength (Kanca, 1992; Gallo & others, 2000). These observations are consistent with the results of this investigation, which demonstrated an interaction between application techniques and the bonding agents tested.

Studies have reported a wide range of shear bond strengths (7 to 25 MPa) of different DBAs following

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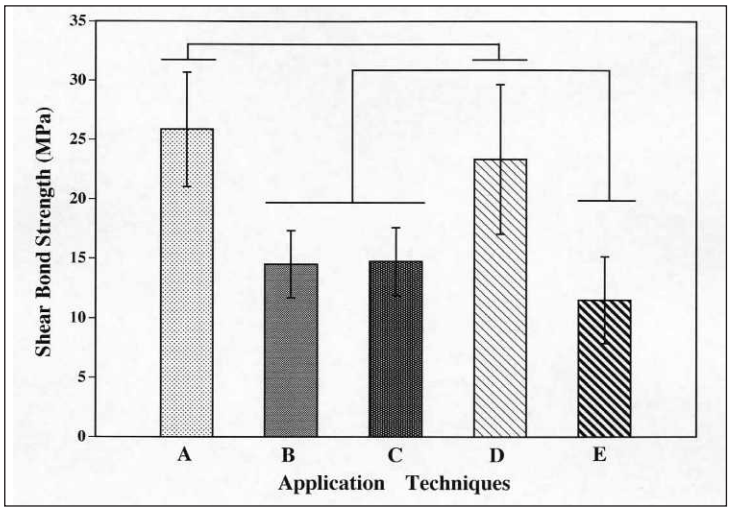

Figure 5. *Summary of shear bond strengths with PQ1. Horizontal line connects groups that are not statistically different.*

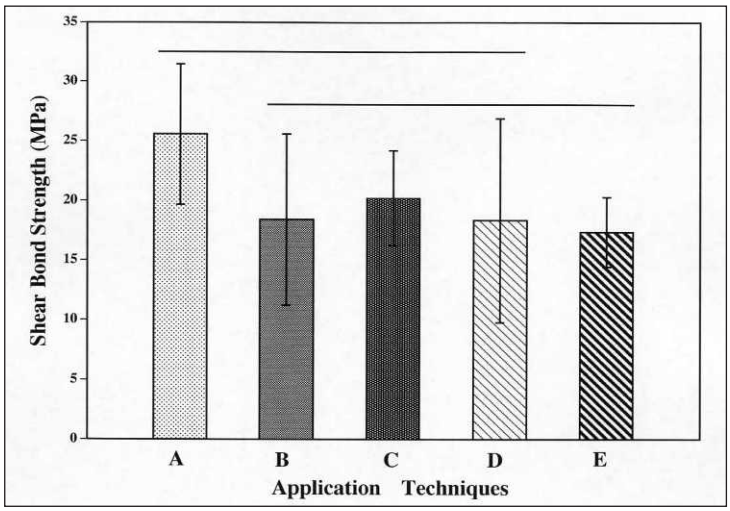

Figure 6. *Summary of shear bond strengths with Optibond FL. Horizontal line connects groups that are not statistically different.*

the manufacturers' directions. In addition, these laboratory-bonding studies prepared the dentin surface by using 150 to 600 grit silicone carbide abrasive papers (Gallo & others, 2000; Gwinnett & Kanca, 1992; Perdigão, Swift & Cloe, 1993). It has been reported that the smear layer produced by different rotary instruments or abrasive paper can affect the bond strengths of resins to dentin (Tagami & others, 1991; Watanabe, Saimi & Nakabayashi, 1994). Therefore, in this investigation, the dentin surface of each specimen was prepared with a medium grit (100 µm) diamond bur followed by a tungsten carbide bur that more closely represents the surface obtained in clinical practice.

In general, DBAs use different organic solvents (acetone and alcohol) or water. These agents promote infiltration of hydrophilic monomers into the exposed collagen network of the dentinal surface to form the hybrid layer. These adhesives are applied to dentin and left undisturbed for 10 to 30 seconds. The treated dentin surface is then subjected to a gentle air stream for 5 to 10 seconds to evaporate the excess solvent carrier and to thin the adhesive layer (Van Meerbeek & others, 1998). This study modified the manufacturers' recommendations using five different techniques to remove the excess solvent and thin the adhesive. Prior to placing the resin composite, the dentin surfaces of the samples subjected to a one-second air stream had a noticeably different surface appearance than those samples subjected to a three-second air stream. The surface appearance after a one-second air stream (Groups A and D) was more consistently smooth, free of ripples and contained an even amount of material remaining for adhesion. This application technique generally led to the highest bond strengths for all the DBAs evaluated.

On the other hand, the surface appearance after a three-second air stream (Group B) was less consistent, containing wave-like ridges of bonding material at the periphery and areas visibly void of adhesive. The surface appearance of the brush-applied and brush-removed groups (Groups C and E) were less consistent and contained large amounts of bonding material in some areas and small amounts in other areas, with ridges of material projecting above the surface. Lower bond strengths were usually associated with these application techniques. These observations are consistent with the results of a study that evaluated the effect of multiple applications of three single-component DBAs on shear bond strength (Swift & others, 1997). The authors concluded that multiple applications of these DBAs caused the formation of thicker adhesive layers and did not improve shear bond strength to dentin.

CONCLUSIONS

This *in vitro* investigation examined the effects of different dentin bonding agent application techniques on resin composite bond strength with various bonding agents. The results led to the following conclusions:

1. There was an interaction between the application technique and bonding agent tested.
2. All bonding agents utilizing the one-second compressed air technique yielded the highest bond strengths ($p<0.01$).
3. Bond strengths from the other application techniques produced lower bond strengths with greater variability among the different DBAs.

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Marginal Adaptation of Dentin Bonded Ceramic Inlays: Effects of Bonding Systems and Luting Resin Composites

B Haller • K Häßner • K Moll

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Clinical Relevance

Marginal quality of Class II ceramic inlays at gingival margins located in dentin can be improved by selecting adequate combinations of bonding system and luting resin composite.

SUMMARY

This *in vitro* study evaluated the marginal adaptation of bonded inlays of lucite-reinforced glass ceramic (Empress) to dentin as influenced by different bonding systems and by luting resin composites (LRCs) with different curing modes. Forty-eight Empress inlays etched with 5% hydrofluoric acid and treated with a silane-coupling agent (Monobond-S) were bonded to two-surface Class II cavities. Two total-etch bonding systems (OptiBond FL, Nexus) and one bonding system with selective enamel etching and a self-conditioning dentin primer (ART Bond) were included in the study. ART Bond was tested with and without the pre-curing of a first layer of adhesive resin selectively applied to the cervical cavity floor (selective double-bond technique). Each bonding system was used in combination with a light-cured resin composite (Prodigy) and a dual-cured LRC (Nexus or Vita Cerec Duo Cement). Marginal integrity was evaluated before and after thermocycling (TC) in a scanning electron microscope (SEM). Dye penetration tests were performed after TC was completed. The median percentages of continuous margin in dentin ranged from 80% to 100% before TC and from 53.5% to 96.1 % after TC. After TC, the influence of the bonding system was more pronounced than that of the LRC. In combination with the LC resin composite, ART Bond with pre-curing was significantly higher and the Nexus bonding system had significantly lower proportions of continuous margin than all the other bonding systems investigated. Swelling of the adhesive along the gingival margins was frequently found with the Nexus bonding system and with ART Bond without pre-curing. Microleakage was detected with all bonding system/LRC combinations, with somewhat lower rates in specimens completed using the selective double-bond technique. With the exception of the Nexus bonding system, post-TC marginal integrity was not influenced by the curing mode of the LRC (LC vs DC). In conclusion, the marginal quality of dentin bonded ceramic inlays can be improved by proper selection of the bonding sys-

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tem/LRC combination. The results of this study indicate the use of the Nexus luting system as directed without substitution.

INTRODUCTION

Bonded inlays of ceramic or resin composite offer excellent aesthetics and reinforcement of the remaining tooth substance (Haller, Posorsky & Klaiber, 1997). Clinical studies have shown that ceramic inlays can be successfully used to restore cavities that are completely surrounded by enamel (Fradeani, Aquilano & Bassein, 1997; Fuzzi & Rappelli, 1999; Reiss & Walther, 2000; Scheibenbogen & others, 1998; Thonemann & others, 1997). However, when replacing Class II restorations, the gingival margins are often located in dentin. Studies have shown that ceramic inlays exhibited more gap formation and leakage at gingival margins in dentin than in enamel (Bronwasser & others, 1991; Kostka & others, 1991; Zuellig-Singer, Krejci & Lutz, 1992). The marginal quality of bonded, tooth-colored inlays depends on various factors such as the bonding system and the luting resin composite (LRC) employed. In direct resin composite fillings, pre-curing the adhesive resin is recommended in order to optimize the effectiveness of the bonding system (Frankenberger & others, 1999; McCabe & Rusby, 1994). However, pre-curing the adhesive resin during the bonding of tooth-colored inlays involves the risk of interfering with the fit of the inlay and compromising complete seating, in particular, when the adhesive resin is highly loaded with filler particles. The marginal quality of bonded, tooth-colored inlays may also be influenced by selection of the LRC as characterized by the type of filler particles and the mode of cure (Krejci, Picco & Lutz, 1990). Dual-cured (DC) LRCs are often preferred for bonding tooth-colored inlays in order to assure optimal polymerization in deep areas of the cavity. DC LRCs have produced better mechanical properties than LRCs which are light cured only (Hofmann & others, 2001). Other investigators have observed a reduction in the degree of polymerization with increased thickness of the restoration, which did not depend on whether the LRCs were light cured or dual cured (el-Mowafy, Rubo & el-Badrawy, 1999; Uctasli, Hasanreisoglu & Wilson, 1994). Adequately light-cured (LC) LRCs have exhibited superior material properties compared to chemically cured materials (Darr & Jacobsen, 1995; Peutzfeldt, 1995). Proper light activation is considered essential in DC LRCs for achieving an optimal degree of polymerization (Braga, Cesar & Gonzaga, 2002; Caughman, Chan & Rueggeberg, 2001; el-Badrawy & el-Mowafy, 1995; Hofmann & others, 2001). However, exposure times recommended by the manufacturers seem insufficient to compensate for the attenuation of light by ceramic inlays (Hasegawa, Boyer & Chan, 1991; Lee & Um, 2001). A disadvantage of DC LRCs is the porosities incorporated during mixing, which may compromise the mechanical properties and promote marginal discoloration. On the other hand, the mixed-in porosities may reduce the polymerization stresses by slowing down the polymerization reaction due to oxygen inhibition and by increasing the free composite surfaces (Feilzer, de Gee & Davidson, 1993). While LC LRCs do not require mixing, the intensity of light transmitted through the inlay must be high enough to ensure an adequate degree of double-bond conversion. Clinically, the quantity of light transmitted through thick inlay sections such as proximal boxes extending from the marginal ridge to the cervical cavity floor may be insufficient for optimal curing of the adhesive resin and the LRC. This *in vitro* study evaluated the effects of (i) different bonding systems, (ii) pre-curing of the adhesive resin and (iii) different LRCs (LC versus DC) on marginal integrity with dentin of bonded glass ceramic inlays.

METHODS AND MATERIALS

Twenty-four human molars free from caries and fillings and stored in 1% chloramine after extraction were selected for this study. In each tooth, two box-shaped Class II cavities (mesio-occlusal and disto-occlusal; Figure 1) were prepared with cervical margins located 0.5 mm apical to the cemento-enamel junction (Figure 2). The dimensions of the proximal cavities were 4 mm in buccolingual and 1.5 mm in the pulpal direction. After finishing the cavities with a diamond-coated finishing bur (mean particle size: 20 µm), a silicon impression was made (President Monobody, Coltène, Altstätten, Switzerland) for the fabrication of a master die. In order to simulate clinical conditions, the cavities were then filled with a resin-based temporary restorative (Fermit, Vivadent, Ellwangen, Germany) and the teeth were stored in saline for seven days. Glass

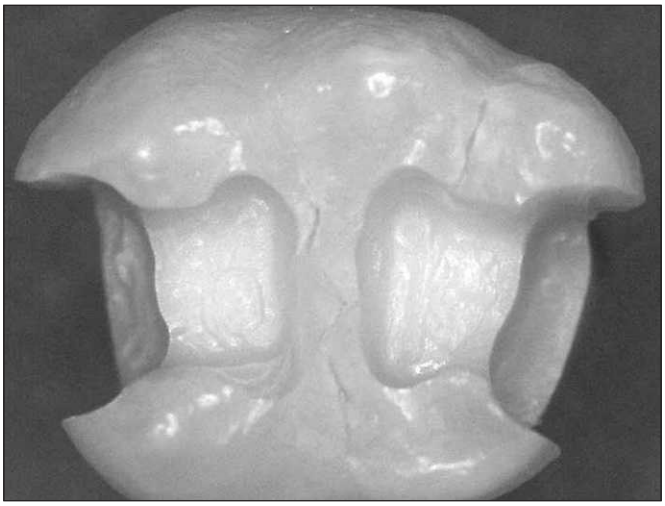

Figure 1. *Occlusal view of test tooth with MO and OD Class II cavity preparations for ceramic inlays.*

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ceramic inlays (n=48; IPS Empress, Ivoclar, Ellwangen) were produced in the dental laboratory according to the manufacturer's instructions. After removing the temporary fillings, the cavities were cleaned with water and pumice using rotating brushes. The inlays were fitted into the cavities, cleaned with ethanol, etched for two minutes with a 5% hydrofluoric acid gel (IPS Ceramic Etch, Ivoclar) and treated with a silane coupling agent (Monobond-S, Vivadent) for one minute. For luting of the inlays, the teeth were mounted in an artificial row of teeth in order to simulate clinical conditions. Two total etch bonding systems, OptiBond FL (Kerr, Karlsruhe, Germany) and Nexus (Kerr), and one bonding system including selective enamel etching followed by dentin treatment with a self-conditioning primer (ART Bond, Coltène), were selected for this study (Table 1). When using ART Bond, two different modes of application were evaluated. In one group, a small amount of adhesive resin was selectively applied to the cervical cavity floor using a disposable brush, thoroughly air-thinned and light cured for 30 seconds using a halogen light-curing unit (Translux CL, Kulzer, Wehrheim, Germany). In a second step immediately prior to inserting the inlays, all cavity walls were covered with adhesive resin that was then cured together with the luting resin composite (LRC). In the other group, a single layer of the ART Bond adhesive resin was applied to the cavities without being pre-cured. One light-curing (LC) resin composite (Prodigy, Kerr) and two dual-curing (DC) LRCs (Nexus, Kerr; Vita Cerec Duo Cement, Vita, Bad Stickingen, Germany) were included in the study (Table 2). Six specimens were prepared for each bonding system/LRC combination (Table 3). Immediately

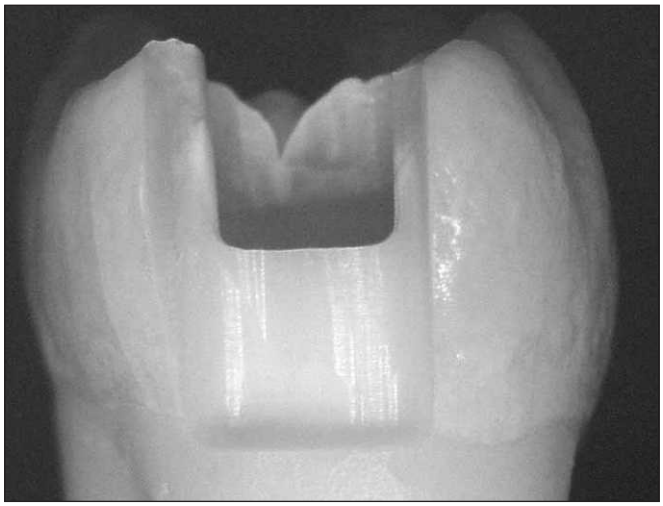

Figure 2. *Proximal view of Class II cavity preparation for ceramic inlay with gingival margin located in dentin.*

Table 1: *Bonding Systems Investigated*

Product (Manufacturer)	Classification (Batch #)	Components	Mode of Application
OptiBond FL (Kerr)	3 steps; total-etching	Etchant (976543): 37.5% PA Prime (604587): HEMA, GPDM, PAMA, CQ, ethanol/water Adhesive (605027): Bis-GMA, HEMA, GDMA, Ba-Al-borosilicate, silicate glass filler, Na2SiF6, CQ	Etchant: apply to enamel (30 seconds) and dentin (15 seconds); water-rinse (15 seconds); remove excess water from cavity with short air blasts without desiccating Prime: scrub onto dentin with microbrush (30 seconds); air dry until dull appearance Adhesive: apply to enamel end dentin, leave undisturbed (5 seconds); spread to thin layer with gentle air stream
Nexus (Kerr)	4 steps; total-etching	Etchant (976543): 37.5% PA Nexus 1 (612339): HEMA, GPDM, PAMA, CQ, ethanol/water Nexus 2 (611255): HEMA, sulfinic acid salts Nexus 3 (609092): Bis-GMA, HEMA, PAMA, CQ, peroxide	Etchant: apply to enamel (30 seconds) and dentin (15 seconds); water-rinse (15 seconds); remove excess water from cavity with short air blasts without desiccating Nexus 1: apply to enamel and dentin; lightly air dry Nexus 2: apply to enamel and dentin; air thin Nexus 3: apply to enamel and dentin; air dry; do not light cure
ART Bond (Coltène)	3 steps; enamel etching with phosphoric acid; self-conditioning dentin primer	Etchant: 35 % PA Primer A: maleic acid, NaF, water Primer B: HEMA, HPMA, PAAC, water Bond: Bis-GMA, TEGDMA (GL 569)	Etchant: apply to prepared enamel (30 seconds); water-rinse (15 seconds); air dry Primer: scrub mixture of Primer A and B (1:1) onto dentin (30 seconds); air dry until dull appearance Adhesive: Only for the "selective double-bond technique": apply small amount selectively to gingival cavity floor in dentin (20 seconds); carefully air thin with gentle air stream; light cure (20 seconds). All specimens: apply to all cavity walls; leave undisturbed (5 seconds); spread to thin layer with gentle air stream

PA, phosphoric acid; HEMA, 2-hydroxyethyl methacrylate; HPMA, hydroxypropyl methacrylate; GPDM, glycerol phosphate dimethacrylate; PAMA, phtalic acid monomethacrylate; CQ, campheroquinone; Bis-GMA, bisphenol glycidyl methacrylate; GDMA, glycerol dimethacrylate; TEGDMA, triethylenglycol dimethacrylate; PAAC, polyalcenoic acid co-polymer

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prior to seating, the inner surfaces of the inlays were coated with a thin layer of adhesive resin. Prior to light curing, excess LRC was removed with a probe and dental floss and the margins were covered with glycerine gel (Airblock, Dentsply DeTrey, Konstanz, Germany). The LRCs were light cured with halogen light (Translux CL) for 60 seconds from the occlusal aspect, 60 seconds from the buccal aspect through a light-reflecting wedge and 30 seconds from the lingual aspect. The intensity (irradiance) of the light-curing unit used in this study was 620 mW/cm^2 as measured with a radiometer (Cure Rite, Caulk Dentsply, Milford, DE, USA). The restored teeth were removed from the artificial tooth row and the restorations were finished and polished using flexible discs (Sof-Lex, 3M, Borken, Germany). Silicone impressions of the restored teeth were taken before and after thermocycling (TC; 1500x 5°C/55°C) for fabrication of the epoxy replica, which were then sputter-coated with gold. The proximal inlay margins located in enamel and the gingival margins located in dentin were investigated in a SEM (Leica Stereoscan 420, LEO-Elektronenmikroskopie, Oberkochen, Germany) at 500x magnification for marginal gaps (Figure 3) and swellings of the adhesive along the margins (marginal swellings; Figure 4). Marginal swellings, as described in previous studies (Thonemann & others, 1995), were only observed along the inlay/dentin interfaces. The length of the observed marginal defects was measured and their percentages calculated from complete lengths of the proximal (buccal plus lingual) enamel margins and gingival dentin margins, respectively. In addition, the maximum width of the luting space at the cervical margins was measured in order to identify potential associations between the maximum width of luting space on one side, and ILRC viscosity and the incidence of marginal defects, respectively, on the other side.

Table 2: *Resin Composites Investigated*

Product (Manufacturer)	Classification	Batch #
Prodigy (Kerr)	Fine-hybrid resin composite restorative; light curing	705800
Nexus (Kerr)	Fine-hybrid luting resin composite; dual-curing	Nexus A: 701488 Nexus B: 702214
Vita Cerec Duo Cement (Vita)	Fine-hybrid luting resin composite; dual-curing	HA 765

Table 3: *Experimental Groups*

Luting System (bonding system/luting resin composite)	Number of Restorations
OptiBond FL/Prodigy	6
OptiBond FL/Nexus	6
Nexus/Prodigy	6
Nexus/Nexus	6
ART Bond (without pre-curing)/Prodigy	6
ART Bond (without pre-curing)/Vita Cerec Duo Cement	6
ART Bond (pre-cured)*/Prodigy	6
ART Bond (pre-cured)*/Vita Cerec Duo Cement	6

*"Selective double-bond technique": includes pre-curing of a first layer of ART Bond adhesive resin selectively applied to the gingival cavity floor

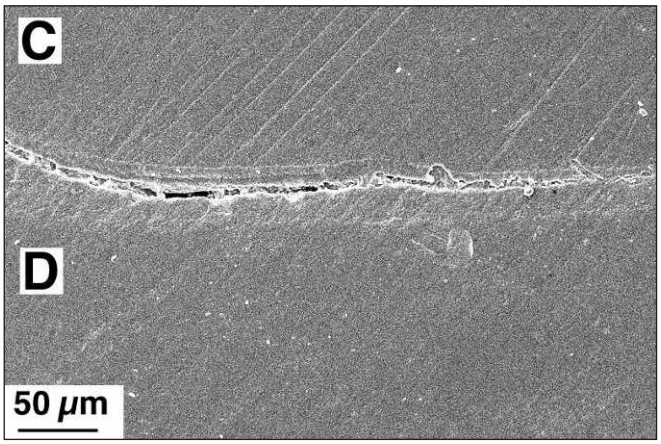

Figure 3. *SEM micrograph of marginal gap between at the gingival dentin margin. C, ceramic inlay; D, dentin. Length of horizontal bar indicates 50 µm (original magnification: 500x).*

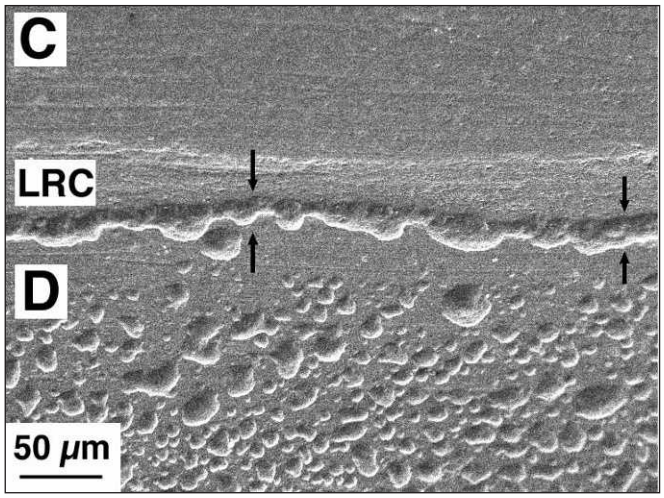

Figure 4. *SEM micrograph of marginal swellings (arrows) at the dentin/luting resin composite interface. C, ceramic inlay; LRC, luting resin composite; D, dentin. Length of horizontal bar indicates 50 µm (original magnification: 500x).*

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For the dye penetration tests, the teeth were sealed with nail varnish, leaving an area about 1 mm from the inlay margins uncovered. They were then immersed in 0.5% basic fuchsin solution for 24 hours at 37°C. The restored teeth were sectioned longitudinally by a cut through the middle of the gingival margins, and the inlay fragments were fractured off the cavities. The depth of dye penetration along the restoration/dentin interface at the cervical cavity floor was determined using an optical microscope at 64x magnification. The quality of dentin sealing was determined by measuring the depth of dye penetration into the dentin tubules towards the pulp.

Statistical analysis of the data is focused on descriptive statistics. Statistical tests are explorative, not confirmative. Hence, no adjustment for multiple testing was used. First, source data and median are presented. The 95% confidence interval of the median ranges for n=6 observations from the highest to the lowest observed value. Therefore, the range of the source data is the 95% confidence interval of the median as well. For some outcome variables the source data were sufficiently distributed without clumpings at zero. For these variables a one way analysis of variance was performed with eight treatment groups and a maximum of luting space as covariate. If the *p*-value of the total model was ≤ 578 0.05, then 12 more detailed pairwise comparisons were tested *t*-test). For n=6 the power is rather low for smaller differences among the groups. However, the authors have some significant results. Therefore, the sample size was sufficient to find major differences among the treatments. Correlations between variables were calculated with the Pearson coefficient of correlation.

RESULTS

At the proximal margins in enamel, only minor gap formation (all medians: 0%) was observed *before TC*. *After TC*, the median percentages of marginal gaps in enamel ranged between 0% and 5%.

In contrast to the situation at the enamel margins, the selection of the bonding system/LRC combination had significant influence on marginal adaptation in dentin. In order to determine the effect of the curing mode of the LRC, the results of LC and DC LRCs where compared when used in combination with the same bonding system (Table 4). In order to identify differences between the bonding systems evaluated, the authors compared groups with the same LRC but different bonding systems (Table 5).

Before TC, the median percentages of continuous (defect-free) margin in dentin (Figure 5) ranged from

Table 4: *Comparisons of Light-Cured and Dual-Cured Luting Resin Composites When Used in Combination with the Same Bonding Systems (t-test). LC Light-Cured, DC Dual-Cured. PC- Without Pre-curing of Adhesive Resin, PC+ with Pre-curing of Adhesive Resin*

Bonding System	Luting Resin Composites Compared: LC vs DC	Before Thermocycling	After Thermocycling	
		% Continuous Margin	% Continuous Margin with Dentin	% Gaps Along Dentin Margins
OptiBond FL	Prodigy vs Nexus	*p*=0.0339	ns	ns
Nexus	Prodigy vs Nexus	ns	*p*=0.0007	*p*=0.0001
ART Bond (PC-)	Prodigy vs Vita Cerec Duo Cement	*p*=0.0445	ns	ns
ART Bond (PC+)	Prodigy vs Vita Cerec Duo Cement	ns	ns	ns

Table 5: *Comparisons of Bonding Systems When Used in Combination with the Same Luting Resin Composites (T-test). LC Light-Cured, DC Dual-Cured. PC- Without Pre-curing of Adhesive Resin, PC+ with Pre-curing of Adhesive Resin*

Luting Resin Composite	Bonding Systems Compared	Before Thermocycling	After Thermocycling	
		% Continuous Margin with Dentin	% Continuous Margin with Dentin	% Gaps Along Dentin Margins
Prodigy (LC)	OptiBond FL vs Nexus	ns	0.0149	ns
Prodigy (LC)	OptiBond FL vs ART Bond (PC-)	ns	ns	0.0111
Prodigy (LC)	OptiBond FL vs ART. Bond (PC+)	0.0215	0.0020	0.0038
Prodigy (LC)	Nexus vs ART. Bond (PC-)	ns	0.0007	0.0007
Prodigy (LC)	Nexus vs ART. Bond (PC+)	ns	<0.0001	0.0002
Prodigy (LC)	ART Bond (PC-) vs ART Bond (PC+)	0.0052	0.0347	ns
Nexus (DC)	OptiBond FL vs Nexus	ns	ns	0.0024
Vita Cerec Duo Cement (DC)	ART Bond (PC-) vs ART Bond (PC+)	ns	ns	ns

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80% (ART Bond without pre-curing/Prodigy) to 100% (ART Bond pre-cured Nita Cerec Duo Cement, OptiBond FL/Nexus), with some significant differences between groups (ANOVA: p=0.0204). Most of the bonding systems performed better in combination with a DC LRC than with the LC resin composite (Table 4). Inlays luted with the LC resin composite Prodigy showed better initial marginal adaptation when bonded with ART Bond (precured) compared to ART Bond (without pre-curing) and OptiBond FL, respectively (Table 5). Initial marginal gap formation ranged from 0% to 2% (median) without significant differences between groups (ANOVA: p=0.4260). In most of the experimental groups, no marginal swellings were detected in four to six out of six samples (median: 0%). In contrast, swellings along the LRC/dentin interface contributed significantly to marginal defects in the case of ART Bond without pre-curing/Prodigy (median: 15%) and Nexus/Prodigy (median: 9.9%).

After TC, significant differences in the percentage of continuous margin were detected between experimental groups (ANOVA: p=0.0003). The proportions of continuous margin (Figure 6) were highest with ART Bond pre-cured/Prodigy (median: 96.1%) and lowest with Nexus/Prodigy (median: 53.5%). The only significant difference between LRCs was detected between Prodigy (LC) and Nexus (DC) when used in combination with the Nexus bonding system (Table 4). In contrast, the influence of the bonding systems on marginal adaptation after TC was distinctly higher than LRC. In combination with the exclusively LC LRC, (i) ART Bond (with pre-curing) showed significantly higher and (ii) the Nexus bonding system showed significantly lower percentages of continuous margin than all other bonding systems investigated (Table 5). Median percentages of marginal gap forma-

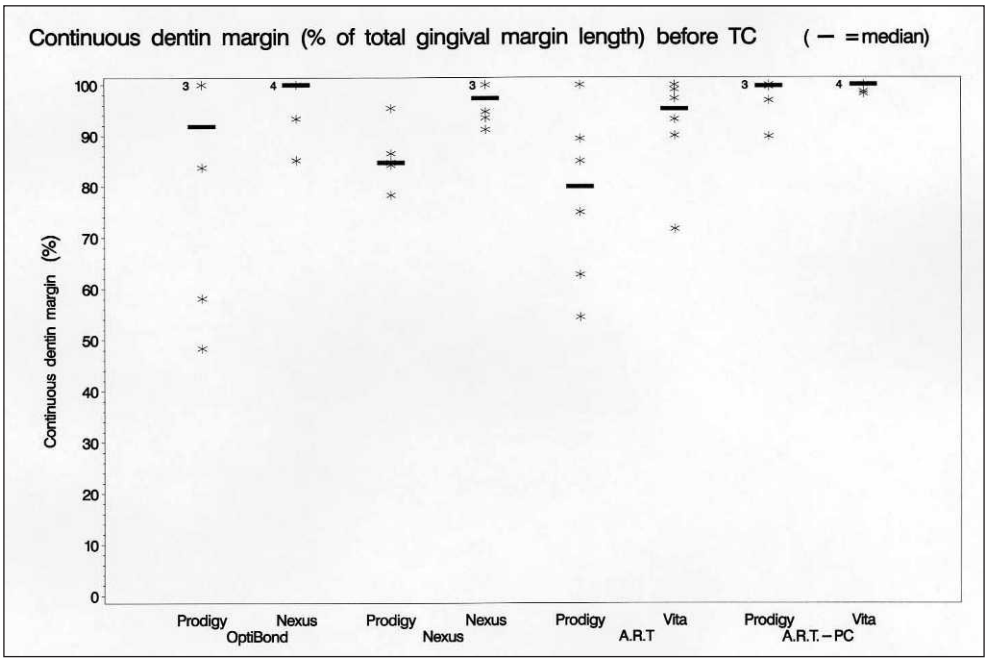

Figure 5. *Results of the quantitative SEM analyses of the gingival dentin margins* before TC*: Proportions of continuous margin in % (n=6; source data, median). ART- PC: ART Bond with pre-curing of first layer of adhesive resin selectively applied to the cervical cavity floor. Numbers beside source data indicate frequency of same value.*

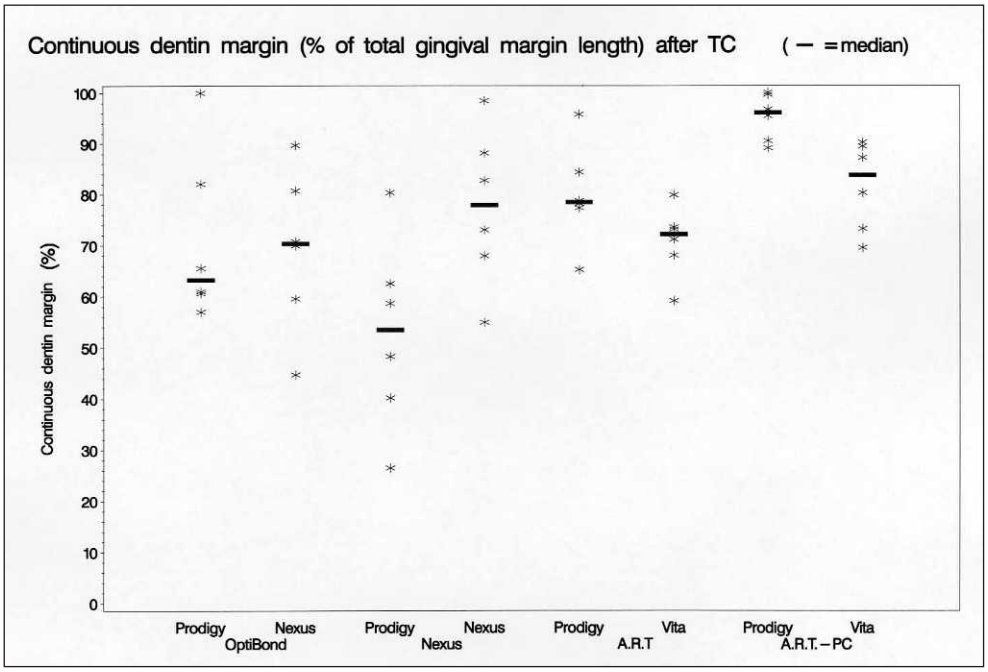

Figure 6. *Results of the quantitative SEM analyses of the gingival dentin margins* after TC*: I. Proportions of continuous margin in % (n=6; source data, median). ART- PC: ART Bond with pre-curing of first layer of adhesive resin selectively applied to the cervical cavity floor. Numbers beside source data indicate frequency of same value.*

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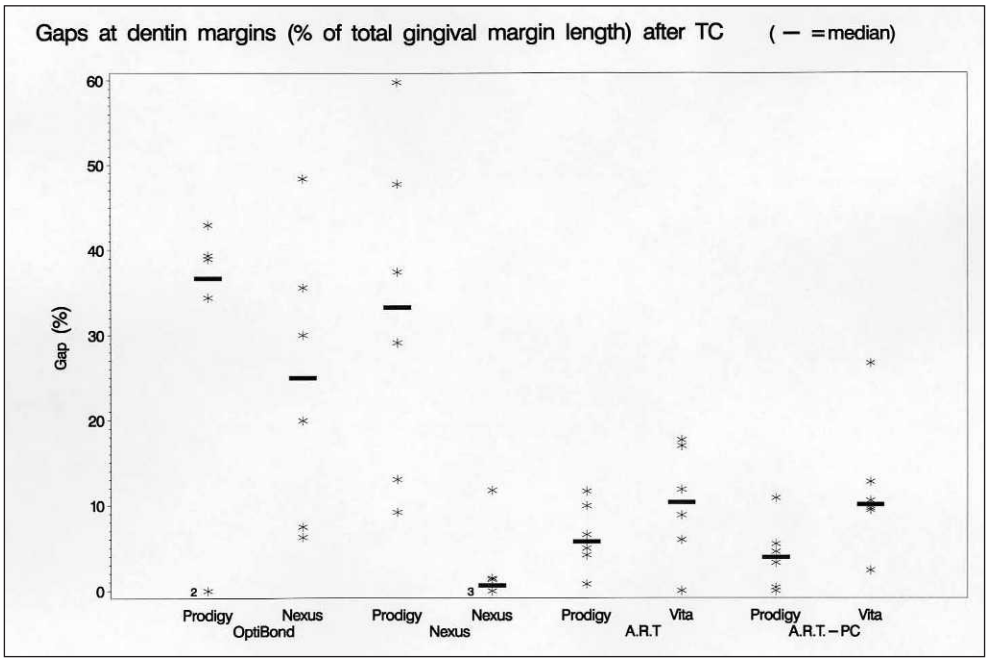

Figure 7. *Results of the quantitative SEM analyses of the gingival dentin margins* after TC: *II. Proportions of marginal gaps in % (n=6; source data, median). ART-PC: ART Bond with pre-curing of first layer of adhesive resin selectively applied to the cervical cavity floor. Numbers beside source data indicate frequency of same value.*

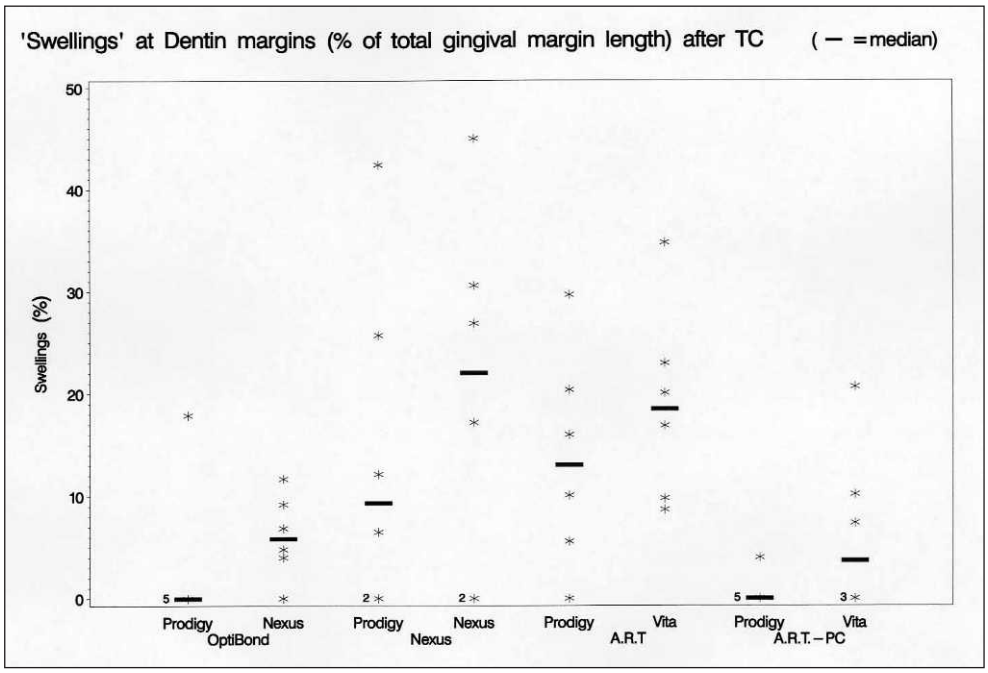

Figure 8. *Results of the quantitative SEM analyses of the gingival dentin margins* after TC*: III. Proportions of marginal swellings in % (n=6; source data, median). ART-PC: ART Bond with pre-curing of first layer of adhesive resin selectively applied to the cervical cavity floor. Numbers beside source data indicate frequency of same value.*

tion (Figure 7) ranged from 0.7% (Nexus/Nexus) to 36.7% (OptiBond FL/Prodigy), with statistically significant differences between groups (ANOVA: p=0.0006). However, the Nexus luting system showed a high incidence of marginal swellings (median: 22.1%). Relatively high percentages of marginal swellings were also observed when using ART Bond without pre-curing (Figure 8). In contrast, five out of six inlays bonded with OptiBond FL/Prodigy and ART Bond (pre-cured)/Prodigy, respectively, showed no marginal swellings. A significant negative correlation was confirmed between the percentage of marginal gaps and the percentage of marginal swellings (r=-0.307, p=0.034).

The lowest degree of dye penetration (Figure 9) was measured in inlays bonded with ART Bond (pre-cured) regardless of which LRC was used (median: 0.15 mm). Considerably more microleakage was observed with OptiBond FL/Nexus (median: 0.93 mm) and ART Bond (without pre-curing)/Prodigy (median: 0.87 mm), although differences between groups were not statistically significant (ANOVA: p=0.4090). Dye penetration into dentin tubules was fully prevented in inlays luted with ART Bond (pre-cured)Nita Cerec Duo Cement. In all other groups using ART Bond, dye penetration into tubules was prevented in four out of six specimens. In contrast, when employing total etching (OptiBond FL, Nexus), dye penetration extended into dentinal tubules in at least four out of six specimens, indicating incomplete sealing of the dentin.

The thickness of the luting space was smallest in inlays bonded with the Nexus luting system (median: 53 µm) and largest when using the Nexus bonding system in combination with the LC restorative Prodigy (median: 115.5 µm). However, differences between groups were not statistically significant, indicating that pre-curing of the ART Bond adhesive resin did not

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increase the thickness of luting space. No correlations were found between the thickness of luting space and the percentage of marginal defects or the extent of dye penetration.

DISCUSSION

Although the efficacy of dentin adhesives has been continuously increased during the last decade (Haller, 2000), perfect marginal integrity of resin-bonded Class II ceramic inlays at gingival margins located in dentin is still critical (Dietschi & Moor, 1999; Manhart & others, 2001). Although some of the bonding system/LRC combinations evaluated in this study showed promising potential, marginal gap formation and microleakage were not completely prevented by any of the luting systems. As a result of the limited sample size (n=6 per group), some of the trends indicated by the results of this study were not confirmed as statistically significant and should be investigated in more detail in future studies. In addition, some differences between the experimental groups may have been more pronounced if mechanical loading of the specimens had been applied. In spite of these limitations, the findings of this study should make the clinician's decision-making easier when it comes to the selecting proper materials and techniques for the bonding of tooth-colored inlays with gingival margins in dentin.

