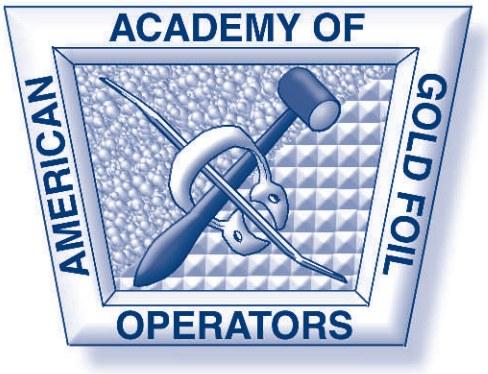
OPERATIVE DENTISTRY



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

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In Memoriam

Dr Floyd Eugene Hamstrom

Floyd Eugene Hamstrom
1911-2004

A true teacher and leader in dentistry was lost on October 11, 2004 when Dr Floyd Hamstrom, age 93, passed away peacefully at the Mountain Glen Retirement Center in Mount Vernon, Washington. His love of his profession was manifest throughout his career as he actively pursued the goal of excellence and patient satisfaction throughout his life. While on this journey, Floyd was a charter member of the Washington Foundation of Dental Education, American Academy of Gold Foil Operators, Academy of Operative Dentistry and a founding member of the American Board of Operative Dentistry, serving as secretary-treasurer for the first five years.

Floyd was born on August 12, 1911 in Quincy, Washington, to Preston and Erika Hamstrom. He received his early education in Seattle and, after graduation from Ballard High School, he enrolled at North Pacific College of Dentistry (now the University of Oregon) in Portland. Following his graduation in 1935, he established his first dental practice in Seattle, Washington. In 1940, he joined the United States Navy, serving through 1945. These years of duty included a term of sea duty aboard the aircraft carrier USS Enterprise. He married Anita Eve Easton on December 20, 1942 and, upon his discharge from the Navy, established his dental practice in Burlington, Washington.

Floyd's genuine concern for his patients and family was manifest in everything he did. Living in the small town of Burlington, he was well known by everyone in the community for his caring nature and professional excellence. When I interviewed with Floyd in 1963 for the privilege of renting an adjoining office in his dental building, he simply wanted to know if I did "rubber dam dentistry." He was emphatic that every patient was important and that excellence could only be accomplished by dentists who were concerned enough with the care of their patients that they would routinely use the rubber dam. His unyielding support of our profession was further exemplified by his active participation in local and national dental societies. He was an Associate Professor Operative Dentistry at the University of Washington from 1953-1956 and he served a three-year term on the Washington State Board of Dental Examiners. Dr Hamstrom served as both president and secretary of the Associated Gold Foil Study Clubs of Washington and British Columbia and was president of the Mt Baker Dental Society. Floyd was a lifetime member of the Washington Gold Foil Study Club and, starting in 1957, was the mentor of the DA Spratley Gold Foil Study Club.

In retirement, Floyd enjoyed fishing, golfing and traveling. He was a 32nd Degree Mason of Lodge #243 in Burlington, Washington, having recently celebrated 50 years of membership. He was also a member of the Skagit Golf and Country Club and the Allen United Methodist Church.

He is survived by two daughters; Sally Dutton of Nevada City, California and Susan Reault of Pullman, Oregon; three grandchildren, Rebecca Dutton, Erik Reault and Kyle Reault; one great-grandchild, Madison Erika Reault, and his nieces Jeanne McGrath of Seattle, Washington and Sharon Beckman of Chicago, Illinois.

Floyd will always be remembered for his dedicated mentoring to all who were associated with him. His legacy is quality dentistry, and all who were "touched" by Floyd will never forget the lessons we learned from him about the utmost importance of quality patient treatment and dedication to the future of our profession.

Richard B McCoy

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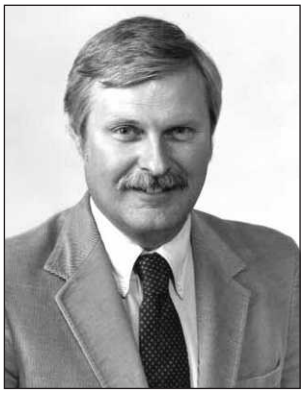
Guest Editorial

Dental amalgam is 50% mercury…or is it?



John W Osborne,
DDS, MSD

How many times have we heard that dental amalgam is 50% mercury? I am not going to debate whether amalgam is 50% or 54% or 39% Hg. The implication of the statement "dental amalgam is 50% mercury" is that all this mercury is readily available to the patient. Frankly, this is a misrepresentation and an exaggeration, either intentionally or naively.

Dental amalgam is a metallic compound. The bonds in this core-matrix complex are very stable and exhibit very high strength characteristics. The weakest portion of the amalgam is the Ag_2Hg_3 matrix (gamma-1 to a dentist or Moschellandsbergite to a geologist). These covalent, ionic and/or intermetallic bonds are very difficult to break. Only very heavy pressure or high heat can potentially cause the bonds in the matrix to come apart.

When chewing, pressures of 30,000 psi or higher are commonly obtained in the oral cavity and the high friction can generate heat in small areas. It is theorized (Mitchell, 2004) that high forces and/or subsequent heat could be the reasons that amalgams release small amounts of mercury during their lifetime. The amount of mercury released is in the range of 1.7 millions of a gram (µg) per day of 12 amalgams (Berglund, 1990).

To illustrate the amount of mercury in and what is coming off of amalgams, I have taken two pictures. Figure 1 is 7.2 grams of mercury in my gloved hand. This is the amount of mercury that would be needed to make 12 average size amalgam restorations, or 12 double spill, 600 mg alloy capsules with a 50% mercury content. Figure 2 is 25 mg of mercury in my gloved hand. This is the amount of mercury that clinical research (Berglund, 1990) has shown to come off 12 amalgams during 40 years of clinical service.

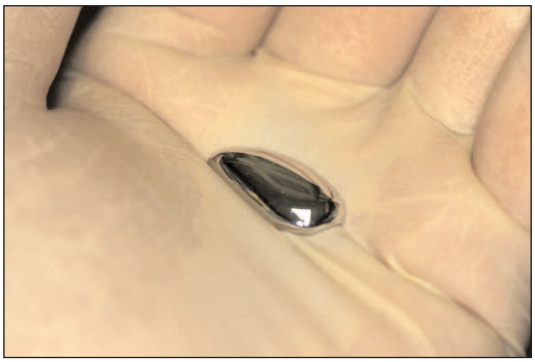


Figure 1.

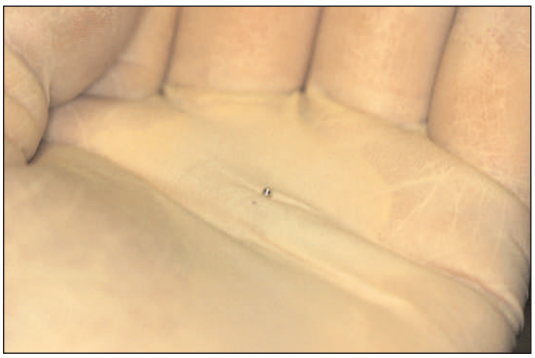


Figure 2.

Dental amalgam is 50±% mercury, but, as a metallic compound, only very small amounts of that mercury are available to the patient over a very long period of time.

John W Osborne

References

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Clinical Research

Three-year Clinical Evaluation of a Compomer and a Resin Composite as Class V Filling Materials

JR Gallo • JO Burgess • AH Ripps • RS Walker
EJ Ireland • DE Mercante • JM Davidson

Clinical Relevance

Silux Plus, a microfilled composite, retained its surface finish better than F2000, a compomer; no other statistically significant differences were found. The retention rate for F2000 and Single Bond was 100%; for F2000, the self-etching primer was 96.6% and for Silux Plus with Single Bond, the retention rate was 90.3%. A compomer can be retained well with a self-etching primer; however, all three combinations were satisfactory at three years.

SUMMARY

The purpose of this study was to evaluate the placement of two restorative materials, including a compomer (F2000, 3M ESPE) and a resin composite (Silux Plus, 3M ESPE), in non-carious cervical lesions using a self-etching bonding agent (F2000 self-etching primer/adhesive) and a fifth generation bonding agent (Single Bond, 3M ESPE) and to evaluate and compare these restorations for marginal discoloration, secondary caries, anatomical form, retention, surface texture and marginal adaptation at baseline and annually for three years. F2000 and Silux Plus were used to restore the teeth with moderate-sized non-carious cervical lesions. F2000 was placed using two different bonding agents: F2000 self-etching primer/adhesive (F2000SE group) and Single Bond (F2000SB group); Silux Plus was placed as a control using Single Bond (SiluxSB group). Thirty restorations of each material/dentin adhesive combination were placed. All restorations were evaluated at baseline and annually for three years using a modified USPHS scale. At the end of the three-year recall, Silux Plus had significantly better surface texture than F2000 ($p<0.0001$). In addition, marginal adaptation significantly worsened over time starting at one year, as compared with baseline, for all groups ($p<0.0001$). When anatomic form was compared between F2000 and Silux Plus, the p-value was 0.085, demonstrating that F2000 was slightly better than Silux Plus.

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Likewise, when comparing marginal adaptation between the F2000SE and SiluxSB groups, the *p*-value was 0.064, demonstrating that F2000 with the self-etching primer had better margins than Silux Plus with Single Bond. No other differences were found among the groups.

INTRODUCTION

Fluoride-releasing restorative materials are generally classified as resin composites, conventional and resin-modified glass ionomers or compomers (Hicks & others, 2002). They can be categorized by their setting reactions into distinct groups of restorative materials, with each group having advantages and limitations. Since a major problem with restorative dentistry is replacement of restorations because of secondary caries (Mjör, 1997; Hicks & others, 2003) and since fluoride is effective in secondary caries prevention (Attar & Onen, 2002; Hicks & Flaitz, 2000), the release of fluoride from restorative materials can be advantageous.

Since their introduction to dentistry in the early 1970s (Wilson & Kent, 1972), the use of glass ionomer restorative materials has increased (Hicks & others, 2003). Several factors have contributed to their acceptance, including biocompatibility, adhesion to tooth structure and their ability to release, absorb and re-release fluoride (Kasuyama, Ishikawa & Fujii, 1993; McLean & Wilson, 1988; Swift, 1986). Since glass ionomers shrink minimally during setting and have a coefficient of thermal expansion similar to tooth structure (Burgess, Norling & Summitt, 1994), restorations made with these materials have little leakage (Schwartz, Anderson & Pelleu, 1990). Unfortunately, conventional glass ionomers have limitations. They are brittle, have poor wear resistance in occlusal load-bearing areas and are technique sensitive, because of their two-stage setting reaction. They are sensitive to moisture during the initial reactive stage and to desiccation as the material hardens after the initial setting. Bayne (1992) reported that tooth sensitivity may occur with glass ionomer restorations because of material degradation and manipulation problems. However, glass ionomer restorations sorb fluoride and release it at a high rate for several days following topical fluoride application (Alvarez, Burgess & Chan, 1994; Gao & Smales, 2001; Perrin, Persin & Sarrazin, 1994).

To improve the physical and clinical handling properties of conventional glass ionomers, resin-modified glass ionomers were developed and introduced to dentistry (Hunt, 1994). These materials are less technique sensitive than conventional glass ionomer restoratives and have the same fluoride release and recharge capability (Gao & Smales, 2001; Burgess & Summitt, 1993; Nunez, Burgess & Chan, 1997; Rothwell, Anstice & Pearson, 1998; Tam, Chan & Yim, 1997). In addition, resin-modified glass ionomers have higher early strength, increased fracture toughness and improved bond strength to tooth structure compared to conventional glass ionomers (Sidhu & Watson, 1995; Attin, Vataschki & Hellwig, 1996).

Compomers are resin composites with glass ionomer fillers (Burgess & others, 1996; Burgess, Walker & Davidson, 2002; Burgess & Xu, 1998). Fluoride is released from aluminosilicate glass fillers. These materials are primarily light-cured fluoride-releasing resin composites that combine the advantages of glass ionomer cements and resin composites. The fluoride release and recharge of compomers, glass ionomers and fluoride-releasing resin composites have been measured (Burgess & others, 1996; Xu & Burgess, 2003). Fluoride-releasing resin composites have the poorest fluoride release and recharge; glass ionomers have the highest fluoride release and recharge, while compomers are intermediate between the two. Glass ionomers are fluoride reservoirs, and the fluoride may be replenished from fluoride sources such as toothpaste, mouth rinses or topical fluoride solutions. These restorative materials are therefore recommended for high caries risk individuals. Unfortunately, glass ionomers are not popular in the United States, where single paste light-cured restorative materials dominate the market.

Clinical trials have reported the favorable performance of compomer restorations in non-carious cervical lesions (Barnes & others, 1995; Van Meerbeek & others, 1994). The clinical success of Dyract (LD Caulk, Milford, DE, USA), a compomer, as a Class V restorative material in non-carious cervical lesions is well-documented (Abdalla, Alhadainy & García-Godoy, 1997; Jedynakiewicz, Martin & Fletcher, 1997; Loher, Kunzelman & Hickel, 1997). Compomers such as Dyract AP have evolved with better mechanical properties and higher fluoride release capabilities. This three-year study evaluated the clinical performance of F2000, a light-cured compomer restorative material used to restore non-carious cervical lesions and compared its performance to Silux Plus (3M ESPE, St Paul, MN, USA), a microfilled resin composite.

METHODS AND MATERIALS

Thirty dental patients were selected for this study from advertisements posted at the Louisiana State University School of Dentistry and in newspapers. Subjects were included in this IRB-approved protocol after they read, understood and signed the consent form allowing their inclusion in the study. Criteria used to select subjects included the presence of non-carious cervical lesions and the absence of severe medical complications, rampant caries, xerostomia and chronic periodontitis.

A compomer and resin composite were used to restore teeth with moderate-sized non-carious cervical lesions. The compomer was placed with two different bonding agents and the resin composite was placed with one bonding agent as a control. Two clinicians inserted 30 Class V restorations of each material/dentin bonding agent combination for a total of 90 restorations. To limit and control the forces received by each restored tooth, all restored teeth were required to be in occlusion and have at least one proximal surface in contact with an adjacent tooth. A local anesthetic, 2% lidocaine with 1:100000 epinephrine, was used when needed to prevent discomfort. If the use of epinephrine was contraindicated, 3% mepivacaine was used. Following rubber dam isolation, cavity preparation (beveling the enamel margin) was done for all restorations using a high-speed handpiece with carbide burs. The lesions were cleaned with a slurry of medium pumice in a rubber cup, rinsed and dried with a cotton pellet to assure a moist surface. In the F2000SB group, the enamel and dentin were etched with phosphoric acid supplied by the manufacturer for 15 seconds, rinsed for 10 seconds and Single Bond was applied followed by the compomer (F2000). In the F2000SE group, the enamel and dentin were covered with F2000 self-etching primer/adhesive followed by the compomer. In the SiluxSB group, the enamel and dentin were etched with phosphoric acid for 15 seconds, rinsed for 10 seconds and Single Bond was applied followed by the resin composite (Silux Plus). Manufacturer's directions were followed for all materials. The compomer and resin composite were inserted using an incremental fill technique beginning at the gingival floor. Additional increments were added to complete the proper anatomical form, as necessary. Each increment was cured for 40 seconds with a Coltolux 4 visible light-curing unit (Coltene/Whaledent, Akron, OH, USA). The output was measured with the unit's built-in radiometer and the output for each restoration was recorded on the data sheet. The power density of the curing unit never dropped below 650 mW/cm^2. Carbide finishing burs used at high speed with water were used to remove gross excess, followed by finishing strips and discs (Sof-Lex, 3M ESPE), where necessary. All polishing was done at slow speed without water spray. All times were measured with a stopwatch to ensure compliance.

Each restoration was evaluated directly and indirectly at baseline (one week after placement), six months and annually for three years. The direct clinical evaluations were made using a modification of the USPHS guidelines (Table 1). Each restoration was evaluated for marginal discoloration, secondary caries, anatomical form, retention, surface texture and marginal adaptation. Impressions were made of the restorations at baseline and at all recalls. The impressions were allowed to set for 24 hours, then poured into dental stone. Separated casts were examined to determine marginal and anatomical breakdown from the previous recall and to resolve any discrepancies. To remove bias, evaluators did not evaluate the restorations they had placed during the clinical trial.

The categorized clinical assessment data was summarized by computing percentages in each category for the three treatment groups. The scores for retention, marginal discoloration and secondary caries were compared among the treatment groups using conditional logistic regression, which allowed the correlation

Table 1: *Modified USPHS Rating Systems Used in Evaluating the Class V Restorations*

Marginal Discoloration	Alpha	No discoloration anywhere along the margin.
	Bravo	The discoloration has not penetrated in a pulpal direction along the margin.
	Charlie	The discoloration has penetrated in a pulpal direction.
Secondary Caries	Alpha	No caries present.
	Charlie	Caries present.
Anatomic Form	Alpha	The restoration is continuous with the existing anatomic form.
	Bravo	The restoration is discontinuous with existing anatomic form, but the missing material is not sufficient to expose dentin or base.
	Charlie	Sufficient material is lost to expose dentin or base.
Retention	Alpha	The restoration is present.
	Charlie	The restoration is absent.
Surface Texture	Alpha	The restoration is as smooth as the adjacent tooth structure.
	Bravo	The restoration is rougher than the adjacent tooth structure.
	Charlie	The restoration is rougher than the adjacent tooth structure and contains pits and fissures.
Marginal Adaptation	Alpha	An explorer does not catch in either direction.
	Bravo 1	The explorer catches in only one direction.
	Bravo 2	The explorer catches and a crevice exists into which the explorer penetrates. Dentin not exposed.
	Charlie	A crevice is present that exposes dentin.

within subjects due to the design (multiple treatments per subjects and/or multiple teeth per patient) to be modeled. Analysis was carried out in LogXact Version 4 (Cytel Software). Scores for marginal adaptation, anatomical form and surface texture were also compared among treatment groups using generalized estimating equations (GEE1) that allowed correlation within subjects due to the design to be modeled. This analysis was done using SAS Version 9, using the GENMOD procedure specifying the binomial distribution and logit link function. Fixed effects were included for treatment and time, while the subject was specified as the random factor.