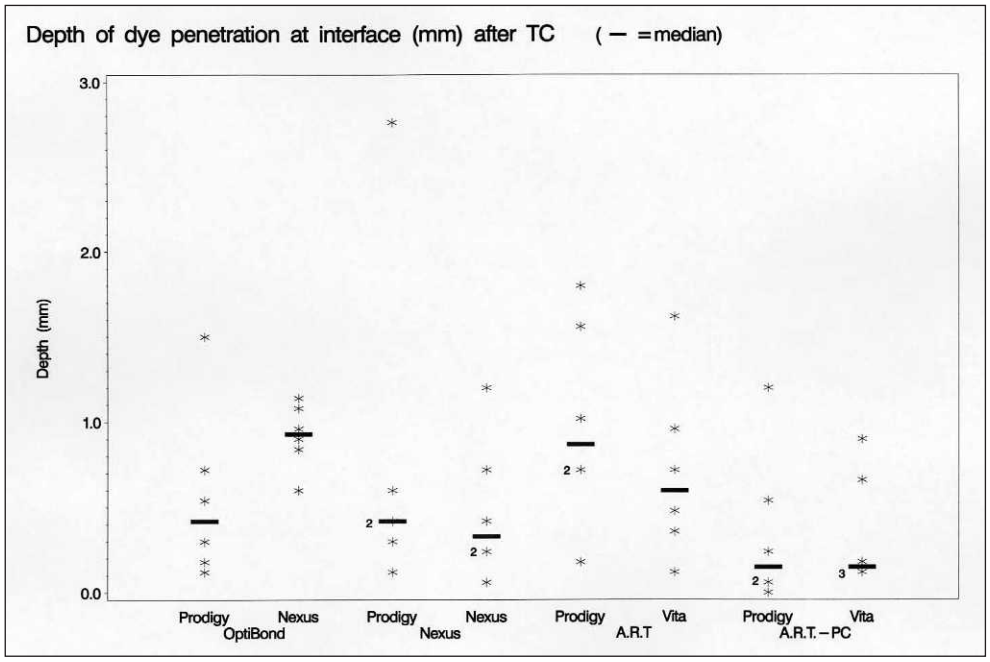

Figure 9. *Results of microleakage tests (*after TC*). Depths of dye penetration along the luting resin composite/dentin interface in mm (n=6; source data, median). ART-PC: ART Bond with pre-curing of first layer of adhesive resin selectively applied to the cervical cavity floor. Numbers beside source data indicate frequency of same value.*

Incomplete transmission of the curing light through ceramic inlays may compromise the polymerization of the bonding system and the LRC (el-Badrawy & el-Mowafy, 1995; Lee & Um, 2001), resulting in sub-optimal dentin bonding. In direct resin composite restorations, separate light curing of the adhesive resin prior to insertion of the filling material is recommended for maximum efficacy of the bonding system (Frankenberger & others, 1999; McCabe & Rusby, 1994). Although tooth-colored inlays are also considered an important factor in bonding, pre-curing of the adhesive resin may increase the luting space and interfere with complete seating of the inlay (Frankenberger & others, 1999; Hahn & others, 2000). In this study, adhesive resin pre-curing was performed in a "selective double-bond technique" in order to minimize the risk of incomplete inlay seating. The procedure includes light curing of a small amount of adhesive resin selectively applied in a thin layer to the cervical dentin floor of the proximal cavities followed by application of a second, overall paint of adhesive resin prior to insertion of the inlay, which is light cured together with the LRC. This approach was only evaluated in combination with the unfilled ART Bond adhesive resin. In the case of OptiBond FL (48% inorganic fillers), the risk of increased film thickness and incomplete inlay seating was rated too high. In the case of Nexus, pre-curing of the adhesive resin (Nexus 3) is not recommended by the manufacturer.

Pre-curing of the ART Bond adhesive resin decreased the incidence of marginal gap formation when used in combination with the LC resin composite. In addition, it considerably reduced the percentage of swelling along the gingival margins and tended to improve the marginal and the internal seal as determined by dye penetration. Although the volume of shrinking resin composite is relatively small in bonded inlays compared to direct fillings, considerable shrinking stresses may occur due to the unfavorable C-factor of the LRC (Feilzer, de Gee & Davidson, 1989). It is important for the survival of the bond between the bonding system and dentin that the shrinkage stress is relieved by an elastic response of the surrounding materials (Kemp-Scholte & Davidson, 1990). Although the beneficial effect of adhesive resin pre-curing can basically be ascribed to an increased degree of polymerization, the formation of an "elastic cavity" wall absorbing some of the contraction stress arising during polymerization of the LRC may have contributed to the improvements

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observed. In accordance with this concept, it has been suggested that the resin dentin hybrid layer can act as a stress-absorbing layer (Uno & Finger, 1995). Hybrid layers produced by phosphoric acid etching are usually thicker than those produced by mild to moderate self-conditioning systems (Prati & others, 1998; Tanumiharja & others, 2000; Tay & Pashley, 2001; Van Meerbeek & others, 1992). However, in this study, the self-conditioning ART Bond system tended to prevent marginal defects more effectively than the total-etch system bonding OptiBond FL.

Marginal swellings along the resin composite-dentin have been reported in previous SEM studies and have been interpreted as an early sign of insufficient adhesion between resin composite and dentin (Manhart & others, 2001; Thonemann & others, 1995). The reason for introducing this criterion was that, although no gaps were visible in these areas, it was not considered appropriate to rate the respective sections of the margin as "continuous." In this study, the incidence of marginal swellings was lower with pre-curing than without precuring of the ART Bond adhesive resin. It was also lower with the highly filled OptiBond FL than with the unfilled Nexus. Therefore, water sorption into unfilled and/or incompletely cured, more or less hydrophilic resin layers may contribute to the phenomenon. Alternatively, the appearance of marginal swellings may be explained by interactions between incompletely cured resin and impression material or by fluid drops or air-bubbles originating from dentinal tubules. Irrespective of their real origin, which remains to be determined, marginal swellings should be considered as a weak link in marginal adaptation or even as an indicator for hidden marginal gaps.

The DC bonding system Nexus showed significantly better marginal adaptation in combination with the respective DC Nexus LRC than with the LC resin composite Prodigy, indicating that all components of the Nexus luting system are carefully reconciled with each other. This includes the self-curing mechanism of Nexus 3, which is initiated by the chemical reaction of its peroxide component with the tertiary amine of the DC LRC. This additional curing mechanism is missing when an exclusive photo-curing LRC (Prodigy) is employed. It is, therefore, recommended that the complete Nexus kit be used rather than combining either the bonding system or the LRC with other products even from the same manufacturer.

In addition to their more complete polymerization reaction (Braga & others, 2002; Caughman & others, 2001; Hofmann & others, 2001), DC LRCs have further advantages over exclusively LC LRCs. It has been demonstrated that lower shrinkage stresses are produced in self-curing resin composites than in LC composites (Feilzer & others, 1993; Kinomoto & others, 1999). The porosities incorporated during mixing of self-curing resin composites contain oxygen that slows down the polymerization reaction and increases the free surface within the resin composite (Feilzer & others, 1993). It is conceivable that the stress reducing mechanisms observed in auto-curing resin composites may also take effect in DC LRCs. However, in contrast to the authors' expectations based on theoretical considerations, no consistent differences in marginal quality were found between the LC restorative and the DC LRCs when used in combination with OptiBond FL and ART Bond, respectively. The only significant difference in the degree of marginal gap formation between LC and DC resin composites was observed in the case of the Nexus bonding system. However, this effect is more likely to be related to the specific chemistry of the Nexus luting system than to different stress-reducing capacities of the resin composites compared. This indicates that the quality of marginal adaptation is determined by the bonding system/LRC combination rather than by the bonding system or the LRC alone. There are several possible explanations for the lack of differences between LC and DC resin composites in this study. First, the incomplete light transmittance through the ceramic inlays may have slowed down the polymerization reaction of the LRC, resulting in a reduction of contraction stresses (Lee & Um, 2001) similar to that observed in chemically curing resin composites (Feilzer & others, 1993). In addition, it has been demonstrated that the polymerization contraction can be compensated to some extent by a vertical movement of the inlay during setting of the LRC (Sorensen & Munksgaard, 1995).

Shrinkage stresses have been shown to negatively correlate with the layer thickness in thin resin composite films (Alster & others, 1997). With a DC LRC also tested in this study (Vita Cerec Duo Cement), the incidence of marginal gaps was found to be independent of width of the luting space ranging from 50 to 1000 µm (Schmalz, Federlin & Reich, 1995). Although the LC resin composite tested in this study tended to produce higher layer thickness than the DC LRCs, this did not result in improved marginal adaptation. Consequently, from a clinical viewpoint, the luting gap should be kept as small as possible in order to avoid the pulling-out of uncured material from the gap during removal of excess LRC.

CONCLUSIONS

Within the limitations of the experimental conditions, the authors draw the following conclusions:

- Achieving a perfect marginal quality with ceramic inlay restorations when gingival margins are located in dentin continues to be critical even when state-of-the art multi-step bonding systems are employed.
- Marginal quality is determined by the bonding system/LRC combination rather than by single factors.

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- The adhesive luting kit Nexus, including a four-step total-etch bonding system and a DC LRC, showed promising marginal quality when applied as a complete system, whereas, the combination with other bonding systems and LRCs, respectively, increased the incidence of marginal defects.
- In Class II cavities with gingival margins in dentin, a "selective double-bond technique," including the pre-curing of a thin layer of adhesive resin selectively applied to the cervical cavity floor, has demonstrated some potential to improve the marginal integrity of ceramic inlays.
- The results indicate that complete sealing of dentin tubules may be more predictable in bonding systems with self-conditioning dentin primers than in total-etch systems. Clinically, this may have an influence on the risk of postoperative sensitivity.

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Shear Bond Strength of Current Adhesive Systems to Enamel, Dentin and Dentin-Enamel Junction Region

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Clinical Relevance

Even though the region approximating the DEJ etched more deeply than enamel or dentin, especially with phosphoric acid gel, bonding to this region is potentially as good as to enamel or dentin.

SUMMARY

This study investigated the bonding of current resin adhesives to the region approximating the dentin-enamel junction (DEJ), where the etch pattern to enamel or dentin may be different. Three kinds of tooth substrates were chosen for testing: enamel, dentin and the DEJ region. A self-etching primer system (Clearfil SE Bond) and two total-etch wet bonding systems (Single Bond and One-Step) were used. Each tooth region was bonded with one of the adhesive systems, and a resin composite and was subjected to a micro-shear bond test. In addition, morphological observations were performed on debonded specimens and etched surfaces using confocal laser scanning microscopy (CLSM). CLSM observations showed that the DEJ region was etched more deeply by phosphoric acid gel than enamel or dentin, suggesting that the action of acid etch seemed to be more intense on the DEJ. However, no statistically significant differences of shear bond strength values were observed between the DEJ region and enamel or dentin, or the adhesive systems used (p>0.05). Bonding to the DEJ is potentially as good as that to enamel or dentin.

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INTRODUCTION

Enamel is a highly mineralized tissue with crystallites arranged in an orderly fashion (Frazier, 1968; Jongebloed, Molenaar & Arends, 1975). Due to its brittleness, enamel requires underlying dentin to transmit and dissipate occlusal forces (Wang & Weiner, 1998), and the dentino-enamel junction (DEJ) has the ability to transfer stress from enamel to dentin (Lin & Douglas, 1994; Xu & others, 1998; Wang & Weiner, 1998). The region of the DEJ has been shown to be rich in organic material (Lin & Douglas, 1994; Mjör, Sveen & Heyeraas, 2001), has less mineral content (Tesch & others, 2001) in the presence of parallel-oriented coarse collagen bundles (Lin & Douglas, 1994) and predominant branches of dentinal tubules (Mjör & others,

2001). Several researchers have distinguished the DEJ from enamel or dentin (Lin & Douglas, 1994; Xu & others, 1998) and regard it as a fiber-reinforced bond that is moderately mineralized (Lin & Douglas, 1994).

Currently, there are two predominant resin-based adhesive system types available for resin restoration—"one-bottle" and "self-etching" adhesives (Nakabayashi, Kojima & Masuhara, 1982; Kanca, 1992; Watanabe, Nakubayashi & Pashley, 1994). These adhesives exhibit bonds strong enough to resist the contraction forces of polymerized resin composite to tooth structure (Nakabayashi & others, 1982; Kanca, 1992; Watanabe & others, 1994). Although many studies have focused on the bonding of enamel or dentin to adhesive materials, little is known about bonding to the vicinity of the region approximating the DEJ. Because of the complex nature of the anatomical structure of the DEJ, its etch pattern may differ from enamel or dentin and may, as a result, affect the bonding of adhesive materials.

This study investigated the bond strength between the DEJ region, enamel and dentin, and three resin adhesive systems. Bond strengths were assessed by means of a micro-shear bond test (Shimada & others, 1999; Shimada, Yamaguchi & Tagami, 2002). In addition to the shear bond test, surfaces were observed for failure mode and the adhesive interface between the tooth and resin and the conditioned tooth surface without any bonding were studied morphologically using confocal laser scanning microscopy (CLSM).

METHODS AND MATERIALS

Three bonding systems were evaluated: Clearfil SE Bond (Kuraray, Osaka, Japan), a self-etching primer system, and two total-etch wet bonding systems that use phosphoric acid etching gel and a one-bottle adhesive, Single Bond (3M ESPE, St Paul, MN, USA) and One-Step (BISCO, Schaumburg, IL, USA). All the materials were used according to the manufacturer's instructions.

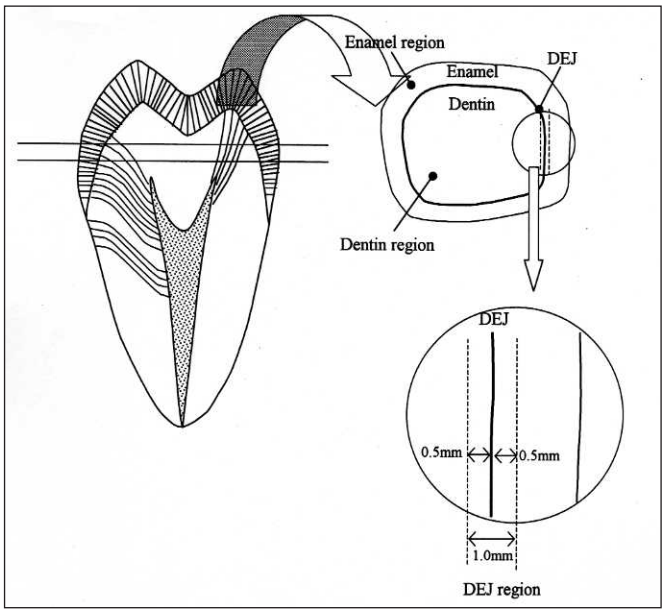

Figure 1. *Tooth regions used.*

Forty-two tooth slices containing enamel and dentin were obtained from extracted human molars that were stored at 4°C in saline. The slices, approximately 1.0-mm thick, were cut parallel to the occlusal surface from the upper middle coronal region using a slowly rotating diamond blade (Struers Minitom, Struers, Copenhagen, Denmark) under water lavage. Three tooth regions were chosen as substrates for testing: enamel, dentin and the DEJ region. The enamel region was defined as enamel located centrally between the DEJ and the outer surface of the tooth, the dentin region was at the outer third of the dentin, approximately 2 mm from the DEJ, and the DEJ region was delineated as being no further than 0.5 mm from the DEJ (Figure 1).

Micro-Shear Bond Test

Thirty slices were randomly selected and assigned to three treatment sequences (10 specimens each) that received one of the following treatments:

Group 1: Etched with Scotchbond Etching Gel for 15 seconds, rinsed for 15 seconds and blot-dried, leaving a moist surface. Two coats of Single Bond Adhesive were applied consecutively, air-thinned and light-cured for 20 seconds;

Group 2: Etched with Uni Etch Gel for 15 seconds, rinsed for 15 seconds and blot-dried, leaving a moist surface. Two coats of One-Step were consecutively applied, air-thinned and light-cured for 20 seconds;

Group 3: Treated with Clearfil SE Bond Primer for 20 seconds and dried. Clearfil SE Bond Adhesive was applied, air-thinned and light-cured for 10 seconds.

Prior to irradiation of the bonding resin, a cylinder of micro bore tygon tubing (R-3603, Norton Performance Plastic Co, Cleveland, OH, USA) with an internal diameter of 0.8 mm and a height of 0.5 mm was cut and mounted on the tooth to restrict the bonding area. A hybrid restorative resin composite, shade A3 (Clearfil AP-X, Kuraray, Osaka, Japan) was placed into the cylinder, and a clear plastic matrix strip was placed over the resin and gently pressed flat and irradiated for 40 seconds. Because the tygon cylinder was bonded tightly to the tooth surface by the bonding resin simultaneously with the photo-curing of the bonding resin, no flash of resin composite extended onto the tooth beyond the base of the cylinder. In this manner, very small cylinders of resin, approximately 0.8 mm in diameter and 0.5 mm in height, were bonded to the surface. The specimens were stored at room temperature (23°C) for one hour prior to removing the tygon tubing, then storing in water at 37°C for 24 hours.

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The micro-shear bond test apparatus is shown in Figure 2. The tooth slice with resin cylinders was attached to the testing device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) with a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA), which, in turn, was placed in a Universal testing machine (EZ-test-500N, Shimadzu, Kyoto, Japan) for shear bond testing. A thin wire (diameter 0.2 mm) was looped around the resin cylinder making contact with half of the cylinder base and held flush against the resin/tooth interface. A shear force was applied to each specimen at a crosshead speed of 1.0 mm/minute until failure occurred.

Ten specimens were tested for each group. The adhesive systems and tooth regions (enamel, dentin and the DEJ region) were the two factors analyzed using two-way analysis of variance (ANOVA) with statistical significance defined as $p<0.05$.

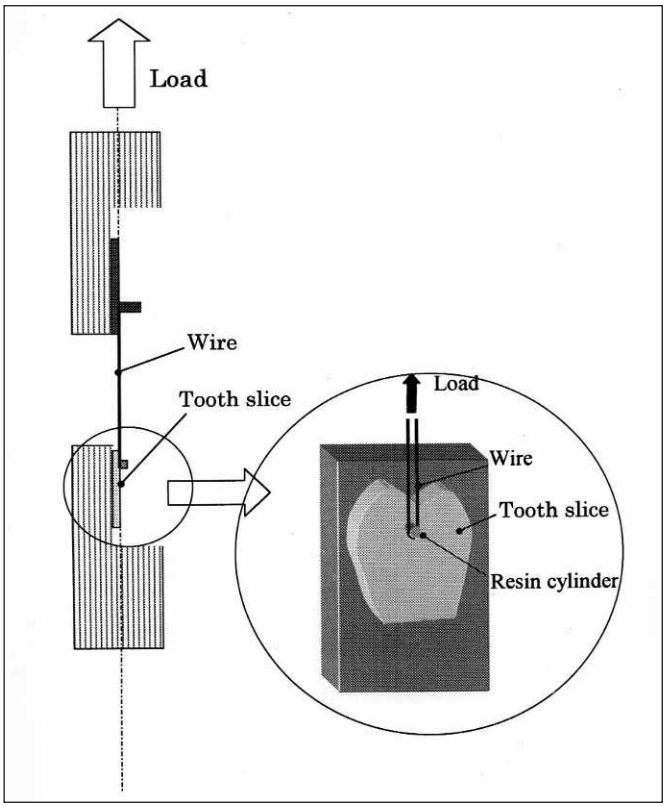

Figure 2. *Schematic diagram of the micro-shear bond test apparatus.*

All the debonded tooth surfaces were examined with an optical microscope at 30x magnification and CLSM (1LM21H/W, Lasertec Co, Yokohama, Japan) to determine the mode of failure. Failure modes were categorized into one of three types: A: 100% adhesive failure between tooth substrate or hybrid-like layer and adhesive resin; C: 100% cohesive failure in tooth substrate; AC: Mixed failure with adhesive failure (A) and cohesive failure in tooth substrate (C). The results of the mode of failure were analyzed using the Kruskal Wallace test with statistical significance defined as $p<0.05$.

Morphological Study Using CLSM

An additional 12 slices were used for the morphological observations. Six slices were divided into three groups (two slices each), corresponding to the adhesive systems, and etched or primed (but not bonded) in the same manner as the shear bond test specimens. The slices treated with the self-etching primer were rinsed for 60 seconds in acetone to remove any crystals or residue from the primer.

The remaining six slices were also divided into three groups and bonded with each of the adhesives in the same manner as that employed for the shear bond test. The bonded specimens were sectioned, ground and polished using wet silicon carbide papers and diamond pastes of decreasing particle size down to 0.25 µm. The etched surfaces and the polished pair of surfaces were viewed under CLSM (1LM21H/W, Lasertec Co, Yokohama, Japan).

RESULTS

Micro-Shear Bond Test

The mean shear bond strength values and standard deviations in MPa and mode of failure are shown in Table 1. The ANOVA indicated that there was no statistically significant variance among the mean bond strength values or no significant interactions between the adhesive systems and tooth regions (two-way ANOVA; adhesive systems, F=0.338, p=0.714; tooth regions, F=1.485, p=0.233; adhesive systems vs tooth regions, F=1.792, p=0.138; one-way ANOVA; F=1.199, p=0.307).

Both one bottle adhesive systems and the self-etching primer system produced similar bond strength values on enamel and dentin, ranging from 37 to 44 MPa. Bonding to the DEJ region was similar to the other bond strength val-

Table 1: *Results of Micro-Shear Bond Test*

		Bond Strength Mean ± SD (MPa)	n	Failure Mode A	AC	C
Single Bond	Enamel	38.0 ± 8.81	10	7	2	1
	Dentin	44.4 ± 3.74	10	2	5	3
	DEJ	37.0 ± 5.98	10	0	6	4
One-Step	Enamel	43.3 ± 11.1	10	6	2	2
	Dentin	38.8 ± 6.49	10	1	2	7
	DEJ	39.5 ± 7.00	10	1	5	4
SE Bond	Enamel	38.0 ± 6.35	10	7	1	2
	Dentin	43.0 ± 6.71	10	2	4	4
	DEJ	39.4 ± 6.62	10	1	6	3

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ues for enamel and dentin, which ranged from 37 to 39 MPa.

However, the mode of failure after shear testing was significantly different among the tooth regions (Kruskal-Wallis, $p<0.0001$), whereas no statistical significance was seen among the adhesive systems (Kruskal-Wallis, $p=0.5271$). Although various failure patterns were observed among the groups, the enamel sites showed adhesive failure between the enamel or underlying hybrid-like enamel and bonding resin (Type A) in most cases. In the case of dentin, cohesive failure in dentin occurred most frequently (Type C), with the remainder showing mixed failure (Type AC). The DEJ region also showed Type C or Type AC failure in most cases.

CLSM Observation

CLSM photomicrographs of enamel, dentin and the region adjacent to the DEJ treated with phosphoric acid gel or self-etching primer are shown in Figure 3. Adhesive interfaces are shown in Figure 4. In the case of phosphoric acid etching, Scotchbond etching gel and Uni Etch Gel showed similar etch patterns, and an application time of 15 seconds was able to remove any surface debris or contamination completely (Figure 3a). The region adjacent to the DEJ was demineralized to a much greater extent and displayed a concave appearance (Figure 4a).

CLSM photomicrographs with the self-etching primer after acetone rinsing revealed that the conditioned surfaces were virtually free of a smear layer (Figure 3b). Nonetheless, the etching effect of the self-etching

Figure 3: *CLSM images of etched tooth surfaces.*

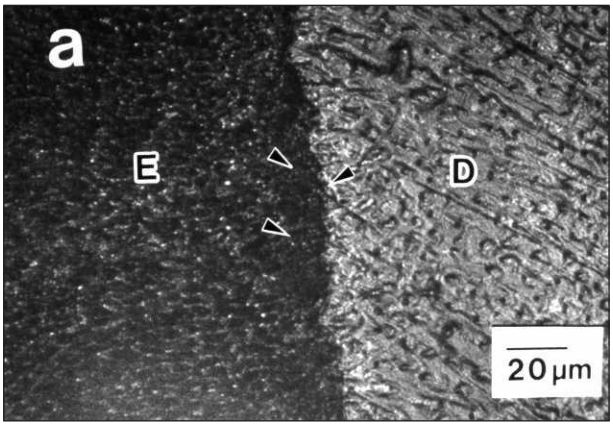

Figure 3a: *The DEJ region etched with Uni Etch Gel for 15 seconds. The smear layer was removed. DEJ region was demineralized to a greater degree than the surrounding enamel and dentin (arrow. E: enamel, D: dentin).*

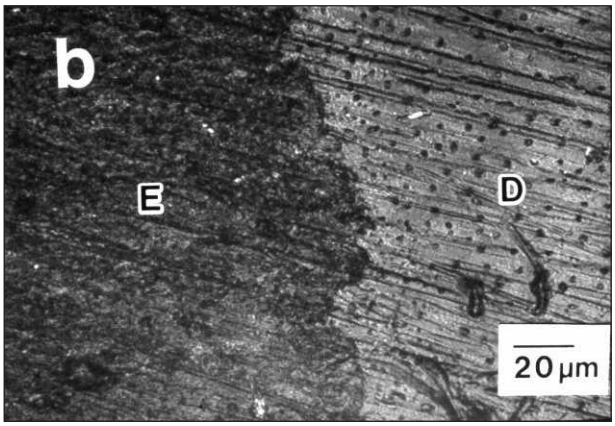

Figure 3b: *The DEJ region treated with Clearfil SE Bond Primer. The smear layer was removed, however, in the vicinity of the DEJ, it was not as deeply etched as in (a) (E: enamel, D: dentin).*

Figure 4: *CLSM images of bonded tooth/resin interfaces.*

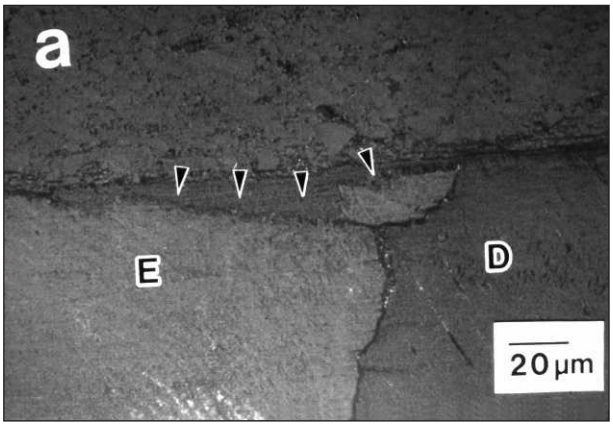

Figure 4a: *One-step. The DEJ region was etched deeply, showing concave appearance (arrow) (E: enamel, D: dentin).*

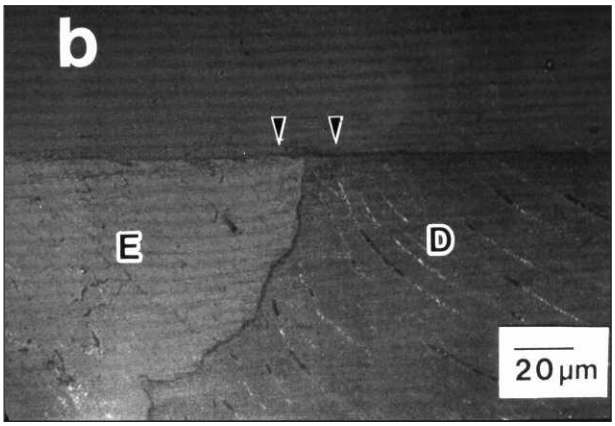

Figure 4b: *Clearfil SE Bond. The depth of etched DEJ region was almost same as the enamel and dentin (arrow) (E: enamel, D: dentin).*

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primer was not as aggressive as phosphoric acid, showing a lesser concavity at the DEJ region (Figure 4b).

DISCUSSION

The region used for bonding at the DEJ vicinity was no further than 0.5 mm from the DEJ on the dentin side. The results of this laboratory study showed that bonding of all the adhesive systems in the region of the DEJ was almost the same as that for enamel or dentin, with no statistical significance observed (Table 1). Urabe and others (2000) compared the fracture toughness of the DEJ region and compared it with enamel and dentin. Their results indicated that the DEJ had a fracture toughness that was between enamel and dentin. While a complex scalloped pattern between enamel and dentin is a noticeable feature of the DEJ, bonding to the DEJ region was attributed to a mixture of dentin and enamel bonding.

Although all three regions produced similar bond strength values, the failure mode was variable; enamel was mostly Type A failure, whereas, dentin and the DEJ region were Types C or AC (Table 1). The anatomical structure and mechanical properties of the DEJ region have been reported previously (Lin & Douglas, 1994; Wang & Weiner, 1998; Mjör & others, 2001; Tesch & others, 2001). The DEJ region has a reduced hardness and is less mineralized than the rest of the coronal dentin (Wang & Weiner, 1998; Tesch & others, 2001). The failure pattern observed on dentin and the DEJ may have been due to the lower elastic moduli compared with the bonding resin. This might have caused an uneven stress distribution on the bonded interface during testing that led to the failure patterns observed (DeHoff, Anusavice & Wang, 1995; Versluis, Tantbirojn & Douglas, 1997; Urabe & others, 2000). While in case of the enamel substrate, which has a higher elastic modulus than bonding resin, adhesive failure (Type A) was the predominant fracture pattern.

Results from the CLSM observations indicated that the DEJ region was more susceptible to acid etching than enamel or dentin (Figures 3 and 4). The relatively low degree of mineralization of dentin and enamel in this region resulted in a deeper etch pattern (Mjör & others, 2001; Tesch & others, 2001). In addition, the collagen fibers or branching of tubules at the DEJ may accelerate the lateral spread of etching solutions (Lin & Douglas, 1994; Mjör & others, 2001). This phenomenon is similar to the lateral spread of early caries that progresses laterally at the DEJ (Roberson & Lundeen, 2002).

Interestingly, the etched dentin surfaces produced almost the same bond strength values as the enamel surfaces. There is no doubt that the high bond strength to dentin or dentin near the DEJ was derived from sufficient impregnation of resin into the etched surface (Nakabayashi & others, 1982; Kanca, 1992). Also, the dentin region used in this study was a peripheral part that corresponded to the highly mineralized mantle dentin, which was less permeable than the inner part of the dentin. The strong bonding might, in part, be attributed to the fact that the peripheral area was more receptive to the bonding resin, as previously reported (Pashley, Michelich & Kehl, 1981; Tagami, Tao & Pashley, 1990). In this study, the micro-shear bond strength of a direct restorative system in the region adjacent to the DEJ was comparable with bond strengths to dentin and enamel.

CONCLUSIONS

The bonding of adhesive systems using phosphoric acid etching with the wet bonding technique and a self-etching primer system to enamel, dentin and in the region adjacent to the DEJ showed little variation among the three substrates. Although the relatively intense action of acid etching of the DEJ region was observed, bonding was not influenced by this regional variation.

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Surface pH and Bond Strength of a Self-Etching Primer/Adhesive System to Intracoronal Dentin After Application of Hydrogen Peroxide Bleach with Sodium Perborate

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Clinical Relevance

Bleaching with hydrogen peroxide caused alteration to dentin surface pH and adversely affected the bond strength performance of a self-etching primer/adhesive system. Delaying bonding for one week is recommended to allow for complete recovery of bleached dentin.

SUMMARY

This study compared the dentin bond strength of a self-etching primer/adhesive system with dentin surface pH with or without bleaching and observed the morphological changes in bleached dentin treated with a self-etching primer. Dentin disks were prepared from the coronal-labial region of 32 human anterior teeth. The pulpal surfaces of the dentin disks were polished with 600-grit SiC paper under running water. The dentin surfaces on all specimens were bleached with a mixture of 30% hydrogen peroxide and sodium perborate in 100% humidity at 37°C for one week. The bleaching agent was then rinsed off with water for 5, 15 or 30 seconds. All specimens were stored in water at 37°C. Half of the five-second rinsing specimens were stored in water for an additional week. Dentin surface pH with or without bleaching was examined using a pH-imaging microscope (SCHEM-100). A self-etching primer/adhesive system (Clearfil SE Bond) was applied to bleached or unbleached dentin according to the manufacturer's instructions. After 24-hour water storage, the bonded specimens were prepared for microtensile testing. Microtensile bond strength (μTBS) to dentin was measured using a universal-testing machine (EZ test, Shimadzu, Japan) at a crosshead speed of 1.0 mm/minute. Data were analyzed by one-way ANOVA and Scheffe's test (α=0.05). The pH values of the dentin surfaces of the 5 and 15 sec-

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ond rinsing groups were significantly higher than the control group ($p<0.05$), while the 30-second rinsing and one-week water storage groups had similar surface pH values to the control group ($p<0.05$). The µTBS of 5, 15 and 30 second rinsing specimens after bleaching were significantly lower than the control specimens ($p<0.05$). However, after one-week of water storage, the µTBS returned to the control group. The application of a bleaching agent increased the pH value of the dentin surface and decreased the bond strength of the self-etching primer/ adhesive system. One-week water storage after bleaching recovered the surface pH and the µTBS to dentin.

INTRODUCTION

The demand for a "perfect white smile" has recently heightened. Bleaching is the most conservative technique to improve the unpleasant appearance of discolored teeth (Goldstein, Haywood & Heymann, 1994). For non-vital, discolored teeth, an intracoronal technique using hydrogen peroxide (up to 35%) with or without sodium perborate has shown clinical success. However, the bleaching mechanism was performed without a comprehensive understanding of the effects on the morphological, mechanical and chemical properties of the tooth surface. Recently, many reports on the bleaching effect on enamel and dentin have become available. Several studies have shown changes in enamel and dentin surface morphology (Titley, Torneck & Smith, 1988a,b; Ernst, Marroquin & Willershausen-Zonnchen, 1996; Zalkind & others, 1996; Smidt & others, 1998) and a reduction in bond strength after bleaching (Torneck & others, 1990a; Titley & others, 1993; Stokes & others, 1992; García-Godoy & others, 1993; Miles & others, 1994; Dishman, Covey & Baughan, 1994; Demarco & others, 1998; Spyrides & others, 2000; Cavalli & others, 2001). The reduction in bond strength after bleaching might result from these morphological changes (Zalkind & others, 1996). Furthermore, the presence of oxygen, which is a breakdown product of hydrogen peroxide, has been related to a reduction in bond strength after bleaching because the residual oxygen might interfere with resin infiltration into the dentinal tubules (Torneck & others, 1990b) and/or inhibit resin polymerization (McGuckin, Thurmond & Osovitz, 1992; Dishman & others, 1994). Oxygen was reported to accumulate within dentin bleached with hydrogen peroxide, since dentin may act as an oxygen reservoir (Titley & others, 1993). On the other hand, a delay of a few weeks for the bonding procedure following bleaching provides time for the adverse effects to dissipate (Demarco & others, 1998; Cavalli & others, 2001).

The bleaching technique for non-vital, discolored teeth has often involved a mixture of 30% hydrogen peroxide and sodium perborate, which is made into a paste that is sealed into the pulp chamber to permit the solution to undergo activation over several days. It has been reported that the optimum pH for hydrogen peroxide in bleaching is 9.5 to 10.8 (Frysh & others, 1993). Recently, Hiraishi and others (2003) evaluated the pH changes on dentin surface with or without saliva contamination using a pH-imaging microscope (SCHEM-100, Horiba Ltd, Kyoto, Japan) and also measured bonding performance to contaminated dentin. They reported that saliva contamination caused a slight increase in dentin surface pH and a reduction in dentin bond strength. SCHEM-100 can perform quantitative multiple-point pH analysis of a solid sample by measuring the pH distribution formed in a thin agar film attached to the sample by the pH-imaging sensor (Hafeman, Parce & McConnell, 1988; Nomura & others, 1997). The pH values at each pH-measuring point are converted to pixels in gray scale mode using imaging analysis software that has been used to characterize features of metals (Nomura & others, 1997), damage to human hair and metabolic activity of *E coli* (Yoshinobu & others, 1997).

This study evaluated the effects of a bleaching agent consisting of 30% hydrogen peroxide with sodium perborate on dentin surface pH and tensile bond strength of a self-etching primer/adhesive system and investigated the morphological changes of the bleached dentin surface treated with a self-etching primer.

METHODS AND MATERIALS

Specimen Preparations

Thirty-seven extracted intact human anterior teeth were used in the study. The teeth were cleaned and kept in water at 4°C until required. Dentin disks were prepared from the coronal-labial region of the tooth using a cutting wheel under water supply. The pulpal surfaces of the dentin disks were polished with 600-grit silicon carbide paper under running water. The pulpal dentin surface was bleached by applying a mixture of 30% hydrogen peroxide (H_2O_2) and sodium perborate ($NaBo_3$-$4H_2O$) in 100% humidity at 37°C for one week (Figure 1), then rinsed with water for 5, 15 or 30 seconds, or five-second rinsing following one-week water storage.

Surface pH Measurement

Eighteen dentin disks were used for pH measurement, and the average pH value of all dentin surface areas was evaluated using a pH-imaging microscope (SCHEM-100, Horiba Ltd) as a control. The dentin specimens were randomly divided into three groups (5-, 15- and 30-second rinsing groups), and after bleaching and rinsing, surface pH values were measured. Half of the specimens of the five-second rinsing group were stored in water for one week, and the surface pH values

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measured again. The pH-imaging sensor is a flat semiconductor sensor with a sensing area of 2.5 x 2.5 cm. The sensor is based on Light Addressable Potentiometric Sensor (LAPS) (Hafeman & others, 1988) consisting of two layers (Figure 2). The upper layer is made of Si_3N_4/SiO_2, in which Si_3N_4 is a proton-sensitive layer able to perceive protons coming from the electrolyte, and the lower layer is made of silicon (Si). According to the semiconductor characteristics of the silicon, an AC photocurrent is induced by the modulated illumination of the silicon side of the sensor when bias voltage is applied between the electrolyte and silicon. Depending on the amount of protons reaching the Si_3N_4 layer, an AC photocurrent flow changes quantitatively, resulting in pH measurement. The current is converted to a grayscale pixel and each pixel is arranged in a pH image using image analysis software (Image Pro Plus, Media Cybernetics, MD, USA). The pH value is correlated to the grayscale of each pixel that composed the image at each measurement point, so that the pH values can be calculated using computer software (EXCEL, Microsoft Corp, Redmond, WA, USA).

A 1.5% agar solution consisting of 0.1M potassium chloride and agar powder was prepared as an electrolyte to make a 1-mm thick agar film on the semiconductor sensor of the SCHEM-100. The bleached and unbleached dentin disks were placed face down on the agar film. If protons were released into the agar film from the

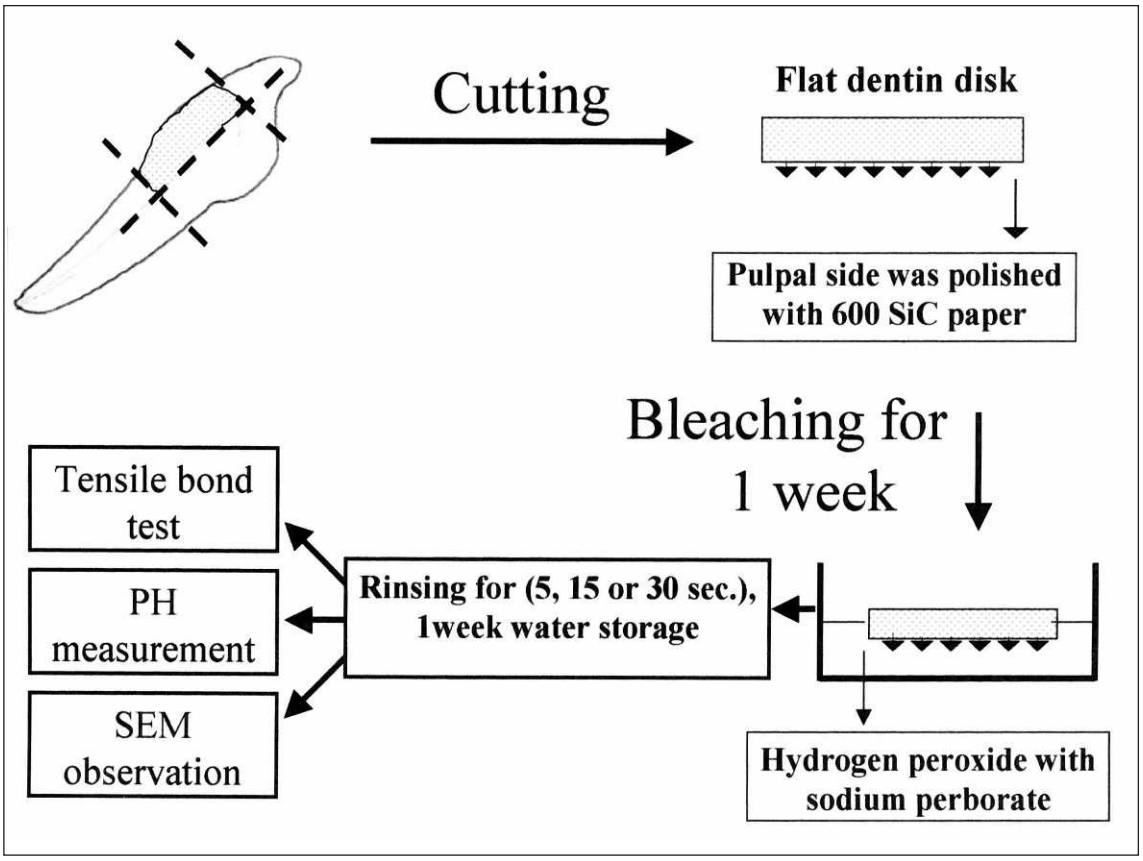

Figure 1. *Schematic of preparation of tooth.*

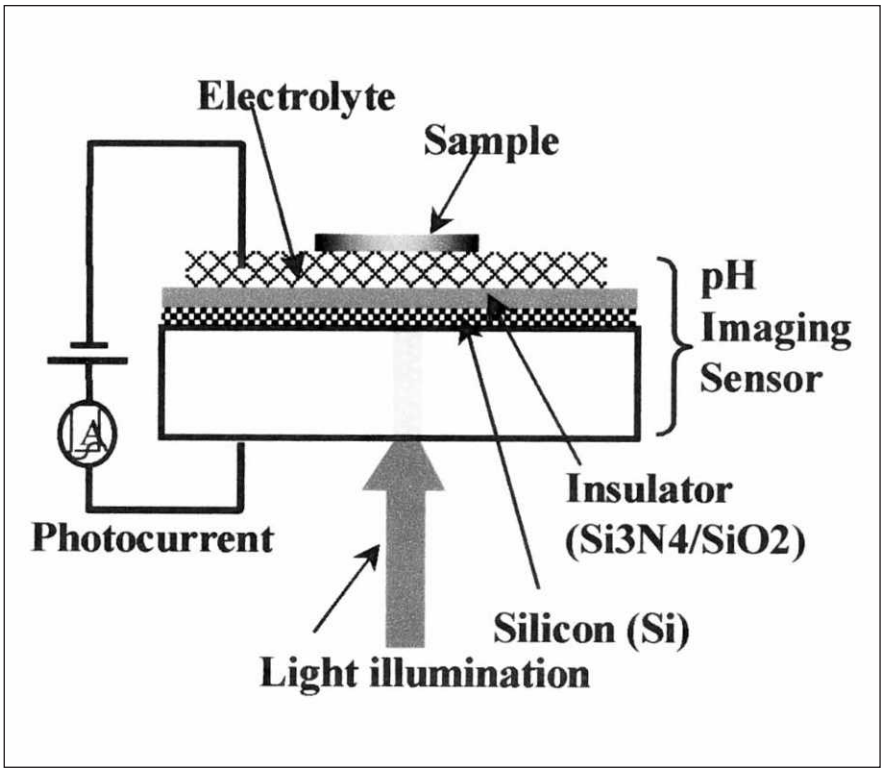

Figure 2. *Schematic illustration of the sensor of SCHEM-100.*

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dentin surface, a pH distribution would be generated. The AC photocurrent varies due to the amount of H^+ or OH^- ions in the agar film, and the pH distribution on the bottom of the agar film is displayed as a pH image.