The study was designed to compare the three treatments in a split-mouth design. Due to the small sample size and correlated data structure (split-mouth design), an analysis was conducted similar to that proposed by Jones and Kenward (1987) and Kenward and Jones (1991) for crossover studies for the measures of retention, marginal discoloration and secondary caries. The primary difference from a crossover trial is that, in the split-mouth model, carryover effects cannot be modeled as separate terms since they are confounded with the treatment effects and, hence, must be considered negligible for meaningful inference. The response structure for marginal adaptation, anatomical form and surface texture permitted the more complex GEE1 repeated measures analysis to be performed. In all analyses, a significant difference among treatments would imply that one of the treatments had a higher probability of a better outcome.

Two separate conditional logistic analyses were conducted on the dichotomized response variables (retention, marginal discoloration and secondary caries), as the sample size was, in general, too small to support analysis on the original ordinal scale (for example, Alpha, Bravo, Charlie). In the first analysis, each response variable was dichotomized based on clinical significance. Response categories of each response variable were classified as having or not having clinical significance. This resulted in classifying only responses in the "Charlie" category as clinically significant for all variables. In the second analysis, each response variable was dichotomized as "Alpha" and "greater than Alpha."

RESULTS

The categorized clinical assessment data summarized over time as percentages for each category and all three treatment groups appears in Table 2. Comparisons among treatments are summarized through *p*-values appearing in Tables 3 and 4 for retention, marginal discoloration and secondary caries. Comparisons involving marginal adaptation, anatomical form and surface texture appear in Tables 5 and 6 (time comparisons). Analyses were adjusted for baseline response levels where possible by including the baseline variable as a covariate. There were no significant differences ($p>0.05$) among groups for retention, marginal discoloration and secondary caries.

For analysis of anatomical form and surface texture, all F2000 restorations, regardless of the bonding agent used, were considered as one group compared to Silux Plus. For the GEE analyses involving marginal adaptation, anatomical form and surface texture, Silux Plus had a significantly smoother surface than F2000 ($p<0.0001$). Marginal significant differences were observed between F2000 and Silux Plus on anatomical form ($p=0.085$, favoring F2000) and between F2000SE and SiluxSB on marginal adaptation ($p=0.0644$, favoring F2000SE). Marginal adaptation significantly worsened over time starting at 12 months, compared with

Table 2: *Percent of Patients in Each Response Category Arranged by Characteristic, Material and Time*

		Alpha					Bravo (/Bravo2)					Charlie				
VARIABLE	**Material**	**BL**	**6 mo**	**12 mo**	**24 mo**	**36 mo**	**BL**	**6 mo**	**12 mo**	**24 mo**	**36 mo**	**BL**	**6 mo**	**12 mo**	**24 mo**	**36mo**
Marg Disc	F2000SE	100	100	93.1	86.2	82.1	0/0	0	6.9	13.8	17.9	0	0	0	0	0
	F2000SB	100	93.6	90.3	93.6	76.7	0/0	6.5	6.4	6.4	23.3	0	0	3.2	0	0
	SiluxSB	100	100	93.6	93.1	92.9	0/0	0	6.4	6.9	7.1	0	0	0	0	0
Marg Adapt	F2000SE	100	93.1	79.3	82.8	78.6	0/0	6.9/0	13.8/6.9	17.2/0	14.3/7.1	0	0	0	0	0
	F2000SB	96.8	90.3	64.5	71.0	63.3	3.2/0	9.7/0	35.5/0	29.0/0	33.3/3.3	0	0	0	0	0
	SiluxSB	96.8	87.1	51.6	55.2	46.4	3.2/0	12.9/0	38.7/9.7	44.8/0	53.6/0	0	0	0	0	0
Anat Form	F2000	98.3	100	95.0	100	96.6	1.7	0	5.0	0	3.4	0	0	0	0	0
	Silux Plus	100	96.8	90.3	93.1	92.9	0	3.2	9.7	0	7.1	0	0	0	6.9	0
Surf Text	F2000	30.0	20.0	45.0	40	50.0	70.0	80.0	55.0	60.0	46.6	0	0	0	0	3.4
	Silux Plus	90.3	83.9	96.8	82.8	82.1	9.7	16.1	3.2	17.2	17.9	0	0	0	0	0
Retention	F2000SE	100	100	100	100	96.6	0	0	0	0	0	0	0	0	0	3.4
	F2000SB	100	100	100	100	100	0	0	0	0	0	0	0	0	0	0
	SiluxSB	100	100	100	93.6	90.3	0	0	0	0	0	0	0	0	6.4	9.7
Sec Caries	F2000SE	100	100	100	100	100	0	0	0	0	0	0	0	0	0	0
	F2000SB	100	100	100	100	100	0	0	0	0	0	0	0	0	0	0
	SiluxSB	100	100	100	100	100	0	0	0	0	0	0	0	0	0	0

baseline, for all groups ($p<0.0001$). No other time trends were noted

DISCUSSION

This study examined the clinical effectiveness of two bonding agents (a two-step total etch or a self-etching bonding agent) and two restorative materials (a compomer or a microfilled resin composite). Our results compare favorably with other studies (Folwaczny & others, 2001a,b) when similar materials were used to restore non-carious cervical lesions. For Dyract, Tyas (1998) reported a 97% retention rate at one year, Folwaczny and others (2001a) reported a retention rate of 90% at three years and Folwaczny and others (2001b) reported a five-year retention rate of 80%. Tyas and Burrow (2002) reported retention rates of 100%, 95%, 87% and 75% at six-month, one, two and three-year recalls, respectively, for non-carious cervical restorations when One-Step (BISCO, Inc, Schaumburg IL, USA) was used as a bonding agent with flowable (AELite Flow and BISCO Glaze) and microfilled (Silux) resin composites. Retention rates showed definite downward trends as the recall time increased. This study established the three-year retention rate of another compomer (F2000), with its self-etching primer as 96%. This retention rate falls within the normal retention rates for modern adhesives.

As restorative materials and adhesives evolve, questions about the compatibility of primer/adhesives and restorative materials continue to arise (Cheong & others, 2003; Pfeifer, Shih & Braga, 2003). For F2000, the retention rates did not vary when the bonding agent was changed from a self-etching adhesive to a total-etch adhesive.

A difference in marginal adaptation was almost statistically significant when SiluxSB was compared to F2000SE. It may be that the beveled margin used with the Silux Plus contributed to its marginal breakdown.

The surface texture of Silux was significantly smoother than F2000 at each recall. In this study, surface roughness was recorded as the roughness of the restorative material when the explorer was dragged across the restorative material and the tooth and was then compared. The Alpha criteria stated that the roughness of the explorer on the restorative material was the same as the natural, unrestored tooth. Bravo stated that the restoration was rougher than the tooth. Silux Plus is a microfilled resin composite with a mean inorganic filler particle size of .04 microns. The mean filler particle size of F2000 is 3.0 microns. This difference was clinically

Table 3: *Tests of treatment effects at three years adjusted for baseline using conditional logistic regression, where response was dichotomized as clinically significant or clinically insignificant. Results are presented as* p-*values from corresponding hypothesis test on fixed effects.*

Outcome	*p*-value
Retention	0.5185
Marginal Discoloration*	1.0000
Secondary Caries*	1.0000

**All responses were equal.*

Table 4: *Tests of treatment effects at three years adjusted for baseline using conditional logistic regression, where response was dichotomized as Alpha or >Alpha. Results are presented as* p-*values from corresponding hypothesis test on fixed effects.*

Outcome	*p*-value
Retention	0.5185
Marginal Discoloration	0.1814
Secondary Caries*	1.0000

**All responses were equal.*

Table 5: *Tests of treatment effects using generalized estimating equations (GEE1) comparing F2000SE, F2000SB, and SiluxSB for marginal adaptation, and compaing the combined F2000 groups against Silux Plus for anatomical form and surface texture. Results are presented as* p-*values from corresponding hypothesis test on fixed effects.*

	Marginal Adaptation	
Treatment	F2000SB	SILUXSB
F2000SE	0.1780	0.0644
F2000SB		0.1721

	Anatomical Form	Surface Texture
Treatment	SILUX PLUS	SILUX PLUS
F2000 Combined	0.085	<0.0001

Table 6: *Tests of time effects using generalized estimating equations (GEE1). All time points compared with baseline. Results are presented as* p-*values from corresponding hypothesis test on fixed effects.*

Measure	Month 6	Month 12	Month 24	Month 36
Marginal adaptation	0.1369	<0.0001	<0.0001	<0.0001
Anatomical form	1.0000	0.1187	0.6063	0.3055
Surface Texture	0.3250	0.3056	0.7323	0.4623

detectable at each recall. ADEPT Report (Albers & Aso, 1998) states that the intrinsic filler particle size of the resin composite determines the ultimate smoothness of the restoration. This clinical study supports that view. In addition, it seems as though the surface finish for the F2000 restorations improved with time. Toothbrushing may influence this by finishing the restorations during brushing. Surface roughness is important, since plaque retention increases with rougher surfaces (Weitman & Eames, 1975). Therefore, rough restorative materials may promote recurrent caries or gingivitis when the restoration is near the gingiva, as in Class V restorations.

CONCLUSIONS

1. Silux Plus, a microfilled composite, and F2000, a compomer, can both be used effectively to restore non-carious cervical lesions, although the microfilled composite retains its surface finish better than the compomer.
2. F2000, a compomer, may be used with a self-etching primer/adhesive or a fifth generation bonding agent successfully.

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Two-year Clinical Performance of Occlusal and Cervical Giomer Restorations

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Clinical Relevance

Beautifil showed promise as a direct-tooth colored fluoride releasing restorative material for occlusal and non-undercut cervical lesions. Reactmer showed mixed promise as a restorative material for cervical cavities.

SUMMARY

This study evaluated the two-year clinical performance of two types of giomers (Beautifil, a surface reaction giomer and Reactmer, a full-reaction giomer), in occlusal (Class I) and cervical (Class V) cavities using the USPHS criteria. Forty-two cervical erosion and carious lesions were restored using Beautifil and Reactmer following manufacturer's instructions. Twenty occlusal cavities were restored with Beautifil. Fifteen patients (mean age 35, ranging in age from 20 to 50 years) participated in the study. The success rate for cervical Beautifil restorations after two years was 80%, while the success rate for cervical Reactmer restorations was 71%. Occlusal Beautifil restorations had a 100% success rate.

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INTRODUCTION

The development of fluoride releasing restorative materials was one of the major achievements in dentistry in past decades. Since the introduction of silicates in the 1950s and conventional glass ionomers in the 1960s, research and development in dental materials and restorative dentistry have been geared toward fluoride release. In 1989, Antonucci and Stansburyn developed the first resin-modified glass ionomers by incorporating a resin component, HEMA, in the polyacrylic acid component. In 1993, polyacid-modified resin composites or "compomers" were introduced. This class of material was the first to attempt to combine both the physical and mechanical properties of resin composites and the fluoride release of conventional glass ionomers. However, it was later found that these materials have minimal fluoride release; they are not fluoride rechargeable and absorb water with time, leading to degradation of the material. They consisted of more resin composites than glass ionomers and,

thus, behaved as such (Attin, Vataschki & Hellwig, 1996).

Most recently, another improvement was made in fluoride releasing materials—the "giomer." Giomer restoratives are touted as the true hybridization of glass ionomer and resin composite. They have the properties of fluoride release and the fluoride recharge of glass ionomer cements along with excellent esthetics, easy polishability and strength of resin composites.

The composition of giomers is based on PRG (Pre-Reacted Glass ionomer) technology. This technology involves the pre-reaction of fluoro-aluminosilicate glass fillers with polyacrylic acid, forming a stable phase of glass ionomer described as "wet siliceous hydrogel." The resulting glass ionomer is then freeze-dried, milled, silane-treated and ground to form the PRG fillers. These fillers are then incorporated into a resin matrix. The final product is composed of a stable phase of glass ionomer suspended in resin matrix (Beautifil Technical Products Profile, 2000). It is supposed that the presence of a pre-reacted hydrogel is responsible for the high levels of fluoride release and the recharge of giomers.

PRG-technology is classified into two categories: F-PRG (full reaction type), where the entire filler particle is attacked by polyacrylic acid, and the S-PRG (surface reaction type), where only the surface of the glass filler is attacked by polyacrylic acid and a glass core remains. The surface reaction type (S-PRG) is exemplified by Beautifil (Shofu Dental Corporation, Osaka Japan) and is indicated for Class I through Class VI cavities. Class VI cavities are those that are on the incisal edge of anterior teeth or on the occlusal cusp heights of posterior teeth (Sturdevant, 1985). Beautifil is recommended for use with its proprietary two-step self-etching F-PRG adhesive system, Imperva FL-Bond. An example of a full reaction type giomer (F-PRG) is Reactmer (Shofu Dental Corporation). Its indication is limited to cervical cavities. The corresponding adhesive system of Reactmer is Reactmer Bond, a one-step, self-etching F-PRG giomer. Both adhesive systems can further be classified as glass-ionomer based adhesives (Tay, Pashley & Hoshiyama, 2002) since they contain ion-leachable fluoroaluminosilicate glass and the adhesion promoting monomer 4-AET (acryloethyltrimellitic acid), which has the ability to form hydrogen bonds with dentin collagen. This suggests some form of chemical bonding (Reactmer Bond Profile, 2000). The properties of materials utilizing PRG-technology include: increased wear resistance and a high level of radiopacity, due to the presence of multi-functional glass fillers; shade conformity, owing to the improved light diffusion and fluorescence of the material, and a high and sustained level of fluoride release and recharge, due to the stable phase of glass-ionomer formed prior to its addition to the resin matrix. Unlike other fluoride containing dental adhesives, the release of fluoride ions in giomers does not cause material degradation since the mechanism of fluoride release is similar to conventional glass ionomers (Roberts & others, 1998).

Several *in vitro* studies have been conducted on the purported properties of this new material. Giomers were found to provide an almost complete seal against bacterial microleakage, cause little mechanical and chemical pulpal irritation and inhibit demineralization. Furthermore, it has been shown that the reuptake of fluoride in giomers can increase the thickness of the caries inhibition zone (Sonoda & others, 2002; Teranaka, 2003; Itota & others, 2003; Okuyama, 2003). In terms of physical properties, the water absorption and expansion of a giomer was found to be greater than compomers (McCabe, 2003).

Despite numerous laboratory testing on giomers, a minimal amount of research has been published relative to the relative newness of the material.

This study evaluated and compare the clinical performance of two giomer restorative materials with their proprietary PRG adhesive systems in cervical and occlusal cavities over a six-month period and after two years.

METHODS AND MATERIALS

The two "giomers" used in this study were Beautifil, with the bonding system Imperva FluoroBond and Reactmer, with the bonding system Reactmer Bond (Table 1).

Samples consisted of 21 cervical Reactmer restorations, 21 cervical Beautifil restorations and 20 occlusal Beautifil restorations. Most cervical lesions were due to abrasion. Only five cervical lesions were due to caries. Patients who participated in the study agreed by signing a written informed consent. The materials were randomly assigned to each patient. All teeth with occlusal cavities included in the study had an antagonist in the opposing arch.

To rule out variations in material properties, all restorative materials were taken from one batch. Appropriate shades were used on each cavity.

Each patient was treated by a single operator. Prior to preparation, the teeth were cleaned using a non-fluoride slurry of pumice and copious amounts of water. Small to moderated pear-shaped occlusal cavities with isthmus widths not more than 1.5 mm and depths not exceeding 2 mm were prepared using #462R (ISO 011) and #364R (ISO 015) diamond points. For the cervical restorations, saucer shaped cavities without any form

of retention or mechanical undercut were prepared with #440S (ISO 013) and #440 (ISO 015) diamond points. Both carious and cervical abrasion/erosion lesions were included, provided that their margins were in enamel and cementum. A non-bevel form of cavity margin was used for all preparations. The restorative materials and their corresponding adhesives were placed according to manufacturer's instructions under rubber dam isolation. To ensure adequate polymerization, a visible light curing machine with a light output not less than 450 mW/cm^2 was used to cure the restorations. Each cavity was filled using the incremental technique and each increment was cured for 40 seconds. Finishing and polishing was done after 24 hours using superfine grit diamond points (Shofu finishing diamond points, Shofu Japan) and a diamond impregnated composite polisher (Compomaster, Shofu Japan). Table 2 shows a summary of the experimental methods used.

Table 1: *Materials Used*

Material	Description	Composition
Reactmer Paste (Lot #129901)	F-PRG restorative material	F-PRG filler, glass filler, UDMA, 2-HEMA Photoinitiator
Reactmer Bond (Lot #099900)	One-step, self-etching tri-curing F-PRG adhesive system	Bottle A: F-PRG filler, fluoroaluminosilicate glass (FASG), new initiators, distilled water, acetone Bottle B: 4-AET, 2-HEMA, UDMA Photo-initiator
Beautifil Paste (Lot #030015)	S-PRG restorative material	S-PRG filler, glass filler, Bis-GMA, catalyst
Imperva Fluoro Bond (Lot # Primer A: 119971 Primer B: 119956 FBBond: 119964)	Two-step, self-etching, F-PRG adhesive system	Primer Bottle A: H_2O, acetone, catalyst Primer Bottle B: UDMA, HEMA, 4-AET, catalyst FB Bond: F-PRG, UDMA, HEMA, 4-AET, catalyst

UDMA = Urethanedimethacrylate; 2-HEMA = 2-hydroxyethylmethacrylate; 4-AET = 4-acryloxyethyltrimellitic acid; Bis-GMA = bisphenol-glycidyl-methacrylate

Table 2: *Experimental Methods*

Gingival Status	: Gingival Index < 1.0 Plaque Index <1.0 (Loe & Silness)	
Control of moisture through use of rubber dam		
Tooth Cleaning	: Slurry of pumice, water	
Cavity Preparation		
Class I	: Burs : Cavity Form	#418R, #F440R, #440S, #440 (Shofu) Small to moderate pear-shaped or saucer-shaped cavities, non bevel
Class V	: Burs : Cavity Form	#418R, #F440R, #440S, #440 (Shofu) Saucer or deep saucer shaped cavities, without retention form, butt joint
Restoration		
Beautifil	: Bonding	Imperva Fluorobond Mix one drop each of Bottle A & B, apply to tooth surface with microbrush, leave for 10 seconds, mild air drying, apply FB-Bond with microbrush, light cure for 10 seconds.
Reactmer	: Bonding	Reactmer Bond Mix one drop each of bottle A & B, apply to tooth surface, leave for 20 seconds, mild air drying, light-cure for 20 seconds.
Filling Instrument	: Composite resin instrument	
Finishing and Polishing	Transparent cervical matrices : After 24 hours Superfine grit diamond points, Compomaster (Shofu)	

Direct/Clinical Evaluation

The restorations were clinically evaluated using the modified Ryge (USPHS) criteria at baseline (24 hours), six months and two years. They were evaluated using visual and tactile inspection.

Evaluations were performed by two examiners trained in the technique. Disagreements between the examiners on a particular score were settled by reevaluating the restoration until a consensus was reached.

Table 3 shows the evaluation items included in the study.

Indirect/ Laboratory Evaluation

Representative replica casts of the restored teeth were made using a silicone rubber based impression material (Exaflex, GC, Japan) and epoxy resin (Stycast 1266, Emerson Cuming). The replicas were observed under scanning electron microscope (S-800, Hitachi, Tokyo, Japan) as an additional evaluation for marginal adaptation, surface texture and wear.

Color photos were taken to serve as a visual record and to assess changes in the color matching and anatomic form over time.

Table 3: *Modified USPHS Evaluation Criteria and Scoring*

CRITERIA	A	B	C	D
Wear (Anatomic Form)	The restoration is continuous with existing anatomic form.	Restoration is discontinuous with existing anatomic form but the amount of lost material is not sufficient to expose dentin.	Sufficient material was lost to expose dentin.	----
Color Match	Restoration matches adjacent tooth in color and translucency.	The restoration mismatches adjacent tooth structure in color and translucency, but the mismatch is within normal range of tooth color and translucency.	The restorations mismatches adjacent tooth structure in color and translucency, and the mismatch is outside the acceptable range of tooth color and translucency.	----
Marginal Discoloration	No discoloration exists anywhere on the margin between restoration and tooth structure.	The discoloration has penetrated along the margin, but has not penetrated in a pulpal direction.	The discoloration has penetrated along the margin in a pulpal direction.	----
Marginal Adaptation	There is no visible evidence of a crevice along the margins. An explorer does not catch when drawn across the margins.	There is visible evidence of a crevice into which the explorer will penetrate, but no dentin is visible.	There is sufficiently deep crevice that the explorer will penetrate into the exposed dentin.	The restoration is fractured, mobile or missing.
Surface Roughness	Surface texture of the restoration is similar to an evaluation model surface finished by Super-fine Sof-Lex disc.	Surface texture of the restoration is similar to an evaluation model surface finished with #600 grit silicon carbide paper.	Surface texture of the restoration is similar to an evaluation model surface finished by #280 grit silicon carbide paper.	----
Post-operative Sensitivity	Patient reports no post-operative sensitivity.	Patient complains of slight post-operative sensitivity, which does not require pulp treatment.	Patient complains of severe post-operative sensitivity, requiring pulp treatment.	----
Secondary Caries	There is no evidence of caries along the margins of the restoration.	There is evidence of caries along the margins of the restoration.		----

Statistical Analysis

The results were analyzed using the Student's *t*-test to determine whether there was significant change in the evaluation items over time.

Table 4: *Distribution of Occlusal and Cervical Cavities Among Teeth Used in the Study*

Cavity Classification		Premolars	Molars	Anteriors	Total
Occlusal Beatuifil		9	11	NA	20
Cervical	Beautifil	11	4	5	20
	Reactmer	17	3	1	21
Total		37	18	6	61

RESULTS

Table 4 shows the distribution of occlusal and cervical cavities among teeth used in the study. Most teeth used in the study were premolars and most cervical cavities were abrasion lesions.

After two years, 20 of the 21 cervical Beautifil restorations and all of the 21 cervical Reactmer restorations were available for recall. There was also a 100% recall rate for the occlusal Beautifil restorations.

In this study, a restoration is considered successful if there is no recorded rating of Charlie or Delta in any clinical parameter examined and if no secondary caries developed. At two years, there was an 80% success rate for Beautifil and only a 71% success rate for Reactmer.

Class V (Cervical Restorations)

After two years, all recalled cervical Beautifil restorations were retained. Figure 1 shows the USPHS rating percentages for cervical Beautifil restorations at base-

line, six months and two years. Alpha and Beta ratings were recorded for most evaluation criteria. The decrease in Alpha ratings for all evaluation criteria at 24 to 30 months was statistically insignificant except for marginal adaptation ($p<0.05$). Marginal adaptation and anatomical form were the criteria that had the most Charlie and Delta ratings at both six months and two years.

Figure 2 summarizes the USPHS ratings of the cervical Reactmer restorations. It is notable that, at six months, four of the 21 Reactmer restorations were already dislodged. The retention rate at six months was a very low 81% compared to the 100% retention rate for Beautifil. At two years, one more Reactmer restoration was lost. Of the remaining 16 restorations, two more failed due to a loss of anatomical form, marginal discoloration, marginal adaptation and secondary caries. Although there was a decrease in Alpha ratings after two years for all evaluation criteria, it was only in the criteria of marginal adaptation that a significant decrease was computed. Marginal adaptation, marginal discoloration and anatomical form were the criteria that had the most Charlie and Delta ratings at both six months and two years.

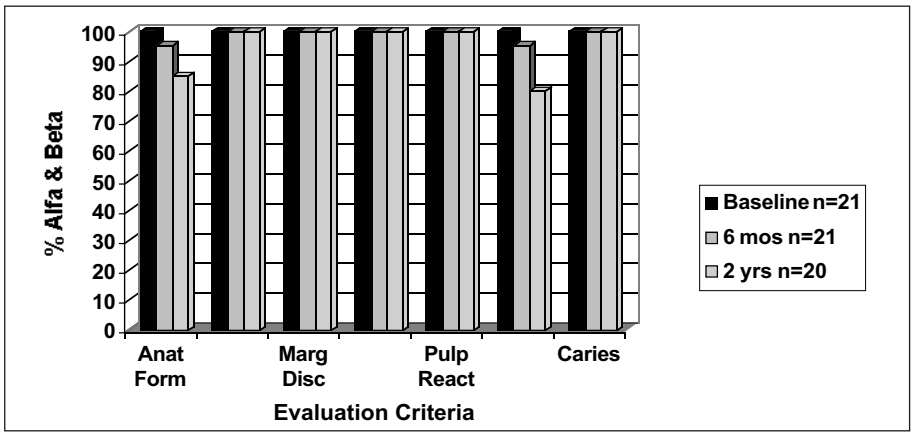

Figure 1. *Alpha and beta ratings for cervical Beautifil restorations at baseline, six months and 24 months.*

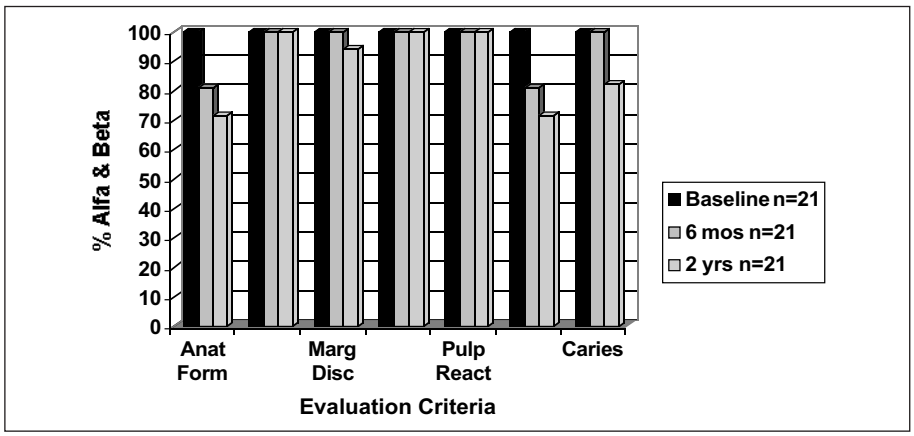

Figure 2. *Alpha and beta ratings for cervical Reactmer restorations at baseline, six months and 24 months.*

Class I (Occlusal) Beautifil Restorations

At 24 to 32 months, all of the 21 occlusal restorations were available for recall.

The occlusal Beautifil restorations exhibited a 100% success and retention rate at six months and two years. There was no incidence of secondary caries. Most of the changes recorded were for anatomical form (Figure 3).

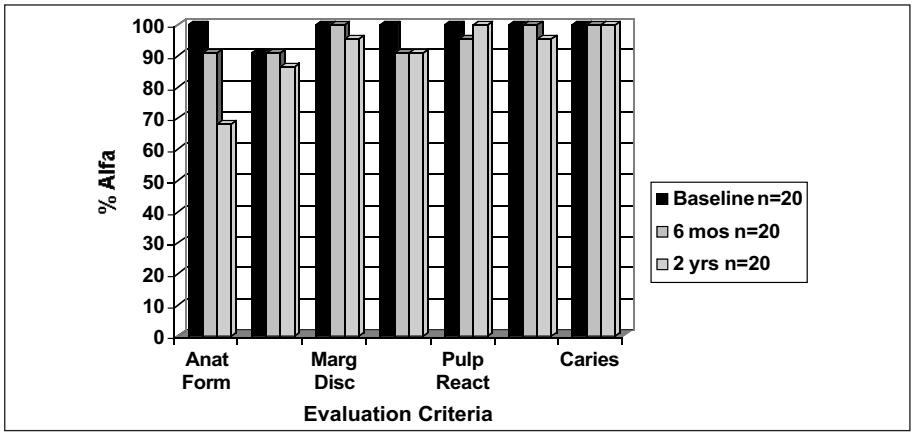

Figure 3. *Alpha ratings for occlusal Beautifil restorations at baseline, six months and 24 months.*

Indirect Evaluation

Representative clinical pictures and corresponding scanning electron micrographs of cervical Beautifil restorations are presented in Figures 4 through 7. Restorations on teeth 24 and 25 (Figures 4A, 4B) were rated clinically as Alpha but SEM pictures showed wear of resin filling at the incisal and cervical margin (Figure 4C).

Figures 5A through 5C show some failures of Reactmer restorations at two years, which include pronounced marginal discoloration at the incisal margin of 43 and the cervical margin of 44 (Figure 5A), expansion of the material resulting in poor marginal adapta-

tion on 35 (Figure 5B) and actual loss of material at the cervical margin of 44 (Figure 5C).

Figure 6 shows an occlusal Beautifil restoration on tooth 37, with good marginal adaptation and no signs of wear at baseline (Figure 6A. However, at two years, slight wear was evident on the same restoration (Figure 6B). Figure 7 is a SEM micrograph of an occlusal Beautifil restoration at two years showing some degree of wear on the occlusal surface of the restoration.

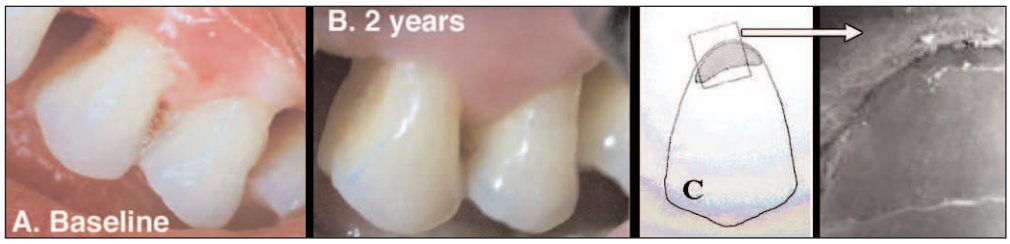

Figure 4*A. Cervical Beautifil restorations on 24 and 25 at baseline.* Figure 4*B. Same restorations after two years. 4C. SEM picture showing slight wear of the material especially at the incisal and cervical margins.*

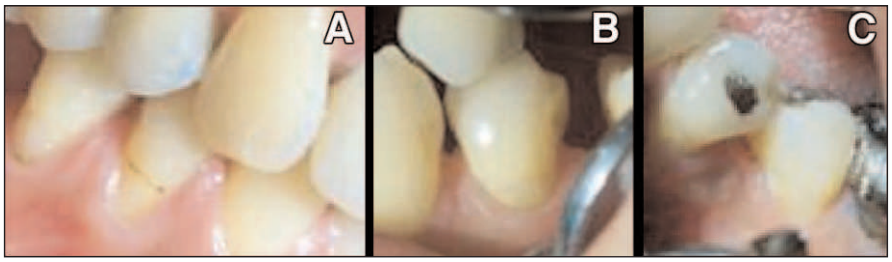

Figure 5. *Failures of Reactmer restorations.* Figure 5*A. Marginal discoloration rated delta at incisal margin of 43 and cervical margin of 44.* Figure 5*B. Failed incisal margins on 35 due to expansion of the material. Figure 5C. Gross loss of material at cervical margin of 44.*

DISCUSSION

Due to the small sample size of the study, comparisons on the retention and success rates of Beautifil and Reactmer in cervical cavities cannot be drawn. The results of this study, however, give a general picture on the overall performance of these two PRG materials in non-undercut cervical lesions after two years.

The retention rate and success rate of Reactmer in this study is lower than that of Beautifil. The retention rates and success rates of both giomers in this study are lower than earlier studies conducted on giomer restorations. These studies reported success rates for cervical Beautifil restorations in the range of 97% to 100% (Matis & others, 2002; Wilson 2003) and for Reactmer restorations at 81% (Matsuo 2003). The difference in the reported retention rates might be due to the vast variation in sample size and operator factors.

The results of this study regarding occlusal Beautifil restorations is similar to the results of a study on the same material by Gordan and others (2003), wherein they found a 100% success rate, with most changes occurring in marginal adaptation, marginal discoloration and anatomy.

A possible explanation for the low retention rate of Reactmer restorations is its high water absorption and solubility. In a study by McCabe (2003), the water absorption of Reactmer was significantly greater than compomers and composites. In that same study, Reactmer also gave a sustained expansion over three months. This hygroscopic expansion or swelling might have caused internal stress within the restorations that caused their dislodgment.

The difference in the resin systems of Beautifil and Reactmer might explain why Beautifil had a better retention rate than Reactmer, despite their both being

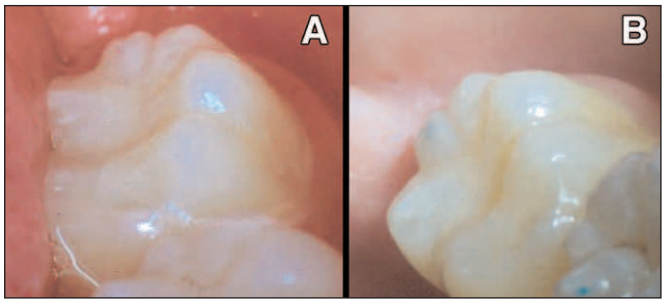

Figure 6*A. Occlusal Beautifil restoration on 37 showing good marginal adaptation at baseline.* Figure 6*B. Same restoration with slight wear at two years.*

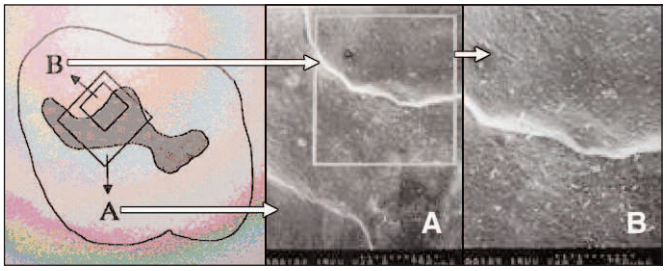

Figure 7. *SEM micrograph of an occlusal Beautifil restoration showing wear on the occlusal margins.*

giomers. Reactmer contains HEMA, which causes water absorption (McCabe, 2003). In addition, Reactmer is a full reaction giomer. The entire glass core is dissolved in polyacrylic acid and, in a way, more of the glass ionomer phase is formed. Thus, it is easier for water to diffuse in and out. On the other hand, the resin of Beautifil does

not contain HEMA and is a surface-reaction giomer. Therefore, there is less water absorption and less expansion.

With regard to the better retention rates for occlusal Beautifil restorations compared to cervical Beautifil restorations, the cavity configuration and different types of stress on the restorations might have had an effect. Although both cavities are high stress or high C-factor cavities, cervical restorations are relatively easier to dislodge since they are subjected to another type of loading stress, abfraction. In this study, most cervical cavities were placed in premolars, the teeth that have the highest incidence of abfraction lesions. Flexion of the teeth in a bucco-lingual direction coupled with the low Young's modulus of the material might have caused easier dislodgment. Moreover, most cervical lesions in this study were toothbrush abrasions due to faulty toothbrushing habits. It is interesting to note, however, that even with the additional stress of faulty toothbrushing, no cervical Beautifil restoration was completely dislodged after two years. This can be attributed to adequate bond strengths and the physical and mechanical properties of the material.

In a study by Matis and others (2002) on the clinical performance of Beautifil restorations in non-restored cervical erosion lesions at 18 months, the criteria that scored the lowest percentages of Alpha ratings were anatomical form (87%), marginal adaptation (95%) and marginal discoloration (90%). In the current study, the same criteria got the lowest percentages for Alpha ratings, indicating that the inherent weakness of the material is in these areas.

Hygroscopic expansion, which is inherent in giomers, is the main cause for marginal deterioration in giomer restorations. A study by Huang and others in 2002 revealed that Reactmer Paste exhibits rapid and extensive expansion, a finding similar to Matis' in 2002 and is clinically evident in this study (Figure 5B). Another study stated that, although bond failure at placement can be decreased by hygroscopic expansion of PRG composite, this expansion is not enough to completely eliminate bond failure (Yap, Shah & Chew, 2003).