In this study, pH image was obtained a spatial resolution of 300 µm. As the pH value of initial agar film was acknowledged prior to placing the sample, changes in the photocurrent following placement of the sample were converted into pH.

Microtensile Bond Test

Fifteen teeth were used to measure bond strength to the pulpal dentin surface. Bleached and unbleached (control) dentin surfaces were treated with a self-etching primer/adhesive system (Clearfil SE Bond, Kuraray Medical Inc, Tokyo, Japan) according to the manufacturer's instructions (Table 1). A resin composite (Clearfil AP-X, Kuraray Medical Inc) was built incrementally up to a height of 5 mm to ensure sufficient bulk for the microtensile bond test (Sano & others, 1994). A resin composite (Clearfil AP-X) was placed on the other enamel surface of all the specimens to make grips for testing and was stored in 37°C water for 24 hours. The resin-bonded teeth were then serially sectioned perpendicular to the adhesive interface in three or four slabs approximately 0.7-mm thick using a low-speed diamond saw under water cooling. The bonded areas were isolated using a superfine diamond bur (c16ff, GC Ltd, Tokyo, Japan) to create an hourglass configuration with a cross-sectional area approximately 1 mm^2. The final width and thickness of the bonded area were measured by means of a digital caliper. The specimens were then attached to the testing device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) with a cyanoacrylate (Zapit, Dental Ventures of America, Corona, CA, USA), which, in turn, was placed in a table-top material tester (EZ-test, Shimadzu Co, Kyoto, Japan) for tensile testing at a crosshead speed of 1 mm/ minute (Sano & others, 1994).

Statistical Analysis

The variations among the studied groups in the first and second experiments were evaluated statistically using one-way ANOVA and Scheffe's test ($p<0.05$).

Ultra Structural Examinations

Four teeth were used to analyze dentin surface morphology after bleaching. Bleached and unbleached samples were treated with the self-etching primer. The primer components were removed by ultrasonic cleaning in 50% acetone. All samples were then dehydrated in ascending grades of ethanol (50%, 75%, 95% and 100% for 30 minutes), followed by immersion in hexamethyldisilazane solution (HMDS) for 10 minutes, placed on filter paper inside a covered glass vial and air dried at room temperature. The samples were then gold sputter coated and observed with a scanning electron microscope (JSM-531, JEOL, Tokyo, Japan). Photographs were taken of the control and exper-

Table 1: *Adhesive System Used in This Study*

Product	Components	Chemical Composition
Clearfil SE Bond	Primer	MDP, HEMA, water, initiator
(Kuraray Medical Inc, Japan)	Bond	MDP, HEMA, dimethacrylates, intiator, microfilter

Table 2: *pH Values of Bleached Dentin*

	Control	5 seconds	15 seconds	30 seconds	1 week (1W)
pH	6.7±0.1(18)[a]	7.7±0.4(6)[b]	7.2±0.3(6)[b]	6.5±0.1(6)[a]	6.5±0.1(6)[a]

Values are mean ± SD (N). Groups identified by different superscript letters are significantly different (p<0.05).

Table 3: *Tensile Bond Strength to Bleached Dentin*

	Control	5 seconds	15 seconds	30 seconds	1 week (1W)
µTBS	39.2±7.8(10)[a]	24.4±4.5(10)[b,c]	19.5±4.8(10)[c]	24.5±4.0(10)[b,c]	36.0±12.9(10)[a,b]

Values are mean ± SD (N). Groups identified by different superscript letters are significantly different (p<0.05).

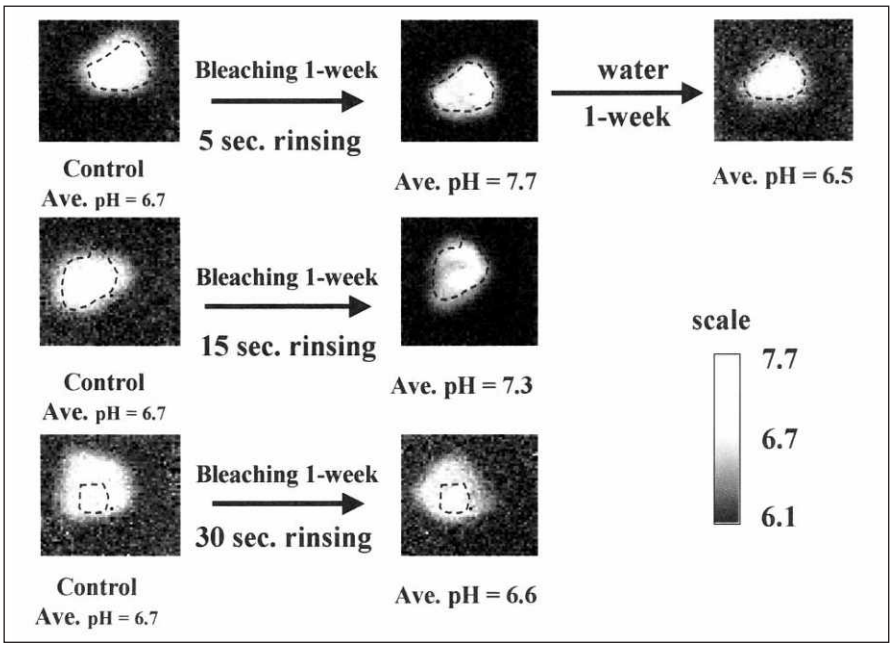

Figure 3. *Representative pH changes of dentin surfaces (bleached and unbleached) with pH explanatory scale. The dotted line represents the location of the specimen.*

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imental specimens at chosen magnifications of 10,000 to observe the differences in surface alterations.

RESULTS

pH of Dentin Surface

Figure 3 shows a representative TIFF pH image of a dentin disk from each experimental group with a pH explanatory scale. Table 2 shows the pH values of dentin surfaces including the means and standard deviations and number of samples. The intact dentin surfaces showed a pH of 6.7. The average pH of the 5 and 15 second groups was 7.7 and 7.2, respectively, and they were significantly higher than the control group ($p<0.05$). No significant differences were found between the pH values of the 30-second rinsing and the control groups ($p>0.05$). Rinsing the dentin surface and waiting for one week produced a pH value of 6.5, which was not significantly different from the control group ($p>0.05$).

Microtensile Bond Test (µTBS)

The results of the µTBS are summarized in Table 3, including means, standard deviations and number of samples. For the control group, the µTBS was 39.2 MPa. In the bleached groups, the µTBS was significantly lower than the control ($p<0.05$)—24.4, 19.5 and 24.5 MPa for 5-, 15- and 30-second rinsing groups, respectively. Delaying bonding for one-week after bleaching resulted in a µTBS equal to 36.0 MPa, which was not significantly different from the control group ($p>0.05$).

SEM Examination

The differences in surface morphology of the unbleached (control) and bleached dentin following rinsing with water for 5, 15 or 30-seconds are shown in Figure 4. For the control group, the smear layer was completely removed after applying the primer. Photographs of bleached samples showed an accumulation of white granular deposits on the dentin surface. For the five-second rinsing group, the dentin surface was contaminated with a large amount of deposit, while a smaller amount was noted on the surface of the 30-second rinsing samples. Deposits were also seen on the dentin surfaces of the samples in the 15-second rinsing group.

DISCUSSION

Many researchers have investigated the effects of bleaching on bond strengths to enamel (Titley & others, 1988b; Titley & others, 1993; Stokes & others, 1992; García-Godoy & others, 1993; Miles & others, 1994; Dishman & others, 1994; Cavalli & others, 2001) and dentin (Torneck & others, 1990b; Demarco & others, 1998; Spyrides & others, 2000). These studies have shown that hydrogen peroxide- and carbamide peroxide-based bleaching agents reduced the immediate bond strength to enamel and dentin. On the other hand, it has been reported that the effects of bleaching agent on bond strength to enamel were reversible (Cavalli & others, 2001) and delaying the bonding procedure for one week after bleaching decreased the adverse effects for bonding to human molar dentin (Demarco & others, 1998) and bovine incisors (Spyrides & others, 2000), using phosphoric acid etchant. These results agree with the findings of this experiment, which used the pulpal dentinal surface of human incisors. Clinically, it is important to evaluate bonding to the pulpal dentin surface after bleaching non-vital, endodontically-treated teeth because the bleaching agent is placed into the pulp chamber. In this study, the µTBS of the self-etching primer/adhesive system in the five-second rinsing group decreased significantly compared to the control group, while the µTBS in the one-week water storage-after-bleaching group recovered a similar value to the control group. The reduction in bond strengths to hydrogen peroxide

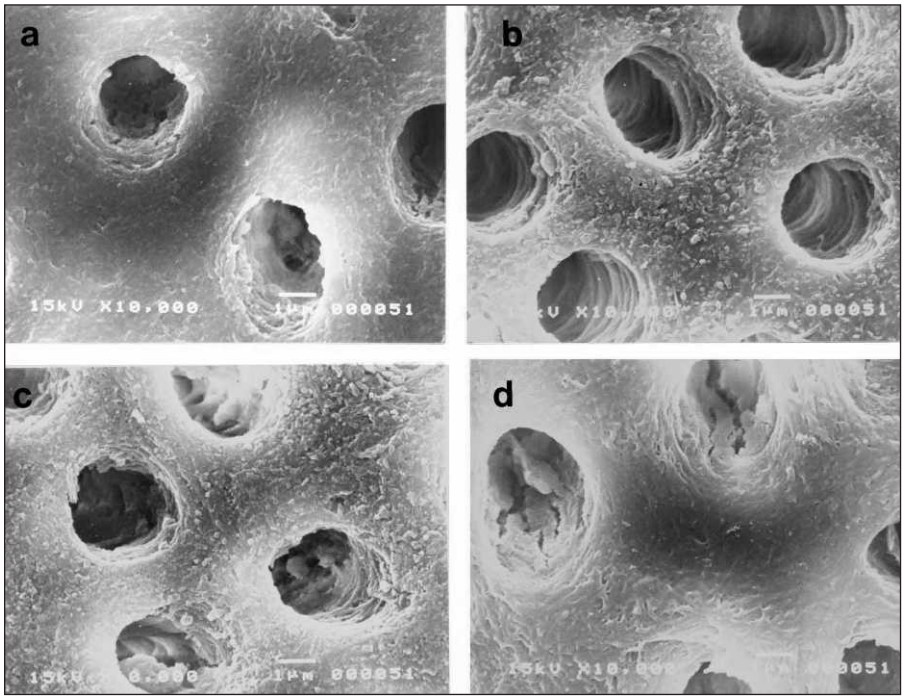

Figure 4: *(a) Scanning electron micrograph of dentin surface applied with the self-etching primer. Smear layer was absent and dentin tubules were seen. (b) Scanning electron micrograph of dentin surface treated with the self-etching primer after bleaching and rinsing with water for five seconds. An accumulation of white granular deposits could be seen on the intertubular dentin. (c) Scanning electron micrograph of dentin surface treated with the self-etching primer after bleaching and rinsing off with water for 15 seconds. A few white granular deposits could be seen on the intertubular dentin comparing with Figure 4b. (d) Scanning electron micrograph of dentin surface treated with the self-etching primer after bleaching and rinsing off with water for 30 seconds. Few white granular deposits could be seen on the intertubular dentin.*

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treated-dentin could have been caused by the release of oxygen, a breakdown product of hydrogen peroxide. The residual oxygen may have inhibited polymerization of the resin that was cured via a free radical mechanism (Demarco & others, 1998) and/or interfered with resin infiltration into dentinal tubules (Tornek & others, 1990b).

In this study, hydrogen peroxide with sodium perborate also caused changes in dentin surface pH. The pH value of the dentin surface after bleaching and rinsing for five-seconds was higher than the control group, and after one-week storage in water, the surface pH returned to a level similar to the control group.

The scanning chemical microscope SCHEM-100 used in this study allows quantitative measuring of the surface pH of a solid sample, and specimen preparation for the SCHEM does not require destruction of the samples by dissolution. The pH distributions formed within an agar film placed beneath the sample can be detected by a pH-image sensor contacting the bottom of the agar film. Therefore, the pH values measured in this study are not actual pH values but are relative to the initial pH of the agar solution. However, the obtained images of pH distribution are thought to show the amount of H^+ or OH^- ions released from the sample subsurface (Nakao & others, 1996). Few studies have reported an analysis of the pH of dental hard tissues using the SCHEM-100 to analyze dental hard tissues with different conditions (Kitasako & others, 2002; Hiraishi & others, 2003).

Hiraishi and others (2003) observed that the dentin surface pH decreased after acid etching and increased after saliva contamination using the SCHEM-100. The pH values revealed the total amount of ions H^+ or OH^- within the mineral and organic phases of the dentin surface. These pH changes might be due to remaining acid conditioner or proteinaceous components of saliva (Hiraishi & others, 2003).

It is important to investigate not only the morphological changes but also any chemical changes in the conditioned dentin surface. In this study, the surface pH value was dependent on the rinsing time after bleaching. The surface pHs of the five-second and the 15-second groups were higher than the control group. These results indicate that the bleaching agent was left on the dentin surface. For the 30-second group, the average pH was similar to the control group; however, some portions were of higher pH value in the pH images of the dentin surface (Figure 3). Hydrogen peroxide was reported to precipitate on the dentin surface and interact with the smear layer (Titley & others, 1988a). The short time rinsing would be not enough to completely remove the bleaching agent from the dentin surface. After one-week storage in water, the surface pH returned to normal. Hydrogen peroxide is extremely unstable in water. Therefore, hydrogen peroxide may lose its activity on the dentin surface but is not absent from the dentin surface during the one-week storage in water.

The pH of the dentin surface would influence etching of the dentin by the self-etching primer. Demarco and others (1998) reported that immediate acid etching by phosphoric acid after bleaching could not remove the smear layer. They indicated that this could be due to a chemical reaction involving hydrogen peroxide (Demarco & others, 1998). In this study, SEM observation showed that there were many granular deposits on the intertubular dentin in the five-second group, and the amount of deposits decreased when rinsing time was longer. In contrast, for the control and one-week groups, the deposits could not be observed. These deposits might be due to a chemical reaction between dentin and hydrogen peroxide. When the dentin surface pH was higher, it would influence the ability of the smear layer removal and demineralization of the underlying dentin by self-etching primer. Moreover, the self-etching primer simultaneously demineralizes and penetrates into the dentin matrix, contributing to a hybrid layer formation. Therefore, the etching effect of the self-etching primer on the dentin surface might have an important role in producing high bond strength to dentin. The higher pH of the dentin surface after bleaching may have been a reason why the μTBS of the self-etching primer/adhesive system to dentin decreased.

CONCLUSIONS

The results of this study suggest that the reduction of bond strength to dentin after bleaching with hydrogen peroxide is related to a higher surface pH and alteration of the etching effect by a self-etching primer. These changes revert back to those of unbleached dentin within one-week water storage.

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Evaluation of Active and Arrested Carious Dentin Using a pH-imaging Microscope and an X-ray Analytical Microscope

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Clinical Relevance

A decrease in pH and mineral loss was significantly correlated in active carious dentinal lesions.

SUMMARY

A pH-imaging microscope was tested on carious teeth to examine the acid-base characteristics of active and arrested carious dentinal lesions and compared with an assessment of mineral loss in carious affected lesions using an x-ray analytical microscope. Extracted human molars, 22 active and 83 arrested carious teeth, were sliced vertically to maximize the presence of visible carious lesions. The pH change on the dentin surface was measured with a pH-imaging microscope, and the mineral loss was measured with an x-ray analytical microscope. The pH value of intact dentin and active and arrested carious dentinal lesions showed 7.0 (n=105), 5.8 (n=22) and 6.6 (n=83), respectively (analyzed by Kruskal-Wallis). The mineral loss was distinguishable in active dentinal lesions (4.92 times as much as intact dentin), while arrested lesions showed a slight mineral loss (2.19 times as much as intact dentin). The changes in pH value and mineral loss were significantly correlated (Pearson's correlation coefficients r=-0.8024) in active carious dentin lesions and weakly correlated (r=-0.1480) in arrested carious dentin lesions. Changes in tooth substance with the progression of the active carious process were reflected in a reduction in pH value and an increase in the amount of mineral loss.

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INTRODUCTION

Caries diagnosis should be accurate and objective in order to distinguish it from intact tooth structures (Ekstrand & others, 1998). However, a significant amount of observer variation exists in conventional clinical examinations by x-ray radiography, probing of pits and fissures and dye solutions (Grondahl, 1979). Many techniques in the detection and quantification of caries have been developed and investigated to examine demineralized or remineralized lesions, such as polarized light microscopy (Hicks & Silverstone, 1984), microhardness (Arends, Schuthof & Jongebloed, 1980; Featherstone & others, 1983), microradiography (Arends & others, 1989; Dijkman, Schuthof & Arends, 1986; Zuidgeest, Heckstiotez & Arends, 1990), light

scattering (Brinkman, ten Bosch & Borsboom, 1988) and passage of an electric current (Longbottom & others, 1996). A new clinical technique for carious detection using quantitative analysis has been limited to a laser-based instrument (Lussi & others, 1999). An alternative method for evaluating carious lesions would play an important role in caries detection if it were non-destructive, sensitive and quantitative. It is important to precisely define the distribution or expansion of a carious lesion, because considerable attention should be paid to preserving natural tooth structures in order to apply a technique of minimum intervention (Mount & Ngo, 2000).

Recently, a pH-value analyzing technique has been introduced into dentistry which used a pH-imaging microscope (SCHEM-100, Horiba Ltd, Kyoto, Japan) (Kitasako & others, 2000; 2002). Preparing for SCHEM-100 does not require samples to be destroyed by dissolution. A pH analysis can be performed by a simplified process of placing flat, solid samples on a semiconductor silicon sensor with photocurrent characteristics (Nomura & others, 1997; 2000). The pH values of carious and intact dentin have been investigated visually and quantitatively. It was reported that carious dentin indicated a lower pH value than intact dentin (Kitasako & others, 2000). Moreover, the pH value changes of active and arrested carious lesions in deep dentinal caries were evaluated with SCHEM-100 and compared by observation using a caries detector solution (Kitasako & others, 2002). There was a significant relationship between pH-imaging characterization and visual inspection using dye staining within active lesions although the arrested lesions were unstainable and impermeable to the dye.

The validity and reproducibility of this pH-analyzing method requires further investigation, and its accuracy should be compared with a representative diagnostic method of detecting dental caries, such as radiographic examination. In this study, an x-ray analytical microscope (XGT-2700, Horiba Ltd, Kyoto, Japan) was used to quantify the mineral content in carious lesions. It combines radioscopic analysis, x-ray penetrating ability and microscopic magnifying ability. This microscope enables the internal structure of a substrate to be determined while the x-rays are transmitted through the substrate and measured by a scintillation detector (Yamamoto, 1996). Moreover, the XGT-2700 is capable of fluorescent x-ray analysis and mapping of the 31 elements between sodium and uranium. The XGT-2700 has been applied on dentinal tissues to evaluate *in vitro* secondary caries formation and inhibition by measuring mineral content and mapping the calcium distribution (Okuda & others, in press).

This study analyzed the correlation between the pH value changes and mineral loss in active and arrested dentin caries.

METHODS AND MATERIALS

Samples Preparation

Human molars with carious dentin were extracted from patients between 18 and 60 years of age. The teeth were extracted because of severe caries or periodontal disease and used for research and educational purposes upon verbal agreement. The teeth were divided into active or arrested caries using the following criteria: active caries—mainly found in young children or adolescents and associated with light brown pigmentation, pain upon chemical stimulation of certain foods, thermal stimulation from a blast of cold air or hot/cold drinks; arrested caries—associated with dark pigmentation, hard or leathery surface and no history of pain (Miller & Massler, 1962; Sarnat & Massler, 1965). Pain or sensitivity was used as the most responsible evidence for selecting teeth after questioning patients about their presence upon certain stimulation. After extraction, the carious activity was confirmed by *in vitro* observation, such as color and hardness. Some lesions could not be fully categorized as active or arrested

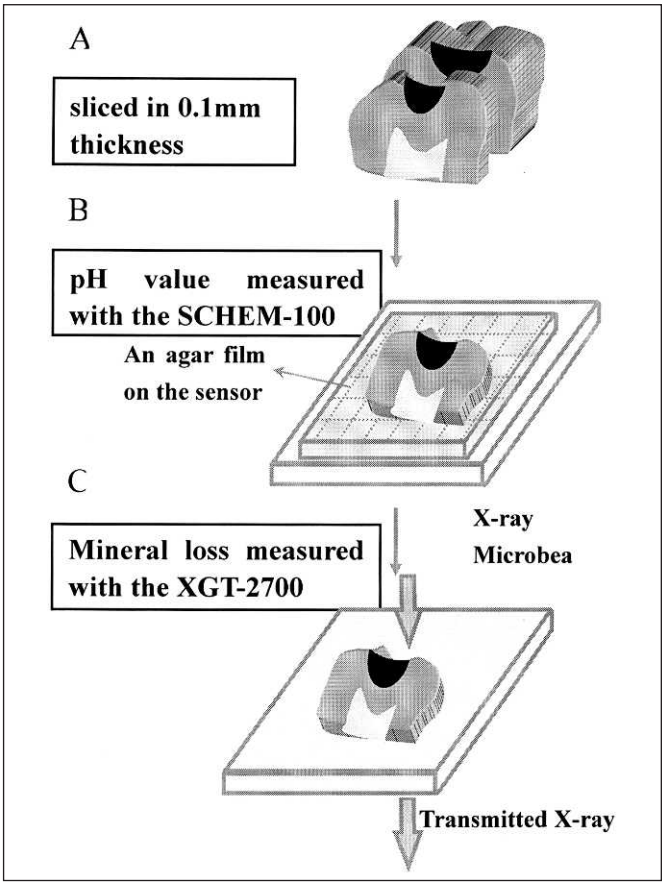

Figure 1. *Scheme of measurement procedure. (A) A tooth with a carious dentinal lesion was sliced vertically to obtain an approximately 1-mm thick sample. (B) The sample was placed on the pH-imaging sensor for pH value measurement with the SCHEM-100. (C) The sample was irradiated by a microbeam and the transmitted x-ray was detected for mineral loss measurement with the GXT-2700.*

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lesions. The authors had to discard teeth that fit into an intermediary group and had a mixture of active and arrested characteristics. Through this process, 22 active and 83 arrested caries teeth were obtained.

The extracted teeth had been frozen in acid-base characteristics for no longer than one month to avoid alterations and were defrosted before use. The center of the carious lesion in the occlusal region was sliced vertically to obtain an approximately 1-mm thick sample using a diamond saw (Leitz Instruments, Heidelberg, Germany). The sliced surface was ground with 600-grit silicon carbide paper under running water for 10 strokes to decrease irregularities and contaminants on the surface.

pH Measurement of Dentin Surfaces with the SCHEM-100

The pH values of active and arrested carious dentin were measured with the SCHEM-100. The pH-imaging sensor is based on a Light Addressable Potentiometric Sensor (LAPS) made of Si_3N_4/SiO_2 and a Silicon (Si) (Hafeman, Parce & McConnell, 1988). For set-up of the SCHEM-100, a 1.5% agar solution consisting of 0.1 M potassium chloride and agar powder was used to make 1-mm thick agar film on the semiconductor sensor (Figure 1). When the dentin sample was placed on the agar film, H^+ or OH^- ions traveled from the dentin surface into the agar film. The AC photocurrent varied due to the amount of H^+ or OH^- ion coming through the agar film from the sample and was converted into a grayscale pixel, with each pixel arranged to pH imaging with image analysis software (Image-Pro Plus, Media Cybernetics, Silver Spring, MD, USA). The pH measurement at multiple points was conducted and the pH distribution displayed as pH images. As the grayscale of each pixel that made up the image was correlated to the pH value at each measurement, the pH value that composed the pH image was examined using EXCEL software (Microsoft Corp, Redmond, WA, USA) (Nakao & others, 1996). The spatial resolution and the pH resolution of the sensor were 200 µm and 0.1 pH, respectively.

The carious lesion or intact dentin was defined in the image analysis as follows: the lowest intensity of the grayscale area was determined to be the carious lesion; the highest intensity in dentin was the intact dentin (Kitasako & others, 2000; 2002).

Mineral Loss Measurement with XGT-2700

Following SCHEM-100 measurement, the mineral loss of all the samples was examined with the XGT-2700. The XGT-2700 was developed to analyze elemental composition and internal structure by combining the analytical ability of a radioscopic analyzer, the penetrating ability of an x-ray and the magnifying ability of a microscope. A fine and high intensity x-ray beam (diameter of less than 10 µm) was irradiated on the sample, and the fluorescent x-rays subsequently generated from the sample were reconstructed to enable a mapping image of the surface elements, while the x-rays transmitted through the sample were simultaneously measured to determine the internal structure by a scintillation detector. The 1-mm thick samples were placed on the stage of the XGT-2700, and their internal structures examined using a 50 kV tube voltage and 1 mA tube current at a resolution of 100 µm.

The mineral loss of a sample was determined from the intensity of the transmitted x-rays, which were converted into TIFF images by Image-Pro Plus in order to calculate the grayscale level that was expressed on a scale between 0 and 255 (0–completely black; 255–completely white). The grayscale values of the TIFF images were associated with the intensity of the transmitted x-rays, with the dark zones representing mineralized zones and the light zones reflecting demineralized zones.

To determine the mineral loss of carious lesions and intact dentin, the lowest and highest pH value point of the pH measurement were used as reference points. The best-fit point of the lowest pH value and the highest pH value in dentin were assumed to be the carious point and intact point on TIFF images of the XGT-2700, respectively. Grayscale values were read on the best-fit points to obtain grayscale values of carious lesion (Gc) and intact dentin (Gi). The fractional change in grayscale (Gc/Gi) was taken as the mineral loss per unit area, because the thickness of the sample might influence the intensity of the transmitted x-ray beam. The grayscale readings of the TIFF images of the XGT-2700 measurement were performed twice in order to ensure accuracy of reading and to estimate the reproducibility of the method. Each Gc and Gi was individually calculated to Gc/Gi and the first and second of Gc/Gi were averaged to Gxgt. Consequently, Gxgt was used as a mineral loss-parameter to represent a progression of mineral loss.

Statistical Analysis

The results of the pH and mineral loss measurements were statistically analyzed among intact dentin, active and arrested dentinal lesions by Kruskal-Wallis one-way analysis of variance (Siegel & Castellan, 1988) and Scheffe's multiple comparison test was used to locate the differences ($p<0.05$). A paired t-test was carried out to assess the agreement between the first and second readings of Gc/Gi. Correlation between the lowest pH value and Gxgt for each group was calculated by Pearson's correlation coefficients. The Gxgt values were plotted with the difference of the lowest pH values and linear regression lines were performed.

RESULTS

Twenty-two active and 83 arrested carious teeth were observed to assess both pH change and mineral loss

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Table 1: *Comparison of Active and Arrested Caries Using SCHEM-100 and XGT-2700*

		SCHEM-100	**XGT-2700**		
Teeth	Age	The Lowest pH	1st Gc/Gi	2nd Gc/Gi	Gxgt
Active carious lesion (n=22)	24.6[a] range 18-31	5.8 ± 0.2[b] range 5.3-6.1	4.85 ± 1.86[c]	5.05 ± 2.27[d]	4.92 ± 1.75[e]
Arrested carious lesion (n=83)	41.4[a] range 30-60	6.6 ± 0.2[b] range 6.2-6.9	2.37 ± 1.30[c]	2.28 ± 1.32[d]	2.19 ± 1.14[e]

Intact dentin showed a mean pH value of 7.0 (range 6.8-7.2) and a standard deviation of 0.1 (n=105).
Means in a column with the same superscript letters were significantly different (p<0.05).

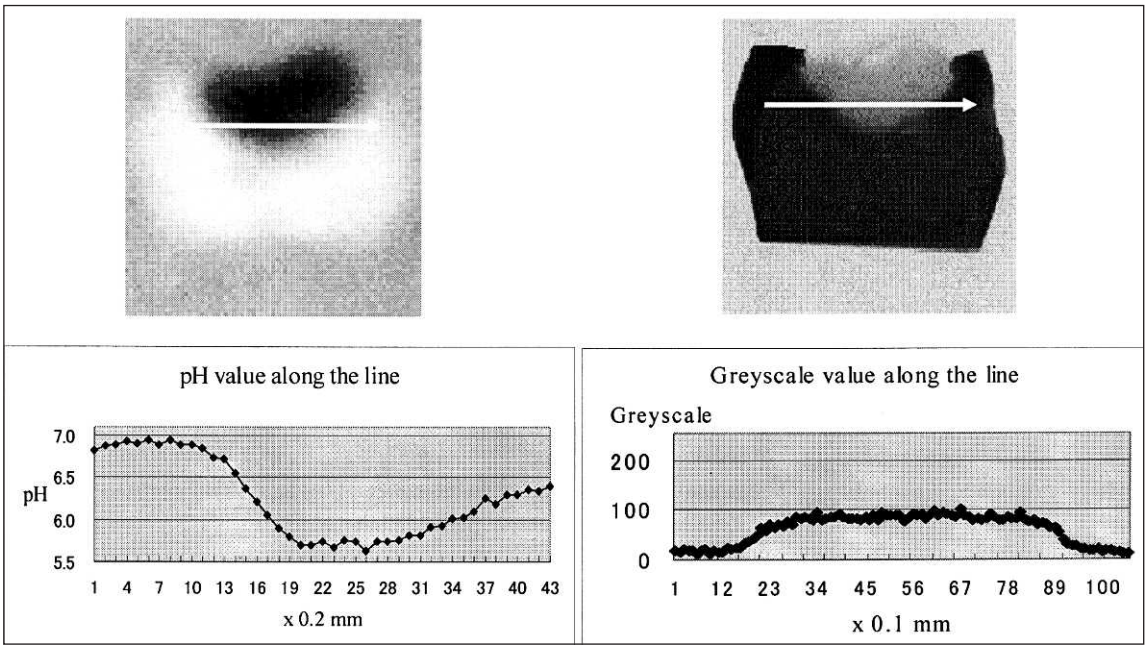

Figure 2: *Representative active carious lesion in TIFF images. Texts were enlarged for readability in the figure. The left TIFF image and graph were on pH value measurement with the SCHEM-100 and the right images were on mineral content measurement with the XGT-2700. A line was dragged to include the lowest and highest pH value points over the dentin area in the left image, then, the best-fit line was reflected in the right TIFF image. The dotted lines in the graphs showed the pH value on the left and the grayscale on the right along the reference lines of each TIFF image. The TIFF image with the SCHEM-100 appeared to distinguish affected lesions, while the TIFF image with the XGT-2700 showed that mineral content decreased accordingly.*

(Table 1). Intact dentin areas in active or arrested carious teeth showed a mean pH value of 7.0 (range 6.8-7.2). The lowest pH value of active carious dentin was 5.8 (range 5.3-6.1), while that of arrested carious dentin was 6.6 (range 6.2-6.9). The Kruskal-Wallis analysis test revealed a significant difference in pH values between intact dentin and active and arrested carious groups ($p<0.05$).

Gxgt represented the mineral loss per unit area. Active carious dentin showed significant mineral loss (Gxg=4.92) compared with arrested dentin (Gxgt=2.19) ($p<0.05$). A paired *t*-test showed no significant difference between the first and second readings of the grayscale, as Gc/Gi proved to be an appropriate reading reproducibility ($p=0.521$).

A representative image of an active carious lesion is shown in Figure 2. The TIFF images with SCHEM-100 indicated a continuous pH change in and adjacent to the carious lesion. The TIFF images with XGT-2700 showed a continuous progression of demineralization of the dentin in the active lesion. The dotted lines in the graphs represent the pH value and grayscale value of mineral content along the reference lines on the TIFF images. For the pH value, the curve declined sharply inward in the caries lesion, indicating that the decrease in pH value was significant.

A representative arrested carious lesion is shown in Figure 3. Although the change in pH value remained less than the active lesion, its distribution corresponded to the carious lesion. The grayscale image with XGT-2700 slightly distinguished the mineral loss.

Figure 4 shows the correlation with regression lines between the lowest pH value and mineral loss (Gxgt). Pearson's correlation coefficients indicated a strong negative correlation (r=-0.8024) in the active caries group but a weak negative correlation (r=-0.1480) in the arrested caries group.

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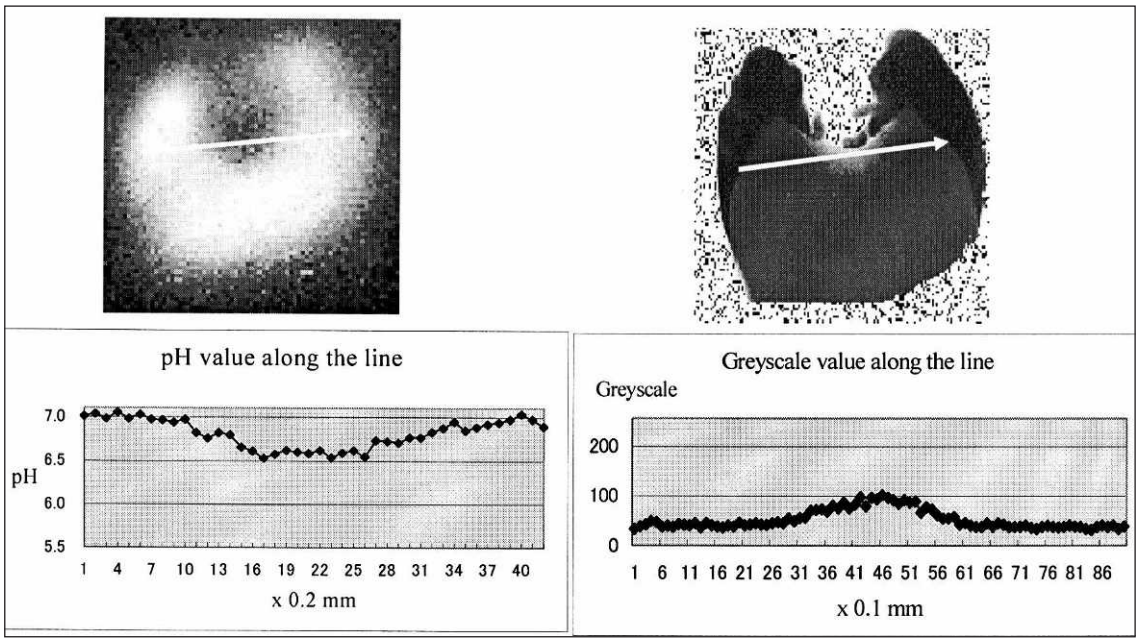

Figure 3: *Representative arrested carious lesion in TIFF images. Note that the instructions are the same as in Figure 2. The TIFF image with the SCHEM-100 revealed an obscure lesion and the TIFF image with the XGT-2700 showed a slight decrease in mineral content.*

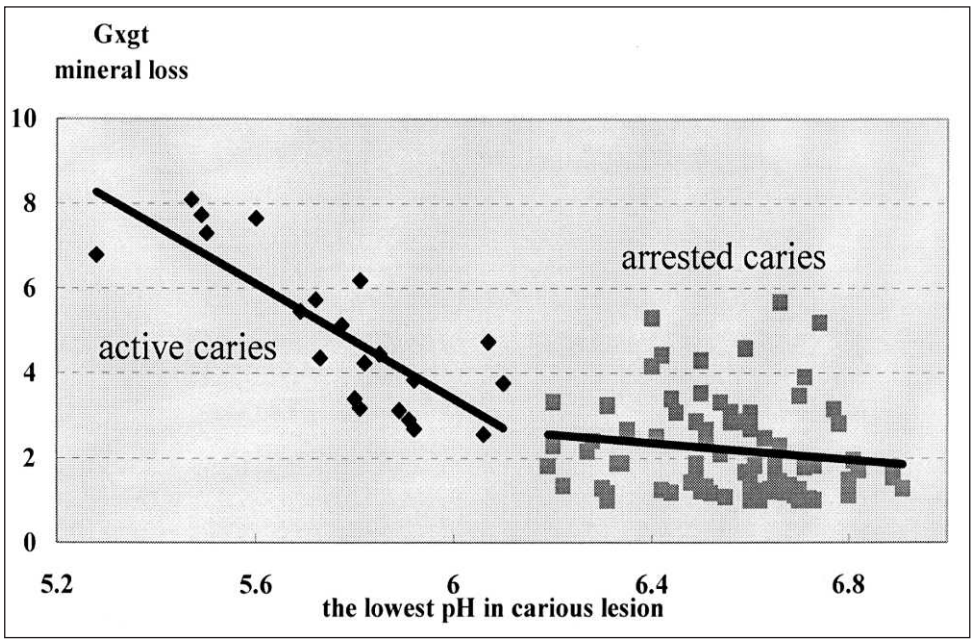

Figure 4: *Notes the correlation between the pH value measurement with the SCHEM-100 and the mineral loss measurement with the XGT-2700 on active and arrested caries. This figure shows a graph of the plotted data of active and arrested caries; Gxgt (Y-axis) vs the lowest pH (X-axis). Gxgt represents the mineral loss per unit area. For active caries, the calculated regression line is Y=-6.7891X+44.109. Note a strong negative correlation of r=-0.8024 and how the data points are scattered close around the calculated line. For arrested caries, the calculated regression line is Y=-0.9789X+8.6188. Note a weak negative correlation of r=-0.1480 and how the data points are scattered away from the calculated line.*

DISCUSSION

Conventional pH value measurement was conducted on saliva or plaque, indicating a close relationship with caries activity (Charlton, 1956; Stephan, 1944). Recently, the pH of carious dentin was examined using an electrode showing that it was lower than sound dentin, especially in active lesions (Hojo & others, 1994). When an electrode was used, samples of carious dentin were collected and suspended, and the pH of the suspension was measured. It had been difficult to evaluate distribution of the pH value on carious lesions until the SCHEM-100 was developed. Specimen preparation for the SCHEM-100 is simple and does not require destruction and dissolution of the specimen. The pH distribution of a flat dentin surface was measured by a quantitative monitoring technique when the sample was placed on an agar film. TIFF images were easy to manipulate in order to observe the pH value distribution over the area of interest on the dentin surface.

In a previous study using the SCHEM-100 for active and arrested carious lesions, a comparison was made between pH value distribution and stainable lesions by a caries detector solution (Kitasako & others, 2002). Although the caries detector solution failed to penetrate into arrested carious lesions, the SCHEM-100 was able to distinguish active and arrested carious lesions. The current study performed another comparison with a pH and mineral-loss examination on active and arrested carious lesions. Statistical analysis between the two methods of the SCHEM-100 and the XGT-2700

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was logical enough to compare the diagnostic accuracy because both were objective and quantitative analysis methods.

The XGT-2700 was used to quantify mineral content over carious lesions. In conventional radiography, x-ray projections are made through samples onto photographic film, where mineral quantification is determined due to the optical density of developed films. On the other hand, XGT-2700 requires no film and captures transmitted x-rays through samples by a scintillation detector so that objective point analysis can be carried out over a mapped image.

In a previous scanning electron microscopic study, it was observed that when carious bacteria reach dentin, acid production by the bacteria decalcifies the dentin (Symons, 1970; Ogushi & Fusayama, 1975). In this study, the TIFF images of XGT-2700, especially in the active lesion, displayed high grayscale in the carious lesion. This finding indicated that a carious lesion corresponded to high mineral loss and a high Gxgt value, which reflected progressive mineral loss of the carious affected lesion.

Correlation between the lowest pH and mineral loss in carious lesions differed in active and arrested lesions. Active lesions showed a strong negative correlation, while arrested lesions had a weak negative correlation. The low pH values of the active lesions indicated that the lesions might be rich in acid products from bacteria. Mineral loss occurs during the progression of dental caries, where the acid products from the bacteria decalcify the dentin (Symons, 1970; Ogushi & Fusayama, 1975). Therefore, when carious affected lesions were observed using XGT-2700, the grayscale value increased in active lesions compared to intact dentin. This finding corresponded to a high Gxgt value and a strong correlation between pH value and mineral loss in active caries.

In arrested lesions, caries activity seemed less influential on dentin. The absence of viable bacteria within the tubules and the limited bacterial activity are associated with arrested dentin lesions (Sarnat & Massler, 1965). Consequently, acid products from bacteria might be scant, which resulted in a higher pH value than in active lesions. Instead, the accumulation of minerals seems to be carried out in arrested lesions by three mechanisms: remineralization of the source of saliva, reprecipitation of dissolved apatite and sclerosis by intratubular calcification and obliteration of the tubules (Sarnat & Massler, 1965). The dentinal tubules are occluded due to the presence of sclerotic dentin and dead tracts (Miyauchi, Iwaku & Fusayama, 1978). Occasionally, a slight increase in mineral content or x-ray opacity appeared in carious dentin (Duke & Lindemuth, 1991; Fusayama, 1979). The increase in mineral content could be due to the progression of remineralization; as a result, a slight mineral loss and low Gxgt value were found. Therefore, a weak negative correlation coefficient was shown in arrested carious lesions in this study.

Previously, the outer and inner layers of carious dentin were outlined in order to establish a reliable clinical guide on removing carious dentin (Fusayama, 1979). The outer layer was bacteriologically infected, irreversibly denatured and hardly remineralizable, while the inner layer remains reversibly denatured (Fusayama, 1979; Ogushi & Fusayama, 1975; Kuboki, Ohgushi & Fusayama, 1977; Shafer, Hine & Levy, 1983). It was suggested that the outer layers should be removed to preserve the remineralizable inner layers (Fusayama, 1979). Therefore, during caries treatment, it is essential to recognize the boundary between the outer and inner layers. Since this study demonstrated the continuous reduction in pH value in and adjacent to the carious lesions, the pH examination seemed an appropriate alternative method for caries detection. The pH analyzing technique is utilized *in-vitro* only at this time because the pH-imaging sensor is not designed for intraoral usage. However, with technological development, it would be possible to use it as a clinical devise for carious diagnosis. If distribution of the pH value displays enough to specify the location of the two layers, it would serve as the standard to improve the restorative procedure and clinical practice. Further studies should focus on the relationship with the pH value and physiological characteristics in carious dentin that pertain to the outer and inner layers.