Aside from hygroscopic expansion, marginal deterioration might have also been caused by any of the following: 1) breakdown of the bond at the margins of the restoration, 2) improper finishing and polishing procedures and 3) fracturing away of thin flashes of the restorative material at the cavosurface margin.

Comparing the marginal adaptation of both materials on incisal/occlusal margins and on cervical margins, no difference was observed. Although inconclusive, this might mean that self-etching, glass-ionomer based 4-AET containing adhesive systems, such as those used in this study, are effective on both enamel and cementum. Unlike earlier compomers that require separate phosphoric acid etching of the enamel, this newer type of adhesive system provides both convenience and adequate bond strengths on enamel and dentin. Some ditching on the gingival margins was observed, although this may be due to inaccessibility of the margins as they are hidden in the gingival sulcus.

Marginal discoloration, such as seen in Figure 5A, could be a consequence of bond failure that allowed for ingress of exogenous stains or was caused by thin flashes of excess material that allowed for the retention of stains.

In terms of wear resistance of the material, this study further affirms what is currently perceived as one of the main drawbacks of direct tooth colored restorative materials, despite the presence of multifunctional glass as fillers that have the same hardness as enamel. SEM evaluation, however, reveals that wear occurs not on the glass filler but on the resin matrix (Figure 6). Although the majority of giomer restorations scored Alpha for wear after two years, the graph for wear shows a downward trend (Figure 1), indicating that wear increases with time. It will be interesting to know how the restorations will rate for wear after three or even five years. One limitation of this study is that no actual measurement of wear rate was conducted to quantify wear. Another notable finding is that in some specimens, there is a difference in clinical and SEM observations. This only highlights the advantage of doing indirect evaluation in addition to clinical evaluation. Clinical evaluation is not sensitive enough to recognize wear, especially in its early stages (Abdalla & Alhadainy, 1997). This is evident in Figures 4B and 4C. Some restorations that were rated Alpha for anatomic form and marginal adaptation might have had some discrepancies that had not progressed sufficiently to be seen clinically.

The very low incidence of secondary caries (one out of 61 restorations) corroborates laboratory findings regarding fluoride release and recharge of the material. One advantage of glass-ionomer based adhesive systems, such as Imperva Fluorobond and Reactmer Bond, is the ability to produce caries inhibition zones on the inner walls of the restoration. Aside from their inherent property of fluoride release and recharge, the uptake of fluoride by the surrounding tooth structure is very possible, because of the supposed permeability of simplified adhesives to fluids. If simplified adhesives are permeable to water, it is also possible that they are equally permeable to fluoride ions from the adhesive and restorative material itself (Tay & Pashley, 2001).

In this study, the giomers, particularly Beautifil, seem to have performed satisfactorily in occlusal and cervical restorations. The clinical performance of Beautifil in cervical restorations appears to be comparable or slightly better than that of compomers and resin-modi-

fied glass ionomers. Several clinical evaluations of these materials revealed retention rates of 97% to 98% at two years (Tay & others, 2002; Abdalla, Mahallawy & Davidson, 2002; Abdalla & Alhadainy, 1997) and 78.5% to 93% at five years (Loguercio & others, 2003). Marginal deterioration and marginal discoloration were also the evaluation parameters that received the most failed ratings for compomers and resin-modified glass ionomers. Reactmer performed rather poorly compared to the other materials. In occlusal restorations, Beautifil appears to perform better than compomers and is comparable to hybrid composites. A recent study by Huth and others (2004) revealed a failure rate of 15.4% at two years for compomers when used in Class I and II cavities, while a study on the clinical performance of a hybrid composite in posterior cavities yielded a success rate of 95% at two years. Although these comparisons are inconclusive, since these results come from different clinical evaluations, they provide some degree of comparison on the clinical performance of these different tooth-colored restoratives.

CONCLUSIONS

Based on the results of this study, it would seem that Beautifil is an acceptable restorative material for cervical and occlusal restorations. Reactmer restorations, on the other hand, seemed to perform poorly in terms of retention. Further improvements are recommended for this material. Clinically, the apparent weaknesses of both PRG materials are in marginal adaptation, marginal discoloration and wear. Water absorption and hygroscopic expansion due to their glass ionomer-like behavior were their biggest drawbacks.

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Two-year Clinical Evaluation of a Posterior Resin Composite Using a Fourth- and Fifth-generation Bonding Agent

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RS Walker • MM Winkler • DE Mercante • JM Davidson

Clinical Relevance

Whether a fourth- or fifth-generation bonding system is used, Solitaire 2 can function successfully as a posterior restorative material, although the fourth-generation material showed a tendency to produce better performance than the fifth-generation material.

SUMMARY

This study evaluated the clinical performance of a posterior resin composite used with a fourth- and fifth-generation bonding agent. Sixty-two Class I and II restorations were placed with half the restorations restored with Gluma Solid Bond (a fourth-generation bonding system, or total etch two-step system) and the other half restored with Gluma Comfort Bond and Desensitizer (a fifth-generation bonding system, or total etch one-step system). Solitaire 2 was used as the restorative material for all restorations. The bonding systems and resin composite were used according to the manufacturer's instructions and all procedures were performed with rubber dam isolation. All restorations were evaluated at baseline, six months and one and two years. A modified USPHS scale was used to evaluate the restorations for marginal discoloration, recurrent caries, anatomic form, marginal adaptation and proximal contact. Statistical analysis revealed that at two years no significant differences were found between the two bonding agents. Overall, Solitaire 2 performed well clinically whether Gluma Solid Bond or Gluma Comfort Bond and Desensitizer was used. It was thus concluded that Solitaire 2 functions successfully when used as a posterior restorative material for at least two years.

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INTRODUCTION

Even though amalgam has been used for more than 150 years with an excellent clinical history, it is being

replaced with resin composite restorations. In 1986, 80% of the dentists practicing in the United States reported using posterior composites (Dental Products Report, 1986). The results of the first clinical trial of posterior composites were published in 1971 (Phillips & others, 1971). Subsequent recalls of the same restorations revealed that resin composite restorations had poor wear resistance (Phillips & others, 1972; Phillips & others, 1973). Early reviews suggested that these materials be limited to conservative bicuspid and first molar restorations where esthetics were critical (Burgess, Summitt & Laswell, 1987). While the physical and mechanical properties of resin composites have improved (Lee & White, 1998; Cobb & others, 2000; Nash, Lowe & Leinfelder, 2001), material and clinical limitations still exist that restrict the use of resin composite as a posterior restorative material. Resin composite restorations are technically difficult, and more time is required to place a resin composite restoration compared to a similarly sized amalgam restoration (Dilly & others, 1985; Leinfelder, Bayne & Swift, 1999; Lopes & others, 2004). In addition, proximal contacts are frequently difficult to obtain (Wilson, Wilson & Smith, 1985; Boksman & others, 1986; Slone, 1994). Contaminants are still detrimental to bond strength (Knight, Barghi & Conn, 1993; Johnson & others, 1993; Gallo, Xu & Burgess, 1998; Abdalla & Davidson, 1998; Van Schalkwyk & others, 2003) and can compound placement difficulties. Post-operative sensitivity with composite restorations has been reported and is thought to be due, in part, to polymerization shrinkage (Civelek & others, 2003) that can produce marginal openings and subsequent leakage (Neiva & others, 1998; Haller & Trojanski, 1998). Polymerization shrinkage can generate forces of 2.4–5.9 MPa for conventional and microfilled composites, which can lead to cracking and crazing at the enamel margins and gap formation at the resin-tooth interface (Davidson, de Gee & Feilzer, 1984; Lutz, Krejci & Oldenburg, 1986; Carvalho & others, 1996; Mehl, Hickel & Kunzelmann, 1997; Ernst & others, 2003). Microleakage occurs through these openings and is more serious with composite than amalgam since composite does not corrode and seal at the margins. It is believed that incremental curing techniques may decrease marginal opening and microleakage resulting from polymerization shrinkage (Gordon & others, 1986; Crim & Chapman, 1986; Eick & Welch, 1986; Podshadley, Gullett & Crim, 1985; Lopes, Baratieri, & Perdigão, 2000; Lopes & others, 2004). Additionally, proper placement of the bonding agent improves its ability to compensate for polymerization forces of the restorative material during light curing (Civelek & others, 2003).

Accurate measurement of composite wear is difficult and many different methods have been used. Most studies have concluded that resin composites exhibit greater loss of anatomic form in occlusal contact areas compared to non-occlusal contact areas (Lambrechts & others, 1984). The wear of early composites ranged from 12-105 microns per year, while in the same studies, amalgam wear ranged from 6–58 microns per year and enamel wear ranged from 3–54 microns per year (Lambrechts & others, 1984; Leinfelder, Wilder & Teixera, 1986; Heymann & others, 1986; Braem & others, 1986; Sturdevant & others, 1986). As resin composite materials have improved, so has wear resistance. Two studies of resin composites have demonstrated wear rates from 4.9 microns for Clearfil Photoposterior (Wendt & Leinfelder, 1992) and 7-8 microns for Heliomolar (Mazer & Leinfelder, 1992). These rates compare favorably to enamel. Other studies have also demonstrated that some posterior composites have wear rates similar to amalgam (Mazer, Leinfelder & Russell, 1992; Dickinson, Gerbo & Leinfelder, 1993). Increased resin composite wear occurs with increased restoration size, more distal placement in the dental arch and placement in stress-bearing areas (Yap, Teoh, & Chew, 2002; Turssi, de Moraes Purquerio & Serra, 2003).

There are several generations of bonding agents currently available. The purpose of this study was to measure the clinical performance of one posterior resin composite placed with two bonding systems: a fourth-generation bonding system (Gluma Solid Bond) and a fifth-generation bonding system (Gluma Comfort Bond and Desensitizer).

METHODS AND MATERIALS

Subjects for this investigation were selected from patients of the Dental School at the Louisiana State University Health Sciences Center (LSU). Each patient recruited had at least two moderately sized carious lesions needing restoration. Patients were excluded according to the following criteria: 1) Patients with severe medical complications (organ transplants, long-term antibiotic or steroid treatment, cancer or compromised immune system) or disabilities that caused an inability to tolerate the time required to complete the restorations or provide adequate oral hygiene; 2) Patients with xerostomia caused by medications or radiation or Sjogren's syndrome patients; 3) Patients with chronic periodontitis, rampant caries or poor oral hygiene which may cause extraction of the restored teeth; 4) Patients with a history of chronic bruxism; 5) Patients unavailable for long-term recall or 6) Patients who could not tolerate the rubber dam that was required for isolation of the tooth during preparation and restoration. Patients were included in this study if they had at least two Class I or II carious lesions or existing restorations needing replacement. The restored teeth had to have both occlusal and proximal contact. Consent for this study was obtained prior to the

initial examination and the initiation of any portion of the study. One of the investigators in the study explained the consent to patients and answered questions.

A single light-cured resin composite (Solitaire 2, Heraeus Kulzer, Inc, Amonk, NY, USA) was used to restore teeth with Class I or II cavity preparations. Sixty-two Class I and II restorations were placed with Solitaire 2 resin composite (37 Class II and 25 Class I). They were placed by four faculty members at LSU School of Dentistry from the Department of Operative Dentistry and Biomaterials. Half the restorations were restored using Gluma Solid Bond (Kulzer), while the other half were restored with Gluma Comfort Bond and Desensitizer (Kulzer). The restorations were placed following the manufacturer's directions for all materials (Table 1). Both bonding agents use a total etch technique (20 second etch with 20% phosphoric acid). The Gluma Solid Bond has a separate primer and adhesive application, whereas the Gluma Comfort Bond has a combined primer/adhesive application. The cavity preparations were done with rubber dam isolation. Prepared interproximal surfaces were pre-wedged with wooden wedges. Isthmus width for the restorations was no greater than 1/2 the intercuspal distance of the tooth. All margins were placed at 90° to the external tooth surface (that is, no bevels were intentionally placed). A sectional matrix band (Composi-Tight, Garrison Dental, Spring Lake, MI, USA) was used, if possible, for Class II restorations.

For interproximal areas, the resin composite was placed incrementally, beginning with an initial increment in the gingival floor, followed by an increment in the facial half of the box and a final increment to fill the box. Occlusal areas were filled in two increments: the first extended from the pulpal floor to the occlusal on a diagonal, followed by a second increment that completed anatomic contour. Carbide finishing burs (OS1, OS2, 7901, Brasseler USA, Savannah, GA, USA) were used to remove gross excess resin composite, followed by finishing strips and disks (Sof-Lex, 3M, St Paul, MN, USA) and the Enhance polishing system (LD Caulk, Milford, DE, USA). All polishing was done at slow speed without water spray. After removing the rubber dam and adjusting the occlusion, all margins were etched for 20 seconds with phosphoric acid, followed by the application of Fortify (BISCO, Chicago, IL, USA), which was light-cured for 20 seconds.

Each restoration was evaluated directly at baseline (one week after restoration placement), six months and annually for two years. The direct clinical evaluations were made using modified USPHS guidelines (Table 2) to evaluate proximal contact, marginal discoloration, secondary caries, anatomic form and marginal adaptation. Slides were used to document any marginal staining of the restorations.

Table 1: *Bonding Procedures*

Gluma Solid Bond	**Gluma Comfort Bond**
Etch tooth for 20 seconds with 20% phosphoric acid, rinse, gently dry leaving dentin moist	Etch tooth for 20 seconds with 20% phosphoric acid, rinse, gently dry leaving dentin moist
Primer (Solid Bond B) applied twice, gently air dried	Adhesive applied for 15 seconds, gently air-thinned
Adhesive (Solid Bond S) applied, gently air-thinned	Light-cured for 20 seconds
Light-cured for 20 seconds	Solitaire 2 placed in 2-mm increments, light-cured for 40 seconds
Solitaire 2 placed in 2-mm increments, light-cured for 40 seconds	

Table 2: *Modified USPHS Rating Systems*

Category	**Score**	**Criteria Used in Rating**
Marginal Discoloration	Alpha	No discoloration anywhere along the margin.
	Bravo	The discoloration has not penetrated in a pulpal direction along the margin.
	Charlie	The discoloration has penetrated in a pulpal direction.
Secondary Caries	Alpha	No caries present.
	Charlie	Caries present.
Anatomic Form	Alpha	The restoration is continuous with the existing anatomic form.
	Bravo	The restoration is discontinuous with existing anatomic form, but the missing material is not sufficient to expose dentin or base.
	Charlie	Sufficient material is lost to expose dentin or base.
Marginal Adaptation	Alpha	An explorer does not catch in either direction.
	Bravo	The explorer catches and a crevice exists into which the explorer penetrates. Dentin not exposed.
	Charlie	The explorer catches in a crevice that exposes dentin.
Proximal Contact	Alpha	Floss snaps through the contact.
	Bravo	Floss passes easier than the contralateral contact.
	Charlie	No contact present.

Table 3: *Percent Response Profiles of Variables by Treatment Group*

		Baseline			6 Months			1 Year			2 Year		
Variable	**Treatment**	**A**	**B**	**C**	**A**	**B**	**C**	**A**	**B**	**C**	**A**	**B**	**C**
Marginal Discoloration	Comfort Bond	100	0	0	97	3	0	93	7	0	83	17	0
	Solid Bond	100	0	0	100	0	0	100	0	0	97	3	0
Secondary Caries	Comfort Bond	100	-	0	100	-	0	100	-	0	97	-	3
	Solid Bond	100	-	0	100	-	0	97	-	3	97	-	3
Anatomical Form	Comfort Bond	100	0	0	97	3	0	93	7	0	93	7	0
	Solid Bond	100	0	0	93	7	0	97	3	0	91	9	0
Marginal Adaptation	Comfort Bond	97	3	0	97	3	0	87	13	0	87	13	0
	Solid Bond	97	3	0	97	3	0	97	3	0	91	9	0
Proximal Contact	Comfort Bond	100	0	0	100	0	0	100	0	0	100	0	0
	Solid Bond	100	0	0	100	0	0	100	0	0	100	0	0

Of the 62 restorations placed, 48 were evaluated at two years (25 from the Solid Bond group, 23 from the Comfort Bond group). Response rates for marginal discoloration, secondary caries, marginal adaptation, anatomic form and proximal contact were compared between treatment groups using conditional logistic regression, which accounted for the correlation within subjects due to the design (multiple treatments per subjects and multiple teeth per patient). Analysis was carried out using LogXact Version 4.1 (Cytel Software). All hypothesis tests were conducted at the α=0.05 level of significance. Most outcome variables were measured on a three-point scale (Alpha, Bravo and Charlie), but, for purposes of analysis, data were transformed to a binary scale by combining the Bravo and Charlie categories (very few responses fell in the Charlie category). Due to the relatively small number of patients (n=18 at year 2) and the lack of variability in the responses (in general, the Alpha response rates remained very high for each time point and treatment group), we were unable to perform an overall repeated measures analysis (such as GEE). This necessitated comparing response rates between the Comfort Bond and Desensitizer and Solid Bond groups at each time point using the conditional logistic model. The study design used was a two-treatment split-mouth design. In all analyses presented, the probability of having a higher ordered response (Bravo or Charlie versus Alpha) is being modeled. Thus, a significant difference between treatments implied that one of the treatments had a higher probability of a worse outcome.

Table 4: *Comparisons of Comfort Bond and Desensitizer with Solid Bond for variables at time points using conditional logistic regression. Results are presented as* p*-values.*

Variable	Baseline	6 Months	1 Year	2 Year
Marginal Discoloration	1.000	0.317	0.250	0.075
Secondary Caries	1.000	1.000	1.000	1.000
Anatomic Form	1.000	0.620	0.548	1.000
Marginal Adaptation	1.000	1.000	0.306	0.667
Proximal Contact	1.000	1.000	1.000	1.000

RESULTS

The categorized clinical data is summarized as percentages for each category and both treatment groups have been arranged by recall visit and are presented in Table 3. Statistical comparisons between groups are summarized with *p*-values in Table 4. No significant differences were found between Solid Bond and Comfort Bond and Desensitizer on any of the outcomes measured at any recall time. One noteworthy comparison that approached significance was marginal discoloration at two years (p=0.0750), where Alpha scores for Comfort Bond and Desensitizer dropped to 83% compared with 97% for Solid Bond. In all, the data indicate high clinical efficacy at two years for all restorations placed regardless of the bonding agent used.

DISCUSSION

This study evaluated the clinical performance of a packable resin composite (Solitaire 2) used for Class I and II restorations with two total etch bonding systems: Gluma Solid Bond, a two-step system and Gluma Comfort Bond and Desensitizer, an ethanol-based one-step system. Overall, the restorations performed well and were successful at the two-year recall, regardless of which bonding agent was used.

Burke and others (2003) placed Solitaire 2 in Class I and II restorations with Gluma Solid Bond or Gluma One Bond, a one-step acetone-based total etch bonding system. They reported no differences between bonding systems. In addition, 99% of the Solitaire 2 resin composite restorations (87 out of 88) performed satisfactorily at one-year recalls. Ernst and others (2002) used Solitaire 2 in Class II restorations with the Gluma Solid Bond system. At two-year recall, they reported 98% of the restorations functioning successfully. The results of these two studies are very similar to the results of the current study.

Achieving adequate proximal contact when using resin composite can be challenging. Adequate proximal contact between teeth is necessary to maintain the health of surrounding tissues (Hancock & others, 1980). Placing resin composite is technique sensitive and it is not unusual for operators to experience difficulty restoring proximal contact because of its handling characteristics, which are different from those of amalgam (Slone, 1994). In this study, adequate proximal contact was achieved for all restorations evaluated, partially because of the sectional matrix system used for the Class II restorations. While it is true that operator skill and experience play a role in successful manipulation of dental materials, the properties of the resin composite also play a major role. Leinfelder and others (1999) concluded that, while there is little evidence to support improved performance of packable composites such as Solitaire 2 over conventional composites, packable composites may allow for more convenient placement in posterior teeth.

Another important issue concerning the longevity of posterior resin composite restorations is the wear of the material. The wear rates of early composites were very high. Resistance to wear of resin composites has greatly improved as advances in the materials have been made. Newer composites have better physical properties, because of changes in filler content and technology and changes in their matrices and polymerization capability (Bayne, Heymann & Swift, 1994). Packable composites are designed specifically for use in posterior teeth. Say and others (2003) evaluated the wear and microhardness of several resin composites. The two packable composites tested exhibited less wear than the other composites tested. Another study also suggested that packable composites have improved wear resistance over some conventional composites (Clelland & others, 2003). In this study, the two-year response rate for anatomic form was 93% Alpha for one group and 91% Alpha for the other group, with no restorations falling in the Charlie group. These results support the conclusion that packable composites can successfully function for at least two years.

This study used two different bonding agents: a fourth-generation system (Gluma Solid Bond) and a fifth-generation system (Gluma Comfort Bond and Desensitizer). Fourth-generation, or two-step systems, generally consist of three components: a conditioner, a primer and a bonding resin. Having three components, fourth-generation systems have more steps than later generation systems. In an effort to reduce the number of steps and time involved in the bonding procedure, manufacturers have introduced bonding systems with a reduction in the number of components. This is the case when comparing the fourth- and fifth-generation systems. In the fifth-generation systems (sometimes called "single-bottle" or one-step bonding systems), the primer and bonding resin are combined. These systems still require a separate conditioning step and some require multiple applications of the primer/adhesive.

The performance of fourth-generation (two-step) bonding systems has been compared to fifth-generation (one-step) systems. In general, the clinical performance of fifth-generation bonding systems is poorer than with the older fourth-generation materials (Sunnegardh & Van Dijken, 2000; Van Dijken, 2000). Ernst and others (2002) evaluated the marginal integrity of fourth- and fifth-generation systems, including Gluma Comfort Bond and Desensitizer and Gluma Solid Bond. The fourth-generation multi-step systems demonstrated better marginal seals than the fifth-generation systems. Koh and others (2001) compared the tensile bond strength of fourth- and fifth-generation systems used with a packable resin composite. They demonstrated that fifth-generation bonding systems produced similar bond strengths to fourth-generation systems. Wilder and others (1998) had similar results, with no difference in bond strengths between fourth- and fifth-generation bonding systems. However, DeMunck and others (2003) investigated the effect of water degradation over a four-year period on fourth- and fifth-generation bonding systems and reported that the long-term tensile bond strengths of fifth-generation systems were significantly reduced compared to fourth-generation systems. Two other laboratory studies that compared microleakage and marginal sealing of fourth- and fifth-generation bonding systems demonstrated no differences between the two (Pilo & Ben-Amar, 1999; Yap, Ho & Wong, 1998).

CONCLUSIONS

Two-year data may not be sufficient to evaluate the longevity of a restorative material and long-term clinical studies are essential to fully and adequately evaluate tooth-colored restorative materials. In this era of increasingly difficult funding, it is challenging to provide long-term clinical evaluations, which are essential, since dental restorative materials have improved to the point that early failures are rare. In order to distinguish between materials, longer trials are absolutely required. At the end of this limited evaluation period of two years, Solitaire 2 was performing well. Although not statistically significant, the fourth-generation bonding agent Gluma Solid Bond performed better than the simpler fifth-generation bonding agent Gluma Comfort Bond and Desensitizer.

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Laboratory Research

Effects of Five Thermal Stressing Regimens on the Flexural and Bond Strengths of a Hybrid Resin Composite

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Clinical Relevance

The value of short- to medium-duration thermal stressing in the *in vitro* evaluation of resin composites remains questionable.

SUMMARY

Thermocycling is commonly employed in laboratory studies to simulate the *in vivo* aging of restorative materials. However, there is little consistency in the regimens used, and some researchers have questioned the clinical relevance and, hence, the necessity of including thermal stressing in *in vitro* protocols. This study examined the effects of five thermal stressing regimens on the flexural and dentin bond strengths of a hybrid resin composite. *Methods:* For flexural strength tests, 95 rectangular specimens (15 mm x 2 mm x 2 mm) were fabricated using a stainless steel split mold, then light cured for 60 seconds. For bond strength tests, 75 caries-free molars were flattened occlusally to expose dentin, then polished through 600 grit SiC paper; dentin surfaces were etched, rinsed and blotted dry. A dentin adhesive was applied and light cured for 30 seconds; resin composite was condensed through a stainless steel split mold (4.3 mm diameter x 3.5 mm high), then light cured for 60 seconds. All specimens were stored in deionized water for 24 hours, then stressed for 100 hours according to one of five regimens: 1) cycled between 5°C and 55°C (9000 cycles; 20-second dwell time); 2) held at 5°C constant; 3) held at 22°C constant; 4) held at 55°C constant; 5) held at 5°C for 50 hours, then at 55°C for 50 hours. Flexural strengths were measured using an Instron 5500R and three-point bending apparatus at a crosshead speed of 0.5 mm/minute. Shear bond strengths were measured using an MTS Bionix 200 at a crosshead speed of 0.5 mm/minute. *Results:* ANOVA revealed no significant differences in either flexural strength or shear bond strength among the five thermal regimens.

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INTRODUCTION

Dental materials researchers have sought for years to improve the clinical relevance of *in vitro* studies. Factors such as material storage media (Pokarier & Gage, 1989), experimental temperature (Tanaka & others, 1995) and duration of testing (Wegner, Gerdes & Kern, 2002) may affect the outcome of physical property testing of dental materials. One testing regimen that has become reasonably well accepted is thermocycling (Brown Jacobs & Thompson, 1972; Crim & Mattingly, 1981; Burger, Cooley & García-Godoy, 1992; Gale & Darvell, 1999). In theory, thermocycling simulates the *in vivo* aging of restorative materials by subjecting them to repeated cyclic exposures to hot and cold temperatures (Burger & others, 1992; Montes-G & Draughn, 1986). In general, physical properties tend to decrease (Draughn, 1981) or remain unchanged (Wendt, McInnes & Dickinson, 1992) following thermal stressing.

Resin composite restorative materials and adhesive systems, in particular, are sensitive to manipulative variables (Tanaka & others, 1995). Thermocycling is thought to be especially relevant to the evaluation of resin-based restorative materials, because it exposes the materials to "the extremes that conform to those in the oral cavity" (Rossomando & Wendt, 1995). According to Crim, Swartz and Phillips (1985), "The value of thermocycling is in its ability to demonstrate the relative effectiveness of different restorative materials and techniques to prevent microleakage at the tooth/restoration interface." Thermocyclic stress may induce a considerable amount of bond fatigue and microleakage at the tooth/restoration interface (Soh & Selwyn, 1992). Marginal leakage is thought to result from the difference in the coefficient of thermal expansion between the restorative material and tooth structure (Crim & Mattingly, 1981; Kidd, Harrington & Grieve, 1978). Water sorption, which may occur separately from thermocycling, has been suggested as influencing the physical properties of materials (Braem & others, 1995) and may, over time, reduce the sealing ability of restorative materials (Peterson, Phillips & Swartz, 1966).

Many studies have investigated the effects of thermocycling on the physical properties of dental materials, and some have questioned its value. Xalabarde and others (1998) compared the microleakage of a filled and unfilled sealant following storage in either distilled water for 48 hours or thermocycling (500 cycles; 5°C/55°C; 30-second dwell time). Thermocycling had no statistically significant effect on microleakage for either material. Wendt and others (1992) reported no significant differences in the microleakage of thermocycled (250 cycles; 5°C/55°C; 15-second dwell time) and non-thermocycled MOD resin composite restorations in molars. Furthermore, the authors suggested that the use of thermocycling to determine the effects of the coefficient of thermal expansion in resin composites is unnecessary. Aguiar and others (2003) found no differences in the microleakage of five restorative materials (one amalgam, one hybrid resin composite and three packable resin composites) following thermocycling (3000 cycles; 5°C/55°C; 60-second dwell time) as compared to non-thermocycled controls stored in 37°C distilled water.

Burger and others (1992) reported no significant differences in resin composite-to-dentin shear bond strengths following thermocycling (6°C/60°C; 30-second dwell time) for 100, 500, 1000, 2000 or 4000 cycles; however, a non-thermocycled comparison group was not included for evaluation. Similarly, other studies (Retief & others, 1988; Mandras, Retief & Russell, 1991) have reported that increasing the number of temperature cycles (from 250 to 1000 cycles) produced no significant differences in the microleakage of resin composite restorations. Mair and Vowles (1989) reported decreased fracture toughness among five of seven resin composite restorative materials following rigorous thermocycling (10,000 cycles; 3°C/60°C; 2.25-minute dwell time) but noted that this decrease was no greater than that caused by prolonged storage (42 days) in room temperature water.

In contrast, other researchers have noted significant effects of thermocycling. Lloyd, McGinley and Brown (1978) reported rapid enamel crack propagation during the first 2000 cycles (24°C/52°C; 30-second dwell time) and slow crack growth thereafter, stating that "*in vitro* thermal cycling tests cause as much damage within a few thousand cycles as appears to occur in *in vivo* observations over several years." The authors suggested that the magnitude of stress induced by rapid thermal changes depends on both the temperature difference between the tooth and the media and the thermal conductivity or heat transfer coefficient (the rate at which a material conducts thermal energy) of the tooth/restoration complex.

Eakle (1986) examined the effect of thermocycling on the fracture strength and microleakage of MOD resin composite restorations in maxillary premolars. Compared to specimens stored in 100% humidity at room temperature, specimens cycled between 5°C and 55°C water baths (480 cycles; 30-second dwell time) demonstrated significantly lower fracture strength. Microleakage, however, was not affected by thermocycling. Both the control and thermocycled groups exhibited extensive microleakage, but there was no significant difference in microleakage between the two groups.

Diaz-Arnold and Aquilino (1989) reported statistically significant decreases in the shear bond strengths of

three of four porcelain repair systems following thermocycling (5°C/60°C; 48 hours; 1776 cycles; 32-second dwell time) as compared to their respective non-thermocycled control groups. Chadwick (1994) reported product-specific variations in compressive strength and abrasive wear resistance, with no clear trends evident among three resin composite restorative materials following thermocycling (37°C/50°C/37°C/5°C or 37°C/60°C/37°C/5°C; 750 cycles; 90-second dwell time). Recent studies reported decreased bond strengths (Wegner & others, 2002; Xie, Dickens & Giuseppetti, 2002; Bishara, Ajlouni & Laffoon, 2003; Titley, Caldwell & Kulkarni, 2003; Kim, Pfeiffer & Niedermeier, 2003) and increased microleakage (Besnault & Attal, 2003; Wahab, Shaini & Morgano, 2003) among several resin-composite-based materials following various thermal stressing regimens.

Peterson and others (1966) compared the marginal leakage of four restorative resin materials. Class V preparations were restored and thermocycled (15°C/45°C or 0°C/60°C; 30-second dwell time) for 10, 50 or 100 cycles. For all materials, leakage among specimens cycled between 15°C and 45°C was similar to that of non-thermocycled specimens; however, leakage increased significantly for all materials when subjected to 0°C/60°C cycling. For both temperature regimens, marginal leakage increased further when the number of cycles increased from 10 to 100.

Momoi and others (1990) reported gradual increases in the marginal leakage of four resin composites thermocycled for up to 47 days (9000 cycles; 37°C/4°C/37°C/60°C; one minute dwell time at 37°C, two minutes at 4°C and 60°C); there were no differences in leakage among the four materials. Non-thermocycled control specimens of each material stored in physiologic saline at 37°C demonstrated no change in leakage over the same time period.

In addition to the effects of thermocycling temperature and duration (number of cycles), the effects of varying immersion media have been investigated. Crim and others (1985) studied the effect of different thermocycling regimens in tap water and fuchsin dye on the microleakage of Class V resin composite restorations in extracted human premolars. There were no significant differences in marginal leakage among the testing regimens. Pokarier and Gage (1989) thermocycled Class V resin composite restorations in artificial saliva or saline (500 cycles; 5°-10°C/45°-50°C; 330 minutes, or approximately 40-second dwell time) and examined them microscopically for marginal gaps. Restorations thermocycled in artificial saliva had significantly fewer marginal gaps than those thermocycled in saline. The authors suggested that the ionic strength of the thermocycling solution might influence marginal gap formation between restoratives and tooth structure.

Several authors have suggested standardized thermocycling regimens based on the temperature fluctuations normally encountered in the oral environment. Unfortunately, few studies have actually recorded *in vivo* observations. Nelsen, Wolcott and Paffenbarger (1952) reported intraoral temperature extremes of 9°C and 52°C following consumption of cold water (4°C) and hot coffee (60°C), respectively. Peterson and others (1966) recorded intraoral temperature fluctuations in two patients, ranging from 15°C to 45°C following consumption of ice water (0°C) and coffee (60°C), respectively. As a result, they suggested that *in vitro* thermal cycling extremes of 15°C and 45°C may be "more indicative of the actual conditions to which a restoration is exposed in the oral cavity."

Michailesco and others (1995) recorded *in vivo* temperatures in restored teeth during a meal. Three subjects ate foods ranging from hot coffee to ice cream. Temperatures were measured on the facial surface of amalgam, at the base of an amalgam and within the root canal of a pulpless tooth. A thermocouple was placed in each location and temperatures were recorded every two seconds over the duration of the meal, approximately 26 to 30 minutes. From a baseline of 35°to 37°C at all measurement locations, temperatures fluctuated within a 30°C range, from 18.9°C to 48.4°C. Fluctuations were greatest (29.5°C) at the base of the amalgam and least (11.8°C) within the root canal. Approximately 30 thermal cycles were recorded during the meal, with a relative change of 5°C for solids and 7°to 10°C for liquids. Based on these findings, the authors suggested a thermocycling range of 17°C to 47°C, with an intermediate phase at 37°C and 30 cycles representing each full meal but made no suggestions regarding immersion or exposure times.

Although it appears that a majority of *in vitro* studies utilize thermocycling to "stress" or "age" dental materials specimens, there is little agreement on the exact regimens that are appropriate or necessary (Crim & Mattingly, 1981; Crim & others, 1985; Michailesco & others, 1995). Research is often conducted using thermocycling regimens that are arbitrary, vary considerably from study to study and fail to simulate *in vivo* conditions (Rossomando & Wendt, 1995; Gale & Darvell, 1999). Michailesco and others (1995) reported an exposure time range in the literature from two seconds to four hours; one study (Buonocore, Sheykholeslam & Glena, 1973) boiled specimens for eight hours. In a review of 130 scientific papers utilizing thermal cycling regimens, Gale and Darvell (1999) calculated the following parameters: the median low temperature was 5°C (mean 6.6°C; range 0° to 36°C); median high temperature was 55°C (mean 55.5°C; range 40° to 100°C); median dwell time was 30 seconds (mean 53 seconds; range four seconds to 20

minutes); median number of cycles was 500 (mean 10,000; range 1 to 1,000,000).

Brown and others (1972) suggested that 3,650 cycles are equivalent to one year *in vivo*. However, they gave no reasoning as to how this number was derived. Gale and Darvell (1999) recommended 20 to 50 cycles per day as "a proposal on the basis that such cycles might occur between 20 and 50 times per day" and recommended 10,000 thermocycles as a number that could be reasonably accomplished in laboratory studies. None of these numbers were based on researched data. Based on their *in vivo* temperature data (described above), Michailesco and others (1995) suggested 30 thermocycles occurred during each meal, which, at three meals per day, equates to about 33,000 thermocycles per year.

This lack of consistency has made it difficult for the dental practitioner to compare the results of one study to those of another or to relate one material to another with regard to its clinical effectiveness and intraoral durability. Therefore, the purpose of this study was to evaluate the effects of five thermal stressing regimens which reflect 1) parameters commonly reported in previous *in vitro* studies and 2) temperature extremes reported in the few *in vivo* studies available on the flexural strength and resin-to-dentin shear bond strength of a hybrid resin composite.

Table 1: *Treatment Groups*

Group	Thermal Stressing Regimen	Temperature	Duration
1	Thermocycled	5°C/55°C	100 hours Dwell time = 20 seconds 9000 cycles
2	Water storage	5°C	100 hours
3	Water storage (control)	Room temperature (22°C)	100 hours
4	Water storage	55°C	100 hours
5	Water storage	5°C/55°C	50 hours @ 5°C, then 50 hours @ 55°C

METHODS AND MATERIALS

A hybrid resin composite (TPH Spectrum, Caulk/Dentsply, Milford, DE, USA) was chosen because of its ability to be used in anterior and posterior applications.