CONCLUSIONS

SCHEM-100 was suitable for pH-value measurement in order to distinguish carious or intact dentin. Especially in active carious dentin, this study demonstrated lower pH and considerable mineral loss, resulting in a significant correlation between a decrease in pH and mineral loss. When a guideline for identifying the outer and inner carious layers would be established, the pH analyzing technique might have the potential to serve as an alternative clinical diagnostic device for carious lesions.

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Effect of Home-Use Bleaching Gels on Fluoride Releasing Restorative Materials

ZC Cehreli • R Yazici • F García-Godoy

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Clinical Relevance

After treatment with bleaching gels, an increase in surface roughness for some materials was noted, while other gels displayed decreased surface roughness values, suggesting that the effects of the gels appear to be material-dependent.

SUMMARY

To evaluate the effects of two home-use bleaching gels (Nite-White and Contrast PM) on the surface roughness of one high-viscosity glass ionomer cement (Fuji IX GP), four polyacid-modified resin-based composites (Dyract AP, F2000, Élan and Compoglass F), two resin-modified glass-ionomer cements (Vitremer and Fuji II LC), a microfilled fluoride-releasing resin-based composite (Tetric) and a microfilled non-fluoridated resin-based composite (Valux) were used as controls. The null hypotheses tested were: (1) the use of bleaching gels would not result in surface roughness values different from those obtained at baseline; (2) differences in the amount of carbamide peroxide present in the gels would not influence surface roughness. Thirty specimen disks (the first 10 used for controls and the balance divided randomly into two groups) were made for each material according to the manufacturers' instructions. Finishing and polishing of the test and control specimens was performed after 24 hours using a sequential series of three (medium, fine and superfine) Sof-Lex disks, keeping the restoration surface wet. For every sequence, 10 strokes were made in one direction using a low-speed handpiece. Ultrasonic cleaning of the polished specimens was performed for two minutes in distilled water to remove any surface debris. Prior to bleaching treatment, the specimens were stored in 37°C deionized water for 24 hours. The specimens were exposed to the bleaching gels for a period that simulated the equivalent of 15 days (eight hours/day) under bleaching treatment. The average surface roughness (R_a) of the control and treated specimens was measured. Three separate R_a measurements along the direction of rotation of the finishing and perpendicular to the finishing direction and edge of the mold were made for each specimen surface. After treatment with bleaching gels, there was an increase in surface roughness for F2000, Dyract AP, Élan, Valux and Tetric, while Fuji IX, Fuji II LC, Vitremer and Compoglass F displayed decreased surface roughness values, suggesting that the effects of the gels appear to be material-dependent. Although Contrast PM contains 5% more carbamide peroxide as the

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active ingredient, the second null hypothesis was rejected for Fuji IX, Vitremer, Dyract AP, Compoglass F, Élan and Valux.

INTRODUCTION

Tooth bleaching is considered a safe treatment (US Department of Health, 1979; Haywood & Heymann, 1991; Howard, 1992; Woolverton, Haywood & Heymann, 1993; Curtis & others, 1996; Matis & others, 1998), and the treatment is rendered in many modalities with a variety of products. It has become a popular treatment to enhance the esthetics of both anterior and posterior teeth.

Some restorative materials used in high caries risk patients are the resin-modified glass-ionomer cements (RMGIC), high-viscosity glass-ionomer cements (HVGIC) and the polyacid-modified resin-based composites (PMRBC). Both RMGICs and PMRBCs are popular in some countries for many types of restorative procedures. Glass-ionomers, including RMGIC, HVGIC and PMRBC may have some type of response to some preventive treatments such as acidulated phosphate fluoride (APF) gels or foams that could alter their surface roughness, micromorphology and hardness (Neuman & García-Godoy, 1992; Triana & others, 1992; Cehreli, Yazici & García-Godoy, 2000; Abate & others, 2001; García-Godoy, García-Godoy & García-Godoy, 2002).

Although bleaching is safe to soft tissues from a procedural standpoint (Curtis & others, 1996), it may not be safe for dental materials with high erosive or degradation characteristics. This may be more significant in the case of home bleaching gels, where patients may not follow the professional recommendation but may alter it by often applying them more frequently in order to maintain a higher concentration of bleach on the tooth and a higher speed of action.

The effect of home bleaching agents on the surface roughness of HVGIC and PMRBC has not been reported. Therefore, this study evaluated the effect of two home-use bleaching gels (Nite White and Contrast PM) on the surface roughness of different restorative materials. The null hypotheses tested was twofold: 1) use of bleaching gels did not influence surface roughness of the test materials and 2) the amount of carbamide peroxide present in the bleaching gels did not bear an affect on surface roughness.

METHODS AND MATERIALS

The bleaching agents used in this study were a 10% Carbamide Peroxide gel (Nite White Excel 10%, Discus Dental, Los Angeles, CA, USA) and a 15% Carbamide Peroxide gel (Contrast PM 15%, Interdent Inc, Culver City, CA, USA) The restorative materials used in the study are presented in Table 1.

Plexiglass molds with disk-shaped specimen wells (10 mm x 2 mm) were used to make the specimens. A total of 30 specimens were made for each material, which were further sub-divided into three groups (n=10) serving as test (Nite White and Contrast PM) and control specimens. The HVGIC and RMGIC materials were applied using a syringe to minimize air entrapment. The molds were first slightly over-filled with the material under evaluation, covered with a 0.002" Mylar matrix strip (Union Broach Corporation, Division of Moyco Industries, Emigsville, PA, USA) and pressed flat with a microscopic glass slide.

All test materials except for Fuji IX GP were then light cured with a light-curing unit (Translux EC, Kulzer, Wehrheim, Germany) for a total of 60 seconds to ensure complete polymerization. The intensity of the light-curing unit was checked before each sample run, using a Demetron 100 curing radiometer (Demetron Research Corp, Danbury, CT, USA) and it was over 600 mW/cm^2. Following light curing with the tip positioned 1 mm away from the samples, the specimens were placed in 37°C deionized water for 24 hours.

For the Fuji IX GP samples, the injected material was left undisturbed for eight minutes. Following removal of the Mylar strip, the surface was protected using the manufacturer's recommended varnish (GC Fuji Varnish, GC Corporation, Tokyo, Japan). Specimens in this group were then kept in 37°C deionized water, the same as the other specimens.

Finishing and polishing of the test and control specimens was performed by one operator after 24 hours using a sequential series of three (medium, fine and superfine) Sof-Lex disks (3M Dental Products, St Paul, MN, USA), always keeping the restoration surface wet. For every sequence, 10 strokes were made using a low-speed handpiece in one direction.

Table 1: *Restorative Materials Used in the Study*

Restorative Material	Material Class	Manufacturer
Fuji IX	HVGIC	GC Corp, Tokyo, Japan
Fuji II LC	RMGIC	GC Corp, Tokyo, Japan
Vitremer	RMGIC	3M Dental Products, St Paul, MN, USA
F2000	PMRBC	3M Dental Products, St Paul, MN, USA
Dyract AP	PMRBC	Dentsply/DeTrey, Konstanz, Germany
Compoglass F	PMRBC	Vivadent, Schaan, Liechtenstein
Élan	PMRBC	Kerr UK Ltd, Peterborough, UK
Valux	MRBC	3M Dental Products, St Paul, MN, USA
Tetric	FMRBC	Vivadent, Schaan, Liechtenstein

HVGIC= High viscosity glass ionomer cement, RMGIC= Resin-modified glass ionomer cement, PMRBC= Polyacid-modified resin-based composite, RBC= Microfilled resin-based composite, FRBC= Fluoride-releasing microfilled resin-based composite

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Ultrasonic cleaning of the polished specimens was performed for two minutes in distilled water to remove any surface debris. Prior to bleaching gel treatments, the specimens were stored in 37°C deionized water for 24 hours.

Cotton applicators were used to bring the bleaching gels into contact with the test surfaces. The specimens were exposed to the gels for a period that simulated the equivalent of 15 days (eight hours/one day) under bleaching treatment. Each eight-hour application was followed by rinsing with deionized water for one minute and subsequent immersion in deionized water at 37°C for 60 minutes to prevent chemical equilibrium formation at the material surface. In the meantime,the controls were stored in 37°C deionized water.

Table 2: *Surface Roughness Values for the Different Groups*

Surface Roughness (Ra) in Micrometers*						
Material	**Baseline**	**SD**	**After Treatment with Nite White**	**SD**	**After Treatment with Contrast PM**	**SD**
Fuji IX	0.84[g]	0.35	0.62[a]	0.12	0.66[a]	0.18
Fuji II LC	0.81[g]	0.25	0.39	0.12	0.53	0.31
Vitremer	1.02	0.25	0.47b	0.11	0.53[b]	0.20
F2000	0.31	0.02	0.59	0.14	0.96	0.44
Dyract AP	0.14[h]	0.01	0.28[c]	0.09	0.36[c]	0.20
Compoglass F	0.24	0.10	0.18[d]	0.06	0.19[d]	0.08
Élan	0.15[h]	0.04	0.22[e]	0.08	0.30[e]	0.19
Valux	0.18[h]	0.03	0.29[f]	0.10	0.33[f]	0.19
Tetric	0.13[h]	0.02	0.17	0.06	0.30	0.11

For each property, identical lettering designates mean values with no statistically significant difference at p*<0.05.*

The average surface roughness (R_a) of the control and treated specimens was measured by one operator to minimize variation, using a Mitutoyo Surftest-402 Surface Roughness Tester (Mitutoyo Corporation, Tokyo, Japan). Three separate R_a measurements along the direction of rotation of the finishing and perpendicular to the finishing direction and edge of the mold were made for each specimen surface. The mean of the readings obtained from these three directions was recorded as the R_a value for each specimen.

Statistical analysis for the resulting R_a data was subjected to Wilcoxon Signed Ranks Test at a significance level of $p<0.05$ for comparison of the control versus the treated groups within each restorative material. Intermaterial comparisons were made using a Mann Whitney U Test, accepting a probability level of $p=0.05$ as significant.

RESULTS

In the control specimens, Fuji IX, Fuji II LC and Vitremer displayed the highest surface roughness values (Table 2) The difference between the R_a values for Fuji IX and Fuji II LC were not significant ($p>0.05$). Compared to the latter three materials, the R_a values for the other test materials were rather low and the difference between the surface roughness values for Dyract AP, Élan, Valux and Tetric were not significant ($p>0.05$).

The results showed that after treatment with bleaching gels, there was an increase in surface roughness for F2000, Dyract AP, Élan, Valux and Tetric, while Fuji IX, Fuji II LC, Vitremer and Compoglass F displayed decreased surface roughness values, suggesting that the effects of the gels appeared to be material-dependent (Table 2). Yet, the difference in surface roughness values between the control and test groups of all materials were statistically significant ($p<0.05$).

Although Contrast PM contains 5% more carbamide peroxide as the active ingredient, the second null hypothesis was rejected for Fuji IX, Vitremer, Dyract AP, Compoglass F, Élan and Valux, as differences in the amount of carbamide peroxide present in the gels influenced the surface roughness of some of the materials tested ($p>0.05$, Table 2).

DISCUSSION

Studies have shown bleaching to be clinically effective for whitening teeth (Gegauff & others, 1993; Small, 1994; Matis & others, 1998, Barnes & others, 1998; Heymann & others, 1998; Gerlach, Barker & Sagel, 2001), including those teeth that are stained (Haywood, Leonard & Dickinson, 1997).

Carbamide peroxide bleaching gels have been shown to produce mercury release from amalgam restorations (Hummert & others, 1993; Robertello & others, 1999) and may slightly roughen resin-based composites (Cooley & Burger, 1991; Bailey & Swift, 1992), although they may have no clinical significance. Interestingly, some studies have reported an increase (Cooley & Burger, 1991), a decrease (Bailey & Swift, 1992) or unchanged (Nathoo, Chmielewski & Kirkup, 1994) composite surface hardness after applying carbamide peroxide gels. This shows that the effect of carbamide peroxide gels may be composite material-dependent.

The effects of different bleaching agents at different concentrations have been evaluated on restorative materials (Cooley & Burger, 1991; Friend & others,

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1991; Bailey & Swift, 1992; Nathoo & others, 1994) and enamel (Lee & others, 1995), showing changes in surface hardness and roughness of the materials tested and in the color of the enamel.

Because glass-ionomer cements and compomers are used in many cases, the effect of some of these at-home bleaching agents on the surface roughness of the restorative materials tested was considered important. Bleaching gel treatment has increased the surface roughness of some test materials, which may contribute to the disintegration of the cement and to harboring cariogenic bacteria in the clinical situation (Neuman & García-Godoy, 1992). On the other hand, in the unbleached, polished condition, Vitremer, Fuji II LC and Fuji IX have generally displayed the highest R_a values of any of the other materials tested. This may also increase further chemical erosion of those materials in the oral cavity. When selecting such restorative materials, the choice of fluoride release and rechargability could be a more important factor. For instance, the recharging of such materials with APF gels will increase the disintegration of the cement, while also increasing their fluoride release (Crisp, Lewis & Wilson, 1980; Stannard & Viazis, 1988).

The mechanism of action of bleaching agents on tooth structures is apparently due to the oxidation of enamel and dentin molecules causing changes in color (Rotstein, Lehr & Gedalia, 1992; Rotstein & others, 1996). This oxidation reaction may also alter the surface pigments of restorative materials. Although the use of bleaching agents is widespread, there is no consensus as to the effects of bleaching agents on enamel or restorative materials.

This study evaluated the effects of two home-use bleaching gels (Nite White and Contrast PM) on the surface roughness of one high-viscosity glass-ionomer cement (Fuji IX GP), four polyacid-modified resin-based composites (Dyract AP, F2000, Élan and Compoglass F), two resin-modified glass-ionomer cements (Vitremer and Fuji II LC), a microfilled fluoride-releasing resin-based composite (Tetric) and a microfilled non-fluoridated resin-based composite (Valux).

After treatment with the bleaching gels, there was an increase in surface roughness for F2000, Dyract AP, Élan, Valux and Tetric, while Fuji IX, Fuji II LC, Vitremer and Compoglass F displayed decreased surface roughness values, suggesting that the effects of the gels appear to be material-dependent. The authors cannot explain why the glass ionomers tested revealed a decreased surface roughness after bleaching. Perhaps the bleaching agents have a greater effect in the composite resin matrix or composition than with the glass ionomers structural integrity (Cehreli & others, 2000).

Although Contrast PM contains 5% more carbamide peroxide as its active ingredient, the second null hypothesis was rejected for Fuji IX, Vitremer, Dyract AP, Compoglass F, Élan and Valux. Therefore, the effects of the different bleaching agents for the different restorative materials have to be tested in the market in order to determine their specific safety.

CONCLUSIONS

After treatment with bleaching gels, there was an increase in surface roughness for some materials (F2000, Dyract AP, Élan, Valux and Tetric), while others (Fuji IX, Fuji II LC, Vitremer and Compoglass F) displayed decreased surface roughness values, suggesting that effects of the gels appear to be material-dependent.

Although Contrast PM contained 5% more carbamide peroxide as the active ingredient, it had no effect on the surface roughness of Fuji IX, Vitremer, Dyract AP, Compoglass F, Élan and Valux.

Further *in vitro* studies, including profilometric and SEM analyses, should evaluate the effects of different bleaching agents on different types of restorative materials in order to draw more meaningful conclusions for the clinical situation.

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Adhesive Permeability Affects Composite Coupling to Dentin Treated with a Self-Etch Adhesive

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Clinical Relevance

The increase in adhesive permeability associated with bonding of a one-step self-etch adhesive to hydrated dentin allows time-dependent water movement through the polymerized adhesive to the adhesive-composite interface. The coupling of dual/chemical-cured composites to dentin is adversely affected by such an adhesive.

SUMMARY

Dentin bonded with one-step self-etch adhesives does not couple well to chemically-cured composites despite the presence of ternary catalysts in some systems. To determine whether additional factors are responsible for the poor coupling of dentin bonded with these adhesives, the authors used a one-step self-adhesive in which the ternary catalyst is incorporated separately in the brush applicator for activating the adhesive. The activated adhesive was bonded to dentin or prepolymerized composites. For microtensile bond strength evaluation, a dual-cured hybrid composite was used for coupling to the bonded substrates and activated using: (1) the light-cured mode (LC), (2) delayed light-activation (DL) and (3) the chemical-cured mode (CC). In addition, the composite polymerized in the CC mode was coupled to the two substrates bonded with (4) additional experimental adhesive versions without the ternary catalyst and (5) without the ternary catalyst and tertiary amine. Silver tracer penetration into bonded interfaces was examined using TEM by replacing the dual-cured composite with a light-cured or a chemically-cured microfilled composite. In the CC mode, the composites coupled poorly to both substrates bonded with the unactivated adhesive (5). The use of activated adhesives allowed for good coupling of the composites polymerized in all curing modes to bonded composite, but only in the LC mode to bonded dentin. Water-filled channels in the form of water trees and water blisters were present in the adhesive layer under all composite-curing modes in bonded dentin. These features were completely absent in the bonded composites, showing that the source of the water was from dentin. Water blisters were also observed along the adhesive-composite interfaces in bonded dentin when the coupling composites were polymerized using the CC or DL modes.

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INTRODUCTION

The incorporation of amphiphilic resin monomers as primer components has long been practiced in the

development of contemporary dentin adhesives. These monomers contain hydrophilic (-OH) and ionic (acidic; -COOH) functional groups for improving the bonding to hydrophilic substrates such as dentin, and methacrylate groups for coupling to the more hydrophobic resin composites (Eick & others, 1997). Priming monomers in total-etch adhesives improve the wetting of the demineralized collagen matrix and resin adaptation within patent dentinal tubules, creating the micromechanical retention that is necessary for optimizing adhesion to acid-etched, smear layer-depleted dentin (Attal, Asmussen & Degrange, 1994). For non-rinsing self-etch adhesives that are designed to bond to smear-layer covered dentin, higher concentrations of acidic resin monomers are often included in the presence of water to render these adhesives acidic enough to etch through the thick smear layer and bond to the underlying intact dentin (Tay & Pashley, 2001).

Simplified-step adhesives are becoming increasingly popular and user-friendly. Bonding procedures are being consolidated so that adhesive application becomes less time-consuming and technique sensitive. For total-etch adhesives, the three cardinal steps of etching, priming and bonding that are used in the conventional three-step systems are combined into single bottle adhesives that prime and bond simultaneously. Likewise, conventional two-step self-etch adhesives have been consolidated into one-step, all-in-one adhesives that etch, prime and bond simultaneously (Inoue & others, 2001). Unlike conventional systems that involve the use of intermediate light-activated resin layers to couple the primed substrates to resin composites, uncured ionic resin monomers in substrates bond using simplified-step adhesives are in direct contact with the coupling composite (Perdigão, 2002).

Although light-cured composites are most commonly used clinically, chemically-cured and dual-cured composites still have wide clinical applications in core build-ups and adhesive luting of indirect restorations and endodontic posts. Incompatibility between adhesives containing acidic resin monomers and chemically-cured composites was first reported for some two-step total-etch adhesives (Hagge & Lindemuth, 2001; O'Keefe & Powers, 2001; Swift & others, 2001). The decrease in microtensile bond strength of chemically-cured composites to dentin was directly proportional to the acidity of these adhesives (Sanares & others, 2001). It is known that acidic resin monomers retard the polymerization of chemical/dual-cured composites that are initiated via peroxide-amine type binary redox catalysts (Yamauchi, 1986). Interaction between acidic resin monomers from the oxygen inhibition layer of polymerized adhesives and the tertiary amines results in the consumption of the latter in acid-base reactions, depriving their capacity to generate free radicals in subsequent redox reactions (Nakamura, 1985). The tertiary amines used in light-cured composites are generally less nucleophilic than those utilized for chemically-cured composites due to the inclusion of electronegative, electron-withdrawing groups. Thus, incompatibility between acidic resin monomers and light-cured composites do not usually occur under the concentration in which these acidic monomers are utilized in self-etch adhesives. Different types of ternary redox catalysts, or co-initiators that are employed to overcome the incompatibility between acidic adhesives and chemical/dual-cured composites, have been reviewed by Ikemura and Endo (1999). One such commonly used ternary catalyst is the sodium salt of aryl sulphinic acid (Yamauchi, Yamada & Shibatani, 1990; Blackwell & Huang, 1987; Nyunt & Imai, 1996; Yamamoto & others, 1996). This reacts with acidic resin monomers to produce phenyl (Ikemura & Endo, 1999) or benzenesulphonyl free radicals (Wang, Chuang & Lee, 1999) that initiates the polymerization of chemically-cured composites. Many two-step total-etch adhesives are now supplemented with an additional bottle of activator solution containing the sodium salt of aryl sulphinic acid to enable them to bond to chemical/dual-cured composites.

As one-step self-etch adhesives contain higher concentrations of acidic resin monomers than two-step total-etch adhesives, they should be more susceptible to adverse acid-base chemical interactions in the absence of ternary catalysts. Some one-step self-etch adhesives available in the market already contain ternary catalysts as integral components of the adhesives to enhance their coupling with chemically-cured composites (AQ Bond [Sun Medical Co Ltd, Shiga, Japan], One-Up Bond F [Tokuyama Corp, Tokyo, Japan], Reactmer Bond [Shofu, Inc, Kyoto, Japan]). However, coupling chemical-cured composites to dentin bonded with these adhesives was still inferior to those achieved using light-cured composites (Tay & others, 2002a).

An alternative mechanism has been proposed to account for the compromised coupling that occurs between one-step self-etch adhesives and chemical/dual-cured composites. It has been shown that these adhesives permit the passage of fluid (Itthagarun & others, 2002) and behave as permeable membranes after polymerization (Tay & others, 2002a; 2003b). They also exhibited intricate patterns of water-filled channels within the adhesive layer that are referred to as water trees (Tay & Pashley, 2003). In the presence of an osmotic gradient, such as that produced by the oxygen inhibition layer of these adhesives, "osmotic blistering" (Pomersheim & Nguyen, 1998) occurs as water permeates via osmosis through the water-filled channels in the cured adhesive layer. The water that migrates to the composite-adhesive interface is trapped by the overlying hydrophobic composite as water blisters (Tay & others, 2001; 2002a). This results in mechanical disruption of the coupling between the adhesive and the

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composites. "Osmotic blistering" occurs in chemically-cured composites due to their slower rate of polymerization that allows sufficient time (two-to-three minutes) for water permeation through the cured adhesives.

The effect of adhesive permeability on compromised composite coupling to dentin is less frequently reported, as its involvement is often masked by a contribution from the accompanying adverse acid-base chemical interactions (Tay & others, 2003b). As it is difficult to draw any definitive conclusion when different commercially available adhesives that contain variable adhesive components are compared, the authors used an experimental one-step self-etch adhesive in which the tertiary amine accelerator and the ternary catalyst are both impregnated in the brush applicator supplied by the manufacturer for activating the adhesive. The authors created three different adhesive versions by eliminating either the ternary catalyst with specially prepared brush applicators, or both components with non-impregnated brush applicators. After confirming that effective coupling of chemical/dual-cured composites could be achieved with the original self-etch adhesive version, the objective was to examine the hypothesis that, in the absence of adverse acid-base chemical interactions, coupling of composites to the one-step adhesive is still compromised by adhesive permeability. The adhesive was bonded either to hydrated dentin, a hydrophilic substrate or processed composite, a hydrophobic substrate. Coupling of composites to these bonded substrates was further performed under different curing modes. The null hypothesis tested was that, in the absence of the contribution from adverse acid-base chemical interaction, bonding of the experimental one-step adhesive is not affected by the curing mode with which composite coupling is performed.

METHODS AND MATERIALS

Adhesive Versions

A one-step self-etch adhesive (Experimental code name RZII, Sun Medical Co—currently being marketed in North America as Brush&Bond by Parkell Inc, Farmingdale, NY, USA) was used throughout the study. The adhesive-base liquid is supplied in a single bottle and contains 4-methacryloxyethyltrimellitic anhydride, triacrylate, monomethacrylate, photoinitiator, stabilizer, acetone and water. The adhesive is activated using brush applicators supplied by the manufacturer. These brush applicators are impregnated with a proprietary amine as the photoaccelerator and the sodium salt of *p*-toulenesulphinic acid as the ternary catalyst. The base liquid was dispensed into a mixing well and stirred with the activator-impregnated brush. The activated adhesive was then applied to the bonding substrates for 20 seconds. Without rinsing, the adhesive was air-dried for three-to-five seconds. A second coat of adhesive was applied in the same manner, then light-activated for 10 seconds.

Table 1: *Microtensile Bond Strengths of the Coupling of a Dual-Cured Composite Under Different Activation Modes to Two Substrates Bonded with an Experimental Single-Step Adhesive*

Versions of the Experimental One-step Self-etch Adhesive	Group	Activation Mode of the Dual-cured Composite	Hydrated Dentin (MPa)*		Processed Composite (MPa)*	
Using manufacturer-supplied brush applicator 1 impregnated with amine and p-TSNa	I	Light-activation	42.3±6.3[A]	(24) [79.2%]	45.3±5.0[a]	(25) [76%]
	II	Delayed light-activation (10 minutes)	25.0±6.4[B]	(27) [100%]	41.3±5.5[a]	(25) [92%]
	III	Chemical-activation	22.5±5.7[B]	(25) [100%]	43.2±4.3[a]	(25) [80%]
Using manufacturer specially-prepared brush applicator 2 impregnated with amine only	IV	Chemical-activation	20.4±6.7[B]	(26) [100%]	44.9±7.5[a]	(25) [72%]
Using normal microbrush applicator without amine and p-TSNa	V	Chemical-activation	**0.3±0.9[C]	(25) [100%]	**5.5±6.6[b]	(25) [100%]

Abbreviation: p-TSNa = the sodium salt of p-toluenesulfinic acid, a ternary catalyst.
1 RZM-025 Brush; lot number 011023
2 RZII-FT Brush; lot number 020513
**Values are means ± standard deviation. For each substrate (each column), groups identified by different letter superscripts are significantly different ($p<0.05$). The numbers of specimens tested are included in parentheses. Percentages of adhesive failure are included in square brackets.*
***Six out of the 25 dentin-composite beams and 11 out of the 25 composite-composite beams exhibited premature failure during preparation for microtensile bond testing. They were assigned null bond strength values and were included in the calculation of mean bond strengths.*

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In addition to the original adhesive, two additional experimental adhesive versions were also produced by the manufacturer using the base liquid of the original version and different types of brush applicators. The first modified version consisted of activating the base liquid using a specially-prepared brush applicators supplied by the manufacturer, which were impregnated with the amine accelerator only (no ternary catalyst). The second modified version consisted of activating the base liquid with a normal microbrush (no amine accelerator and no ternary catalyst). These modified adhesive versions were also used in the same manner as the original unmodified version.

Bonding Substrates

The substrates to be bonded were divided into two categories: dentin and processed composite. Bonding to dentin was performed on the occlusal surfaces of deep coronal dentin from extracted, noncarious human third molars. They were stored in a 1% chloramine T solution at 4°C and used within one month following extraction. The occlusal enamel of each tooth was removed using a slow-speed saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water-cooling. The tooth surfaces were polished with 180-grit silicon carbide (SiC) papers to create bonding surfaces with clinically-relevant, thick smear layers (Koibuchi, Yasuda & Nakabayashi, 2001). The teeth were bonded in their normal hydrated status as they were retrieved from the storage medium.

For bonding to processed composites, a heat- and light-activated hybrid resin composite (Tescera, BISCO Inc, Schaumburg, IL, USA) was used to produce void-free, composite blocks. Layers of composite 5-mm thick were dispensed into 2x2 cm flat Teflon molds (Electron Microscopy Sciences, Fort Washington, PA, USA). The molds containing the uncured composite were placed inside a composite inlay processing chamber (Nitro-Therma-Lite, BISCO Inc) and light-activated under pressurized nitrogen maintained at 551.6 kPa (80 psi) for one complete cycle at 125°C for 10 minutes. The bonding surface of each block was ground flat with 180-grit SiC paper. The adhesives were applied to the processed composites in exactly the same way as they were used on hydrated dentin.

Composite Curing Modes

For microtensile bond strength evaluation, a dual-cured, hybrid resin composite, Bis-Core (BISCO Inc), was used for coupling to the substrates that were bonded using the original adhesive version. Coupling of the composite to the bonded dentin and the bonded processed composite was performed under three different modes of activation (Table 1):

1. Light-Cured Mode

Only the light-cured base syringe of the composite was used. The composite was hand-mixed for 20 seconds under ambient light in the same manner as when both the base and catalyst components of the dual-cured composite were mixed. The rationale was to introduce the same amount of air when the composite was used in both the light-cured and the chemically-cured modes. The composite was applied in five 1-mm increments, with each increment light-activated for 40 seconds at a curing intensity of 600 $mWcm^{-2}$ immediately upon placement.

2. Delayed Light-Activation

The rationale for delayed light-activation was to simulate the slow rate of polymerization of a chemically-cured composite, but avoiding the adverse chemical interaction between acidic resin monomers in the oxygen inhibition layer of the adhesive and the basic tertiary amine accelerator in a chemically-cured composite (Tay & others, 2001). The first 1-mm increment of the hand-mixed composite from the base syringe was applied over the cured adhesive and left in complete darkness for 10 minutes before light-activation. After polymerization, the rest of the increments were applied and light-activated immediately and separately.

3. Chemically-Cured Mode

Composite from the base and catalyst syringes were hand-mixed for 20 seconds in the manner previously described. The mixed composite was applied in bulk to the bonded adhesive surfaces and polymerized under complete darkness for 20 minutes before re-exposure to ambient light.

In addition to the three groups mentioned above, the composite was also coupled in the chemically-cured mode to substrates bonded with the two modified adhesive versions (Table 1):

4. Chemically-Cured Mode—Adhesive version without ternary catalyst, and

5. Chemically-Cured Mode—Unactivated adhesive version without proprietary amine and ternary catalyst.

Microtensile Bond Strength Evaluation

Twenty teeth and five processed composite blocks were used for bond strength evaluation. After storage in distilled water at 37°C for 24 hours, the bonded teeth were sectioned occluso-gingivally into 0.9-mm thick serial slabs using the Isomet saw under water-cooling. The bonded composite blocks were similarly sectioned into 0.9-mm thick slabs. The slabs were further sectioned into 0.9 x 0.9 mm composite-dentin beams according to the technique for the "non-trimming" version of the microtensile test reported by Pashley and others (1999). After examination under a stereomicroscope at 20x, beams containing peripheral enamel or imperfections at the bonded interface or composite were discarded, yielding 24-27 beams per group. Each beam was stressed to failure under tension using a universal testing machine (Model 4440, Instron Inc, Canton, MA,

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Figure 1: *Coupling of a light-cured composite (LC) using the immediate light-activation mode to different substrates bonded with the manufacturer-supplied, original one-step self-etch adhesive (Group 1). A and B. Dentin; C. Composite.*

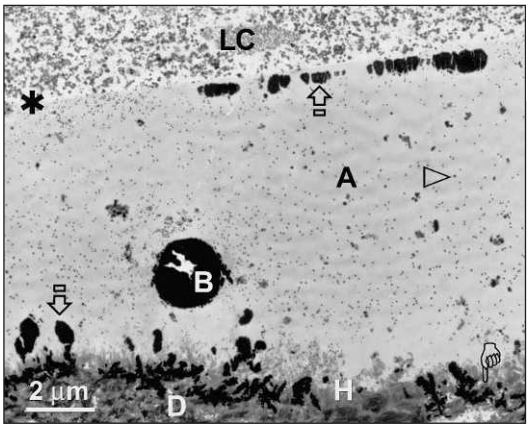

Figure 1A. *An overall view of the silver-impregnated resin-dentin interface. Nanoleakage of silver (pointer) could be seen in the hybrid layer (H). Permeability of the adhesive layer (A) was manifested by the presence of water trees (open arrows), silver-impregnated spherical water blisters (B) and isolated silver grains (open arrowhead) after immersion in ammoniacal silver nitrate. However, the adhesive-composite interface was intact (asterisk) and without silver deposits when the composite was light-activated immediately. D: dentin.*

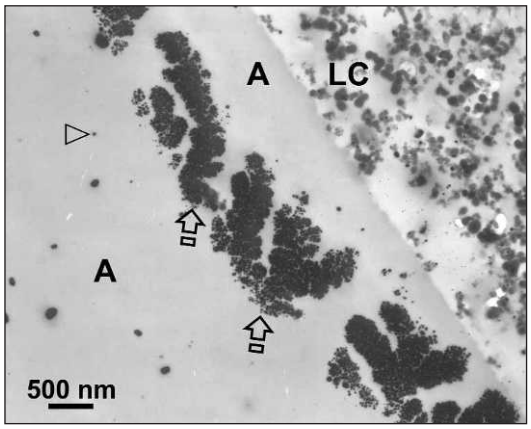

Figure 1B. *A high magnification view of the intact adhesive (A)-composite (LC) interface that was devoid of silver deposits, despite the presence of numerous water trees (open arrows) beneath this interface. Isolated silver grains (open arrowhead) could also be identified within the adhesive layer.*

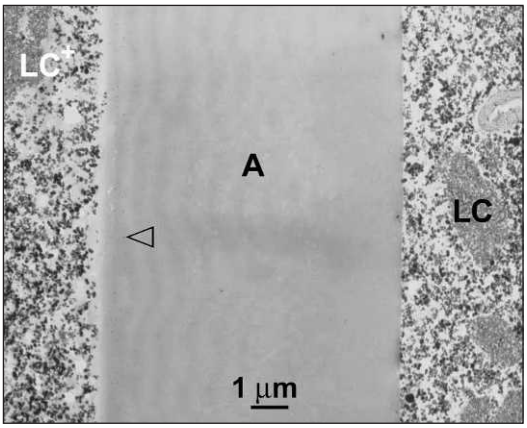

Figure 1C. *In contrast with bonded dentin, the adhesive layer (A) in the bonded composite (LC) was almost completely devoid of silver deposits when it was coupled to a light-cured composite (LC+). Some isolated silver grains (open arrowhead) could be found along the adhesive-composite interface.*

USA) at a crosshead speed of 1 mm/minute. Failure modes were recorded as adhesive, mixed or cohesive failures in either dentin or resin. For each type of substrate, bond strength data from the five experimental groups were statistically analyzed using the Kruskal-Wallis one-way analysis of variance on ranks test followed by Dunn's multiple comparison tests. Statistical significance was set in advance at α=0.05.

Ultrastructural Examination of Silver Tracer Penetration

Ten additional human third molars and five composite blocks were used for transmission electron microscopy (TEM). They were bonded with activated and unactivated adhesives in the manner previously described. To avoid damage to the diamond knife by glass fillers, the dual-cured composite was replaced by a light-cured composite and an experimental self-cured composite. A commercially available light-cured, microfilled composite with pre-polymerized fillers (Metafil CX, Sun Medical Co Ltd) was used to facilitate ultramicrotomy. The experimental chemically-cured composite (Sun Medical Co Ltd) has the same resin and filler composition as the light-cured composite. These composites were mixed and overlaid on the bonded dentin as before.

After storage in distilled water at 37°C for 24 hours, specimens from the 10 subgroups were sectioned into 0.9 mm-serial slabs. The widest slab from each tooth was coated with nail varnish applied 1 mm away from the bonded interface. One slab was also selected from each bonded composite block. They were immersed in a 50 wt% ammoniacal silver nitrate solution (pH=9.5) for 24 hours according to the diamine silver impregnation protocol reported by Tay and others (2002c). The ammonium ions in this basic tracer solution were complexed to the silver ions in the form of diamine silver (I) ions ($[Ag(NH_3)_2]^+$). The use of this tracer solution allowed the authors to identify, apart from nanoleakage within hybrid layers (Sano & others, 1995; Pioch & others, 2001), water-filled channels (water trees) within adhesive layers (Tay & Pashley, 2003) and regions that were thought to represent bound water that are affiliated with hydrophilic resin domains within the resin-dentin interfaces (Tay, Pashley & Yoshiyama, 2002). After reduction of the diamine silver ions, the silver-impregnated slabs were processed according to the TEM protocol reported by Tay, Pashley and Yoshiyama (2002). Undemineralized, epoxy resin-embedded, 90-120-nm thick sections were examined unstained using a TEM (CM100, Philips, Eindhoven, The Netherlands) operated at 80 kV.

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Figure 2: *Coupling of a light-cured composite (LC) using the delayed light-activation mode to different substrates bonded with the manufacturer-supplied, original one-step self-etch adhesive (Group 2). A. Dentin; B. Composite.*

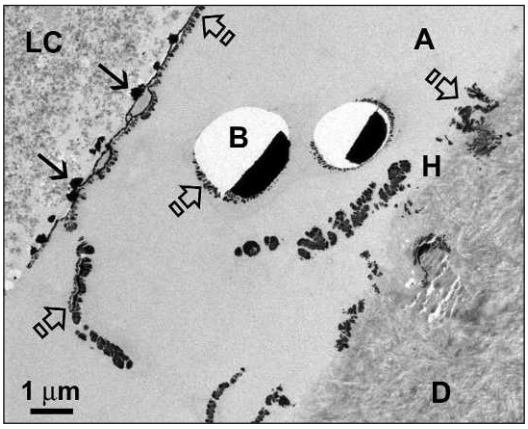

Figure 2A. *Apart from the features previously observed, the adhesive-composite interface was characterized by the presence of water blisters. Both fine (arrow) and large water blisters (B) were also found along the adhesive-composite interface. The large blisters were formed by the coalescence of the water blisters (pointers) residing within the adhesive (A) that were located above the dentinal tubules (T). Open arrows: water trees; H: hybrid layer; D: dentin.*

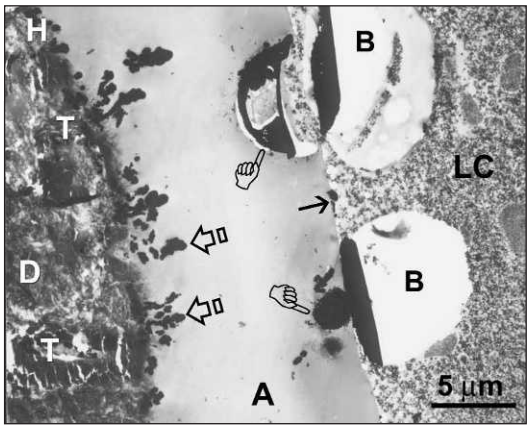

Figure 2B. *In contrast with bonded dentin, the adhesive layer (A) in the bonded composite (LC) did not exhibit any notable silver deposits after delayed light-activation of the composite (LC+) that was placed over the cured adhesive. The adhesive-composite interface (pointer) was intact and completely devoid of water blisters.*

RESULTS

Microtensile bond strengths of composites coupled under different curing modes to substrates bonded with the three adhesive versions are shown in Table 1. When the original adhesive version was used, bond strengths to hydrated dentin were significantly higher ($p<0.05$) when the composites were light-activated immediately (Group 1), compared with those activated using delayed light-activation (Group 2) or the chemical-activation mode (Group 3). No significant difference could be detected among these three groups when processed composite was used as the bonding substrate ($p>0.05$).

When chemically-activated composites were coupled to both types of substrates, there was no difference between adhesive versions with (Group 3) or without (Group 4) the sodium salt of *p*-toluenesulfinic acid as the ternary catalyst. Conversely, bond strengths of both substrates were significantly lower ($p<0.05$) in the unactivated adhesive version in which both the tertiary amine photoaccelerator and the ternary catalyst were excluded (Group 5).

When the light-cured composite was activated immediately upon placement on bonded hydrated dentin (Group 1), three patterns of silver deposition were identified within the adhesive layer (Figure 1A), which were also observed universally in the other four groups. They consisted of a) Isolated silver grains randomly distributed within the entire adhesive layer, b) Interconnecting silver-filled water channels (water trees) that were predominantly located along the surface of the hybrid layer and a zone beneath the composite that probably represented the original oxygen inhibition layer of the adhesive and c) spherical silver-filled water blisters that were sometimes surrounded by an additional peripheral corona of water trees. Despite the extensive silver deposition within the adhesive layer, adhesive-composite interfaces were all intact and completely devoid of silver penetration (Figure 1B).

In addition to the three silver deposition patterns, resin-dentin interfaces coupled with the light-cured composite using the delayed light-activation mode were characterized by the presence of blisters along the adhesive-composite interface. Fine blisters were associated with water trees in the adhesive layer (Appendix), whereas, large blisters were formed by the coalescence of the water blisters within the adhesive (Figure 2A).

Blisters were also found along the adhesive-composite interface when the chemically-cured composite was coupled to hydrated dentin bonded with the original version of the experimental adhesive (Figure 3A). A higher magnification view depicted the communication that existed between the water trees within the adhesive and the fine water blisters within the composite (Figure 3B).

The extent of interfacial silver tracer penetration was strikingly different between bonded dentin and bonded composite when the original adhesive version was used for bonding. Similar features were observed in Groups 1, 2 and 3. The adhesive layer could be devoid of silver deposits (Figure 2B) or isolated silver grains (Figure 1C) or water trees (Figure 3C) that were predominantly found beneath the coupled composite layer. No water blisters were observed within the adhesive or along the adhesive-composite interface.