Flexural Strength: Ninety-five rectangular resin composite specimens (TPH Spectrum, Shade B1, Caulk/Dentsply) (15 mm x 2 mm x 2 mm) were prepared utilizing a stainless steel split mold and were covered with a glass slide. The specimens were polymerized for 60 seconds using four overlapping Luxor Lamps (ICI Dental, Macclesfield, UK) light curing units. Light intensity was measured using a radiometer (Model 100, Demetron Research Corp, Danbury, CT, USA) following every 10 specimens and was verified to be at least 350 mW/cm^2. Each specimen was sanded to remove surface irregularities, measured using digital calipers (Mitutoyo Corp, Tokyo, Japan) at six points along its length for dimensional consistency and adjusted to approximate the proper dimensions.

Shear Bond Strength: Seventy-five caries-free human third molars were mounted in phenolic rings (Buehler Ltd, Lake Bluff, IL, USA) with autocuring acrylic resin. The occlusal surface of each tooth was ground with a water-cooled model trimmer (Whip-Mix Corp, Louisville, KY, USA) to create a flat dentin surface with no remnants of enamel. The teeth were then polished with wet 240-, 400- and 600-grit silicon carbide abrasive paper (3M Dental Products, St Paul, MN, USA) on a water-cooled abrasive table (Handimet II, Buehler Ltd). Dentin surfaces were rinsed, etched for 15 seconds with 34% phosphoric acid, rinsed and blotted to a moist glossy surface (per manufacturer instructions). A dentin adhesive (Prime and Bond NT, Caulk/Dentsply) was applied and light cured for 10 seconds. Resin composite (TPH Spectrum, Shade B1, Caulk/Dentsply) was condensed onto the dentin surface through a stainless steel split mold (4.3 mm diameter x 3.5 mm high) with a large amalgam condenser, then light cured for 60 seconds. During specimen fabrication, all teeth remained submerged in water except for the three to four minutes required for the actual bonding procedure.

All specimens (flexural and bond strength) were stored in deionized water at 22°C for 24 hours to prevent dehydration and allow for additional cure of the material prior to testing. Next, specimens were randomly assigned to five treatment groups (flexural strength: n=19; shear bond strength: n=15) and thermally stressed in deionized water for 100 hours according to one of five regimens (Table 1): (1) cycled between 5°C and 55°C (9000 cycles; 20 second dwell time); (2) held at 5°C constant; (3) held at 22°C constant (control); (4) held at 55°C constant; (5) held for 50 hours at 5°C, then 50 hours at 55°C.

Flexural strengths were measured using an Instron 5500R (Instron Corporation, Canton, MA, USA) biomaterials testing machine with a 5 kN load cell and three-point bending apparatus at a crosshead speed of 0.5 mm/minute. The shear bond strengths were measured using a Bionix 200 (MTS Systems Corporation, Eden Prairie, MN, USA) biomaterials testing machine with a 1 kN load cell and knife-edge probe at a crosshead speed of 0.5 mm/minute. All specimens were tested in

random order using a computer-generated random number table.

Statistical Analyses: Data analysis was completed using the SPSS 8.0 computer software (SPSS Inc, Chicago, IL, USA). Mean (± standard deviation) flexural and shear bond strengths were calculated for each treatment group. For each property, category means were compared using a one-way Analysis of Variance ($\alpha = 0.05$).

RESULTS

Results are presented in Table 2 and in Figures 1 and 2. Mean flexural strengths ranged from 121.3 (± 15.3) MPa to 127.5 (± 15.0) MPa, while mean resin-to-dentin shear bond strengths ranged from 8.2 (± 4.6) MPa to 11.1 (± 3.7) MPa. ANOVA revealed no statistically significant differences in either flexural strength (p=0.741) or shear bond strength (p=0.352) among the five treatment groups.

DISCUSSION

This study differs from most previous research in that it evaluated not only the effect of a single thermocycling regimen, but also the effects of varying thermal stress exposures on the physical properties of a resin composite. Results indicated no significant differences in either flexural strength or resin-to-dentin shear bond strength of a hybrid resin composite subjected to five different thermal stressing regimens. These findings are consistent with others reporting no degradation in resin-to-dentin bond strengths (Retief & others, 1988; Mandras & others, 1991; Burger & others, 1992), fracture toughness (Mair & Vowles, 1989) or microleakage (Rossomando & Wendt, 1995; Li, Burrow & Tyas, 2002; Aguiar & others, 2003) following increased

Table 2: *Flexural and Shear Bond Strengths of Resin Composite (MPa)**

Treatment Group	Thermal Regimen (100 Hours)	Flexural Strength (MPa) n=19		Shear Bond Strength (MPa) n=15	
		Mean	Std Dev	Mean	Std Dev
1	Thermocycled (5°/55°C)	121.3	15.3	8.7	4.8
2	5°C	124.8	14.7	9.9	3.4
3	22°C (control)	125.0	15.4	8.2	4.6
4	55°C	127.0	17.0	8.6	4.7
5	50 hours @ 5°C, then 50 hours @ 55°C	127.5	15.0	11.1	3.7
		p=0.741		p=0.352	

One-way ANOVA (α< 0.05). No significant differences among treatment groups.

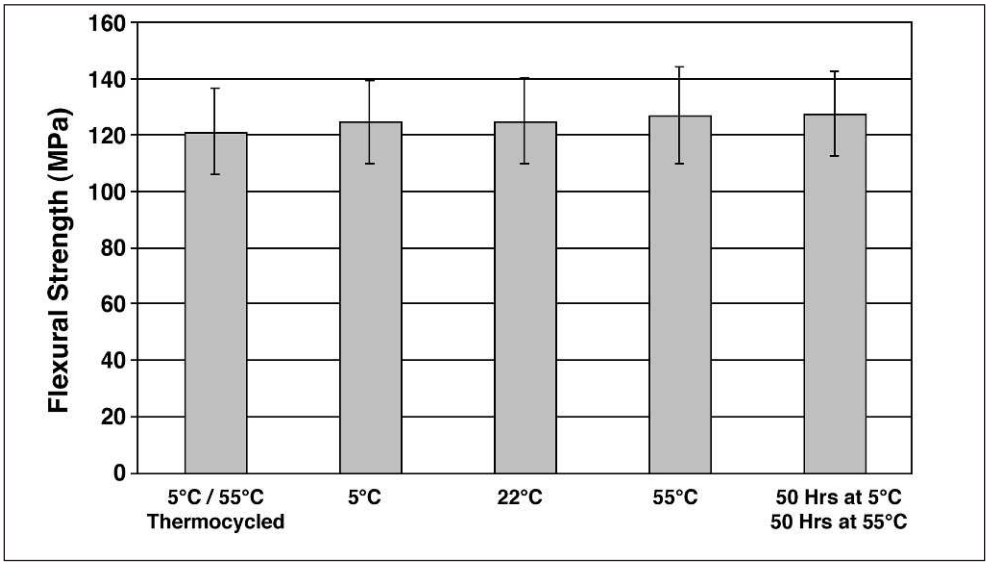

Figure 1: *Mean (± SD) flexural strengths of resin composite following thermal stressing (n=19).*

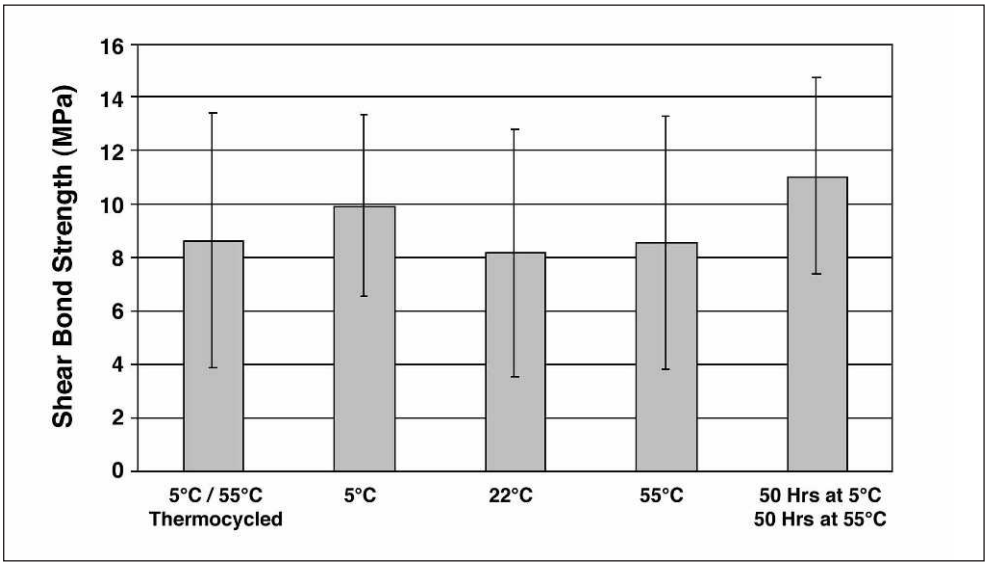

Figure 2: *Mean (± SD) shear bond strengths of resin composite following thermal stressing (n=15).*

thermal stressing. While other studies have reported the diminished physical properties of restorative materials following thermocycling compared to non-thermocycling, only two (Peterson & others, 1966; Momoi & others, 1990) reported a worsening of detrimental effects (increased microleakage) following exposure to increasingly severe thermal stressing regimens (increased temperature extremes or cycling duration). No other study comparing the relative effects of varying thermal regimens on contemporary resin composite materials has been found.

This study, however, must not be interpreted as suggesting that the thermal stressing of dental materials produces no ill effects or that some form of thermal stressing is unnecessary in *in vitro* protocols. This study did not measure 24-hour flexural or bond strengths; therefore, definitive conclusions regarding the absolute effects of thermal stressing on these properties cannot be made. However, since few studies have reported improvements in physical properties following either thermocycling (Tanaka & others, 1995; Xi & others, 2002; Bishara & others, 2003) or extended water immersion (Mair & Vowles, 1989; Wegner & others, 2002), the authors considered a comparison of the relative effects of varying thermal environments to be of greater importance than a comparison of pre- and post-thermal stressing properties.

Although the previous literature appears to be equivocal, studies that have reported the measurable effects of thermal stressing have generally utilized immersion (dwell) times of greater than 30 seconds (Lloyd & others, 1978; Eakle, 1986; Diaz-Arnold & Aquilino, 1989; Mair & Vowles, 1989; Chadwick, 1994). The clinical relevance of *in vitro* dwell times, however, remains largely unproven and open to speculation. Rossomando and Wendt (1995) have suggested that dwell time is important only if the restorative material is thermally conductive, and recommended short (10-second) durations, while Soh and Selwyn (1992) recommended dwell times of 60 to 90 seconds. The dwell time utilized in the current study (20 seconds) was selected to maximize both the exposure time during each cycle and the number of cycles that could be accomplished during the 100-hour experimental period; however, 20 seconds may be insufficient to induce degradation in physical properties that may occur with more extreme exposures.

It is of interest to note that the American National Standard/American Dental Association (ANSI/ADA) (2003) and International Organization for Standardization (ISO) (2001) Specifications for direct filling resins and dental adhesion vary depending on the physical property evaluated. For example, for flexural strength evaluation, material specimens should be stored in 37°C distilled water for 24 hours prior to testing; for water sorption evaluation, specimens should be stored in 37°C water for seven days. For adhesion and microleakage testing, specimens should be stored in 37°C for 24 hours. ISO Specification 11405 states, "Thermal cycling between 5°C and 55°C may be used as an accelerated aging test. Longer periods of water storage may be necessary to show durability of the bond." Recommended parameters include 1) 500 cycles, 2) an immersion time of at least 20 seconds in each bath and 3) a transfer time between baths of 5 to 10 seconds; for long-term evaluations, ISO recommends storage in water at 37°C for six months prior to testing. However, results of this study indicate no differences in either flexural or bond strengths among specimens stored at 5°C, 22°C or 55°C, suggesting that, at least for studies employing relatively short storage periods, storage temperature may not be a critical factor. Therefore, in light of this study and previous literature, we suggest that these guidelines be viewed as minimum parameters when thermocycling is to be included in laboratory protocols. As others (Wendt & others, 1992; Rossomando & Wendt, 1995) have suggested, the choice of appropriate thermal stressing parameters will likely depend upon both the materials and physical properties chosen for evaluation.

CONCLUSIONS

Under the conditions of this study, there were no differences in either the flexural strength or the resin-to-dentin shear bond strength of a hybrid resin composite when subjected to five different thermal stressing regimens. The practical value of short- to medium-duration thermal stressing in the evaluation of restorative dental materials remains in question. Further study is needed to delineate the relative contributions of temperature, dwell time and cycling duration to the deterioration of dental material properties.

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Disclaimer

The opinions expressed in this article are the private views of the authors and should not be construed as reflecting official policies of the US Army, US Navy, Department of Defense or the US Government.

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Hybrid Layer Thickness and Morphology: The Influence of Cavity Preparation With Er:YAG Laser

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Clinical Relevance

The thinner, irregular hybrid layer found when a cavity is prepared with a LASER may have a negative effect on bonding.

SUMMARY

Dentinal surfaces prepared with Er:YAG laser have significantly different characteristics from those prepared with conventional instruments. Different hybrid layer morphologies and thicknesses occur, which may result in differences in the quality of restorations placed on dentinal surfaces prepared with a diamond bur when compared with using an Er:YAG Laser. This study compared the hybrid layer thickness and morphology formed utilizing Scotchbond Multipurpose Plus (SBMP) on dentin prepared with a diamond bur in a high speed handpiece and dentin prepared with an Er:YAG laser. Flat dentin surfaces obtained from five human teeth were treated with the two methods and then with the dentin adhesive system according to the manufacturer's instructions. After a layer of composite was applied, the specimens were sectioned, flattened, polished and prepared for SEM observation. Ten different measurements of hybrid layer thickness were obtained along the bonded surface in each specimen. Results showed that SBMP produced a 3.43 ± 0.75 µm hybrid layer in dentin prepared with a diamond bur. This hybrid layer was regular and constantly found. In the laser group, the dentin adhesive system produced a 1.54 ± 0.35 µm hybrid layer that was very irregular and not found constantly. Statistical analysis of variance ($p \leq 0.05$) showed that there was a statistically significant difference between the groups. These data indicate that the Er:YAG laser, with parameters used in the experiment, is not a preparation method that allows for a thick hybrid layer formation, which

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is in opposition to using a diamond bur in a high speed turbine.

INTRODUCTION

One of the greatest challenges faced by dentistry today is obtaining an ideal restorative material. Characteristics of such a material include biocompatibility, fluoride release and recharge, adhesion to both enamel and dentin, esthetics, ease of use and an affordable cost, among others. This quest has been facilitated by Buonocore's (1955) studies on enamel acid etching development, and later, with the studies on hybrid layer development by Nakabayashi, Kojima and Masuhara (1982).

The hybrid layer appears to play a critical role in adhesive dentistry. In studies by Nakabayashi and Saimi (1996), a good quality hybrid layer is very important as a barrier against demineralization caused by the action of cariogenic agents. Krejci and others (1999) have also stated that an adhesive system should promote a perfect marginal seal, exhibit stability under occlusal load, and provide protection against secondary caries, marginal staining and post-treatment sensitivity. According to these authors, there is a close relationship between hybrid layer morphology and obtaining an excellent marginal seal. Therefore, tests evaluating the junction layer micromorphology and hybrid layer thickness should be carried out in order to evaluate an adhesive system. A significant amount of literature and research dealing with morphology evaluation and hybrid layer thickness is available (Ferrari, Cagidiaco & Mason, 1994; Uno & Finger, 1996; Ferrari & others, 1999; Nakajima & others, 2000).

Perdigão and others (1994) have stated that the dentinal substrate type could influence the adhesion mechanism and hybrid layer formation. If this is true, the manner in which the substrate is prepared for adhesion becomes critical. In other words, the preparation technique may influence the final restorative result. In studies evaluating preparation effectiveness, laser has compared favorably with conventional preparation methods using a high-speed turbine handpiece. Wright, McConnell and Keller (1993) carried out a pilot study comparing the microleakage of composite restorations in teeth prepared conventionally and those prepared with Er:YAG laser, with an energy of about 300 mJ and a frequency of 2 Hz. The authors concluded that there was no significant statistical difference between the studied groups, indicating that the use of Er:YAG laser had no significant influence on microleakage in adhesive filling procedures.

Other authors (Niu & others, 1998; Blankenau & others, 1999; Barceleiro & others, 2002) have conducted similar studies using different Er:YAG laser parameters but with similar results. Niu and others (1998) compared the microleakage of composite restorations in teeth prepared conventionally or with Er:YAG laser with an energy of about 200 mJ and a frequency of 10 Hz. Barceleiro and others (2002) used an energy of 400 or 500 mJ and a frequency of 8 Hz and also compared the microleakage of composite restorations. In all these studies, the authors showed that use of Er:YAG laser had no significant influence on adhesive procedures. Visuri and others (1996) evaluated the adhesion force of composite restorations in teeth prepared conventionally and those prepared with Er:YAG laser with an energy of about 350 mJ and a frequency of 6 Hz through shearing or traction tests and saw improvement in shear bonding after laser treatment.

On the other hand, other studies (Wigdor & others, 1993; Rechmann, Goldin & Hennig, 1998; Hossain & others, 1999; Tokonabe & others, 1999) have shown that dentinal surfaces prepared with Er:YAG laser present significantly different characteristics from those prepared with conventional rotary instruments. These studies have also shown that different parameters will produce different tissue characteristics. Hossain and others (1999) showed that there is a linear relation between the ablation rate and the used energy. The question exists, then, how can it be possible to have similar adhesion results (Wright & others, 1993; Visuri & others, 1996; Niu & others, 1998; Blankenau & others, 1999; Barceleiro & others, 2002) in different dentinal substrates (Wigdor & others, 1993; Rechmann & others, 1998; Hossain & others, 1999; Tokonabe & others, 1999).

Based on this question and using Krejci and others' (1999) statement that tests evaluating the junction layer micromorphology and hybrid layer thickness should be carried out in order to evaluate an adhesive system, the authors concluded that these tests should also be carried out in order to evaluate a cavity preparation method.

This study compared the hybrid layers formed between an adhesive system and dentin developed with two different cavity preparation methods—the diamond bur in a high speed handpiece used as a control method and the Er:YAG laser, using high energy parameters used as a test method.

METHODS AND MATERIALS

Five extracted mandibular molars were selected for this study. All the teeth were free from caries and previous restorations. The samples were cleaned with a periodontal curette and cleaned with a fine flour of pumice using a rubber cup in a low-speed handpiece for 30 seconds. The samples were then stored in 37°C distilled water that was changed every seven days. These teeth were longitudinally sectioned into four parts by means of a mesio-distal cut and a facio-lingual cut

using a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under a coolant water flow. After the initial sections were cut, the occlusal surface was removed by means of a horizontal cut, using a low speed diamond saw 1 mm below the DEJ.

After fragments had been obtained, they were randomly separated and divided into the following groups:

Group I–High Speed Turbine (Control) (n=5)

One section from each tooth was separated into an individual container containing distilled water and assigned a group number (B1 to B5). A #1013 diamond bur (KG Sorensen, Alphavile, Barueri, São Paulo, Brazil), placed in a high speed handpiece (Kavo) under abundant water spray, was used on the flattened occlusal surface of each section. The surface was prepared using random movements for 10 seconds, simulating the bottom of an occlusal cavity. One diamond bur was used for each dentin section.

Group II –Er:YAG Laser (n=5)

One section from each tooth was separated into an individual container containing distilled water and assigned a group number (L1 to L5). Throughout the study, the numbering of each section corresponded to the same section in each group. For example, the sectioned numbered L1 in this group corresponded to B1 in the previous group. The Fotona Twinlight Er:YAG laser (Fotona, 1210 Ljubljana, Slovenia) device was used to irradiate the previously flattened occlusal dentin sections. Laser was applied by means of a handpiece kept at a standardized distance and fixed with an orthodontic thread fastened to the handpiece. Scanning movements were randomly carried out for 10 seconds simulating the bottom of an occlusal cavity prepared with the mentioned laser according to the parameters described in Table 1.

After all occlusal surfaces had been prepared with a diamond bur or Er:YAG laser, all sections were conditioned with 37% phosphoric acid (Ivoclar-Vivadent AG, FL-9494, Schaan, Liechtenstein/Lot 0070799) for 15 seconds. After application of the conditioning gel, the surfaces were rinsed with distilled water for 15 seconds, then gently dried with oil and dust-free air for two seconds. The Scotchbond Multi-Purpose Plus (3M ESPE, St Paul, MN, USA) adhesive system was then applied according to the manufacturer's instructions. A summary of the instructions follows: A thin layer of the Primer (3M ESPE/Lot 9XD) was applied with the help of a brush and left undisturbed on the conditioned surfaces for 30 seconds. The solvent was then removed from the surface with oil and dust-free air jets for five seconds and a thin layer of the Adhesive (3M ESPE/Lot 9LE) was applied over it. The adhesive layer was light-cured for 20 seconds with a 3M light source (intensity = 400 mW/cm^2, evaluated by means of a radiometer, every 10 uses). The next step was to apply a 1-mm thick microhybrid composite layer, Fill Magic (Vigodent S/A 21041-150 RJ Brazil/Lot FM 04898), with A1 shade, in a unique increment covering all the occlusal surface, which was then light-cured for 40 seconds (Ferrari & others, 1999). The samples were then kept in distilled water for seven days.

After storage, a transverse section was made 5 mm below the tooth/composite interface by means of a diamond saw (Isomet); the roots of the sections were set apart, and the remaining portion was sectioned along the long axes through the middle of the composite, using the same diamond saw under abundant water spray. Two sections were obtained that were formed by enamel and dentin, the adhesive system and microhybrid composite. The two sections were hand-polished on wet 600-grit silicon carbide paper (Norton, Guarulhos, SP, Brazil), then polished on a felt wheel placed in a polishing device (Prazis). An alumina (AP-Paste SQ—Struers, São Paulo, SP, Brazil) polishing paste with 1 µm-sized particles was used until no grooves were observed with a 50x magnifying glass. After these procedures had been carried out, sections were again conditioned in distilled water.

After seven days, one section of each previously formed pair was gently decalcified with 37% phosphoric acid (Vivadent/Lot 0070799) for 10 seconds, rinsed with distilled water and deproteinized with 3% sodium hypochlorite for 60 seconds (Ferrari & others, 1999). After time had elapsed, the sections were rinsed with distilled water, placed on aluminum stubs and sputter-coated with gold (Edwards Coater S150B Sputter Coater, EMIF, University of Rhode Island, Kingston, RI, USA).

Table 1: *Fotona Twinlight Er:YAG Laser Parameters*

Wave Length	2.940 ηm
Pulse Energy	400 mJ
Frequency	8 Hz
Operation Mode	Pulse
Operation Distance	10 mm (Focal distance)
Beam Diameter	0.8 mm
Guiding Light	Diode laser (670 ηm with a 1 mW power)
Coolant	Spray air/water

Table 2: *Samples Distribution*

Group	Cavity Preparation Method	Number of Samples	Number of Measurements
I	Diamond bur	5	50
II	Laser	5	50
Total		10	100

Table 3: *Hybrid Layer Thickness in Group I (Diamond bur)*

Sample	Hybrid Layer Thickness (µm)									
B1	5.215	4.880	2.086	2.436	3.131	3.478	3.942	4.637	2.781	3.856
B2	2.552	3.538	2.544	2.300	3.410	4.635	2.604	3.710	3.345	4.023
B3	3.278	3.128	2.997	3.154	3.872	4.157	2.465	3.118	4.927	3.788
B4	2.665	3.600	3.477	3.015	3.245	3.592	3.592	4.404	4.410	3.824
B5	2.278	2.658	2.478	3.109	3.199	3.478	3.561	4.101	3.907	3.145

Table 4: *Hybrid Layer Thickness in Group II (Er:YAG Laser)*

Sample	Hybrid Layer Thickness (µm)									
L1	1.365	1.452	1.784	2.108	1.921	1.787	2.007	1.678	1.898	1.731
L2	1.159	1.622	2.318	1.275	1.549	1.395	1.789	1.656	1.439	1.437
L3	1.189	1.464	1.649	1.467	1.193	1.310	1.203	0.828	0.646	0.731
L4	1.446	1.795	1.385	0.997	1.205	1.364	1.474	1.964	1.823	1.742
L5	1.159	1.858	1.973	1.858	1.738	1.627	1.491	1.738	1.639	1.970

Table 5: *Mean Results in the Groups*

Group	Number of Samples	Number of Measurements	Mean Thickness (µm)	Standard Deviation
I	5	50	3.43	0.74
II	5	50	1.54	0.35

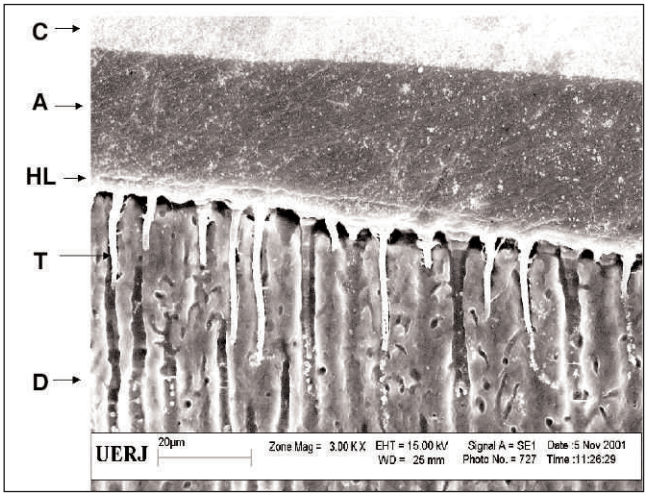

Figure 1. *Portion of the hybrid layer formed in sample B1. C = Composite; A = Dentinal Adhesive; HL = Hybrid Layer; T = Adhesive Tag; D = Dentin (magnification 3000x)*

After the metalizing procedure, the samples were evaluated under LEO 1450VP (LEO Electron Microscopy LTD, Cambridge, Cambridgeshire, UK) Scanning Electron Microscopy. Microphotographs of the hybrid layers were taken at standard magnifications (3000x). In each sample, 10 measurements of the hybrid layer thickness were done in this study as follows: Six measurements in the outer part of the hybrid layer were done—three on each side and four in the central part of the same layer. These measurements were carried out with the help of LEO-32 software (version 3.0) from LEO Electron Microscopy LTD, which is a component of the Electronic Microscope set. This software allows for measurements of distance between two points on an image, with a 2% error margin.

Table 2 shows sample distribution in the two groups in relation to cavity preparation modes.

RESULTS

The data derived from the two groups are indicated in Tables 3 (Group I—Diamond bur) and 4 (Group II – Er:YAG Laser).

The measurement data were statistically treated through the variance analysis with the SPSS for Windows release 5.0 program. The variance analysis ($p \leq 0.05$) showed that there was a significant statistical difference between the combined effects of the two treatments that had been carried out. The Tukey and Student-Newman-Keuls ($p \leq 0.05$) tests separated the treatment into two homogeneous and distinct groups, Group I ≠ Group II.

In Table 5, the average results obtained in the two groups can be observed after they had been statistically treated.

Figures 1 and 2 show differences between the hybrid layer thickness and morphology. These figures were obtained from fragments of the same tooth (Samples B1 and L1).

DISCUSSION

The high speed handpiece is the primary method for cavity preparation in dentistry. The alternative methods for cavity preparation, among them the Er:YAG laser, still present many deficiencies, while preparations are not standardized. A number of authors have recommended techniques and have noted some advantages for Er:YAG laser use, with emphasis on pain, noise and pressure reduction, and have considered the possible use of such preparation procedures in Class I, II, III, IV and V direct restorations (Chappell & others, 1994; Ferrari & Davidson, 1996). However, no author has reported the possible use of Er:YAG laser with indirect restorations, which require more defined cavitary preparations. It is because of this limitation, when recommending such procedures, that the high-speed turbine is still the most widely used method for cavity preparation in dentistry.

Experiments comparing the adhesive fillings microleakage data carried out in cavities prepared with different preparation methods are quite abundant in the literature and, in general, all work has shown that the studied methods present statistically similar results (Wright & others, 1993; Visuri & others, 1996; Niu & others, 1998; Blankenau & others, 1999; Barceleiro & others, 2002). Nonetheless, according to Krejci and others' (1999) description, tests that only evaluate bonding forces would be important for an adhesive system quality evaluation. However, these studies should not be used as the sole or primary parameters for recommending an adhesive system. According to these authors, a perfect marginal sealing would seem more important, therefore, marginal adaptation tests would have a greater clinical value. According to them, there is a close relationship between the hybrid layer morphology and a perfect marginal sealing; therefore, tests that evaluate the hybrid layer micromorphology and evaluate the hybrid layer thickness should always be carried out to evaluate an adhesive system quality.

The hybrid layer is extremely dependent on the dentinal substrate over which the layer is being produced (Perdigão & others, 1994). Because of this, the manner in which the dentin is prepared may be important. Studies by Wigdor and others (1993) and Tokonabe and others (1999) indicate that preparations prepared with a laser show lack of the smear layer and present open dentinal tubules after preparation, which is quite different from preparations carried out with diamond points.

This study followed the methodology used by Ferrari and others (1999). All steps that were carried out, including tooth preparation and storing, tooth cutting, samples preparation to visualization in SEM, among others, were based on the mentioned work. The only exception to the Ferrari and others (1999) study was in the dentinal substrate preparation method (diamond point and laser) and the adhesive system used.

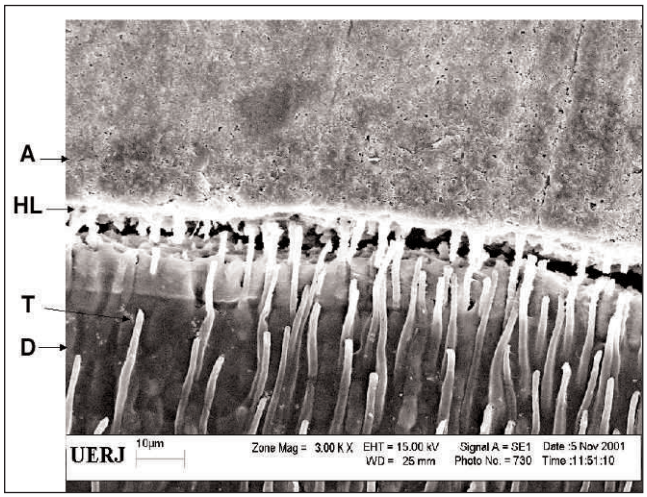

Figure 2. *Portion of the hybrid layer formed in sample L1. A = Dentinal Adhesive; HL = Hybrid Layer; T = Adhesive Tag; D = Dentin (magnification 3000x)*

In this work, tooth fragments were used, making up two groups that always originated from the same tooth, according to the description in the Method and Materials section. The aim of this procedure was to standardize the dentinal substrate where the superficial treatment had been carried out, so that comparisons could be done using the same dentin pattern. This contrasts with many other studies where experiments are carried out in multiple teeth that could exhibit different dentin characteristics.

The average result analysis obtained in Group I (3.43 ± 0.74 µm) showed similarities with the existing literature (Chappell & others, 1994; Ferrari & others, 1994; Ferrari & Davidson, 1996; Prati & others, 1999). Their studies indicated that, when using the adhesive system utilized in this study, the hybrid layer thickness average results varied between 3 and 5 µm.

A simple comparison between the average results obtained in Groups I (3.43 ± 0.74 µm) and II (1.54 ± 0.35 µm) shows that the Er:YAG laser, with the parameters used in the experiment, is not a preparation method that allows a thick hybrid layer formation when compared to using a diamond in a high speed turbine. Table 5 indicates that the groups were statistically heterogeneous, Group I being greater than Group II. However, more important than the comparison between the obtained measurements was the fact that, in the samples belonging to Group II, the hybrid layer was too irregular and its measurement was more difficult to achieve than in Group I. Unlike what happens in Group II, the hybrid layer could be observed all the time in Group I, in a very even way, and the measurements were also easily done.

Mello (2001) described the Er:YAG laser, when used according to certain parameters on healthy dentin, as promoting fusion between the dentinal components and a later solidification, leading to a solid structure formation with a mineral composition that is still unknown. According to Mello (2001), it is known, for example, that the structure is extremely acid resistant and presents completely altered collagen fibers composition mixed in the formed structure. It is believed that the structure is more mineralized and could be a poor substrate for the hybrid layer formation, which would agree with Perdigão and others' (1994) statement that a more mineralized structure is a poor substratum for adhesion.

With the applied methodology, it was not possible to find out the cause of the obtained results in Group II, both regarding measurement results and in relation to the formation inconstancy. It is believed that the obtained results may relate to the following explanation: in the place where there is a perpendicular laser beam incidence, an acid resistant structure formation will occur, as has already been described, thus, in this place, the hybrid layer formation would suffer damage. In the region around the laser beam incidence, a lower temperature increase would occur, promoting a less stressing alteration in the collagen fibers structure, and the formed structure would be less acid resistant, leading to an irregular, not so thick hybrid layer formation. This explains the hybrid layer formation inconstancy; the observed hybrid layer forming over the peripheral region of the laser beam incidence.

When looking for a better hybrid layer quality formation over dentin prepared with the Er:YAG laser, it is believed that some changes in the operation parameters could generate different results. Additional studies are in order, including alterations in parameters used by the laser device, which would promote less alterations in the dentinal structure, use acid etching for a longer period or use more concentrated acids, trying to promote a greater demineralization of the dentinal structure altered by laser or use adhesive systems different from the adhesive system used in the experiment. Another suggestion would be to elaborate on works that would try to explain, using all parameters used in this experiment, how a hybrid layer so different from what is commonly found can obtain microleakage results or adhesion force results similar to the those found in tests that use substrates where adhesion occurs, as was described by Nakabayashi and others (1982). That is, how would it be possible that works using the same parameters here (Barceleiro & others, 2002) have microleakage results statistically similar between the group prepared with a high speed diamond bur and the group prepared with the Er:YAG laser.

CONCLUSIONS

Through analysis of the results obtained in this *in vitro* study, it was possible to conclude that:

1. The use of Scotchbond Multi-Purpose Plus adhesive system on dentin prepared with a high speed diamond bur produces a hybrid layer with an average thickness of 3.43 ± 0.74 µm, with an even and continuous layer;
2. The use of Scotchbond Multi-Purpose Plus adhesive system on dentin prepared with Er:YAG laser produces a hybrid layer with an average thickness of 1.54 ± 0.35 µm, with an irregular and inconsistent layer;
3. The preparation using a high speed diamond point allows for a thicker hybrid layer formation than the one done with the Er:YAG laser.

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The Microtensile Bond Strength of Fuji IX Glass Ionomer Cement to Antibacterial Conditioned Dentin

MG Botelho

Clinical Relevance

One percent concentration antibacterial dentin conditioners can be left *in situ* without affecting the bond strength of GIC to dentin.

SUMMARY

Introduction: Adding antibacterial agents to a dentin conditioner used for a glass ionomer cement (GIC) has been shown to be antibacterial; however, it is not known whether this antimicrobial conditioning agent affects the bond strength to dentin *in situ*. This study applied GIC to antibacterial conditioned dentin without rinsing and determined whether there is an affect on the material's bond strength.

Materials and Methods: Chlorhexidine acetate (CX), benzalkonium chloride (BC) and cetrimide (CT) were added to Dentin Conditioner (DC) (GC Corp, Japan) at 1% and 5% concentrations. Molars were sectioned coronally to expose dentin, onto which 50 µl of the test conditioners was applied for 20 seconds with a gentle scrubbing action and the residual liquid was blotted dry, as would occur under "field" conditions when performing atraumatic restorative therapy. To serve as the control, the DC was left *in situ* and compared to the DC that was washed off. Proportioned Fuji IX GIC (GC Corp, Japan) was built-up on the prepared dentin surface and varnish was applied and stored for 24 hours. An annular saw was used to create sticks of GIC bonded to dentin, with a bonding area 1 mm². After 24 hours, the specimens were tested to failure in a Universal testing machine at a crosshead speed of 1 mm/minute.