When the adhesive version that is devoid of the ternary catalyst was used (Group 4), the ultrastructure of bonded dentin and composite interfaces were similar to those observed in Groups 2 and 3, with the exception that water trees were present throughout the entire adhesive layer (not shown). Conversely, when both the ternary catalyst and the amine were absent from the adhesive (Group 5), the chemically activated composite

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Figure 3: *Coupling of a chemical-cured composite (CC) to different substrates bonded with the manufacturer-supplied original one-step self-etch adhesive (Group 3). A and B. Dentin; C. Composite.*

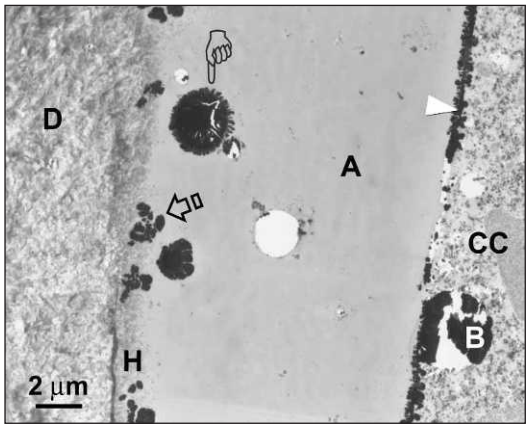

Figure 3A. *Similar to Group II, large water blisters (B) were seen along the adhesive-composite interface. At this magnification, a discontinuous line of silver deposits could also be identified along this interface (solid white arrowhead). Pointer: silver-filled water blisters within the adhesive; A: adhesive layer; Open arrow: water tree; H: hybrid layer; D; dentin.*

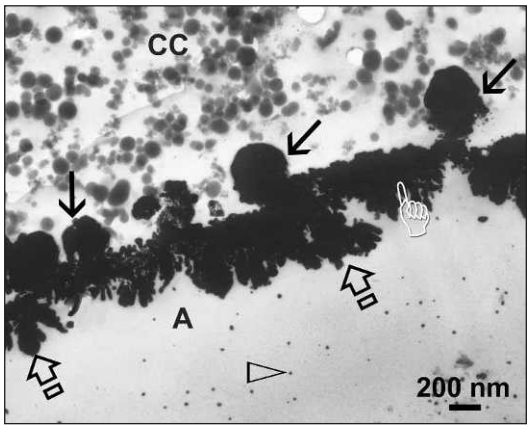

Figure 3B. *At a high magnification, the line of silver deposits (pointer) was formed by the direct communication between water trees (open arrows) in the adhesive (A) and fine water blisters within the chemically-activated composite.*

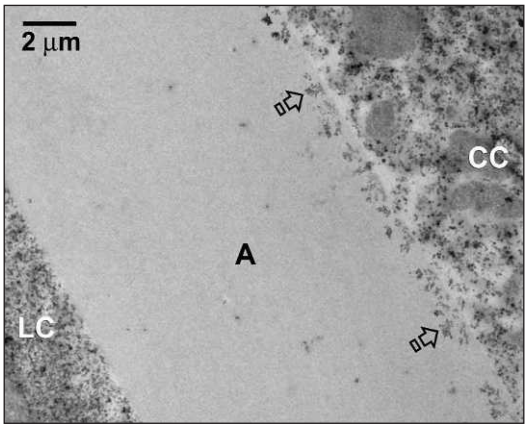

Figure 3C. *When the chemically-activated composite (CC) was coupled to the bonded light-cured composite (LC), very small water trees (open arrows) were seen beneath the chemically-activated composite. However, the adhesive-composite interface was intact and was completely devoid of water blisters.*

coupled poorly to the bonded dentin (Figure 4A) and the bonded composite (Figure 4B). Thick, diffuse patches of silver were present along the adhesive-composite interfaces, which also extended into the composite. Apart from the presence of water trees and water blisters (not shown), the entire adhesive layer was full of silver clusters that consisted of a rosette of silver grains that had formed around the isolated silver grains observed in the other four groups (Figure 4C).

DISCUSSION

There are three clues to indicate that, in this particular one-step self-etch adhesive, compromised coupling of chemically-cured composites cannot be attributed to adverse acid-base chemical interaction between acidic monomers and tertiary amines. First, the compromised bonding results were substrate dependent, affecting only hydrated dentin but not the processed composites. Second, a similar decline in bond strength was also observed with delayed light-activation of the composites. The light-cured composites were activated in a delayed light-activation mode to simulate the slow rate of polymerization of chemically-cured composites. Tertiary amines employed in light-cured composites are generally less nucleophilic than those utilized for chemically-cured composites. Thus, incompatibility between light-cured composites and acidic adhesive monomers do not usually occur under the concentration in which they are present in self-etch adhesives. Finally, coupling of the chemically-cured composites in bonded dentin and bonded processed composite was not further compromised by the absence of the adhesive ternary catalyst. This is rather unusual for one-step self-etch adhesives, as they are more acidic than total-etch, single-bottle adhesives (Tay & others, 2002a). The authors speculate that an excess of tertiary amine may be present in the brush applicator of the original adhesive which prevents their total deactivation by the adhesive acidic monomers.

As the microtensile strengths of bonded dentin are still affected by curing modes of the coupling composites in the absence of adverse chemical interactions (in the delayed light-activation group), the authors have to reject the null hypothesis and assert that adhesive permeability adversely affects composite coupling to the one-step self-etch adhesive in a time-dependent manner. When composite coupling was performed on bonded dentin using the chemical-activation and delayed light-activation modes, blisters were present along adhesive-composite interfaces that act as stress raisers when the specimens were challenged under tension. This resulted in the low bond strengths and exclusive adhesive failures. The ultrastructural features of inter-

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Figure 4: *Coupling of a chemical-cured composite (CC) to different substrates bonded with the modified one-step self-etch adhesive version that is devoid of the amine and ternary catalyst (Group 4). A. Dentin; B and C. Composite.*

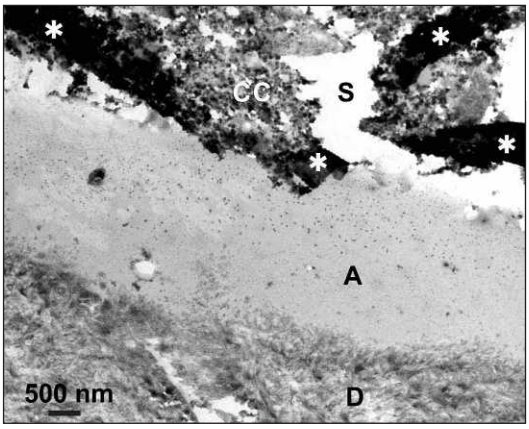

Figure 4A. *The adhesive (A) was sufficiently polymerized to support the overlaying of the chemically activated composite (CC). However, the poorly coupled composite (CC) was partially torn during ultramicrotomy, leaving behind spaces (S) among the severed composite segments. Diffuse patches of silver (asterisks) could be identified along the adhesive-composite interface. D: dentin.*

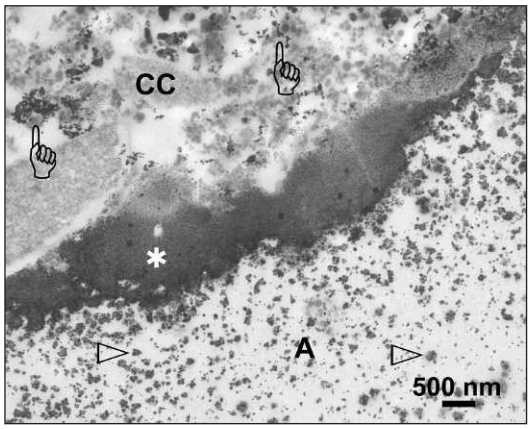

Figure 4B. *The chemically-cured composite (CC) was also poorly coupled to the bonded light-cured composite (not shown). Thick, diffuse silver patches (asterisk) were recognized throughout the adhesive-composite interface, with silver deposits (pointers) also located within the chemically-activated composite. Within the adhesive layer (A), an extensive collection of segregated silver clusters (arrowheads) could be identified.*

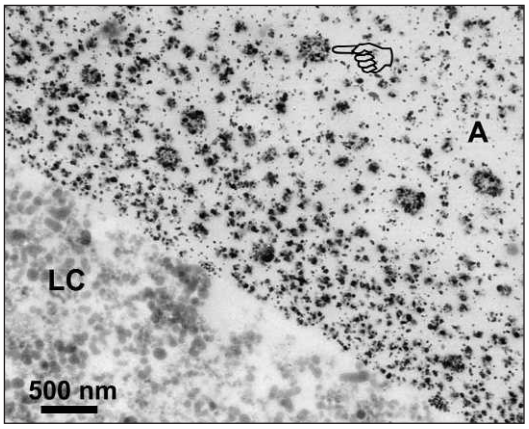

Figure 4C. *At a higher magnification, each of these clusters consisted of a rosette of silver grains that was condensed along the periphery of a central silver grain (pointer).*

facial blisters in these non-stressed specimens corresponded with the scanning electron microscopical appearance of fractured blisters, in the form of a honeycomb appearance, from specimens that were subjected to microtensile bond testing (Tay & others, 2001). The interfacial blisters in this study were smaller in size as the time-lag for activation of the light-cured composites was reduced from 20 minutes (Tay & others, 2001, 2002a) to 10 minutes. This, however, enabled the authors to preserve the integrity of these interfacial blisters without delaminating the adhesive-composite interface during laboratory specimen preparation. It is interesting to observe that interfacial blisters were completely absent when the light-cured composites were activated immediately upon placement, despite the morphologic evidence of increased permeability that is represented by the different forms of silver tracer deposition within the cured adhesives. This suggested that adhesive permeability and secondary interfacial blister formation are inter-related issues. However, the former must pre-exist within the adhesive before the latter can occur during the time-lag in composite polymerization. These two issues need to be discussed separately.

Although permeability of dentin adhesives is not a well-understood phenomenon, permeability of polymers to water and electrolytes has been investigated extensively in film coatings with important industrial applications (Banker, Gore & Swarbrick, 1965; Fales, Springer & Vanderborgh, 1986). The concentration of water that can be taken up by these polymers depends on the type of polar or ionic function groups that are present in the polymer, and the chain length (the number of methylene groups) to which these functional moieties are attached to the monomer molecules. In the study of diffusion of electrolytes in hydrated polymer systems, Zaikov, Iordanskii and Markin (1988) noted that the distinction between hydrophobic and hydrophilic systems is based on their equilibrium water content at ambient temperature and 100% relative humidity (>10 wt% for hydrophilic polymers). Polymers produced at low relative humidity from neat hydrophilic resins exist in a dry, glassy state. However, a considerable amount of water may be trapped in various forms when polymer films are produced by solvent (Abdel-Aziz & Anderson, 1977) or latex casting (Steward, Hearn & Wilkinson, 1995a). Although porosities do exist in dry polymers (pore size *ca* 1-15 Å) due to the presence of a free-volume that accounts for the segmental mobility of the polymer chains (Kanaya & others, 1999; Schmidt & Maurer, 2000), hydrated polymers are far more porous by virtue of their increased water content. Thus, hydrated polymers differ in water sorption and diffusional characteristics from dry hydrophilic polymers that are polymerized from neat resins. One of the most well known examples is the variation in pore size between pure, glassy poly(2-hydroxyethyl methacry-

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late) and hydrogels of poly(2-hydroxyethyl methacrylate) that are polymerized in the presence of different concentrations of water (Chirila & others, 1993).

The difference in diffusional characteristics between dry and hydrated hydrophilic polymers can be readily appreciated when the acetone-based, one-step self-etch adhesive is applied to the bonded composite or the hydrated dentin. Although both types of specimens are bonded at ambient relative humidity (around 70%), the hydration status of polymerized adhesive depends on whether water can be effectively removed from the solvented adhesive. The adhesive was applied to the two substrates in the same manner and air dried to the same extent before light-activation. In the bonded composite, water that is included in the adhesive to effectuate demineralization of dental hard tissues can be effectively removed by the acetone solvent, leaving only trace amounts within the oxygen inhibition layer of the adhesive. In this situation, there is no additional source of water. Conversely, when the adhesive is applied to dentin, water is readily available as the self-etch adhesive etches through the smear layer and smear plugs into the underlying hydrated intact dentin. This allows water to enter the hydrophilic polymer during its polymerization. Such a difference was manifested ultrastructually by the different extent of silver deposition in the adhesive layer of the two types of bonded substrates.

Water in hydrophilic polymers exits as a spectrum of intermediate states, from strongly bound to free, each of which is characterized by its specific energy level and mobility (Zaikov & others, 1988). Three states can be readily distinguished in nuclear magnetic resonance studies: 1) the primary state, consisting of bound, strongly ordered water molecules that form the first hydration sphere of a polar functional group via hydrogen bonding; 2) the secondary state, consisting of less ordered hydrated water associates that are still influenced by the functional group but that are structurally different from that of the initial hydration sphere by the presence of a large number of broken hydrogen bond and 3) the tertiary state, consisting of unbound water that is structurally identical to that of unperturbed bulk water (Hamilton & others, 1988). The existence of bound water in hydrophilic polymers accounts for the formation of hydrogels, while the presence of free water in the form of continuous water-filled channels is common in highly swollen, weakly cross-linked hydrophilic polymers. Depending on whether water is initially present, and the state of water that exists, permeability of electrolytes through polymer membranes changes from a predominantly Fickian diffusion type mechanism to a mechanism of predominantly convective transport through the water-filled channels (Zaikov & others, 1988; Steward, Hearn & Wilkinson, 1995a, b, c).

The authors speculate that both the water trees (Tay & others, 2002; Tay & Pashley, 2003) and the water blisters observed in this study are morphologic manifestations of unbound water within the polymerized adhesive. Water treeing, in its original context, is a water-induced deterioration phenomenon that occurs in polymer insulation of electrical cables after aging. These microscopic tree-like channels are formed by the oxidation of hydrophobic polymers into more hydrophilic moieties, with moisture condensation along the self-propagating tracks that eventually lead to cable failure (Dissado & Fothergill, 1992). It is possible that the water trees observed in polymerized dentin adhesives were formed by a similar mechanism of moisture condensation along tracks molded by the hydrophilic resin domains of the resin matrix. The occurrence of water blisters within the adhesive layer is unique to this experimental adhesive and has not been observed before in other commercially available one-step self-etch adhesives (Tay & Pashley, 2003). Unlike water trees that are confined within the resin matrix, water blisters, being spherical in configuration, may represent distinct aqueous phases that separate out of the adhesive. Due to the presence of water soluble monomers and/or electrolytes in the adhesive, some of the free water may be drawn osmotically out of these blisters and back into the resin matrix, resulting in the corona of short water trees that were occasionally seen around the interphasic boundary of the water blisters (Figure 3A). Irrespective of their mechanisms of formation, both the water trees and water blisters observed in this study are possible channels for rapid water transport that may occur through the adhesive layer.

The authors have also previously reported of the existence of isolated silver grains in polymerized dentin adhesives after immersion in ammoniacal silver nitrate solution (Tay & others, 2003a). They may represent polar and ionic functional groups of the hydrophilic resin monomers to which water molecules are structurally bound as first hydration spheres. The additional rosette of silver grains, which were clustered around a single silver grain in the adhesive layer of Group 5 (Figure 4C), may further represent the secondary hydrated water associates. The very low bond strengths observed for both types of bonded substrates in this group suggested that polymerization of the adhesive is incomplete in the absence of the tertiary amine. Light-activated polymerization proceeds via the generation of free radicals from the activation of a photosensitizer such as camphorquinone, with the free radicals transferred from the activated photosensitizer to the accelerator on dissociation (Monroe, Weiner & Hammond, 1968). Chain propagation proceeds very slowly in the absence of the amine accelerator, resulting in incomplete polymerization of the adhesive. Insufficient cross-linking of the polymer or subsequent relaxation of the

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incomplete polymerized matrices may have produced increased mobility of the polymer chains (Zaikov & others, 1988). This probably permitted a more rapid rate of water sorption, with the formation of secondary hydrated water associates around the original primary hydration spheres. Similar silver clusters were also observed in well-polymerized dentin adhesives after six months of aging in water (Tay & others, 2003a).

Detection of silver tracer within the adhesive layer only provides evidence for its permeability potential. This does not, however, explain why interfacial blisters were only present when free-flowing composites were subjected to a time-tag before gelation of the resin matrices (chemical and delayed light-activation). Although diffusional water transport via hydration spheres affiliated with the polar and ionogenic function groups may be important in hydrolytic degradation of the adhesive (Sano & others, 1999), it is probably too slow to account for the rapid development of the interfacial blisters that occur during the polymerization of a chemical-cured composite. Moreover, the interfacial blisters were shown to be directly associated with the water trees and water blisters located within the adhesive layer (Appendix). This indicates that rapid water transport is a more likely mechanism for the formation of these interfacial blisters. The authors further speculate that the motive force is derived from osmosis (Zentner, Rork & Himmelstein, 1985), and that the manifestation of interfacial blisters corresponds to a phenomenon in the resin paint-coating industry known as "osmotic blistering."

Blister initiation and growth via osmosis is common in resin-coated metal systems, as they are one of the first signs for corrosion that eventually causes delamination of the coating (van der Meer-Lerk & Heertjes, 1975). In conventional osmotic blistering, interfacial contamination in the form of trapped ions between the metal and resin surface results in the coating acting as a semi-permeable membrane, allowing water transport from the environment into the interface. Using mathematical models to predict blister growth in resin-coating systems, Pomersheim and Nguyen (1998) showed that changes in blister radii, surface areas, heights, volumes and osmotic pressures all varied directly as a fractional power of time. This corresponded with the observations of this study in that interfacial blisters were absent when light-cured composites were immediately light-activated, but they were present after delayed light-activation. The time-dependent manner in which osmotic blistering occurs also accounts for the exponential decrease in microtensile bond strengths of some one-step self-etching systems bonded to hydrated dentin, with increasing time-lag periods in which the overlaying composites were light-activated (Tay & others, 2001).

In the context of dentin bonding, differential water movement across the cured adhesive layer may occur in the presence of increased concentrations of dissolved electrolytes, such as calcium and phosphorus ions derived from the self-etching process, uncured, water-soluble hydrophilic resin monomers, or dissolved collagen/proteoglycans components that are concentrated along the oxygen inhibition layer of the cured adhesive. This concentration difference may establish an osmotic pressure gradient, causing water movement from a region of low solute concentration (dentinal tubules in hydrated dentin substrate) to a region of high solute concentration (the adhesive-composite interface). This, in turn, may result in osmotic blistering of microscopic water droplets beneath the uncured hydrophobic composites. The interfacial blisters observed in Groups 2, 3 and 4 of this study are essentially negative impressions of the water droplets that extruded out of cured adhesive interfaces.

CONCLUSIONS

The increase in adhesive permeability associated with the bonding of an experimental one-step self-etch adhesive to hydrated dentin allows for time-dependent water movement through the polymerized adhesive to the adhesive-composite interface. The coupling of dual/chemical-cured composites to dentin is adversely affected even when a ternary catalyst is incorporated in the adhesive. Thus, apart from adverse chemical interactions, increased adhesive permeability also contributes to the reported incompatibility between one-step self-etch adhesives and chemically-cured composites. Based on the results obtained with bonding to processed composites, the authors speculate that as long as ternary catalysts are used with these adhesives, no compromised bonding will occur on bonded enamel due to its lower water content (Dibdin, 1993). Further studies should be performed to validate this hypothesis.

Acknowledgements

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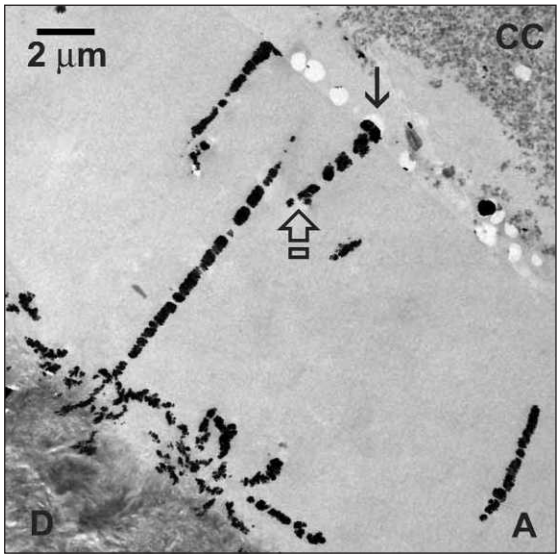
APPENDIX



TEM micrograph showing the communication between water trees (open arrow) and interfacial blisters (arrow) when the dual-cured composite were coupled to bonded dentin using the chemical-activation mode (CC). The micrograph is digitally enhanced to remove sectioning and image acquisition artifacts and was not included in the Results. D: dentin; A: one-step, self-etch adhesive.

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Effect of Power Density on Shrinkage of Dental Resin Materials

TG Oberholzer • CH Pameijer
SR Grobler • RJ Rossouw

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Clinical Relevance

Exposure at a lower power density causes less polymerization shrinkage in dental resins and could clinically improve the quality of the bond between the tooth and the restoration.

SUMMARY

This study compares volumetric changes and rates of shrinkage during different stages of polymerization of dental resin composites and compomers exposed to the same total energy by using two different combinations of power density and exposure duration. A hybrid composite and its equivalent flowable and a compomer and its equivalent flowable were exposed using a halogen curing unit set at 400 mW/cm² for 40 seconds and 800 mW/cm² for 20 seconds: delivering 16 J/cm² in both cases. Volumetric changes were recorded every 0.5 seconds using a mercury dilatometer. Ten replications per test condition were performed and the data were subjected to ANOVA. Statistically significant differences in shrinkage values and rates among different power densities were determined by means of paired *t*-tests at a 95% confidence level. Significantly more shrinkage ($p<0.05$) was found for the higher filled materials, Z250 and Dyract AP, when higher power density was used. However, no significant differences were found between their flowable counterparts when exposed to various power densities. Of the four materials, only Dyract AP exhibited no significant difference in shrinkage rate when various power densities were used. All the other materials exhibited significantly higher rates ($p<0.05$) at the higher power density.

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INTRODUCTION

Light-curing dental materials that adhere to tooth structure have revolutionized modern restorative dentistry. However, perfect adaptation should be obtained during the setting reaction of a material. Unfortunately, materials such as resin composites and compomers do not meet this requirement. The primary problem is that the polymerization reaction of the matrix phase causes dimensional instability of the restorative material. The conversion of monomer molecules into a polymer network is accompanied with closer packing of the molecules, which leads to bulk contraction (Koran & Kurschner, 1998). As most composites and compomers contain camphorquinone as the photo-initiator, a substantial power density of approximately 470-nm wavelength is required for complete polymerization (Miyazaki, Fukuishi & Onose, 1999). The optimal duration of light exposure and intensity for resin composites and compomers are, as yet, unknown.

Studies (Feilzer & others, 1995; Miyazaki & others, 1999) have reported that marginal adaptation is more effectively preserved when the polymerization process is slower. These studies assert that slower polymerization causes an improved flow of molecules in the material, which is associated with less shrinkage. However, it must be taken into account that complete polymerization is important in order to obtain sufficient surface hardness, which is accomplished only by means of a satisfactory period of high intensity illumination (Feilzer & others, 1995; Lovell, Newman & Bowman, 1999; Althoff & Hartung, 2000). Photo-energy radiated from the curing light is directly equal to the intensity and the period of radiation. Althoff and Hartung (2000) reported that in order to reduce the exposure time by half, the intensity of the curing light had to be doubled. Therefore, lengthening the exposure period or increasing the intensity results in increased energy density, increased degree of conversion and, unfortunately, more volumetric shrinkage. This could give rise to increased tension on the bond (Miyazaki & others, 1999). Uno and Asmussen (1991) recommend that low intensity light be used in order to bring about lower polymerization speed, thus, counteracting the tension, which is compensated for by a longer exposure duration.

For the sake of convenience and for economic reasons, it is in the best interest of a practicing dentist to minimize the clinical exposure time of resin-type restorations. Over the past few years, the industry has focused on reducing the resin exposure time by using stronger curing lights and/or altering resin composition and photoinitiator concentration (Rueggeberg, Ergle & Lockwood, 1997). The impact these new devices and new composite formulations have, however, are complex and not yet fully understood. Recently, a number of manufacturers have introduced light-curing units capable of producing various levels of power density and variable modes of application.

This study compared polymerization shrinkage in dental composites and compomers using different power densities but with the same energy density. The research hypothesis was that if the power density was doubled but the same total energy levels maintained, the rates of cure and the total shrinkage at various times would not increase for any material tested.

METHODS AND MATERIALS

Four light curing materials were tested, comprising two resin composites (color A3) and two flowable composites (color A3) (from the same companies) (Table 1). Ten readings for each of the materials were taken with the light source set at 400 mW/cm^2 for 40 seconds and 800mW/cm^2 for 20 seconds, respectively, at a temperature of 25°C ±10. The light source for this experiment was a Spectrum 800 (Dentsply/Caulk, Milford, DE, USA). The power density of the light source was veri-

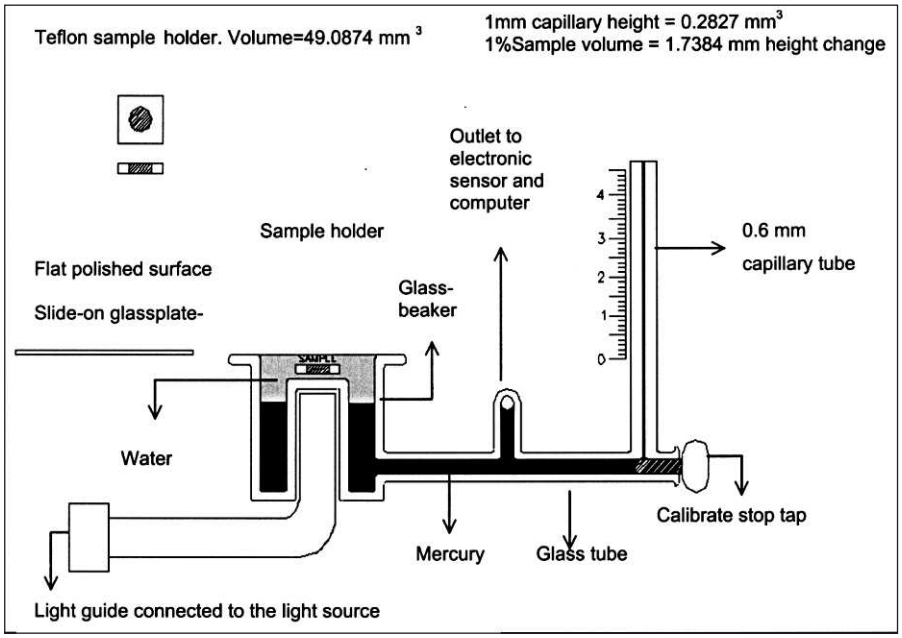

Figure 1. *Schematic image of the glass parts of the dilatometer and the teflon sample holder (top left).*

Table 1: *Materials Tested and Their Filler Composition as Supplied by the Manufacturers*

Material	Manufacturer	Batch # (Expiration Date)	Filler Particle Size	Filler Content/Weight	Filler Content/Volume
Z-250 (Composite)	3M Dental Products, St Paul, MN, USA	20000717 (2003-04)	0.6 µm	80%	60%
Filtec-flow (flowable composite)	3M Dental Products, St Paul, MN, USA	20000417 (2003-04)	1.5 µm	68%	47%
Dyract AP (compomer)	Dentsply/Caulk, Milford, DE, USA	0101001343 (2003-01)	0.8 µm	73%	49%
Dyract-flow (flowable compomer)	Dentsply/Caulk, Milford, DE, USA	0101001077 (2002-07)	1.6 µm	60%	38%

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fied using a hand-held dental curing radiometer (Dentsply/Caulk).

Volumetric changes were determined by a dilatometer with an average coefficient of variation of less than 1.22% as described by Oberholzer and others (2002). Any change in volume of the sample resulted either in expansion or contraction, which is registered as a change of the liquid height in the capillary. A diagram of this system is shown in Figure 1.

Instead of manually viewing and recording the liquid height, the column pressure was measured electronically with a pressure sensitive transducer and recorded by a computer that also stored the data. The relationship between shrinkage and the pressure transducer output had been correlated and calibrated (Oberholzer & others, 2002). The computer also recorded the temperature and controlled the light source. Since it is important that dilatometers operate at a constant temperature, the apparatus was placed in an incubator that maintained a temperature of 25°C ±10 while readings were taken.

The samples were randomly selected and a standard procedure followed for each specimen. A thin layer of silicon grease was applied to the edge of the glass beaker, which was filled to the brim with distilled water. The sample to be tested was prepared by placing the Teflon specimen holder on a lubricated glass cover slip, completely filling the hole with the material, using a resin disposing gun and scraping the excess off with a glass plate (volume=49.087 mm^3, thickness=2 mm). The specimen holder was then placed on the platform of the glass beaker with the open end facing upward and away from the curing light. The glass cover plate was placed on the beaker and checked to make sure that it was properly sealed and that no air was trapped. A clamp was placed on the cover plate to ensure that it would remain properly sealed. The height of the column of mercury in the capillary tube and the computer was adjusted until both had a "0" reading. The computer was then activated to register a reading that was monitored for five seconds. If the computer found the system unstable, it would reject the test and request that the container be sealed again. Once the system was stable, the computer activated the light curing apparatus for 20 or 40 seconds, respectively, at the chosen intensity and readings were taken at half-second intervals.

The data were tabulated and the mean and the standard deviation were calculated. Shrinkage rates for the four materials were compared using ANOVA at a pre-set alpha of 0.05. Statistically significant differences between different power densities were determined by means of paired *t*-tests at a 95% confidence level ($p<0.05$). For each recording of a specific material, the following parameters were established: (i) the mean values for the slope (%/time) of the shrinkage curves (rates), (ii) the mean value of shrinkage after an exposure period of 20 and 40 seconds measured at 40 seconds (iii) and

Table 2: *Rates of Polymerization Shrinkage (%/time) Using Different Power Densities*

Material	Power Density mW/cm²	Mean Values(%/time) (sd)	p=
Z 250	400	0.162 (0.010)	0.008
	800	0.206 (0.022)	
Filtec Flow	400	0.124 (0.024)	0.000
	800	0.185 (0.017)	
Dyract AP	400	0.119 (0.022)	0.527
	800	0.127 (0.024)	
Dyract Flow	400	0.161 (0.032)	0.017
	800	0.203 (0.025)	

N=10 specimens per test condition
Vertical lines indicate statistically different values (p<0.05)

Table 3: *Volumetric Shrinkage of Restorative Materials at 35- and 55-Seconds After Light Exposure Was Initiated*

Material	Power Density (mW/cm²)	Mean Values(%) at 35 Seconds (sd)	p=	Mean Values (%) at 55 Seconds (sd)	p=
Z 250	400	0.991 (0.071)	0.004	1.011 (0.088)	0.009
	800	1.304 (0.108)		1.317 (0.104)	
Filtec Flow	400	2.305 (0.112)	0.425	2.463 (0.137)	0.695
	800	2.385 (0.193)		2.505 (0.204)	
Dyract AP	400	1.183 (0.096)	0.001	1.303 (0.1110	0.003
	800	1.541 (0.114)		1.652 (0.147)	
Dyract Flow	400	3.326 (0.120)	0.892	3.417 (0.141)	0.528
	800	3.318 (0.087)		3.469 (0.132)	

N=10 specimens per test condition
Vertical lines indicate statistically different values (p<0.05)

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the mean value of shrinkage measured after 60 seconds of measurement.

RESULTS

Table 2 shows the values for the rates of shrinkage obtained during polymerization of the four materials when exposed at a power density of 400 mW/cm² for 40 seconds and 800 mW/cm² for 20 seconds. It was found that only the compomer (Dyract AP) showed no significant difference in the rate of shrinkage with respect to power density ($p<0.05$). All the other materials showed a significantly higher shrinkage rate with increased power density.

Descriptive statistics of polymerization shrinkage of the four materials tested after 40 and 60 seconds, respectively, are shown in Table 3. Generally, more polymerization shrinkage was demonstrated when a higher power density was applied. After 40 and 60 seconds, significant differences in shrinkage were shown for the higher filled materials (Z250 and Dyract AP) when higher power densities were used ($p<0.05$). However, no significant differences were found for the two flowables.

Graphic illustrations of the volumetric changes that took place in the different materials when exposed at various power densities are shown in Figures 2-5. The contraction rate as shown by the gradients of all the curves is at its highest during the first 20 seconds of exposure. With the composite (Figure 2), the polymerization process was nearly completed after 20 seconds of exposure with both power densities, while shrinkage could still be demonstrated in the other materials (Figures 3-5) even after approximately 40 seconds.

DISCUSSION

Yap and Seneviratne (2001) performed studies on the influence of light energy density on the effectiveness of composite cure. They found that optimal cure at the bottom surface of a 2-mm deep Z100 restoration could be achieved using a 20 second exposure at 600 mW/cm² and 40 seconds at 400 mW/cm². Based on these findings, it was assumed that in this experiment the degree of conversion

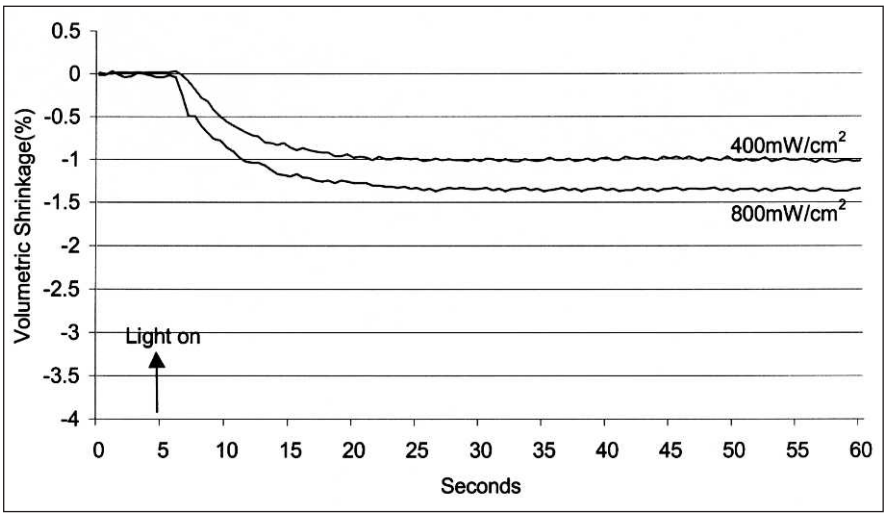

Figure 2. *Polymerization shrinkage curves for Z 250 exposed at different power densities.*

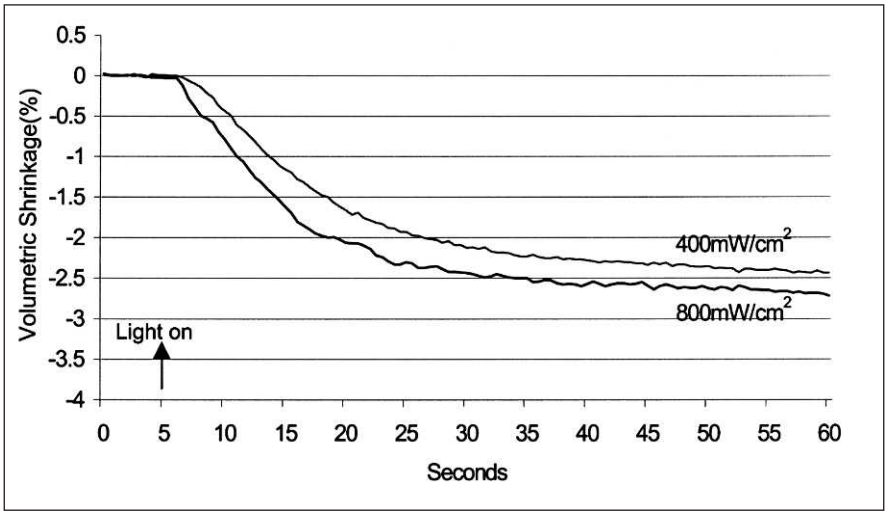

Figure 3. *Polymerization shrinkage curves for Filtec Flow exposed at different power densities.*

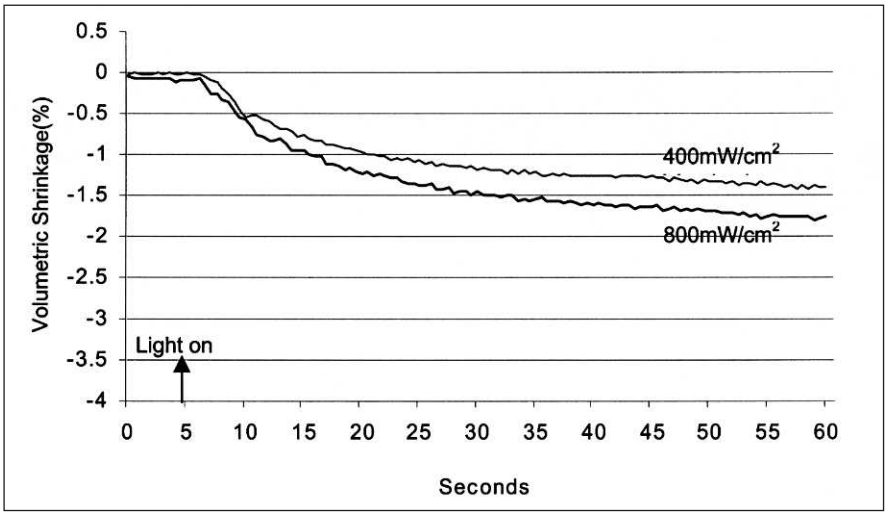

Figure 4. *Polymerization shrinkage curves for Dyract AP exposed at different power densities.*

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should be adequate and equivalent for both power densities (16 Joules).

The results obtained showed that after 40 and 60 seconds, the two higher filled materials, Z250 and Dyract AP, shrink significantly more ($p<0.05$) when exposed at 800mW/cm² than at 400 mW/cm². For these materials the research hypothesis was thus rejected. This corresponds with the findings of other authors, including Uno and Asmussen (1991); Koran and Kurschner, (1998); Silikas, Eliades and Watts (2000); Miyazaki and others (1999). A contributing factor in the amount of polymerization shrinkage that occurs is the number of monomers per volume of a resin material (Venhoven, de Gee & Davidson, 1993). Thus, to reduce polymerization shrinkage, the number of polymerizable monomers at a certain stage should be reduced. According to Ferracane and others (1997) and Lovell and others (2001), exposure at lower power densities may lead to this. However, it must be kept in mind that a high monomer conversion remains important for optimal physical characteristics and that the intensity of the curing light cannot be reduced indefinitely (Rueggeberg, Caughman & Curtis Jr, 1994). Rueggeberg and others (1994) found that a longer exposure time could compensate for a lower intensity in terms of monomer conversion. However, they found that using a curing light with an intensity of more than 578 mW/cm² and an exposure time of more than 60 seconds had no significant advantages.

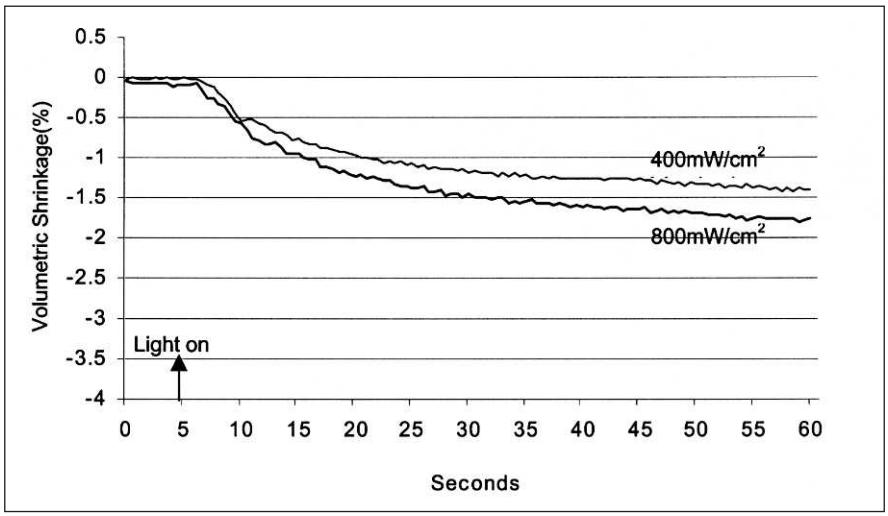

Figure 5. *Polymerization shrinkage curves for Dyract Flow exposed at different power densities.*

Conversely, the different power densities had no significant effect on the amount of shrinkage found for the two "flowable" materials, Dyract Flow and Filtec Flow (Tables 3 and 4). For these materials, the research hypothesis was thereby sustained. A probable explanation for the difference is that shrinkage of a material with a higher monomer concentration during polymerization will be enhanced by the lower filler contents as less light attenuation occurs when the filler particles are reduced (Ferracane & Greener, 1986). The photosensitizer concentrations in the matrix phase may also have an effect on the responsiveness to particular power densities. Rueggeberg and others (1997) and Silikas and others (2000) found that the higher the photosensitizer concentrations, the more rapid the polymerization. Furthermore, the ratio between the particle size and filler content is different for the "flowable" materials on the one side compared to the others.

The three-dimensional molecular structures of the monomers in a material might also play a role. For example, Z-250 and Dyract AP also contain UDMA, where the urethane part can be a large molecule and may be extended with an aliphatic or even an aromatic group. It is therefore possible that in this experiment the polymerization process of the flowable materials was already nearly completed at the lower power density, and that applying a higher power density could not induce more polymerization. This was probably not the case with the higher filled materials, where a higher power density caused more polymerization shrinkage. This finding coincides with those of Goldman (1983); Davidson and Feilzer (1997) and Rees, O'Dougherty and Pullen (1999), that the addition of monomers causes polymerization shrinkage to increase, while increasing filler content generally causes less polymerization shrinkage. Thus far, it has been generally accepted that this is applicable to all resins. The results of this study, however, show that a difference in shrinkage is demonstrated when the influence of high and low power densities on materials and their flowable counterparts is compared.

Although the total energy remained the same, it was found that contraction rates were generally higher when exposed at a power density of 800 mW/cm² compared to 400 mW/cm² (Figures 2-5). Therefore, in this case, the research hypothesis for contraction rates was rejected. This result corresponds with the findings of Sakaguchi, Douglas and Peters (1992), Bouschlicher, Vargas and Boyer (1997) and Dennison and others (2000).

Sakaguchi and others (1992) demonstrated a linear relationship between power density and polymerization shrinkage. They found that the higher the power density of the curing light, the sooner polymerization takes place and the greater the contraction rates. Clinically, this finding is of great importance, as the bond between the tooth structure and the restoration can be compromised at this early stage, possibly causing microleakage. A further disadvantage of high intensity exposure is that quicker termination of the polymerization process may occur due to the enhanced formation of certain ter-

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minating radicals. This may lead to further tension within the restoration (Althoff & Hartung, 2000). Rapid polymerization also generates reactive heat within the material, thus, enhancing the polymerization process. This process, known as the "Trommsdorff effect," causes a vast increase in the viscosity of the material (Goracci, Mori & de' Martinis, 1996).

CONCLUSIONS

This work is based on a study of four commercially available, well-researched materials. It is probable that the conclusions will also apply to a wider range of materials with comparable formulations. Within the conditions and limitations of this study, the following conclusions may be drawn:

1. Higher filled materials show more polymerization shrinkage when exposed to the same total energy but at higher power densities, while this finding is less significant for "flowable" materials.
2. Exposure to the same total energy, but using a lower power density, causes less and slower rates of shrinkage in the dental resins tested.

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Wear and Microhardness of Different Resin Composite Materials

EC Say • A Civelek • A Nobecourt
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Clinical Relevance

From a clinical standpoint, the wear property of a resin composite used for a posterior restoration is the most important variable. The use of a three-body abrasive wear test is a feasible method for selecting the composite material for non-stress bearing areas in the posterior region.

SUMMARY

This study determined the three-body abrasive wear resistance of two packable composites (P-60; Solitaire 2), an ion-releasing composite (Ariston AT), a hybrid composite (Tetric Ceram) and an ormocer (Admira). The study also looked at the correlation between wear resistance and hardness of the composites. Three-body wear testing was performed using an ACTA wear machine with 15 N contact force using millet seed as the third body. Wear depth (µm) was measured by profilometry after 200,000 cycles. The hardness test was performed using a digital microhardness tester (load: 500 g; dwell time: 15 seconds). The data were analyzed by using Kruskal Wallis ($p<0.05$). There were statistically significant differences among the three body abrasive wear of the composites. The ranking from least to most were as follows: Filtek P-60< Solitaire 2 < Ariston AT < Tetric Ceram < Admira. Filtek P-60 showed the highest microhardness value. No other significant differences in hardness were observed among the different resin composites (P-60>AristonAT=Tetric Ceram= Solitaire 2= Admira).

The results of this study indicate that there are significant differences in the wear resistance of the resin composites. The correlation between hardness and wear was significant with a correlation coefficient of r:–0.91. A significant negative correlation exists between hardness and three-body wear of resin composites.

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INTRODUCTION

Wear is a loss of material that occurs through contact of two or more surfaces (Craig, 1997). In general, there are four types of wear of dental materials (abrasive, adhesive, fatigue and corrosive) and three terms for wear of teeth (erosion, abrasion and attrition) (Smith, 1989). Abrasive wear occurs when a soft surface comes into contact with a harder one. Corrosive wear is the physical removal of a protective layer and is, therefore, related to the chemical activity of the wear surfaces. By fatigue, wear stresses are produced by free particles leading to the formation of surface or subsurface cracks. Adhesive wear is characterized by the formation and

disruption of microjunctions. Any of these mechanisms may operate alone or in combination with others. The wear process in the oral cavity is characterized by loss of the original form of teeth or restorative materials (Craig, 1997).

Resin composites have been available in the dental market for about four decades; however, their weak resistance to wear is still a major problem in clinical practice for restorations involving posterior occlusion. The first generation resin composite materials contained fillers with a mean diameter of 30-50 µm and long-time investigations have indicated that the wear resistance of those materials were not sufficient for use as amalgam substitutes in posterior teeth (Phillips & others, 1973; Leinfelder & others, 1980).

Newer resin composites have increased strength, owing to a higher filler content, an improved filler technology, modifications in the organic matrices and a greater degree of polymerization that improves their mechanical and physical properties (Bayne & Heymann & Swift Jr, 1994).

The new class of resin composites, high viscosity, packable composites whose handling has been modified to mimic the condensability of amalgam and ormocers (organically modified ceramics) that consist of organic-inorganic copolymers and inorganic silanated filler particles, are specifically designed for posterior restorations where wear resistance is very important.

Clinical wear of resin composites occur at the occlusal contact (OCA-wear; two body wear) and contact-free areas (CFA-wear; three-body wear). Three-body abrasive wear has been considered the main wear mechanism active in contact-free areas, resulting in a generalized loss of form (Lutz & others, 1984).

Assessment of the wear resistance has been conducted through *in vivo* and *in vitro* methods (Leinfelder, Beaudreau & Mazer, 1989; de Gee & Pallav, 1994; Condon & Ferracane, 1996). Due to complexity of the wear mechanism, correlation of the *in vivo* and *in vitro* findings is often difficult (Mair & others, 1996). However, *in vitro* tests are still necessary to test newly developed materials, as the clinical trials are expensive and time consuming. One *in vitro* wear test developed for three body abrasive wear that can test dental materials under conditions designed to simulate the clinical situation is the Acta wear machine described by de Gee, Pallav and Davidson (1986).

This study compared three-body abrasive wear resistance and the microhardness of two packable composites, an ion releasing composite, a hybrid composite and an ormocer and investigated the possible relationship between three-body abrasive wear and surface hardness.

METHODS AND MATERIALS

Table 1 lists the composite materials investigated and their composition. Included are two packable composites (Filtek P-60; Solitaire 2), an ion-releasing composite (Ariston AT), a hybrid composite (Tetric Ceram) and an ormocer (Admira).

Table 1: *Technical Profiles of the Composite Resins Evaluated*

Material Category	Brand Name	Filler Volume%	Filler Weight%	Average Filler Size (µm)	Resin System	Filler Type	Manufacturer
Packable resin-based composite	Filtek P 60	61	83	0.6	Bis-GMA, UDMA, Bis-EMA(6)	zirconia/ silica	3M Dental Products, St Paul, MN, USA
	Solitaire 2	66	75	0.7, 5, 8	UDMA, Bis-GA, TEGDMA, Tetrafunctional monomers	Ba-Al-B-F-Si glass, porous SiO_2	Heraus Kulzer, Wehrheim, Germany
Ion-releasing	Ariston AT composite	59	79	1.3	Bis-GMA, UDMA, Dimethacrylate	Ytterbium-trifluoride, Ba-Al-Fluorosilicate glass, silica, Alkaline glass	Ivoclar Vivadent, Schaan, Liechtenstein
Hybrid composite	Tetric Ceram	60	78	0.7	Bis-GMA, UDMA, TEGDMA	Barium glass, Ytterbium-trifluoride, Ba-Al-Fluorosilicate glass,silica	Ivoclar Vivadent, Schaan, Liechtenstein
Ormocer	Admira	56	78	0.7	Organic	SiO_2 polymers, Polysiloxanes (inorganic-organic unit)	Voco, GmbH, Cuxhaven, Germany

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Wear Testing

Experiments were carried out with the ACTA wear machine. The compartments of the metal sample holding wheel of the ACTA wear machine were filled with resin composites (l=14, w=10, h=3 mm). The composite specimens were cured in 2-mm increments using a Visiolux (105608, 3M Dental Products Division, Germany) light-curing unit. Five specimens of each of the five materials were fabricated. The sample holding wheel was mounted on the fixed axis of the wear machine and a diamond grinding wheel was mounted on the other axis to create a cylindrical composite surface. The grinding procedure of the samples was carried out under water using 600 grit. After storage in water at 37°C for 24 hours, the metal sample holding wheel was mounted in the wear machine together with a stainless steel antagonist wheel that had approximately half the diameter of the sample wheel and was 2.5-mm smaller in width. The antagonist wheel was pressed against the sample wheel by a force of 15 N. To simulate the sliding action of the antagonist teeth, surface speeds of the two wheels differed by 20%. Then, the two wheels were allowed to rotate in the food slurry that consisted of a mixture of rice and shells of millet seeds (pH=7). After 25,000 cycles (three hours) of the sample wheel, the worn surfaces were measured by profilometry and were used as the base line tracings. The actual wear trace in µm (vertical loss of substance) was measured after 200,000 cycles (55 hours) by profilometry in comparison to the non-worn surfaces. During the measuring procedure the composite samples were always kept wet.

Hardness Testing

To measure for Vickers Hardness, composites (4-mm long, *4-mm wide and *4-mm deep) were applied in self-cured acrylic resin embedded in metal molds. The composites were light polymerized in 2-mm increments with a Visiolux (105608, 3M Dental Products Division, Germany) light curing unit according to the manufacturer's curing times. Five specimens were made for each composite. A glass slide was placed over the composites and pressure was applied to extrude the excess material. After light polymerization, the specimens were immersed in distilled water and stored at 37°C for 24 hours (the time necessary to obtain water sorption). Prior to the hardness measurement, the samples were polished flat using a sequence of 400-800-1200 grit silicon carbide paper and alumina polishing paste (0.5µ). After the specimens were blotted dry, they were subjected to the digital microhardness tester (Shimadzu MHT,HMV-2, Japan) and a 500 g load was applied through the Vickers indentor with a dwell time of 15 seconds. Three indentations were made and measured at different points on each specimen and an average value was determined from each specimen.

Statistical Evaluation

Kruskal Wallis analysis of variance was performed on wear and microhardness test data at a significance level of 0.05 using computer software (SPSS 8.0, SPSS Inc, Chicago IL, USA).

To obtain the correlation between wear and microhardness, optimized box-cox transformation of the wear variable was used and a linear relationship between the modified variable and the hardness was performed.

RESULTS

Table 2 and Figure 1 show the mean three-body abrasive wear values (µm). Filtek P-60 had the least three-body wear. The ranking for composite wear from least to most were as follows: P-60 < Solitaire 2 < Ariston AT < Tetric Ceram < Admira. Admira was significantly less wear resistant compared to all the other composites tested (Kruskal Wallis; $p<0.05$).

The mean microhardness (VHN) values are shown in Table 2 and Figure 2. Filtek P-60 was significantly harder than all the resin composites (Kruskal Wallis; $p<0.05$). No other significant differences in hardness were observed among the different resin composites (P-60 > AristonAT=Tetric Ceram=Solitaire 2=Admira).

The correlation between hardness and wear was significant, with a correlation coefficient of r:–0.91 ($p<0.0001$) indicating a relatively strong relationship between the variables. This indicates that higher surface hardness was associated with less three-body abrasive wear.

DISCUSSION

The occlusal loss of composite is caused by two-body wear at occlusal contact points and generalized three-body wear from movement of abrasive food slurries during mastication. Two-body wear is primarily the result of fatigue from cyclic loading (Pallav, 1996; Wu & others, 1984; de Gee & Pallav, 1994), while three-body wear involves the process of resin matrix loss between filler particles and subsequent dislodgement of the filler. Filler loading, size, interparticle spacing, degree of polymerization (Jørgensen & others, 1979; Bayne, Taylor & Heymann, 1992), filler/matrix cracking caused by water sorption and hydrolytic degradation of the filler surface

Table 2: *Mean Three-Body Wear and Microhardness Values of Resin Composites*

Material	Three-Body Wear (mm)	Microhardness (VHN)
P-60	50.8 (± 0.80)	88.00 (± 2.71)
Solitaire 2	61.6 (± 1.70)	59.86 (± 1.75)
Ariston AT	66.2 (± 2.20)	63.40 (± 6.99)
Tetric Ceram	81.1 (± 1.50)	62.81 (± 5.87)
Admira	103.2 (± 2.60)	58.33 (± 4.03)

Figures in brackets are standard deviations.

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(Sarrett, Sölderholm & Batich, 1991; Sarrett & Ray, 1994) are important for protecting the resin matrix from wear and, therefore, reducing three-body wear. All of these variables participate in the wear process, however, at present, it is not clear which plays the most important role.

By reducing the filler particle size and increasing filler loading, wear resistance has been improved (Suzuki & others, 1995; Condon & Ferracane 1997). In this study, the packable resin composite Filtek P-60, which has the highest filler content and the lowest particle size, has demonstrated significantly improved wear resistance compared to the packable composite Solitaire 2, ion releasing composite Ariston AT, hybrid composite Tetric Ceram and ormocer Admira. However, the average particle size did not influence the wear properties of the other resin composites in this study. Although Solitaire 2 and Ariston AT have a larger average filler size than Tetric Ceram and Admira, they showed significantly less wear behavior. With respect to the inorganic fillers, for the same particle size distribution, glass fillers are more hydrolytically unstable than fillers containing silica (Söderholm, 1983). Barium-containing glass fillers have been shown to be more radiopaque; however, they leach significantly more than pure quartz filler and their degradation rate is higher in saliva-simulating solutions than in distilled water (Söderholm, Mukherjee & Longmate, 1996; Söderholm, Yang & Garcea, 2000). The zirconia/silica containing resin composite Filtek P-60 showed better wear resistance than barium glass filler containing the composites Solitaire 2, Ariston AT and Tetric Ceram. Although the hydrolytic degradation of the filler particles could not be proven to be the primary cause for the clinical wear process, it is important for selection of the materials. From a clinical viewpoint, using a silica filled composite has been recommended instead of a glass-filled composite that leaches more filler elements and degrades faster (Söderholm & others, 2001). The barium glass-filled composites Solitaire 2, Ariston AT and Tetric Ceram have almost the same filler type and filler weight; however, Solitaire 2 showed superior wear resistance compared to the others in spite of its relatively large filler size. It has been suggested that finer glass particles dissolve faster than coarser particles and create more interfaces that affect filler degradation (Kalachandra, 1989). Moreover, Solitaire 2 contains porous SiO_2 filler that integrates part of the resin matrix within the porosities of the bodies of the filler particles. This results in a firm bond of the filler particles to the matrix, which also may play a role in wear resistance (Knobloch, Kerby & Seghi, 1999). As mentioned above, in terms of the wear process, the type of fillers seem to be more important than the size. However, other factors, such as filler shape, proportion and size of the largest fillers may also play a role in three-body abrasive wear (Manhardt & others, 2000).

Ferracane and others (1997) indicated that the abrasive wear rate depends on the degree of cure of the resin

Table 3: *Results of Statistical Analysis*

Subject	Differences
Three-Body Wear	P-60 < Solitaire 2 < Ariston AT < Tetric Ceram < Admira
Microhardness	P-60 > Ariston AT = Tetric Ceram = Solitaire 2 = Admira

Results of Kruskal Wallis (p<0.05) > indicates statistical significance

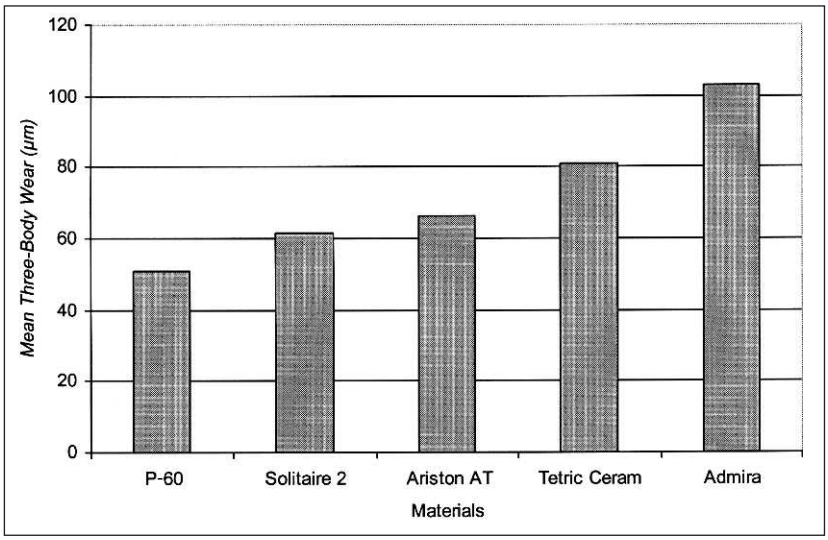

Figure 1. *Mean three-body wear values of resin composites.*

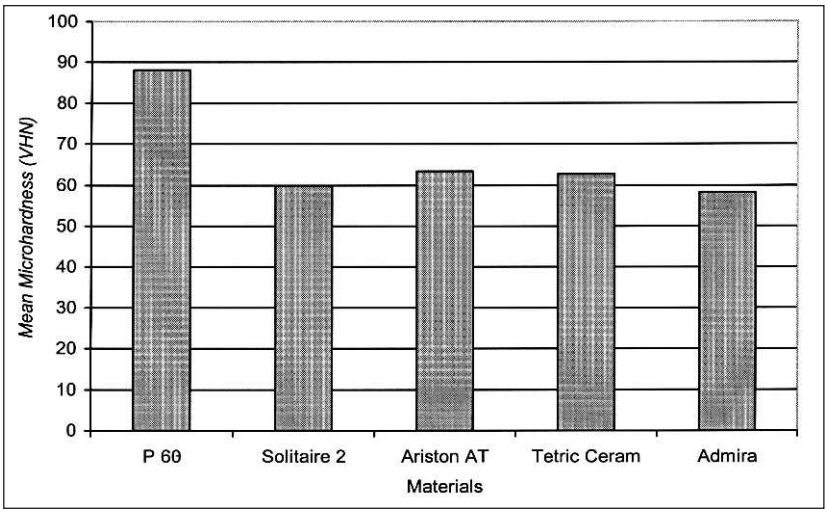

Figure 2. *Mean microhardness values (VHN) of resin composites.*

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matrix. For optimum physical and mechanical properties of the restoration, the residual monomer content should be minimized and the percentage of conversion to polymer maximized (Willems & others, 1993). It has been claimed that in Filtek P-60, the majority of TEGDMA has been replaced with UDMA and Bis-EMA(6). Both of these resins have a higher molecular weight and fewer double bonds per unit, which improves the degree of cure of the polymer matrix and, therefore, wear resistance (3M ESPE, 1998).

The degree of wear among the resin composites Solitaire 2 and Tetric Ceram found in this study is in agreement with the findings of Sunnegardh-Grönberg, Peutzfeldt and van Dijken (2002). The hybrid composite Tetric Ceram showed significantly more three-body abrasive wear than the packable composite Solitaire 2. In a previous study of the two-body sliding wear test of several resin-based restorative materials, Ariston pHc showed significantly higher wear rates than Tetric Ceram (Manhart & others, 2000). This study showed contrary results, with higher three-body abrasive wear rates for Tetric Ceram compared to Ariston AT. In addition to adding filler particles to the matrix of the composite, the degree of bonding of the filler particles to the matrix appears to have a significant effect on increasing the wear resistance of the resin composites (Venhoven & others, 1996). Although Ariston AT contains relatively more hydrophilic monomer in its organic matrix than Tetric Ceram, it showed significantly less three-body wear rates. Ariston AT is recommended, especially in the anterior region. The results of this study suggest that the ion-releasing composite Ariston AT may be used in the posterior region but not in non-stress bearing areas.

The ormocer matrix has been suggested to exhibit significantly less wear than composite matrices (Bauer, Kunzelmann & Hickel, 1995; Kunzelmann, Mehl & Hickel, 1998). In a previous study using a fatigue simulator, the ormocer Definite demonstrated significantly improved wear resistance compared to Tetric Ceram and Ariston (Manhardt & others, 2000). This study showed contrary results with higher wear rates for ormocer Admira compared to Tetric Ceram and Ariston AT.

According to ADA guidelines (ADA, 1989), the wear rate of an acceptable resin composite used as an "unrestricted" amalgam-replacement material should be $\leq 50\mu$ after two years and $\leq 100\mu$ after four years of clinical performance. However, there are no acceptable definite quantitative wear rates for *in vitro* wear tests of resin composites for the posterior region. In this study, the wear rates of the different types of resin composites are higher than previously reported with hybrid resin composites (Z100 and Charisma) (Peutzfeldt & Asmussen, 2000) tested with the ACTA wear machine, which showed excellent clinical performance after six years (Busato & others, 2001). The low wear resistance of the tested composites might be related to the ingredients and preparation of the slurry. In addition, comparison of the test results among *in vitro* studies and between *in vivo* and *in vitro* studies is often difficult to determine due to wear evaluation methods (visual, profilometric, microscopic; laser measurements) (Söderholm & others, 1992; Söderholm & others, 2001), variations in preparation designs among clinicians, finishing and polishing procedures and variations in food habits among the populations (Söderholm & others, 2001). Therefore, the primary purpose of this study was to rank the three-body wear resistance of the tested composites under *in vitro* conditions. However, whether this ranking will correlate with clinical performance of the resin composites should be confirmed with long-term *in vivo* studies, as the majority of the wear occurs in the first years after placing the restorations that decreases with time (Braem & others, 1986).

The relative importance of a microhardness test lies in the fact that it throws a light on the mechanical properties of a material (Braem & others, 1989). A positive correlation has been established between the hardness and inorganic filler content of resin composites (Boyer, Chalkly & Chan, 1982). Increased filler levels result in increased hardness numbers (Chung, 1990). In this study, the packable resin composite Filtek P-60, which had the highest filler content (83% by weight) and zirconia/silica, as the inorganic particle, showed significantly higher surface hardness than all the other composites tested. No other significant differences in hardness were observed among the other resin composites. Composites with harder filler particles exhibit higher surface hardness (Craig, 1997); however, the bond of the filler particles to the polymer matrix also affect their hardness values.

It has been shown that the degree of polymerization of the resin matrix influences the hardness of the composites. An increased conversion rate of carbon double bonds led to improved physical properties and higher hardness values (Asmussen, 1982; Ferracane & Greener, 1986). Hardness values of resin composites have also been considered to provide an indication of their wear resistance properties. However, the complexity of the wear process of resin composites has led to conflicting reports regarding the correlation between the hardness of a material and its wear resistance. Yap, Teoh and Tan (2001) observed a negative correlation between hardness and wear of resin composites. However, Lappalainen, Yli-Urpo and Seppa (1989) and Harrison and Draughn (1976) reported no relationship.

For the resin composites tested, a significant negative correlation was observed between hardness and three-body abrasive wear. This indicates that composites with higher surface hardness are less prone to three-body abrasive wear. This result seemed to be influenced by the tendency of one material, Filtek P-60, which showed

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lower wear rates at higher hardness values. Therefore, the significant correlation between hardness and wear observed in this study might be interpreted with some caution, as it can only be supportive for some materials. Whether this relationship can be extended into the clinical situation should be confirmed with *in vivo* wear studies.

CONCLUSIONS

Since an accurate ratio between clinically observed wear and that measured in the laboratory seldom exists, the primary purpose of this study was to rank the three-body wear resistance of the tested composites under *in vitro* conditions. Based on the results of this study, it was found that the different kinds of resin composites (ormocer, hybrid, ion releasing, packable) differ in their wear resistance. The packable composite P60 showed the least three-body abrasive wear and the highest microhardness values, while the ormocer Admira demonstrated the worst wear resistance and the lowest microhardness. A significant negative correlation was observed between three-body abrasive wear resistance and surface hardness; however, it should be taken into consideration that this result can be supportive for some materials only.

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Polymerization Shrinkage and Microleakage in Class II Cavities of Various Resin Composites

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M Soyman• EC Say

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Clinical Relevance

Ormocer and bonding-flowable-hybrid composite restorations showed less microleakage than ion releasing and hybrid composites lined only with bonding agent at cementoenamel junction in Class II cavities.

SUMMARY

This study investigated the polymerization shrinkage of restorative materials and microleakage in the proximal box of Class II restorations. Twenty caries free extracted human molars were prepared on the mesial and distal, making 40 slot cavities. Groups (n=10) were classified as: Group 1: Single bond/Filtek Z-250; Group 2: Single Bond/Filtek Flow/Filtek Z-250; Group 3: Admira Bond/Admira; Group 4: Ariston Liner/Ariston. Dentin bonding systems and resin composites were applied according to the manufacturer's instructions. The specimens were stored in distilled water for one week at 37°C, thermocycled (5°-55°C x 2000), stained with 50% aqueous silver nitrate, immersed in a diluted developer solution (24 hours), rinsed and sectioned in two pieces with a low speed saw (Isomet). Dye penetration (Score 0-4) was determined separately at the occlusal enamel margin and cementoenamel junction. The volumetric polymerization shrinkage of materials was determined with the apparatus of Watts and Cash (Watts & Cash, 1991) for 300 seconds. Polymerization shrinkage results were as follows: Filtek Flow (3.5%±0.1) > Admira (2.1%±0.1) = Ariston AT (2.3%±0.1) > Filtek Z-250 (1.8%±0.1) (One-way Anova, post hoc Bonferroni test at *p*=0.05 level). No statistical difference was found at the enamel margins for any of the materials (Score 0-1). At the cemento-enamel junction, all test groups showed significantly higher microleakage when compared to the enamel margins (Kruskal-Wallis test, Mann-Whitney U). Dye penetration results at dentin were as follows: Filtek Flow + Filtek Z-250= Admira < Ariston AT=Filtek Z-250.

It was concluded that enamel showed no high dye penetration scores and microleakage was not influenced by the material. Microleakage at dentin could not be eliminated with any adhesive restoration. The ormocer and bonding-flowable-hybrid composite restorations showed less

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microleakage than the ion-releasing and hybrid composites lined only with bonding agent at the cementoenamel junction in Class II cavities.

INTRODUCTION

Amalgam remains one of the most commonly used restorative materials in the posterior region (Jackson & Morgan, 2000). However, improvements in adhesive techniques, the introduction of light–cured composites and the esthetic demands of patients have driven the development of hybrid composites (Jackson & Morgan, 2000; Cobb & others, 2000). These resins, along with a mixture of particles, were recommended for universal use in anterior teeth and the posterior region (Baratieri, 1998; Meiers, Kazemi & Meier, 2001).

Resin composites have been improved during the past several years, especially with regard to their physical properties (Lee & White, 1998; Perry & others, 2000; Cobb & others, 2000; Nash, Lowe & Leinfelder, 2001). Recently, manufacturers have increased the filler load, changed filler types and resin formulations and introduced new hybrid, ion-released, packable and ormocer composites (organically modified ceramics). Significant advantages, such as esthetics, relatively low thermal conductivity, wear resistance and easy handling have been obtained (Manhart & others, 2000). However, a major disadvantage of resin-based materials is polymerization shrinkage that causes gap formation, which enables bacterial penetration (Haller & Trojanski, 1998). Microleakage is defined as the passage of bacteria, fluid, molecules or ions between the cavity wall and the restorative material (Kidd, 1976). Microleakage primarily results in postoperative sensitivity, marginal staining, recurrent caries and/or possible loss of the restoration (Aboushala, Kugel & Hurley, 1996; Haller & Trojanski, 1998; Roulet & Degrange, 2000; al-Ehaideb & Mohammed, 2001).

Microleakage is still a major focus of many researchers (Tung, Estafan & Scherer, 2000b; Meiers & others, 2001; Beznoz, 2001). Several studies have found wide gaps occurring in conventional Class II composite restorations and have reported that the marginal adaptation of these restorations may present a problem with regard to microleakage (Aboushala & others, 1996; Davidson & Feilzer, 1997; Tung, Estafan & Scherer, 2000a). Assessment of microleakage is usually determined by dye penetration tests (Derhami, Coli & Brannström, 1995; Reeves & others, 1995; Dejou, Sindres & Camps, 1996; Friedl & others, 1997; Zivkovic 2000). In the absence of clinical data, laboratory microleakage studies are an acceptable method to evaluate adhesive restorative materials for adequate marginal adaptation (Brackett & others, 1995).

This study determined the polymerization shrinkage of various resins and evaluated the microleakage of different resin composites (applied with their corresponding bonding agents) in Class II restorations at enamel and dentin, *in vitro*.

METHODS AND MATERIALS

Microleakage Study

Twenty non-carious molars were used in this study. The teeth were brushed, cleaned with scalers and stored in distilled water. Each tooth received two Class II cavity preparations, including an occlusal margin in enamel and a cervical margin in dentin or cementum on the mesial and distal surface. One operator prepared all 40 cavities. A fissure diamond bur (ISO 806 314 157524 012, Komet, Germany) was used in a high-speed water-cooled handpiece to prepare the cavities. The Class II slot cavities on the mesial and distal surfaces were prepared using the following dimensions: 4 mm in the buccolingual (width) and 1.5-2 mm in the pulpal direction (mesiodistal depth). The height (occluso-gingival length) of the cavity depended on the cemento-enamel junction (CEJ), approximately 3.75-4 mm. The cervical wall was located just below the cemento-enamel junction (0.5 mm). The axial wall was parallel to the long axis of the tooth. The approximal enamel margins of the teeth were beveled 0.5-1 mm (acurata, ISO 806314,

Table 1: *Materials Used in This Study*

Material Category	Brand Name	Manufacturer	Resin System	Filler Type	Average Filler Size (μm)	Filler Volume (%)
Hybrid	Filtek Z-250	3M ESPE Dental St Paul, MN, USA	TEGDMA, UDMA, Bis-EMA(6)	Zirconia/Silica	0.6	60
Flowable	Filtek-Flow	3M ESPE Dental St Paul, MN, USA	TEGDMA, Bis-GMA Dimethacrylate	Zirconia/Silica	1.5	47
Ormocer	Admira	Voco, GmbH Luxhaven Germany	Dimethacrylate polysiloxan	Silicium-Dioxide	0.7	56
Ion Releasing Composite	Ariston AT	Vivadent, Schaan Liechtenstein	Bis-GMA, UDMA, Dimetacrylate	Alkaline glass, Ba-Al-Fluorosilicate Ytterbiumtrifluoride, Silicondioxide	1.3	59

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1665004014,D-94169 Thbg, Germany) to improve the bonding surface (Opdam & others, 1998; Lee & White, 1998; Dietrich & others, 2000); however, the occlusal and cementum margins did not receive bevels. After finishing the cavity preparation, the teeth were divided into four groups. Acid etching, dentin bonding systems and composites were applied according to the manufacturer's instructions. The cavities were restored with the following bonding agents and resin composites: Single Bond (3M ESPE)—Filtek Z-250; Single Bond—Filtek Flow–Filtek Z-250 (3M ESPE); Admira Bond—Admira (Voco); Ariston Liner-Ariston AT (Vivadent) (Table 1).

The resin composite restorations were placed using a four light-curing incremental technique (Figure 1). The halogen dental curing device (XL 3000; 3M, St Paul, MN, USA) was tested for adequate light intensity (680 mW/cm^2). Finishing and polishing was accomplished using polishing discs (Soflex XT, 3M Dental) within five minutes after light curing. The specimens were stored in distilled water for one week at 37°C. After thermocycling (5°-55°C x 2000), the teeth were sealed with two coats of nail varnish and immersed in 50% aqueous silver nitrate in darkness for two hours. The teeth were immersed in a diluted developer solution for 24 hours, rinsed and two sections per restoration were made with a low-speed saw (Isomet). Two dye penetration determinations were made per restoration area (two at enamel and two at CEJ). The teeth were examined using a binocular microscope (Leica MZG, United Kingdom) coupled with a video camera (Sony 3 CCD MVA 41-A, Japan). The definition and a scheme of the dye penetration scores were given in Table 2 and Figure 2.

Statistical differences within groups were determined using the Kruskal-Wallis test and paired comparison was made using the Mann-Whitney U test. Statistical significance was at $p<0.05$.

Volumetric Polymerization Shrinkage Determination

Five samples were prepared for each group as described by Tolidis, Nobecourt and Randall (1998). Volumetric polymerization contraction was measured using the apparatus of Watts and Cash (1991) at 300 seconds. This apparatus consists of a linear vertical displacement transducer (LVDT), a light cure device and a computerized recording system. Polymerization shrinkage was recorded by means of data-logging software. The testing procedure was as follows: A brass ring was placed on a glass microscope slide and filled with the restorative material. The brass ring used to prepare the composite samples had an internal diameter of 19 mm and was 2-mm deep. A glass coverslip was put on the ring to flatten the resin material. The LVDT, which was held in contact with the tested resin composite sample, exerted approximately a 7g of force during polymerization. The visible light unit

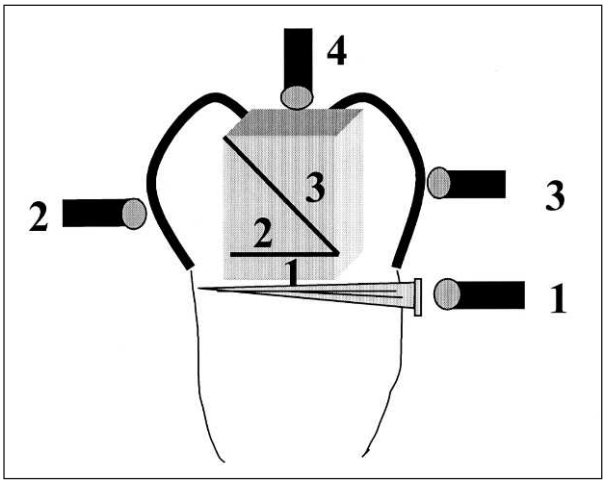

Figure 1. *Four-sited light curing incremental technique.*

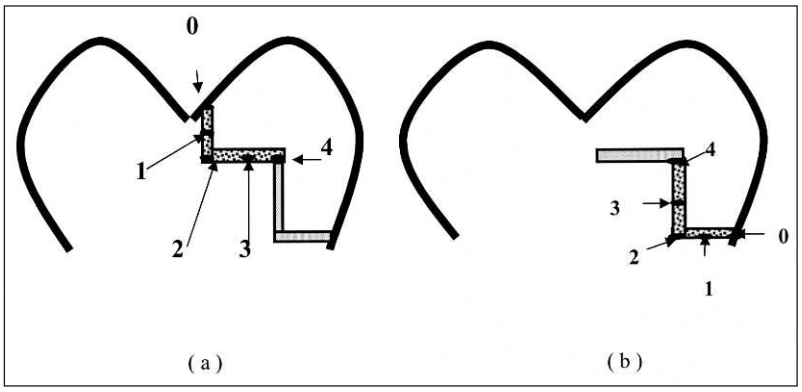

Figure 2. *Scheme of dye penetration scores at enamel (a) and at dentin (b).*

Table 2: *Definition of Dye Penetration Scores at Enamel and Dentin*

Dye Penetration Scores (Enamel)	
Scores	
0	No penetration
1	Dye penetration less than or up to one half of the cavity depth
2	Dye penetration greater than one half of the cavity depth
3	Dye penetration involving the 1/2 of base of the cavity
4	Dye penetration involving more than half of the base of the cavity
Dye Penetration Scores (Dentin)	
Scores	
0	No penetration
1	Dye penetration involving the 1/2 of base of the cavity
2	Dye penetration involving the base of the cavity
3	Dye penetration involving the half of the axial wall
4	Dye penetration involving more than the half axial wall

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(XL 3000, 3M) was applied from the bottom of the testing samples and cured the material (recommended by the manufacturer) during loading. Dimensional changes were monitored during light polymerization and after the curing procedure for 300 seconds (post-irradiation period).

Polymerization shrinkage data was analyzed with One way Anova, post hoc Bonferroni test at p=0.05 level.

RESULTS

Microleakage at the enamel and cementoenamel junction (CEJ) was evaluated separately. Tables 3 and 4 list the microleakage scores at enamel and dentin. Figure 3 showed the dye penetration scores at dentin.

Microleakage at the enamel margins showed no significant differences among all treatment groups and all scores were either 0 or 1. Filtek Z-250 did not present any microleakage at the enamel margin (Table 3).

All test groups showed a significant increase at CEJ when compared to enamel microleakage. The group restored with Filtek Z-250 and Ariston AT were scored as 3 and 4 at CEJ (Table 4). The restorations with Filtek Flow and Admira showed significantly less dye penetration than Ariston AT and Filtek Z-250 at dentin (p<0.05). Table 5 shows the percentage of microleakage scores. All groups except Admira showed more dye penetration at CEJ compared to enamel and the difference was found to be statistically significant.

Polymerization Shrinkage Results

Polymerization shrinkage results were as follows: Filtek Flow (3.5%±0.1) > Ariston AT (2.3%±0.1) = Admira (2.1%±0.1) > Filtek Z-250 (1.8%±0.1).

Table 3: *Dye Penetration Scores at Enamel*

Scores	Materials			
	Filtek Z-250	Filtek Flow+ Filtek Z-250	Admira	Ariston AT
0	20	18	14	15
1	-	2	6	5
2	-	-	-	-
3	-	-	-	-
4	-	-	-	-
Mean±Sd	0.00±0.00[a]	0.15±0.36[a]	0.30±0.47[a]	0.25±0.44[a]

Materials identified with the same superscript letter are significantly not different; p>0.05, Mann Whitney U test)

Table 4: *Dye Penetration Scores at Dentin*

Scores	Materials			
	Filtek	Filtek-Flow+ Z-250	Admira Z-250	Ariston AT
0	-	6	8	5
1	12	14	10	6
2	3	-	2	4
3	2	-	-	3
4	3	-	-	2
Mean±Sd	1.80±1.15[a]	0.70±0.47[b]	0.70±0.65[b]	1.55±0.31[a]

Materials identified with the different superscript letters are signaficantly different; p<0.05, Mann Whitney U test.)

Table 5: *Percentage of Dye Penetration Scores*

Enamel Scores (%)	**0**	**1**	**2**	**3**	**4**
Filtek Z-250	100	-	-	-	-
Filtek Flow+ Filtek Z-250	85	15	-	-	-
Admira	70	30	-	-	-
Ariston AT	75	25	-	-	-
GCM Scores (%)	**0**	**1**	**2**	**3**	**4**
Filtek Z-250	0	60	15	10	15
Filtek Flow+ Filtek Z-250	30	70	-	-	-
Admira	40	50	10	-	-
Ariston AT	25	30	20	15	10

DISCUSSION

This study determined the polymerization shrinkage of various resin composites and evaluated microleakage of these materials in Class II cavities. It evaluated the performance of the bonding and composite restoration system together, not the performance of the bonding system or resin composite, separately. The reaction between the cavity wall and bonding system during thermal changes and occlusal forces in the mouth are important factors from the viewpoint of microleakage and durability of the restorative (McCoy & others, 1996). Therefore, the test groups were thermocycled (5°-55°C x 2000) but the occlusal forces could not be simulated.

The results of this study showed that bonded tissue is a more important factor in microleakage than the applied material. The dye penetration scores indicated that enamel is a more reliable substrate than dentin in direct adhesive Class II restorations, because of its homogenous structure and hydophobic character (Schwartz, 1996).

Microleakage at enamel margins was not evident in most of the investigations (Munro, Hilton & Hermesch, 1996;

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al-Ehaideb & Mohammed, 2001; Beznos, 2001). In this study, enamel margins also demonstrated a very good seal. Microleakage at enamel margins showed no significant differences among treatment groups. This indicates that the resin composite material did not influence microleakage at the enamel-restorative interface. This study also showed that using an adhesive system and the corresponding resin composite do not completely eliminate microleakage when the cavity margins are at the cementoenamel junction (Figure 3). Filtek Z-250 (with Filtek Flow) and Admira showed significantly less dye penetration than Ariston AT and Filtek Z-250 without Filtek Flow at dentin. The authors reported that microleakage at dentin depends on the parallel, oblique or crosscut configuration of the dentinal tubules (Derhami & others, 1995).

In order to prevent microleakage, attention has to be given to the mechanism that may relieve polymerization shrinkage and occlusal forces (Unterbrink & Liebenberg, 1999; Choi, Condon & Ferracane, 2000; Tolidis & others, 1998). Several layers of low viscosity bonding agents, one layer of high viscosity bonding agents, flowable composites and different resins with distinct physical properties were recommended by the manufacturers to avoid microleakage (Ferdianakis, 1998; Reeves & others, 1995, Choi, Condon & Ferracane, 2000). An important factor in avoiding microleakage seems to be the capacity of the bonding agent to compensate for the polymerization forces of the restorative material during light curing. In this situation, bonding agents may show different performance with various resin composites. The differing results between Group 1 (without flowable composite) and Group 2 (with flowable composite) support this suggestion. Single Bond showed different microleakage scores when Filtek Flow was used with Filtek Z-250 (Group 2) compared to applying hybrid composite after Single Bond directly, without any flowable resin (Group 1). The use of Filtek Flow decreased the amount of microleakage significantly. The results of another investigation also revealed that flowable resin composites caused the greatest reduction in microleakage (Ferdianakis, 1998). It was noted that the low elasticity modulus of flowable composites provide flexibility for the bonded restoration (Labella & others, 1999; Park, Krejci & Lutz, 1999). Lining might lead to a more equal distribution of the stresses over the adhesive interface (Davidson & Feilzer, 1997) and reacts as a stress breaker (Haller & Trojanski, 1998; Beznoz, 2001).

The bond strength of restorative materials to dentin can also be improved by cavity design and the size, shape and position of increments of resin composite placement (Carvalho & others, 1996; Davidson & Feilzer, 1997; Andersson-Wenckert, van Dijken & Horstedt, 2002). The influence of the composite curing procedure is also an important factor on microleakage

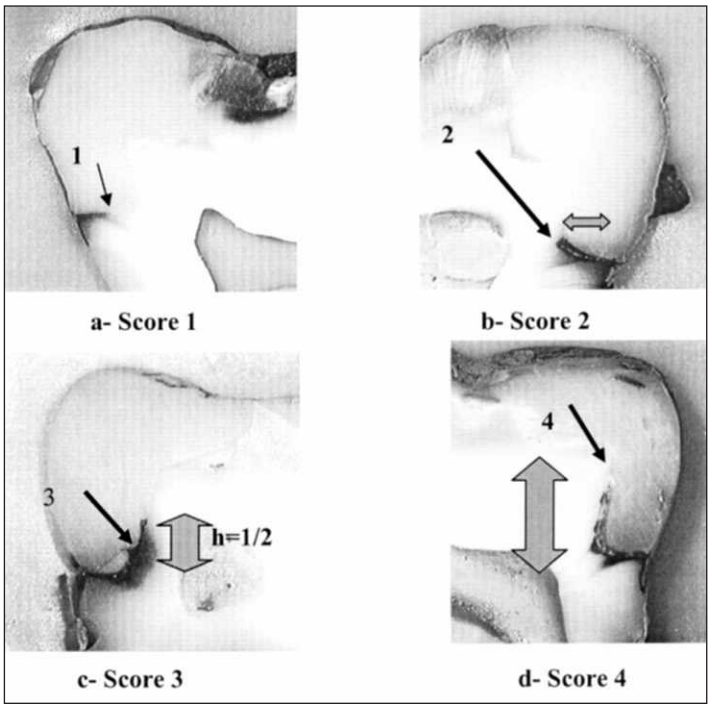

Figure 3. *Dye penetration scores at dentin.*

(Haller & Trojanski, 1998). Therefore, a four-sited light curing technique was used in this study (Figure 1). The four light-curing technique and translucent wedges were used to direct polymerization shrinkage towards the enamel margin and avoid gap formation at dentin.

All tested materials in this study were not sticky and provided some resistance to "condensation." Ormocers consist of organic-inorganic copolymers and inorganic silanated filler size (Manhart & others, 2000). They are designed for the posterior region and mimic the condensability of amalgam as claimed by the manufacturers. Stickiness can result in voids and porosity (Jackson & Morgan, 2000). The authors determined that voids caused by improper placement technique can lead to microleakage and staining (Ferdianakis, 1998; Opdam & others, 1996). Voids were observed in the sectioned teeth restored with Ariston AT. The voids were primarily in the restoration body, which did not influence microleakage at the cavity margin. However, these voids may increase staining and water sorption, which may have undesirable influences on the durability and esthetics of the restoration.

Other influencing factors on polymerization shrinkage are resin formulation, amount of filler, filler type and size (Feilzer, de Gee & Davidson, 1988; Fitchie & others, 1995). The higher molecular weight of the resin results in less shrinkage (Technical Product Profile Filtek P60, Z250, 1998). The formulations of the applied resins are shown in Table 1. Bis–GMA was replaced in Filtek Z-250 with TEGDMA and UDMA, and in Admira with

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dimetacrylate polysiloxan. These higher molecular weight materials (Filtek Z-250 and Admira) shrinked less than Ariston AT and Filtek Flow. Filtek Flow has a resin formulation similar to Filtek Z-250 but shrinks more because of the low filler content (47%). Filtek Z-250 has the highest filler load, 60% and polymerization shrinkage is the lowest, 1.8%. Flowable and Ormocer resin showed the greatest amount of *in vitro* polymerization shrinkage, but these groups did not result in the highest microleakage scores. The polymerization shrinkage apparatus evaluated the shrinkage of restorative materials without cavity factor or bonding influences. No relationship was observed between *in vitro* polymerization shrinkage and dye penetration scores. That means that *in vitro* polymerization shrinkage of the filling materials does not simulate shrinkage during the light curing procedure in the cavity, which caused microleakage *in vivo*. There is a need for clinical observations and a determination technique of polymerization shrinkage directly in cavities.

The influence of the loading cycle on microleakage and the determination of dye penetration localization (cavity wall-bonding or bonding-resin composite) should be investigated in future studies.

CONCLUSIONS

It was concluded that enamel is well sealed with adhesive systems and dye penetration at enamel is not influenced by resin composite material. However, microleakage at dentin cannot be eliminated with any adhesive restoration. Restorative materials differ in their microleakage scores at dentin. Ormocer and bonding-flowable-hybrid composite restorations show less microleakage than ion released and hybrid composite lined only with bonding agent at the cementoenamel margin in Class II cavities.

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Effect of Different Intensity Light Curing Modes on Microleakage of Two Resin Composite Restorations

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AJS Santos • JR Lovadino

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Clinical Relevance

Restorations light cured with low initial intensity polymerization showed lower leakage means, even when high intensity was used after low intensity.

SUMMARY

This *in vitro* study evaluated the marginal leakage of two resin composites light cured in four different polymerization modes. Standardized Class V cavities were prepared on the buccal enamel surface of 80 sound, freshly extracted inferior bovine incisors. The teeth were randomly divided into eight experimental groups: two resin composites (Z250 Charisma) and four polymerization modes (conventional–680 mW/cm²/30 seconds; soft start–380mW/cm²/10 seconds + 680 mW/cm²/20 seconds; plasma arc curing–1480mW/cm²–3 seconds; combined– 380mW/cm²–10 seconds + 1480mW/cm²—3 seconds). All specimens were thermocycled (1,000 cycles at 5°C and 55°C) with a dwell time of one minute at each temperature and immersed in a 2% methylene blue solution for more than 12 hours. The specimens were then washed and prepared for spectrophotometric analysis to quantify the dye concentration. Soft start and combined polymerization modes presented better results and were statistically different from conventional and plasma arc curing polymerization methods for both resin composites. There were no statistical differences between the two resin composites.

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INTRODUCTION

The major problem of light cured composites and compomers is the development of shrinkage during polymerization (Luo & others, 2002; Lambrechts, Braem & Vanherle, 1987). Conversion of the monomer molecules into a polymer network is accompanied by a closer packing of the molecules, causing contraction in the composite (Friedl & others, 2000; Feilzer, de Gee & Davidson, 1990). Such contraction creates mechanical stresses in the resin composite that can disrupt the marginal seal between the composite restoration and dentin or enamel (Feilzer & others, 1990). Polymerization shrinkage leads to some clinical problems, such as marginal fading, restoration fractures, solubility of the bonding system and marginal leakage (Koran & Kürschner, 1998). Leakage is characterized by the invasion of acids, enzymes, ions, bacteria and their products into the margins of the restoration (Kidd, 1976), causing postoperative sensitivity, recurrent caries, inflammation and later, pulp necrosis (Gordon & others, 1986; Jedrychowski, Bleier & Caputo, 1998; Tarle & others, 1998).

Polymerization shrinkage has three didactical phases, the pre-gel phase, gel point and post-gel phase. However, the only phase capable of disrupting the marginal seal between the composite restoration and dentin or enamel is post-gel (Koran & Kürschner, 1998; Friedl & others, 2000) because, in the phase before the gel point, the monomers can still move or slip into new positions within the resin matrix without causing stress at the interface (Koran & Kürschner, 1998). With an increase in the number of monomers converted into polymers, the flow gradually decreases, while resin composite stiffness increases and the material becomes sufficiently strong to exert forces or stress in the bond system (Feilzer & others, 1990; Friedl & others, 2000). Post-gel polymerization results in clinically significant stress in the composite-tooth bond and surrounding tooth structure (Yap, 2000), leading to an adhesion failure (Koran & Kürschner, 1998).

Adhesive bond strength, restorative material elasticity modulus, cavity design, light intensity and curing time are some factors influencing the marginal quality of restorations (Unterbrink & Muessner, 1995; Friedl & others, 2000). Some polymerization techniques have been developed in an attempt to decrease the stress caused by polymerization shrinkage.

Initial polymerization with low intensity light followed by a final cure at high intensity light may result in improved marginal integrity without losing the achievable material properties (Mehl, Hickel & Kunzelmann, 1997). The aim of this technique is to prolong the time prior to reaching the gel point by using low light curing intensities and increase the flow capability of the material. Subsequently, high light intensities are necessary for complete polymerization and optimal mechanical properties (Friedl & others, 2000). Another technique that prolongs the time span prior to reaching the gel point is the pulse activation technique (Luo & others, 2002), a method similar to the soft-start technique. This technique differs from soft-start by using a waiting interval between the initial low intensity and the final exposure to the high intensity light. However, these two techniques have long cure times that are inconvenient for the patient, impractical with children, uncomfortable for the dentist, and they make treatment more expensive due to the extra time spent in the chair (Peutzfeldt, Sahafi & Asmussen, 2000).

A completely different method of providing extremely high levels of irradiance is plasma arc curing (PAC) units (Rueggeberg, Ergle & Mettenburg, 2000). Light is emitted from glowing plasma composed of a gaseous mixture of ionized molecules and electrons (Peutzfeldt & others, 2000). Curing by PAC light units occurs very quickly. The high light curing can polymerize the resin composite in less time, which is compensated for by the intensity. However, high light intensities do not allow enough flow for reducing internal stress, thus, contributing to high polymerization shrinkage (Mehl & others, 1997; Althoff & Hartung, 2000) and increased leakage (Althoff & Hartung, 2000).

This study evaluated the marginal leakage of two resin composites using four polymerization modes: conventional, soft-start, plasma arc curing and combined (soft start + plasma arc curing).

METHODS AND MATERIALS

One week after extraction, 80 sound, inferior bovine incisors were cleaned, polished and examined under a light microscope (4x) in order to exclude those with cracks. The teeth were stored in distilled water at 5°C for less than one month prior to conducting the restorative procedures. Cubic blocks 5-mm in size were obtained from the buccal surfaces. These dental blocks were included in epoxy resin to facilitate handling.

Standardized square Class V cavities (3 mm) were prepared in the middle of the vestibular surface of each tooth using a #3100 diamond bur (KG Sorensen Ind Com Ltda—Barueri, SP, Brazil—13400). The cavities were made with a water-cooled high-speed turbine by using a standard cavity preparation device. A new bur was used for every five preparations. The cavity preparations were

Table 1: *Experimental Groups (Charisma—Dentsply/Caulk, Petropolis, RJ, Brazil 25665010; Z250—3M-ESPE; Degulux Soft Start Curing Light Device—Degussa–Hüls AG—Hanau, Germany 1364; Apollo 95E—DMD, West Lake Village, CA USA)*

Groups	Resin Composite	Light Curing Intensity	Light Curing Unit
Group 1	Charisma	680mW/cm^2-30 seconds	Soft Start – Degussa
Group 2	Z250	680mW/cm^2-30 seconds	Soft Start – Degussa
Group 3	Charisma	380mW/cm^2-10 seconds + 680mW/cm^2-20 seconds	Soft Start – Degussa
Group 4	Z250	380mW/cm^2-10 seconds + 680mW/cm^2-20 seconds	Soft Start – Degussa
Group 5	Charisma	1480mW/cm^2-3 seconds	Apollo 95E
Group 6	Z250	1480mW/cm^2-3 seconds	Apollo 95E
Group 7	Charisma	380mW/cm^2-10 seconds +1480mW/cm^2-3 seconds	Soft Start – Degussa/ Apollo 95E
Group 8	Z250	380mW/cm^2-10 seconds +1480mW/cm^2-3 seconds	Soft Start – Degussa/ Apollo 95E

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rinsed for 10 seconds with air/water spray and air dried for 10 seconds. The same operator performed all cavity restorations.

Each tooth was acid-etched with 35% phosphoric acid (Scotchbond—3M ESPE, St Paul, MN, USA) for 15 seconds. The cavities were rinsed for 15 seconds and gently air dried for 10 seconds. Two layers of adhesive system (Single Bond—3M-ESPE) were applied and light cured for 20 seconds. All specimens were randomly restored according to the experimental groups (n=10) (Table 1).

After restoration, all specimens were stored in distilled water at 37°C for 24 hours and polished with flexible aluminum oxide disks (Sof-Lex Pop-on—3M ESPE) under water spray. All specimens were kept in water at 37°C for 24 hours. They were then thermocycled 1,000 times (5° ± 2°C and 55° ± 2°C) with a dwell time of one minute at each temperature.

The interface between the block and the epoxy resin of all specimens was then protected with cianocrylate adhesive Superbond (Henkel Loctite Adhesives, LTDA, Itapevi-SP, Brazil). The blocks were immersed in a 2% methylene blue solution for 12 hours at 37°C and the specimens were rinsed in tap water and dried. A surface layer of the composite restorations was abraded with Sof-Lex to remove possible superficial dye penetration in the restorative material.

Dental blocks were removed from the resin cylinders and prepared for spectrophotometric analysis according to the methodology described in Aguiar and others (2002) and Aguiar, Ajudarte and Lovadino (2002). A one-way ANOVA and Tukey-Kramer test was performed on the data at 0.05 confidence level.

RESULTS

Results of the leakage test are presented in Figure 1. ANOVA showed no statistically significant differences between the two materials tested. There were statistically significant differences among the four polymerization modes. The Tukey test (p<0.01) was applied to individual comparisons (p<0.01). The test showed that conventional and PAC polymerization modes presented the highest leakage means, which were statistically different from the other two techniques. The soft start and combined polymerization modes presented no significant differences (Figure 1).

DISCUSSION

Under the experimental conditions of this study, the results showed that the soft start and combined polymerization modes presented the lower leakage means, which were statistically different from the conventional and PAC polymerization modes. A low intensity light can prolong the time of flow capability of the composite, thus, decreasing stress during polymerization (Friedl & others, 2000). However, a high intensity light is necessary for complete polymerization and optimal mechanical properties (Friedl & others, 2000). Burgess and others (1999) demonstrated that polymerization with a low initial intensity light, followed by a high intensity light, does not reduce the mechanical properties of the resin composite. As a result, conventional intensity was used for at least 20 seconds in this study, following the manufacturer's instructions for the composite used.

The marginal adaptation of the restoration strongly depends on the initial curing intensity and the relationship between the initial and final curing intensity (Friedl & others, 2000). Mehl and others (1997) and Price and others (2000) showed that a light intensity lower than 280 mW/cm^2 cannot activate enough initiator molecules to start an adequate reaction, and the final cure of this nearly unpolymerized material then corresponds to an immediate full intensity curing.

The results showed that conventional and PAC polymerization modes presented the highest means of leakage, which was statistically different from the other groups. The conventional technique does not allow

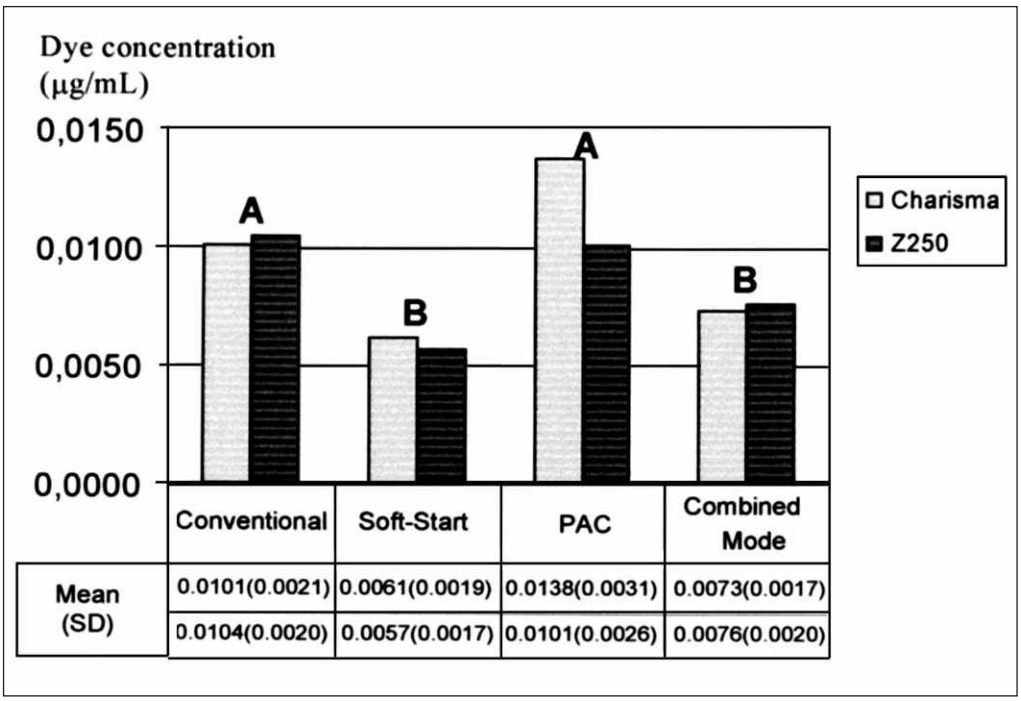

Figure 1. *Results of microleakage (mg/mL) for the experimental groups. Same capital case letter were not statistically different for the same polymerization method (p<0.05).*

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enough flow of the composite to reduce stress at the interface of the restoration (Yap, 2000). The same explanation could be used to explain the results of the PAC, but according to Peutzfelt and others (2000), Rueggeberg and others (2000) and Park, Krejci and Lutz (2002), the time recommended by the manufacturers of the plasma arc curing Apollo 95E (three seconds) for curing the resin composite is less than that necessary to result in optimum irradiated resin composite properties. When this curing unit is used with this time, the remaining double bonds of the resin composite are higher when compared with other units and the flexural strength and elasticity modulus is lower (Peutzfelt & others, 2000). However, according to Peutzfelt and others (2000), contraction of the composite polymerized with Apollo 95E was almost the same as the composite polymerized with conventional light intensity (480mW/cm^2). This similar contraction may produce similar stress at the interface. Furthermore, if resin composites light cured with Apollo 95E have not been adequately polymerized, and the elasticity modulus is lower than that of other satisfactorily polymerized resin composites, the Apollo restorations could present problems when submitted to thermocycling (Brackett, Haisch & Covey, 2000). The temperature difference of the thermocycling test can increase gaps formed during polymerization (Rossomando & Wendt, Jr, 1995), mainly in restorations not polymerized adequately by Apollo 95E (Brackett & others, 2000).

When the resin composite was light cured with a low intensity, followed by a high intensity (PAC, in this study), this low intensity, apart from decreasing the leakage means, started the polymerization reaction. When added to the high intensity, even for three seconds, this low intensity may have helped the resin composite become adequately polymerized, thus, improving the physical properties of the restorations. If the initial intensity was sufficient to start the curing reaction when added to the final intensity, it could produce high power intensity that would produce a degree of conversion (DC) adequate for the restoration (Burgess & others, 1999). However, the disadvantage of this technique is that it used two light-curing units that can be unviable clinically. However, it is important to emphasize that if the initial intensity allows the resin composite to flow, thus decreasing the stress of the shrinkage polymerization, it does not decrease the total DC of the resin composite since an appropriate intensity is used.

This study suggests that the best way to cure the resin composite and decrease polymerization shrinkage stress without decreasing the physical properties is by using an adequate, low initial intensity polymerization technique, followed by a light intensity capable of complementing the resin composite curing. This low intensity can be obtained by using a light curing unit with a soft start or ramp device (Friedl & others, 2000) or by placing the tip of the light curing unit at a distance capable of reducing the intensity (Mehl & others 1997; Dennison & others, 2000), but allowing the initiator molecules of the resin composite to be activated to start an adequate reaction.

CONCLUSIONS

Within the limits of this study, it can be concluded that:

1. No light curing technique or resin composite used was able to eliminate leakage;
2. An initial low intensity is very important to preserve the marginal integrity of the restoration, even when a high intensity is used to complement the polymerization.

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Microtensile Bond Strengths of an Etch&Rinse and Self-Etch Adhesive to Enamel and Dentin as a Function of Surface Treatment

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Clinical Relevance

Cavity preparation methods, such as diamond sonoabrasion, laser irradiation and air abrasion, may affect bonding performance of etch&rinse and self-etch adhesives to enamel and dentin. Irrespective of the cavity preparation technique, separate acid-etching as part of an etch&rinse approach remains recommended to achieve adequate bonding to enamel.

SUMMARY

In light of the current trend towards "minimal invasive" dentistry, diverse cavity preparation techniques have been introduced as an alternative or addition to common bur instrumentation. This study investigated whether diamond sonoabrasion (SonicSys Micro, Kavo), air abrasion (Prep Start, Danville) and Er:YAG laser irradiation (Fidelis) produce surfaces at enamel/dentin that are equally receptive to bonding as traditional mid-grit diamond-bur (Komet) and 600-grit SiC-paper prepared surfaces, of which the latter two served as controls. An etch&rinse adhesive (OptiBond FL, Kerr) applied with and without prior acid-etching and a self-etch adhesive (Clearfil SE, Kuraray) were employed to bond the restorative composite (Z100, 3M ESPE) to the diversely prepared enamel and dentin surfaces. The microtensile bond strength (µTBS) was determined after 24 hours of storage in water at 37°C. The results indicated that the manner of preparation of enamel and dentin prior to bonding procedures significantly influenced the bonding effectiveness of both the etch&rinse and the self-etch adhesive. Using an etch&rinse adhesive, separate acid-etching of air-abraded and Er:YAG-irradiated enamel and dentin surfaces remains mandatory. Bonding to diamond-sonoabraded and air-abraded enamel and dentin was, in general, not different from bonding to conventional diamond-bur prepared surfaces, whereas, bonding to Er:YAG-irradiated

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enamel and dentin surfaces in general resulted in a significantly lower bonding effectiveness compared to bonding to diamond-bur prepared surfaces.

INTRODUCTION

Tooth cavities are commonly prepared by means of rotary instruments equipped with either diamond or tungsten carbide burs. While prepared relatively fast, bur-prepared cavities are often, however, larger than dictated by the extent of the actual caries. In addition, beveling of margins for adhesive purposes in critical approximal regions using burs is often not feasible. Also, iatrogenic bur damage of healthy adjacent teeth has been reported to be responsible for a substantial amount of new caries lesions (Moopnar & Faulkner, 1991; Qvist, Johannessen & Bruun, 1992; Lussi, 1995; Lussi & Gygax, 1998; Wicht & others, 2002). All these drawbacks, associated with conventional rotary cavity preparation techniques, along with the current trend toward "minimal invasive" (Degrange & Roulet, 1997) or "minimum intervention" dentistry (Tyas, Anusavice & Frencken, 2000), have lately led to the introduction of new tools or the revival of already existing techniques that, until recently, were considered somewhat less practical. These techniques intend to facilitate the preparation of micro-cavities with minimal sacrifice to sound tooth substrate.

One such new cavity preparation method involves using a modified sonic air-scaler handpiece equipped with a diamond-coated working tip activated through ultrasonic frequency oscillations. These diamond tips exist in different forms, enabling direct occlusal or lateral access to the initial caries lesion through sonoabrasion. The tips are only coated with diamonds at one side. The highly polished, non-active side allows the tip to be supported on the approximal site of the neighboring tooth without damaging it. Diamond sonoabrasion is, therefore, particularly recommended for minimal cavity preparations of proximal lesions in anterior and posterior teeth (Hugo, 1999a,b; Yip & Samaranayake, 1998). In addition, they are very handy to bevel enamel/dentin margins at difficult accessible areas, such as the approximal cervical and lateral box margins.

Air abrasion is a cavity preparation technique first introduced in the 1940s by Dr Robert Black (Goldstein & Parkins, 1994) that lately has re-gained interest. Its reappearance must largely be attributed to significantly improved air abrasion units released on the market (Manhart & others, 1999). The major benefit of this technique is that kinetic energy, generated by a high-velocity stream of aluminum oxide particles, can be utilized to prepare hard tissue (dentin/enamel), while having little effect on soft materials such as gingival tissues. In addition, this abrasive technique increases patient comfort by reducing pressure, heat, vibration and noise, as these are commonly experienced during mechanical preparation of teeth when rotating burs are used (Roeder & others, 1995).

Recently, the use of laser technology has also been introduced as an alternative to traditional mechanical rotating instruments for cavity preparation (Hibst, Keller & Stainer, 1988). In particular, the Erbium:YAG laser with an ultra-short square pulse technology has been advocated to prepare micro-cavities. Thanks to an ablation process that involves micro-explosions, hard tooth substrate can be effectively removed, while cooling with water prevents cracking or melting of enamel and dentin, thereby, protecting the underlying pulp tissue.

This laboratory study determined the microtensile bond strength (µTBS) of two representative adhesives to diamond sonoabraded, air-abraded and laser-irradiated enamel and dentin, as compared to their µTBS to bur-cut and SiC-paper prepared enamel and dentin (controls). The hypothesis tested was that diamond sonoabrasion, air abrasion and laser irradiation are equally effective to pre-treat enamel and dentin for bonding as traditional acid etching of bur-cut tooth surfaces.

METHODS AND MATERIALS

Ninety non-carious molars were stored in 0.5% chloramine solution at 4°C and used within one month after extraction for µTBS determination. The molars were randomly divided into 18 experimental (nine enamel and nine dentin experimental groups) and 12 control groups (six enamel and six dentin control groups) (Figure 1). All teeth selected were first mounted in gypsum blocks to ease sample manipulation. The µTBS to enamel and dentin was determined using one of the protocols described by Pashley and others (1999). A representative etch&rinse adhesive, Optibond FL (Kerr, Orange, CA, USA) and a self-etch adhesive, Clearfil SE (Kuraray, Osaka, Japan) were applied to successively diamond sonoabraded, air-abraded, laser-irradiated, bur-cut and SiC-paper-prepared enamel and dentin (Table 1 and Figure 1). The etch&rinse adhesive OptiBond FL was used with (AE) and without (NAE) 37.5% phosphoric acid conditioning. At each moment during specimen processing, care was taken to prevent dehydration of the samples.

Enamel Specimen Preparation

Lingual and/or buccal enamel of the human third molars was flattened using a high-speed, medium-grit (100 µm) diamond bur (842, Komet, Lemgo, Germany) mounted in the MicroSpecimen Former (University of Iowa, Iowa City, IA, USA). The exposed enamel surface served as the bur-cut control. For the SiC-paper prepared samples (control), a standard smear layer was also produced by wet-sanding the enamel surface with

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600-grit silicon carbide sandpaper for 60 seconds. For the diamond-sonoabraded specimens (SonicSys Micro, Kavo Dental, Biberach, Germany), a new "space shuttle"-shaped diamond tip of the SonicSys system was applied to the SiC-paper prepared enamel surface for one minute in as uniform a manner as possible. For the air-abrasion samples, the surface received a standard SiC-paper smear layer prior to uniform abrading for 10 seconds following three different directions by means of Prep Start (Danville, San Ramon, CA, USA) with 27 µm aluminum oxide particles at 6.5 psi air pressure. The distance of the nozzle tip to the dentin surface was kept constant to about 2 mm, with the nozzle held at 45° to the enamel surface. After air abrasion, the specimens were thoroughly rinsed with a vigorous water spray for 15 seconds to remove residual aluminum particles from the surfaces. For laser-irradiated surfaces, a standard SiC-paper prepared smear layer was first produced prior to being uniformly irradiated using a commercially available Er:YAG laser (Fidelis, Fotona Medical Lasers, Ljubljana, Slovenia) with a sapphire tip in constant slight contact with the surface. The settings for enamel "conditioning" were 10 Hz, 120 mJ and short pulse mode (pulse length is 250 µs). Subsequently, the adhesives were applied on the diversely prepared enamel surfaces strictly following the manufacturers' instructions (Table 1). After curing the adhesive using an Optilux 500 (Demetron/Kerr, Danbury, CT, USA) device with a light output not less than 550 mW/cm^2, the surface was build up with Z100 (3M ESPE, St Paul, MN, USA) in three-to-four layers to a height of 5-6 mm.

Dentin Specimen Preparation

The occlusal third of the molars was removed using an Isomet slow-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). The dentin surfaces were controlled for the absence of enamel and/or pulp tissue using a light microscope (Wild M5A, Heerbrugg, Switzerland). For the bur-cut samples (control), the teeth were mounted in a chuck and a standard smear layer was produced by removing a thin layer of the surface with a high-speed diamond bur (842, Komet) using the MicroSpecimen Former. For all other experimental and controls groups, the respective dentin surfaces were prepared as described above for the enamel specimens. Subsequently, the adhesive and composite were applied again, strictly following the manufacturers' instructions (Table 1). Settings for the laser "condition-

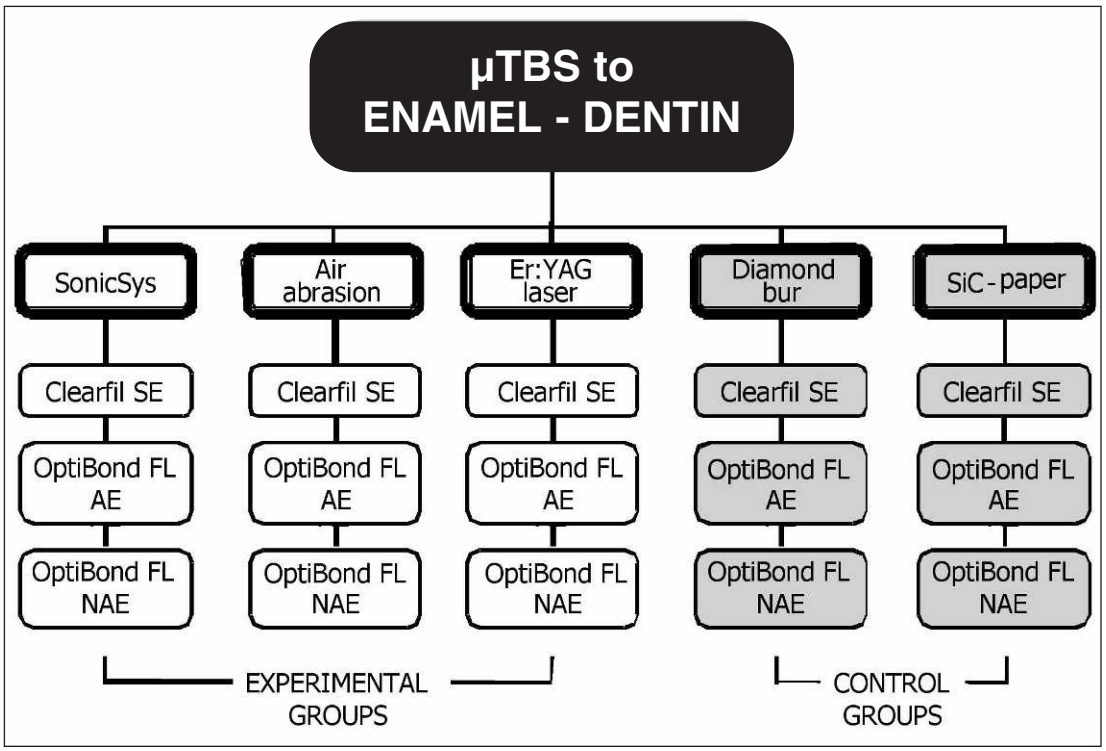

Figure 1. *Flow diagram presenting the experimental set-up. AE = Acid-etch; NAE = No acid-etch.*

Table 1: *List of Adhesives Investigated*

Code	Product Name	Adhesive Component	Application Procedure	Batch #
C-SE	**Clearfil SE Bond** Kuraray, Osaka, Japan	Primer	Applied for 20 seconds; Gently air blown.	[00125C]
		Bond	Applied and light cured for 10 seconds.	[00069A]
O-FL	**OptiBond FL** Kerr, Orange, CA, USA	Kerr Gel Etchant	Applied for 15 seconds, rinsed for 15 seconds and gently air dried for five seconds.	[104634]
		OptiBond Primer	Applied for 30 seconds with continuous scrubbing, gently air dried and light cured for 20 seconds.	[103962]
		OptiBond Adhesive	Applied in a thin, even layer.	[104369]

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ing" of dentin were 10 Hz, 80 mJ and short pulse mode (pulse length was 250 µs).

µTBS Testing

After bonding procedures, all specimens were stored for 24 hours at 37°C in tap water. The teeth were then sectioned perpendicular to the bonding surface using the Isomet saw to obtain rectangular samples about 2x2 mm wide and 8-9 mm long. These specimens were mounted in the pin-chuck of the MicroSpecimen Former and trimmed at the biomaterial-tooth interface to a cylindrical hour-glass shape with a diameter of about 1.2 mm using a cylindrical extra-fine grit (15 µm) diamond bur (835KREF, Komet) in a high-speed handpiece under air/water spray coolant. The specimens were then fixed to a Ciucchi's device with cyanoacrylate glue (Model Repair II Blue, Sankin Kogyo, Otahara, Japan) and stressed at a crosshead speed of 1 mm/minute until failure in an LRX testing device (LRX, Lloyd, Hampshire, UK) using a load cell of 100N. The µTBS was expressed in MPa as derived from dividing the imposed force (in N) at the time of fracture by the bond area (in mm^2). When the specimens failed prior to actual testing, the mean µTBS was determined from the specimens that survived specimen processing with an explicit note of the number of pre-testing failures (ptf). The mode of failure was determined at a magnification of 50x using the stereomicroscope.

Pairwise Kruskal-Wallis analysis and Dwass-Steel-Chritchlow-Fligner multiple comparisons were used to determine statistical differences in µTBS between the experimental and control groups for each adhesive group separately (C-SE, O-FL AE, O-FL NAE) at a significance level of 0.05. Furthermore, the effect of acid-etching using OptiBond FL and the adhesive (Clearfil SE versus OptiBond FL AE) were pair-wise assessed using two-sided Mann-Whitney U tests for the diversely prepared enamel and dentin surfaces. In order to compensate for multiple testing, the significance level α for the latter statistical analysis was adjusted to 0.01 following a Bonferroni correction.

Fe-SEM Evaluation

Selected µTBS samples of each experimental and control group that exhibited a representative failure mode and a µTBS close to the average value were processed for Fe-SEM. The authors employed common procedures for SEM specimen preparation, including fixation in a 2.5% glutaraldehyde in cacodylate buffer solution, dehydradation in ascending concentrations of ethanol and chemical drying using HMDS (the protocol has been described in detail by Perdigão & others, 1995). After mounting on aluminum stubs, the specimens were coated with a thin gold layer using a gold sputtering device (Sputtering device 07 120, Balzers Union, Liechtenstein) prior to examination in the Philips XL-30 Fe-SEM (Eindhoven, The Netherlands).

Twenty additional human third molars were used for examination using Fe-SEM of unbonded enamel and dentin surfaces prepared following the three experimental and two control conditions. The specimen surfaces were prepared in exactly the same way that the µTBS was determined and were further processed for SEM following the methodology described above.

RESULTS

The mean µTBS and standard deviations are summarized per experimental group in Table 2 and graphically presented in Figures 2-3, respectively, for Clearfil SE and Optibond FL, when bonded respectively to enamel and dentin. The p-values comparing the effects of acid-etching (solely OptiBond FL) and the adhesive (Clearfil SE versus OptiBond FL AE) are mentioned in Table 3. The results from failure analysis are summarized in Tables 4 and 5, respectively, for Clearfil SE and Optibond FL.

When bonded to enamel, the µTBS ranged from on average 10.0 MPa for OptiBond FL bonded to non-etched, Er:YAG-irradiated enamel to an average 37.8 MPa for OptiBond FL bonded to etched, air-abraded enamel (Table 2, and Figures 2 and 3). Clearfil SE bonded equally effectively irrespective of experimental or control conditions (Table 2 and Figure 2). Separate acid-etching of enamel prior to applying OptiBond FL significantly increased ($p<0.01$) the µTBS in all experimental and control groups with the exception of bonding to etched versus non-etched, air-abraded enamel, which was only nearly significantly different (Tables 2 and 3, and Figure 3). Following acid-etching (AE), the µTBS of OptiBond FL bonded to laser-irradiated enamel was significantly lower than that obtained when OptiBond FL was bonded to all other experimental and control enamel surfaces (Table 2). Among the latter groups, no significant differences were found. When enamel was not separately acid-etched (NAE), again, the µTBS of OptiBond FL bonded to Er:YAG-irradiated enamel was significantly lower than OptiBond FL bonded following all other experimental and control conditions with the exception of sonoabrasion (Table 2), which, however, was not significantly different from all other experimental and control conditions. Pre-testing failures only occurred for OptiBond FL bonded to non-etched, sonoabraded enamel (Table 2), indicating that when these failures would have been taken into account (µTBS=0 MPa), bonding to non-etched, sonoabraded enamel and non-etched, laser-irradiated enamel should be considered significantly less effective than the remaining three conditions.

When bonded to enamel, failure analysis revealed the highest amount of adhesive failures when Clearfil SE was bonded to bur-cut enamel (Table 4). This was also characteristic of the relatively high amount of mixed failures (including enamel) when Clearfil SE was bonded

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to laser-irradiated enamel (Table 4). For OptiBond FL, the relatively high amount of adhesive failures when bonded to acid-etched, laser-irradiated and bur-cut enamel is noteworthy (Table 5). Also, when Optibond FL was bonded to unetched enamel surfaces, the amount of adhesive failures significantly increased, confirming the less optimal bonding effectiveness when no separate acid-etching was carried out. Finally, all samples of OptiBond FL bonded to non-etched, Er:YAG-irradiated enamel were recorded as mixed failures, including enamel (Table 5).

When bonded to dentin, the µTBS means ranged from 6.9 MPa for Optibond FL when applied to non-etched, laser-irradiated dentin to 59.6 MPa for Optibond FL when applied to etched, bur-cut dentin (Table 2). Clearfil SE bonded least effectively to laser-irradiated dentin that was significantly different from all other experimental and control conditions (Table 2 and Figure 2). The µTBS of Clearfil SE to air-abraded dentin was significantly higher than all other experimental and control conditions except when bonded to SiC-paper prepared dentin and bur-cut enamel (nearly significantly different). Bonding of Clearfil SE to diamond-sonoabraded dentin was equally effective as when

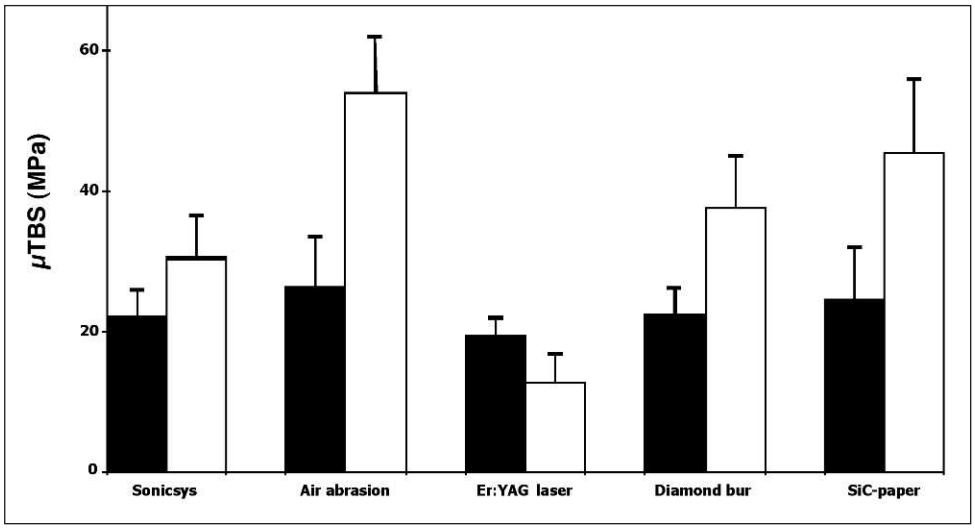

Figure 2. *Bar plot (+95% confidence interval) of µTBS results for Clearfil SE. Black bars represent results to enamel, white bars to dentin.*

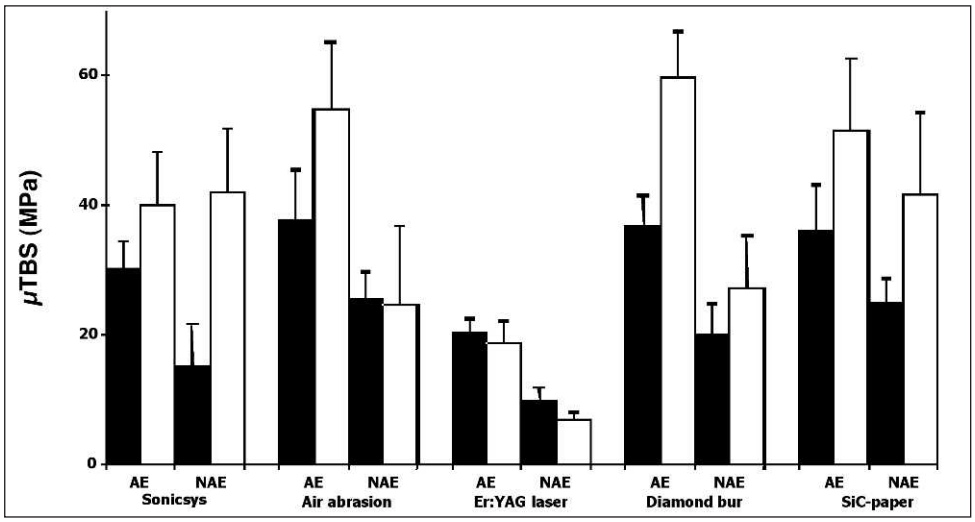

Figure 3. *Bar plot (+95% confidence interval) of µTBS results for Optibond FL. Black bars represent results to enamel, white bars to dentin.*

Table 2: *µTBS to Enamel and Dentin*

ENAMEL		SonicSys	Air Abrasion	Er:YAG Laser	Diamond Bur	SiC-Paper
Clearfil SE	µTBS (MPa) ± SD	22.0 ± 7.0[a]	26.3 ± 11.1[a]	19.2 ± 4.7[a]	22.4 ± 6.8[a]	24.5 ± 11.4[a]
	ptf/n	0/12	0/9	0/12	0/12	0/9
OptiBond FL (AE)	µTBS (MPa) ± SD	30.3 ± 7.5[A]	37.8 ± 12.9[A]	20.3 ± 4.0[B]	36.9 ± 9.8[A]	36.2 ± 11.5[A]
	ptf/n	0/12	0/11	0/12	0/17	0/10
OptiBond FL (NAE)	µTBS (MPa) ± SD	15.1 ± 10.7[a,b]	25.6 ± 7.2[b]	10.0 ± 3.2[a]	20.1 ± 8.1[b]	24.9 ± 7.3[b]
	ptf/n	2/12	0/11	0/11	0/11	0/13
DENTIN		**SonicSys**	**Air Abrasion**	**Er:YAG Laser**	**Diamond Bur**	**SiC-Paper**
Clearfil SE	µTBS (MPa) ± SD	30.4 ± 10.7[a]	54.1 ± 13.0[b]	12.7 ± 6.9[c]	37.7 ± 10.5[a]	45.5 ± 16.6[a,b]
	ptf/n	0/12	0/10	0/11	0/8	0/10
OptiBond FL (AE)	µTBS (MPa) ± SD	39.9 ± 13.4[A]	54.9 ± 16.3[A,B]	18.6 ± 5.5[C]	59.6 ± 16.8[B]	51.6 ± 19.6[A,B]
	ptf/n	0/10	0/10	0/9	0/21	0/12
OptiBond FL (NAE)	µTBS (MPa) ± SD	41.9 ± 16.7[a]	24.6 ± 17.6[a,b]	6.9 ± 1.2[b]	27.3 ± 13.6[a,b]	41.8 ± 21.2[a]
	ptf/n	0/11	3/11	5/9	0/11	0/11

SD = Standard deviation; ptf = pre-testing failures; n = total number of specimens measured; Data marked with same superscript are not significantly different from one another.

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bonded to bur-cut and SiC-paper prepared dentin, but significantly less effective than bonding to air-abraded dentin. Separate acid-etching significantly increased the µTBS of Optibond FL when bonded to air-abraded dentin, Er:YAG-irradiated and bur-cut dentin, but not to sonoabraded and SiC-paper prepared dentin (Tables 2, 3 and Figure 3). Following acid-etching (AE), OptiBond FL had the least effective bond to laser-irradiated dentin, while bonding to air-abraded dentin was not significantly different from both control conditions. The use of sonoabrasion resulted in a µTBS of Optibond FL to dentin that was only significantly lower than Optibond FL bonded to diamond-bur cut dentin (Table 2, and Figure 3). When enamel was not separately acid-etched (NAE), bonding of Optibond FL to Er-YAG irradiated dentin was least effective, although its µTBS was only significantly different from Optibond FL bonded to diamond-sonoabraded and SiC-paper prepared dentin (Table 2). However, when the high number of pre-testing failures of Optibond FL to Er:YAG irradiated dentin (five out of nine specimens) would have been included (µTBS=0 MPa), bonding of Optibond FL to non-etched, Er:YAG irradiated dentin would be significantly worse compared to all other experimental and control groups with the exception of bonding to non-etched, air- abraded dentin. Also, for the latter, three out of 11 specimens failed prior to being measured.

When bonding to dentin, the failure analysis of Clearfil SE revealed that (as when bonded to enamel)

Table 3: p-*Values Comparing OptiBond FL Applied with (AE) and without Acid-Etching (NAE) and Clearfil SE Versus OptiBond FL (AE) (Mann-Whitney U Test)*

	AE vs NAE		C-SE vs O-FL	
	Enamel	**Dentin**	**Enamel**	**Dentin**
SonicSys	0.0026	0.9177	0.0068	0.1072
Air Abrasion	0.0192	0.0031	0.0562	0.8534
Er:YAG	<0.0001	0.0028	0.7987	0.0674
Diamond bur	0.0001	<0.0001	0.0002	0.0008
SiC-paper	0.0099	0.2351	0.0350	0.3136

The significance level _ was set at 0.01 to correct for multiple comparison testing (Bonferroni correction); p-*values underlined are smaller than 0.01 and thus indicate significant difference;* p-*values underlined with a dashed line indicate nearly significant difference.*

Table 4: *Failure Analysis of Clearfil SE*

ENAMEL	**SonicSys**	**Air Abrasion**	**Er:YAG Laser**	**Diamond Bur**	**SiC-Paper**
Adhesive failure	0	0	2	4	0
Mixed failure including enamel	0	2	8	1	3
Mixed failure including resin	12	7	2	7	6
DENTIN	**SonicSys**	**Air Abrasion**	**Er:YAG Laser**	**Diamond Bur**	**SiC-Paper**
Adhesive failure	2	0	5*	1	0
Mixed failure including dentin	1	5	3	5	5
Mixed failure including resin	9	5	3	2	5

**Micro-cohesive in dentin*

Table 5: *Failure Analysis of OptiBond FL*

ENAMEL-Acid Etch	**SonicSys**	**Air Abrasion**	**Er:YAG Laser**	**Diamond Bur**	**SiC-Paper**
Adhesive failure	0	0	11	9	2
Mixed failure including enamel	3	2	1	0	2
Mixed failure including resin	9	9	0	8	6
ENAMEL-No Acid Etch	**SonicSys**	**Air Abrasion**	**Er:YAG Laser**	**Diamond Bur**	**SiC-Paper**
Adhesive failure	8	2	0	5	4
Mixed failure including enamel	0	3	11	4	0
Mixed failure including resin	2	6	0	2	9
DENTIN-Acid Etch	**SonicSys**	**Air Abrasion**	**Er:YAG Laser**	**Diamond Bur**	**SiC-Paper**
Adhesive failure	2	0	6*	3	0
Mixed failure including dentin	6	5	0	7	6
Mixed failure including resin	2	5	3	11	6
DENTIN-No Acid Etch	**SonicSys**	**Air Abrasion**	**Er:YAG Laser**	**Diamond Bur**	**SiC-Paper**
Adhesive failure	1	3	4*	9	2
Mixed failure including dentin	6	1	0	1	7
Mixed failure including resin	4	4	0	1	2

**Micro-cohesive in dentin*

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characteristic of all adhesive groups is the relatively high amount of adhesive and mixed failures, including dentin (Table 4). Again, when Optibond FL was bonded without acid etching, the number of adhesive failures increased with the exception of the sonoabrasion group (Table 5).

Comparing the bonding effectiveness to enamel of the two-step self-etch adhesive Clearfil SE with that of the three-step etch&rinse adhesive OptiBond FL (including acid-etching), Clearfil SE bonded significantly worse ($p<0.01$) to diamond sonoabraded and diamond-bur prepared enamel than OptiBond FL (Table 3). No significant difference in bonding of Clearfil SE and OptiBond FL was found when applied to Er:YAG-irradiated enamel, with bonding to air-abraded and SiC-paper prepared enamel approaching significant difference. At dentin (Table 3), Optibond FL bonded only significantly better to diamond-bur prepared dentin than Clearfil SE. In all other experimental and control conditions, Clearfil SE did not bond significantly differently from OptiBond FL.

Representative examples of failure modes imaged by Fe-SEM are shown in Figures 4-8. Fe-SEM photomicrographs of enamel and dentin subjected to the three experimental and two control tooth tissue preparation tools are shown in Figures 9-13.

DISCUSSION

In order to measure the bonding effectiveness to diversely prepared enamel and dentin surfaces, two commercially available adhesives that represent a three-step etch&rinse (OptiBond FL) and a two-step self-etch (Clearfil SE) approach were selected (Table 1; Van Meerbeek & others, 2001; 2003). Both adhesives have repeatedly been documented to consistently provide high bond strengths to enamel and dentin (Tay & others, 2000a; Armstrong, Keller & Boyer, 2001; Inoue & others, 2001b). Special attention was given to correct application procedures, in particular, to apply both adhesives to tooth substrates that were prepared in a standardized way, as previously described in detail (De Munck & others, 2002b). As a tool to prepare specimens for µTBS testing, the authors used the MicroSpecimen Former developed at the University of Iowa (Armstrong & others, 2001). This device greatly reduces the technique sensitivity of specimen preparation since any hand manipulation is excluded (De Munck & others, 2002a). It also enables the preparation of cylindrical µTBS specimens that optimally distribute internal stress and cause them to fail at the interface itself (Armstrong & others, 2001). In order to prepare a relatively flat surface using the SonicSys system, the air-abrasion unit and the Er:YAG laser, flat

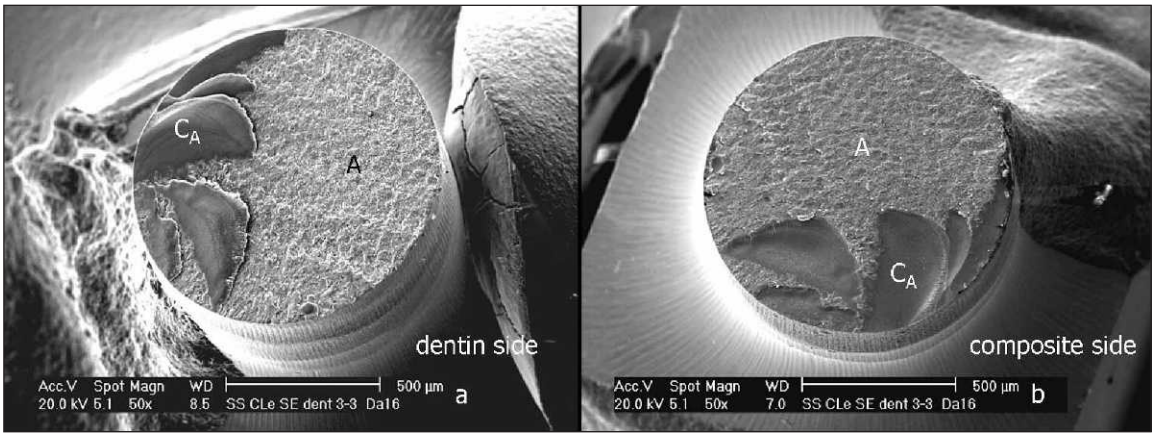

Figure 4. *Fe-SEM photomicrographs of a fractured dentin-adhesive interface with the dentin side in (a) and the composite side in (b) when Clearfil SE was bonded to diamond sonoabraded dentin. The specimen failed mixed adhesively (A) at the interface and cohesively within the adhesive resin (C_A).*

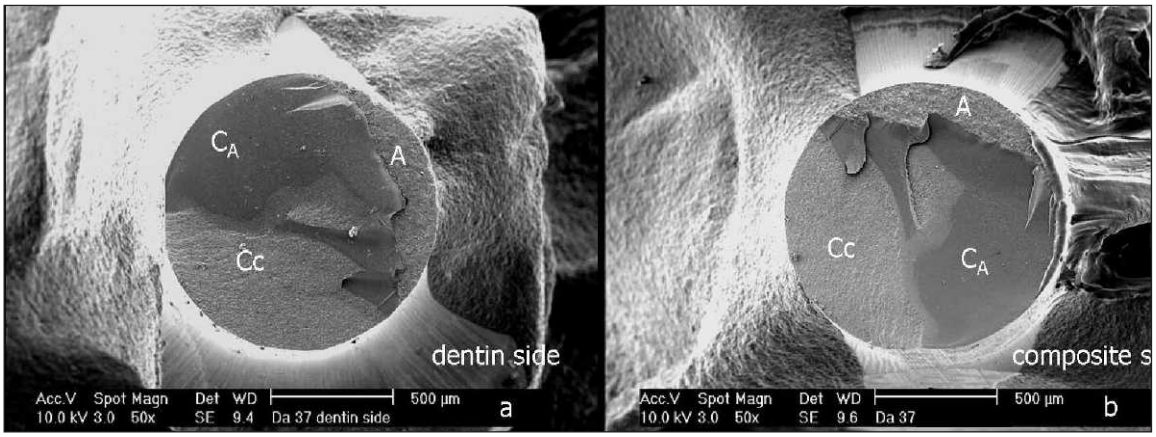

Figure 5. *Fe-SEM photomicrographs of a fractured dentin-adhesive interface with the dentin side in (a) and the composite side in (b) when Clearfil SE was bonded to air-abraded dentin. The interface failed mixed adhesively (A) at the interface and cohesively within the adhesive resin (CA) and within the resin composite (C_C).*

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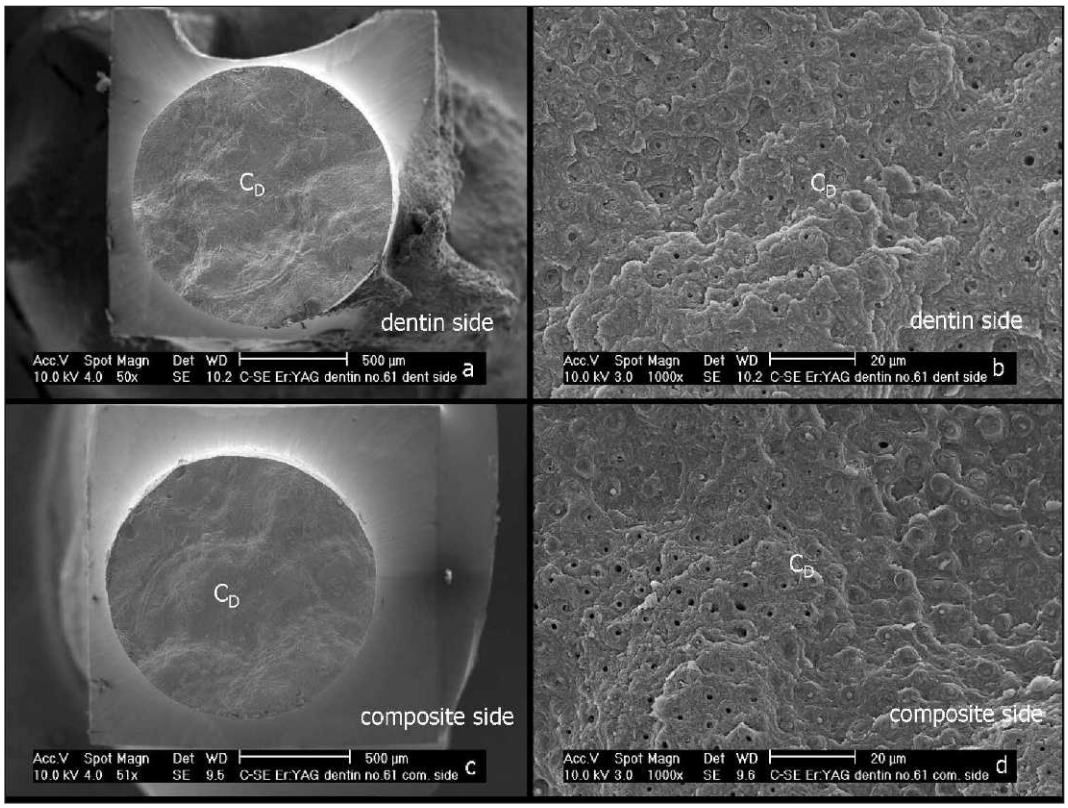

Figure 6. *Fe-SEM photomicrographs of a fractured dentin-adhesive interface with the dentin side in (a) and (b), and the composite side in (c) and (d) when Clearfil SE was bonded to Er:YAG-irradiated dentin. The interface failed uniformly cohesively within dentin (C_D).*

enamel and dentin surfaces were initially prepared using the diamond saw and/or wet sanding with 600-grit SiC-paper. Only a small layer was subsequently removed through respective diamond sonoabrasion, air abrasion and Er:YAG-laser irradiation. In this way, the authors expect to have prevented surface enlargement by irregularities and, thus, to have avoided a false-positive increase of bond strength. The Fe-SEM images confirmed that the enamel and dentin surfaces were uniformly prepared following the three experimental and two control conditions (Figures 9-13).

The results obtained in this study clearly demonstrate that separate acid etching prior to applying an etch&rinse adhesive is indispensable for enamel and dentin, by which the hypothesis advanced was rejected (Table 3). The only exceptions were the bonding of Optibond FL to diamond sonoabraded and to SiC-paper prepared dentin. A plausible explanation for the latter is that sonoabrasion and the use of 600-grit SiC paper produces a relatively thin smear layer (Figures 9 and 13). The primer of OptiBond FL is acidic (pH=1.78, measured using Inolab pH Level 2, WTW, Welheim, Germany) (De Munck & others, 2002a). Consequently, due to a self-etch effect induced by the primer, itself, a thin, submicron hybrid layer is produced on a 600-grit SiC-paper prepared dentin surface (unpublished observations). The rather thin smear layer produced by sonoabrasion and wet sanding with 600-grit SiC-paper did not obstruct the formation of a hybrid layer. As the thickness of the hybrid layer has been shown not to influence bond strength (Inoue S & others, 2001b), the mild self-etch effect of the OptiBond FL primer that resulted in hybrid-layer formation most likely explains that separate acid-etching of dentin did not result in a significantly higher µTBS in the case where OptiBond FL was applied to sonoabraded or SiC-paper-prepared dentin.

Comparing the bonding effectiveness of the conventional three-step etch&rinse adhesive OptiBond FL to the two-step, self-etch adhesive Clearfil SE to enamel (Table 3), the µTBS of Optibond FL exceeded Clearfil SE in all groups except Er:YAG-irradiated enamel, to which both adhesives bonded equally effectively. Due to the Bonferroni correction applied because of multiple comparison testing, significant differences were only found for sonoabrasion and diamond-bur preparation at the level of $p<0.01$ (Table 3). However, the p-values for air abrasion and SiC-paper preparation approached statistically significant differences. All together, the authors can, therefore, conclude that the "etch&rinse" approach using phosphoric acid remains the more reliable bonding technique for enamel, though the difference between the etch&rinse and self-etch approach becomes smaller (Inoue & others, 2000; Van Meerbeek & others, 2001, 2003; De Munck & others, 2002a). At dentin, only the diamond-bur preparation appeared to have hindered adequate micromechanical bonding through hybridization. This is not unexpected considering the regular-grit diamond the authors applied results in a relatively thick smear layer (Figure 12), as compared to the superficial smearing effect resulting from the other tissue preparation techniques and 600-grit SiC-paper, in particular. Such bur-dependent bonding effectiveness has repeatedly been reported for so-called "mild" self-etch adhesives (Inoue & others, 2000; Tay & others, 2000b; Inoue & others, 2001a; Van Meerbeek & others, 2001; De

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Munck & others, 2002a), while the "strong" self-etch and etch&rinse adhesives, thanks to their higher etching aggressiveness, are rather insensitive to the tooth surface preparation mode.

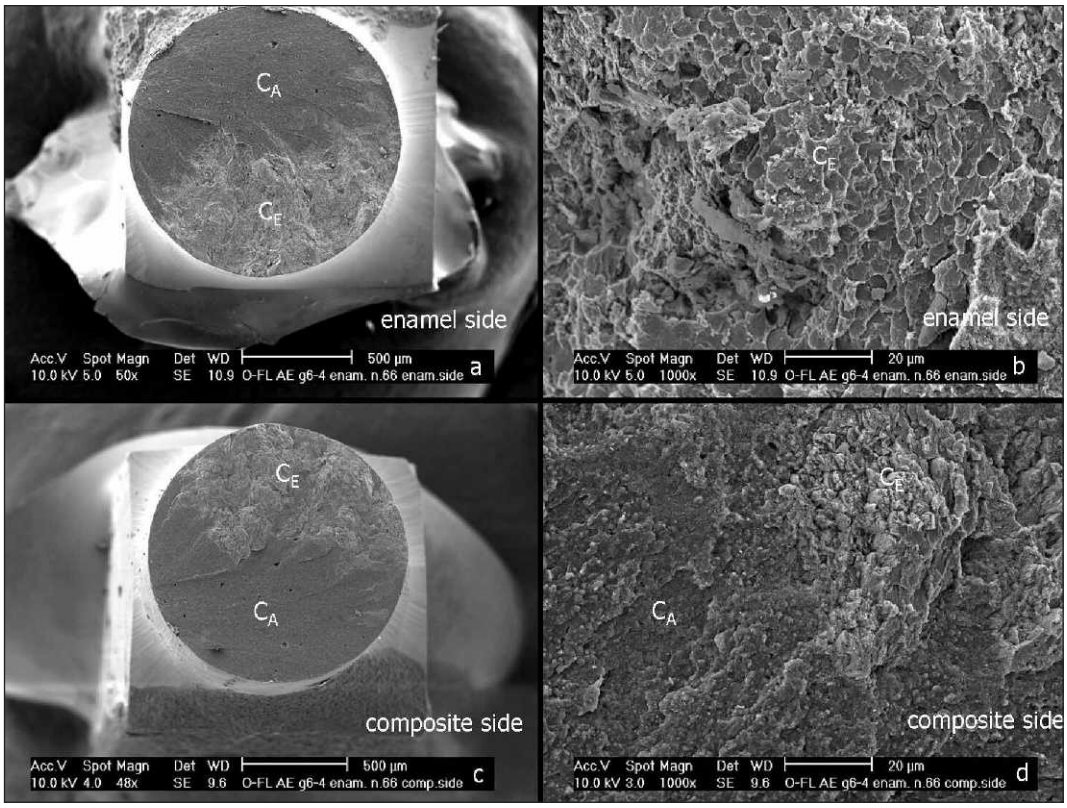

Figure 7. *Fe-SEM photomicrographs of a fractured enamel-adhesive interface with the enamel side in (a) and (b), and the composite side in (c) and (d) when OptiBond FL was bonded to acid-etched, Er:YAG-irradiated enamel. The interface failed cohesively within enamel (CE) and within the particle-filled adhesive (C_A).*

Diamond Sonoabrasion (SonicSys)

Except for OptiBond FL bonded to acid-etched dentin, diamond-sonoabrasion resulted in tooth surfaces that are equally receptive to bonding compared to conventional diamond-bur prepared surfaces.

No data with regard to bond strength of adhesives to diamond sonoabraded enamel and dentin are available in the literature. In a study by Setien and others (2001), SonicSys Approx (Kavo) was investigated among other cavity preparation devices. The authors of that study concluded that the way the cavity was prepared did not affect the sensitivity of the composite restoration for microleakage at the (acid-etched) enamel and dentin margins. Since SonicSys Approx only differs from SonicSys Micro with regard to the shape of the instruments' active end, the authors of this study believe that the latter data can be extrapolated to the SonicSys Micro tested in this study. Consequently, this latter study substantiates the bond strength data gathered in this study using diamond sonoabrasion.

Air Abrasion

Bonding to air-abraded enamel and dentin was not significantly different from bonding to conventional diamond-bur prepared tooth surfaces for either adhesive tested. Moreover, as mentioned above, the mild self-etch adhesive Clearfil SE bonded even significantly better to air-abraded dentin than to the coarse, thick, smear layer produced by the diamond bur.

In the literature, great controversy exists regarding whether air abrasion produces a micro-retentive surface that is sufficiently receptive to bonding so that acid etching is no longer needed. Several studies showed that air abrasion alone yielded results equal to or superior to acid etching. Laurell, Lord and Beck (1993) reported no significant difference in shear bond strength to enamel between an acid-etched and solely air-abraded surface. Likewise, Keen, von Fraunhofer and Parkins (1994) obtained similar shear bond strengths to air-abraded and acid-etched (35% phosphoric acid) enamel, and even higher bond strengths to dentin obtained with air abrasion compared with acid etching. Also, Doty and others (1994) concluded that air abrasion technology had the potential to prepare enamel bonding surfaces equal to those obtained from acid etching.

However, many papers reported the contrary. Roeder and others (1995) reported that acid etching of enamel and the priming of dentin was necessary to achieve maximum bond strengths to air abraded tissues. In addition, according to a study by Berry and Ward (1995), tensile bond strengths of resin composite to acid-etched, air-abraded enamel were significantly greater than those of unetched, air-abraded enamel. Also, a shear bond strength study by Bae and others (1996) showed that "air abrasion" conditioning was less effective than conventional acid etching. Canay, Kocadereli and Akça (2000) found that enamel preparation through solely sandblasting using a micro-etcher resulted in a significantly lower bond strength than conventional acid-etch-

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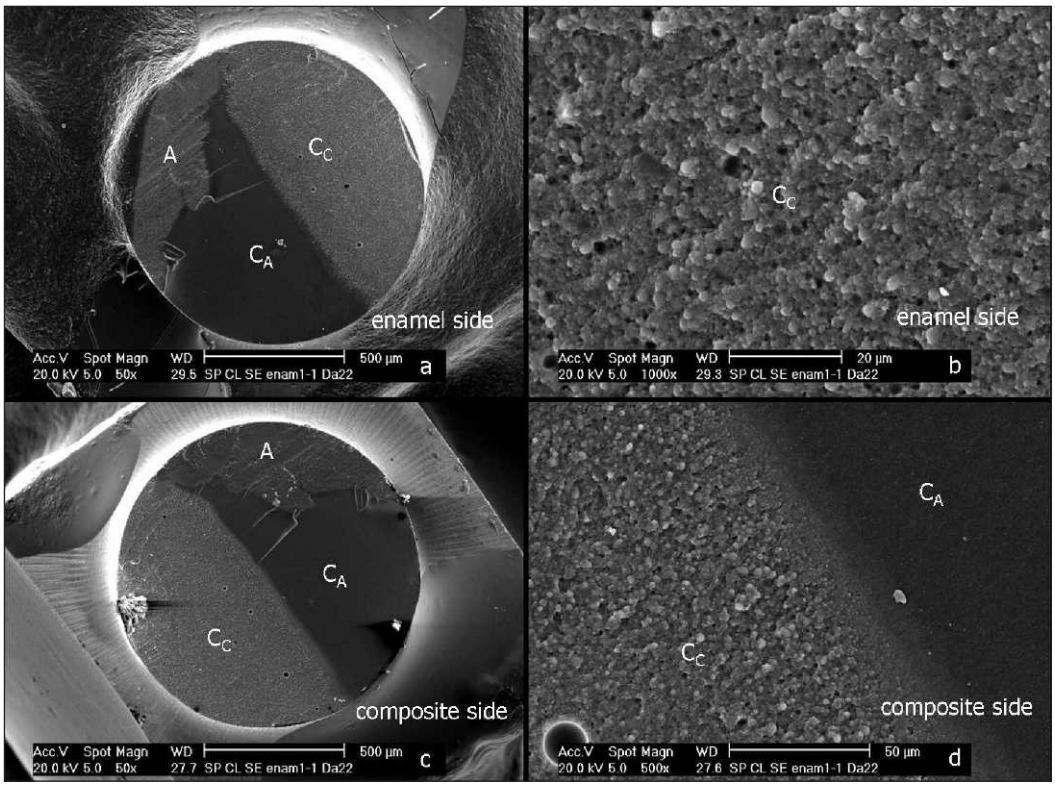

Figure 8. *Fe-SEM photomicrographs of a fractured enamel-adhesive interface with the enamel side in (a) and (b), and the composite side in (c) and (d) when Clearfil SE was bonded to SiC-paper prepared enamel. The interface failed mixed adhesively at the interface (A), cohesively within the adhesive (C_A) and cohesively within the resin composite (C_C).*

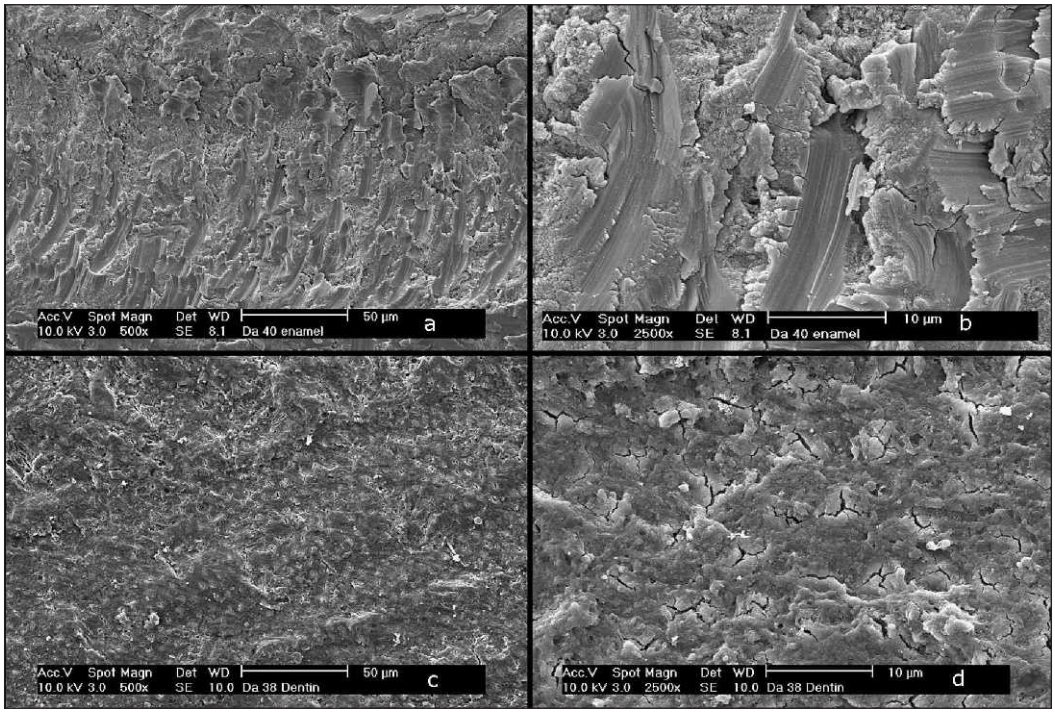

Figure 9. *Fe-SEM photomicrographs of diamond sonoabraded enamel in (a) and (b), and of diamond sonoabraded dentin in (c) and (d). Note the clear marks at the enamel surface resulting from sonoabrasion. At the dentin surface, sonoabrasion resulted in a relatively thin smear layer with the dentin tubules almost completely closed.*

ing and, therefore, should not be advocated for clinical use as an enamel conditioner. Finally, Valentino and Nathanson (1996) concluded that air abrasion could be effective for dentin bonding when used in conjunction with acid etching but should not be used as a sole surface treatment prior to bonding composite to enamel or dentin. All the latter studies agree with the present results, suggesting that it remains mandatory to separately acid-etch air-abraded enamel and dentin when applying an etch&rinse adhesive.

Er:YAG Laser

Thanks to the ablation process that involves micro-explosions of tooth substrate and the greater volatilization of intertubular compared to peritubular dentin (Armengol & others, 1999a), the Er:YAG laser exposes a typical imbricate patterned surface (Kameyama & others, 2001) with open dentin tubuli (Figure 11). Moreover, this ablation process leaves no hydroxyapatite-depleted collagen on the surface. Consequently, when the laser-conditioned tissue is not separately acid-etched, collagen is not exposed and, consequently, no hybrid layer can be formed (Kataumi & others, 1998), also eliminating the need for an hydrophilic dentin primer to penetrate within the organic tissue (Armengol & others, 1999b). Despite this different approach, laser manufacturers commonly argue that at low-energy settings enamel and dentin can be conditioned with the

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Er:YAG laser when replacing conventional acid-etching as pretreatment for adhesive procedures. However, bond strengths to Er:YAG-irradiated tooth substrate reported in the literature are often confusing and even contradictory.

Kataumi and others, (1998) observed resin-dentin interfaces and found microcracks below the hybrid layer, indicating that subsurface damage was caused by Er:YAG irradiation. These findings confirm the authors' previous study that demonstrated using Fe-SEM resulted in significantly more microcracks at laser-irradiated rather than at fractured dentin surfaces (De Munck & others, 2002b). Very conclusive evidence of deficient hybridization to Er:YAG lased dentin was provided in a recent study using transmission electron microscopy by Ceballo and others (2002). The results showed a 3-4 µm severely altered dentin subsurface beneath which collagen fibrils appeared to have lost cross-banding and were fused together, eliminating interfibrillar spaces and, thus, impeding hybridization.

This subsurface damage caused by laser irradiation can also exceed the thickness of the hybrid layer, leaving a weakened substrate without resin reinforcement underneath. This, in particular, could explain the cohesive fractures of dentin and enamel frequently observed in this and in other studies (Martinez-Insua & others, 2000; Kameyama & oth-

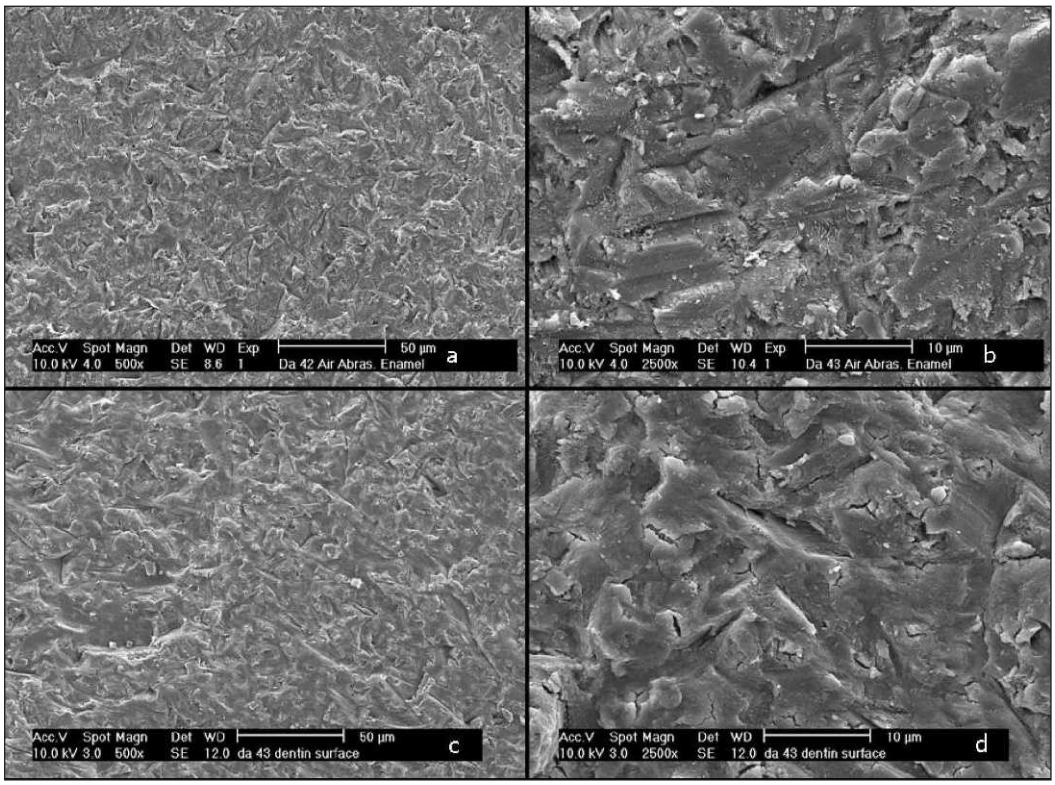

Figure 10. *Fe-SEM photomicrographs of air-abraded enamel in (a) and (b), and of air-abraded dentin in (c) and (d). Note at dentin that air abrasion resulted in an irregular surface without any patent tubules.*

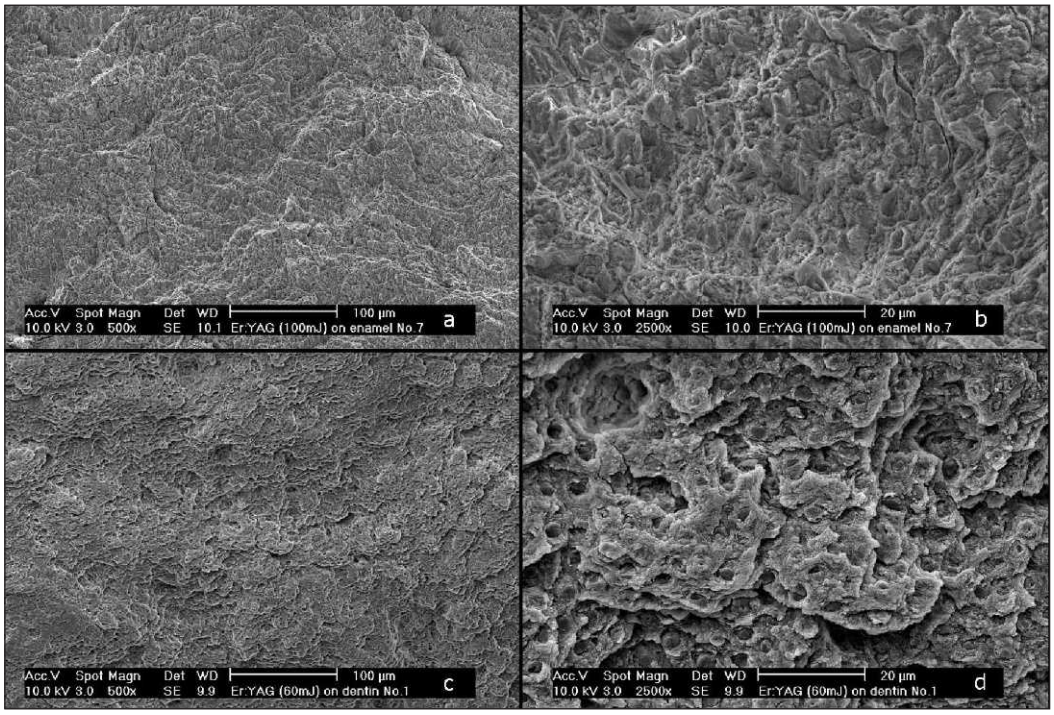

Figure 11. *Fe-SEM photomicrographs of Er:YAG-irradiated enamel in (a) and (b), and of Er:YAG-irradiated dentin in (c) and (d). (a,b) The surface is very rough and irregular. The enamel prisms are clearly visible, but appear to have been partially melted at their exposed ends. Micro-cracks in between enamel prisms can be observed. (c,d) Dentinal tubules are clearly visible and are not covered by a smear layer. The surface is very rough and irregular with a typical imbricate patterned topography. Also a lot of microcracks can be observed.*

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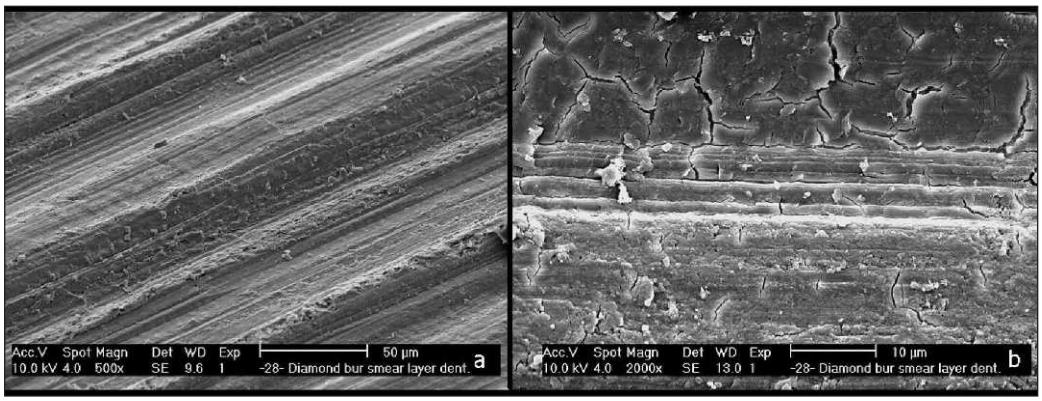

Figure 12. *Fe-SEM photomicrographs of diamond-bur prepared dentin. Note the rather coarse scratches across dentin as part of a rather thick smear layer produced by the diamond bur. All dentin tubules are closed.*

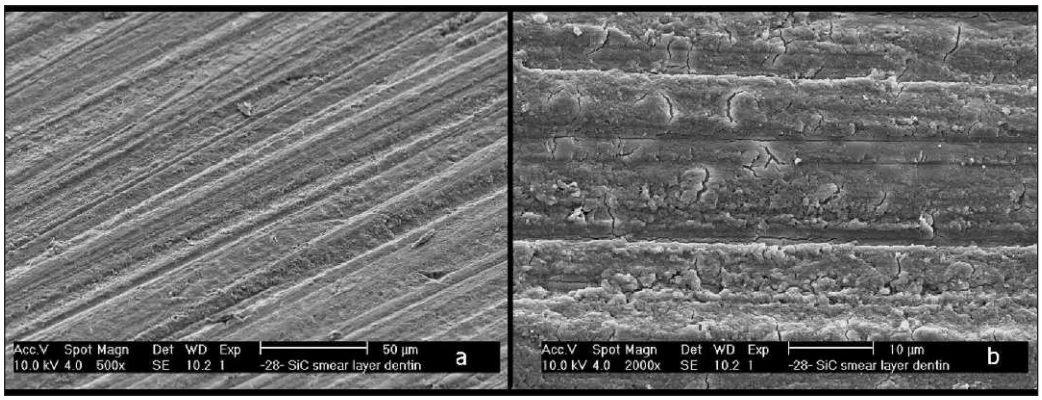

Figure 13. *Fe-SEM photomicrographs of SiC-paper prepared dentin. Note the scratches produced by wet-sanding dentin with SiC paper. The scratches are less irregular and deep than those produced by the diamond bur in Figure 12. All dentin tubules are closed.*

ers, 2000). A tensile bond strength study of Clearfil SE applied to Er:YAG irradiated bovine enamel and dentin revealed that treatment with phosphoric acid, air-scaler, ultrasonic scaler and air-abrasion after Er:YAG irradiation improved bond strength (Eguro & others, 2001, 2002). All these surface treatments remove a small layer of the surface, so that they may eliminate possible drawbacks related to the aforementioned surface alterations and, thus, support the hypothesis that Er:YAG irradiation causes subsurface damage, compromising the hybridization effectiveness.

CONCLUSIONS

The manner of preparation of enamel and dentin surfaces prior to bonding procedures significantly influences the bonding effectiveness of the etch&rinse and the self-etch adhesive. Using an etch&rinse adhesive, separate acid-etching of sonoabraded, air-abraded and Er:YAG-irradiated enamel and dentin surfaces remains mandatory. Bonding to diamond-sonoabraded and air-abraded enamel and dentin is, in general, not different from bonding to conventional diamond-bur prepared surfaces, whereas, bonding to Er:YAG-irradiated enamel and dentin surfaces, in general, results in significantly lower bonding effectiveness as compared to bonding to diamond-bur prepared surfaces. Subsurface damage initiated by Er:YAG ablation is most likely the major reason for the decrease in µTBS and, thus, might compromise clinical bonding on the long-term.

Acknowledgements

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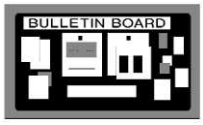

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Corrections

In ***Operative Dentistry***, 2003, 28-4, pgs 325-326, a line was missing from the editorial, *Focus*. The paragraph should read:

The dental profession has an enviable track record for working to eliminate dental caries, but we can't rest on the laurels of fluoride. For years we have lived with the debate as to whether dentists are really doctors or just technicians who place restorations. Obviously, we need to be an amalgam of both. From my perspective, good dentists are a combination of diagnostician, healer, technician and artist… but if we want to truly be health care providers, our goal of excellence must be focused on the elimination of dental disease and not just treatment of its ravages.

Also in the same issue of ***Operative Dentistry***, 2003, Dentin Bonding: Effect of Degree of Mineralization and Acid Etching Time by GC Lopes, LCC Vieira, S Monteiro Jr, M Caldeira de Andrada and CM Baratieri, which appeared on pgs 429-439, Figure 5 on pg 434 was incorrectly shown. The correct figure and legend follow.

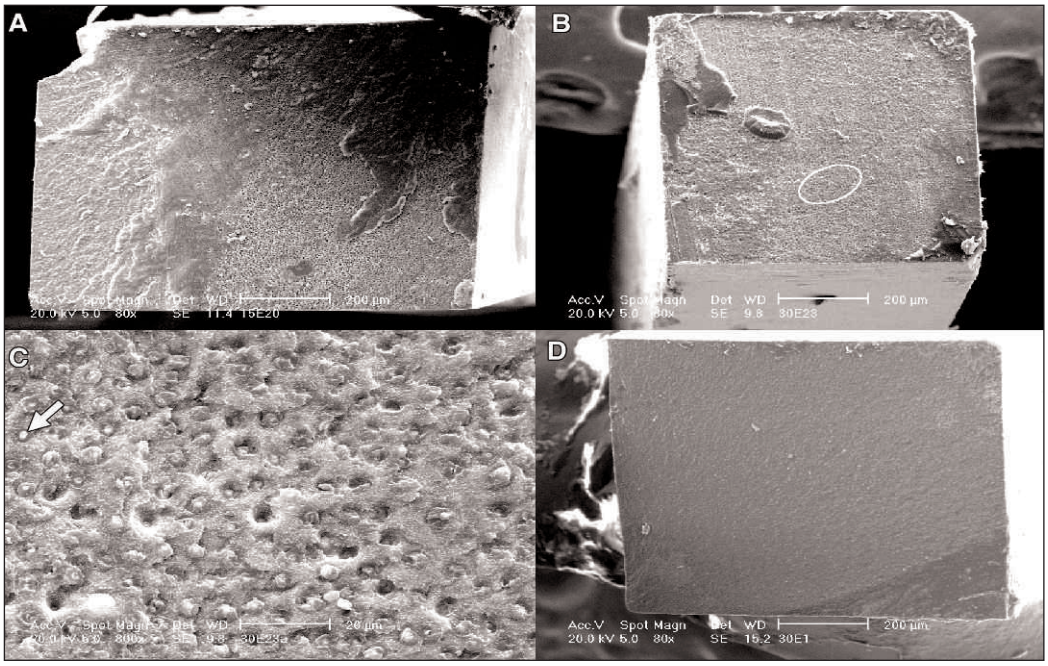

Figure 5. *SEM of the fractured surface sclerotic dentin. (A) Mixed failure typically in G15S. (B) Mixed failure in G30S. (C) Higher magnification of the area indicated by the circle in Figure 5B. Note the presence of hybridization next to the tubule walls (arrow), but having the central portion containing mineral deposits which were not dissolved during 35% PA etching for 30 seconds. (D) Complete cohesive failure in adhesive in G30S.*

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