Results: Five percent CX-DC was not tested, as it formed a precipitate. Results in MPa: DC-not washed, 9.3 ± 2.4; DC-washed, 9.3 ± 2.5; 1%BC-DC, 8.8 ± 2.5; 1%CX-DC, 8.7 ± 2.7; 1%CT-DC, 8.2 ±1.7; 5%CT-DC, 8.1 ± 2.7; 5%BC-DC, 5.4 ± 1.0. One-way ANOVA showed that there was a significant difference between the test groups ($p<0.05$), and Tukey's studentized range test showed that only 5% BC-dentin conditioner left *in situ* was significantly different from the other groups.

Conclusion: Under the conditions tested, only the 5% BC-DC left *in situ* affected the bond strength of Fuji IX to dentin.

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INTRODUCTION

Glass ionomer cements (GICs) are considered the material of choice when dental caries and cavity preparation are managed with the use of dental hand instruments when performing atraumatic restorative therapy (ART). However, the use of dental hand instruments are known to be inefficient at removing infected dentin (Terashima & others, 1969) and, in such situations, residual caries is likely to be restored over. This also occurs when decay is close to the pulp and a decision is made to leave the carious tissue *in situ* in an attempt to preserve tooth vitality.

A cause for concern in such situations is that the infected dentin that is incarcerated under a dental restoration has the potential for secondary caries. This is, in fact, the greatest cause of GIC restoration failure (Burke & others, 1999; Mjör, 1997). Therefore, the use of an antibacterial in the GIC restorative process may help to reduce or eliminate cariogenic bacteria that may contribute to secondary caries and failure of the restoration. The combination of cationic disinfectants with GIC powder (Botelho, 2003) and GIC dentin conditioner (Botelho, 2002) have been shown to have an antimicrobial effect *in vitro*. However, the addition of antibacterial agents with the GIC powder has been shown to have an adverse effect on the material's compressive strength (Botelho, 1998). There are benefits to delivering antibacterial agents with dentin conditioner, as this allows targeting of the antimicrobial to potentially infected dentin, while at the same time minimizing any impact the agent may have on the physical properties of the GIC material. However, it is not known whether such an antibacterial conditioning agent may affect the bond strength of GIC to dentin.

GIC dentin conditioners are considered a useful step in the GIC restorative process, as they have been shown to increase the bond strength of GIC materials to dentin surfaces (Galun, Saleh & Lewinstein, 1994; García-Godoy, 1992; Hewlett, Caputo & Wrobel, 1991; Joynt & others, 1990; Peutzfeldt, 1996; Peutzfeldt & Asmussen, 1990; Powis & others, 1982; Prati, Nucci & Montanari, 1989; Tanumiharja, Burrow & Tyas, 2000; Tay & others, 2001) and the clinical retention rate of GIC restorations (Ngo, Earl & Mount, 1986). However, other studies have shown little or no difference in the bond strength of GIC to conditioned dentin (Aboush & Jenkins, 1987; Caples, McInnes-Ledoux & Weinberg, 1988; Hewlett & others, 1991; Peutzfeldt, 1996).

Polyacrylic acid has been used as a dentin conditioner for GIC, as it creates a clean surface by removing the smear layer and surface contaminants without opening the dentin tubules too widely (Powis & others, 1982). Polyacrylic acid is also thought to enhance wetting of the tooth surface to a water containing cement by preactivating the calcium and phosphate ions in the dentin, thereby making them more available for ion exchange with the GIC (Watson, 1999). Given that polyacrylic acid is an integral component of the GIC, any dentin conditioner residue present at the time of restoration may be expected to have little effect on the nature of the bonding interface.

Recently, microtensile bond strength testing has become increasingly used for *in vitro* dental bonding investigations; it reduces the number of teeth required for testing, thereby reducing multiple variables, and the testing of smaller bonding areas produces higher bond strength values and lower scatter of data (Sano & others, 1994). This study compared the effects of different antibacterial dentin conditioners on the microtensile bond strength of a GIC to dentin.

METHODS AND MATERIALS

GC Dentin Conditioner (GC Corp, Tokyo, Japan) is a 10% polyacrylic acid solution chosen because it is the recommended conditioner for use with Fuji IX, (GC Corp), a GIC intended for use in ART. Antibacterial agents that have been shown to be effective against oral cariogenic bacteria (Botelho, 2000) were chosen for this investigation: chlorhexidine acetate (CX), benzalkonium chloride (BC) and cetrimide (CT) (Sigma, St Louis, MO, USA). The antibacterial conditioners were prepared to 1% and 5% concentrations in the dentin conditioner from 20% w/v sterile aqueous solutions and stored at 4°C. The 5% chlorhexidine acetate was observed to form a discreet, fine, white precipitate and therefore was not investigated at this concentration.

Recently extracted third molars from adults of both genders were stored in 0.5% chloramine-T and used within two months following collection. The dentin surfaces were prepared by removing the occlusal enamel by sectioning the crown of the tooth using an in-house custom made slow speed circular saw with a diamond impregnated disc. The root tip was then sectioned 2 mm from the apex and a dental milling machine (Meteaux Precieux, Neuchatel, Switzerland) was used to prepare a 2.4-mm diameter hole up the long axis of the root into which a stainless steel threaded screw was cemented with a zinc polycarboxylate dental cement (Denstply DeTrey, Konstanz, Germany). This was done to facilitate subsequent finishing and sectioning of the tooth for the creation of the test specimens.

The tooth was then held by the cemented screw in the chuck of a lathe, and the coronal dentin surface turned with a tungsten carbide cutting tool at slow speed under water to create a flat dentin surface perpendicular to the mounting screw. This would then allow sectioning of the specimens so that the bonding interface would be at right angles to the length of the test beams, thereby ensuring tensile loading would occur during mechanical testing.

Visual inspection with a stereomicroscope was then performed to determine if any residual enamel was present on the dentin surface to ensure no enamel bonding would occur. Enamel at the periphery of the tooth was removed using a diamond bur in a dental air turbine handpiece under water coolant for similar reasons. The tooth was then placed back in the lathe and, while rotating, a 180 grit silicon carbide paper was applied to the bonding surface to create a smear layer.

A metal matrix strip was then retained around the tooth with a Siqveland matrix band to support the GIC during setting. A Brinkman micropipette (Brinkmann Instruments Inc, Westbury, CT, USA) was used to apply 50 µls of the test conditioners to the prepared dentin surface for 20 seconds. A gentle scrubbing action was performed with a mini-sponge held in college tweezers and then gently blot dry with lint free tissue. This was performed to allow the residual antibacterial dentin conditioner to be left *in situ* as might occur in the "field" setting when providing ART restorations.

Fuji IX was weighed out to the recommended powder-liquid ratio, mixed by hand, then loaded into the matrix band to a height of approximately 5 mm. The excess was then compressed gently with a transparent Mylar matrix strip and allowed to set for four minutes in a humidor. The matrix band was removed and GC dental varnish (GC Corp, Tokyo, Japan) applied, then the specimen was placed in a 5 mL plastic vial at 100% humidity.

After 24 hours, the mounting screw of the test specimen was secured to the mounting arm of the Microslice 2 annular saw (Ultra Tech, Santa Anna, CA, USA) and positioned so that the cutting plane was on the long axis of the mounting screw. The tooth was then carefully sectioned under water cooling to create 1-mm wide slices into the tooth-GIC specimen. The mounting screw was then loosened and the tooth rotated 90° and sectioned again to produce sticks of GIC bonded to dentin with a bonding area 1 mm^2. The mounted specimen was then removed and sectioned on a custom-made circular saw through the tooth to release the test specimens. Given the different sizes of the tooth samples, different numbers of test specimens were produced for testing.

Immediately after sectioning, the GIC bonded specimens were secured with cyanoacrylate in a Bencor Multi-T testing jig (Danville Engineering, San Ramon, CA, USA) and tested at a crosshead speed of 1 mm/minute until failure in a universal testing machine (Instron 4440, Instron, Canton, MA, USA), after which the force in Megapascals was calculated.

The specimens were then examined for the nature of their fracture under a stereomicroscope (Nikon Corporation, Kawasaki, Japan) and kept for scanning electron microscopic (SEM) examination. The specimens evaluated by SEM were dehydrated, coated with gold/palladium and examined using a scanning electron microscope (Cambridge Stereoscan 440, Cambridge, UK) operating at 10-20kV.

A one-way Anova was performed to identify whether there was a significant difference between the test groups and a Tukey's post-test analysis was used to identify differences between the groups with a significance level set at $p<0.05$.

RESULTS

There was no difference in the mean bond strength of the dentin conditioner whether it was rinsed or left *in situ* to dry, with both test groups having a mean of 9.3 MPa (Table 1). A one-way ANOVA showed that there was a significant difference between the test groups ($p<0.05$), and Tukey's studentized range test showed that only 5% BC-dentin conditioner left *in situ* was significantly different (Table 1).

Chlorhexidine gluconate was initially selected, as it is available as an aqueous solution and therefore thought simplest to combine with the dentin conditioner. However, it was noted that the Chlorhexidine gluconate solution formed a fine white precipitate when combined at 1% concentration with the Dentin Conditioner, and the 5% Chlorhexidine gluconate-Dentin Conditioner produced a solidified blue gel with a white precipitate within a few minutes. Chlorhexidine acetate was then combined with the dentin conditioner, as chlorhexidine hydrochloride is not very water-soluble. However, the 5% Chlorhexidine acetate-Dentin Conditioner produced a noticeable white precipitate; at the 1% concentration, no precipitate was visible. Based on these findings, the 5% CXA-dentin conditioner was excluded from investigation and only the 1% CXA-dentin conditioner was investigated.

Visual examination of the debonded specimens under stereomicroscope showed that the test specimens failed

Table 1: *The mean tensile bond strength of dentin bonded glass ionomer (SD). Groups with no significant difference between them are joined with a vertical bar (p>0.05). The number of specimens per tooth varied, given the different size of the tooth samples.*

Test Groups	# of Samples	Tensile Bond Strength MPa (SD)
Dentin conditioner not washed	30	9.3 (2.4)
Dentin conditioner washed	25	9.3 (2.5)
1% BC-dentin conditioner	31	8.8 (2.5)
1% CXA-dentin conditioner	27	8.7 (2.7)
1% CT-dentin conditioner	30	8.2 (1.7)
5% CT-dentin conditioner	27	8.1 (2.7)
5% BC-dentin conditioner	24	5.4 (1.0)

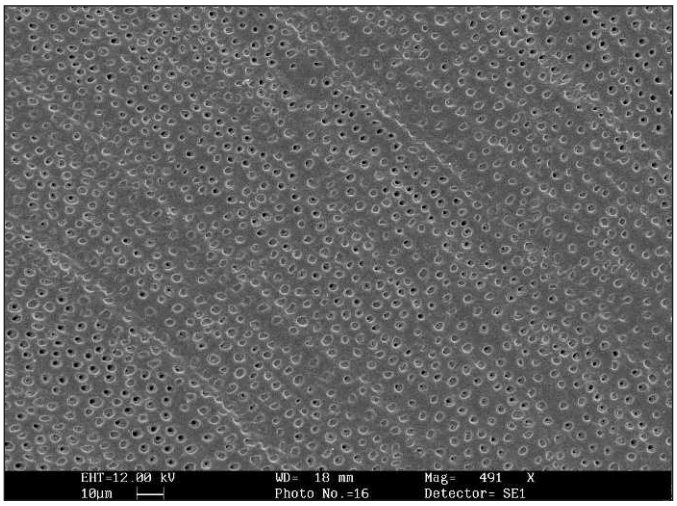

Figure 1. *SEM view of 1% CX-dentin conditioned surface that was allowed to dry* in situ*. The dentin surface appears clear of debris with open, partial and completely occluded dentinal tubules.*

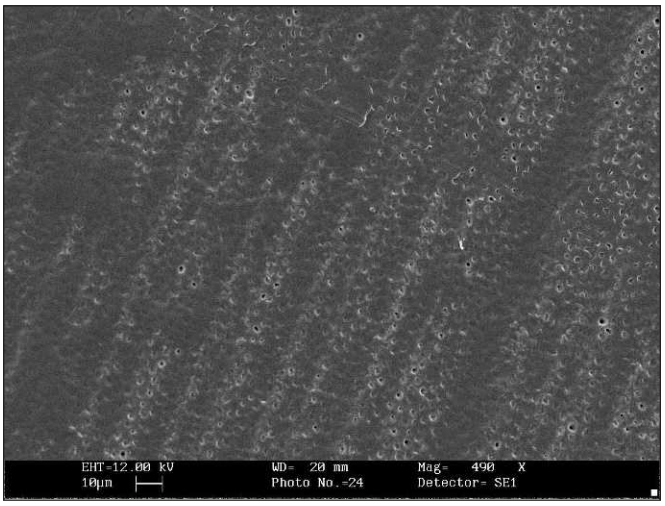

Figure 2. *SEM view of 1% CT-dentin conditioner allowed to dry* in situ*, also showing an unclean dentin surface with some open and partially open dentinal tubules.*

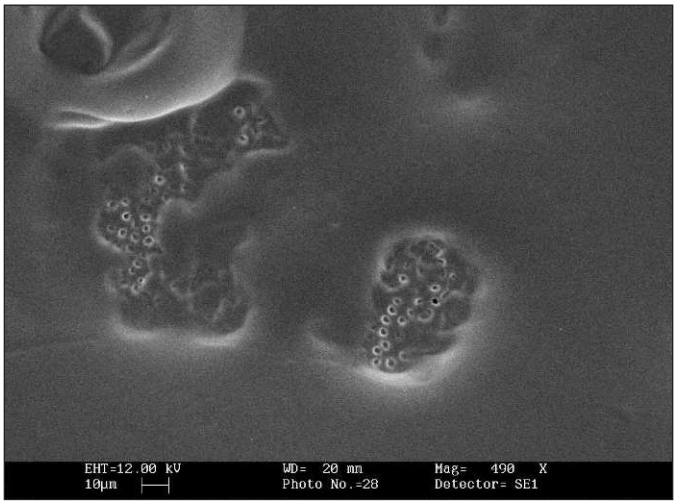

Figure 3. *This SEM view shows a thick residual layer of 5% BC-dentin conditioner with small areas of dentin showing open dentinal tubules.*

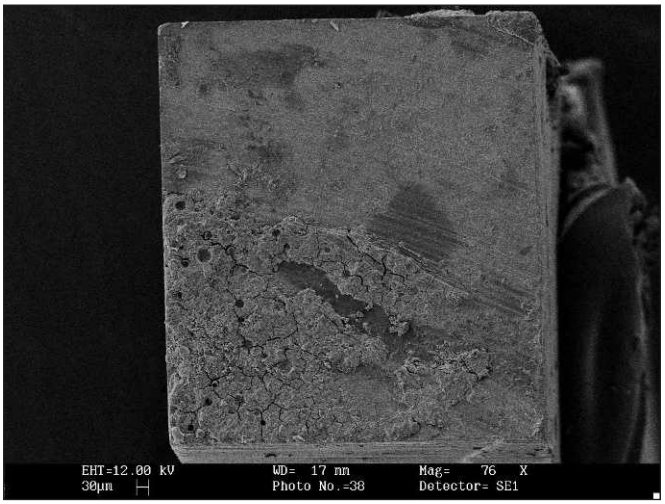

Figure 4. *This SEM shows a debonded specimen after 5% BC-dentin conditioner application with a significant area of adhesive failure in the top right hand side with dentin exposed and an amorphous coating likely to be a residual conditioner. The lower left half of the specimen shows residual GIC, indicating a cohesive failure.*

predominantly, cohesively apart from the 5% BC-dentin conditioner where observable areas of adhesive failures occurred.

The use of the Dentin Conditioner and 1% CX-dentin conditioners which were blot dried from the surface of the dentin, produced surfaces that appeared smear layer free (Figure 1). The use of 1% CT-dentin conditioners (Figure 2) revealed coverage of the dentin surface with an amorphous layer and only some dentinal tubules were apparent. This covering is presumed to be either the residual smear layer, remnants of the antibacterial conditioner or a combination of both.

The application of 5% BC-dentin conditioner with a gentle scrubbing action for 20 seconds and subsequent blotting dry revealed a residual viscous coating on the dentin surface that was visible with the naked eye. Under SEM examination, the thick adherent mass is visible with some areas of dentin exposed (Figure 3). Close inspection of the bonding interface of the fractured specimen of the 5% BC-DC showed significant areas of adhesive failure (Figure 4).

DISCUSSION

While *in vitro* strength testing gives no indication of clinical performance (Anusavice & de Rijk, 1990), it is a useful screening stage to compare experimental variables before *in vivo* testing. The advantages of microtensile bond strength testing are that multiple specimens can be produced from one tooth, no matrix is needed to limit the area for bonding, as sectioning of the tooth

determines the area of bonding, and there is a lower variance of data results (Cardoso, Braga & Carrilho, 1998; Pashley & others, 1999). The lower variance is thought to be due to a reduction in flaw density at the interface, both in the dental material and dentin substrate, thereby allowing higher bond strengths to be achieved with smaller specimens (Cardoso & others, 1998; Phrukkanon, Burrow & Tyas, 1998; Sano & others, 1994); this is the basis of Griffith's defect theory (Griffith, 1920). The small size of the specimens also allows for better stress distribution during test loading so that there are fewer complex cohesive failures that can occur in tooth tissue when compared to conventional testing (Pashley & others, 1995).

Table 2: *A comparison of studies reporting on the microtensile bond strength MPa (SD) of GIC to dentin. (Applied dentin conditioner left* in situ *and not washed off is marked with an asterisk.)*

	Fuji IX	Other GICs	Area of Bonding
Tanumiharja & others, 2000	8.5 (2.9) Fuji IX GP capsulated		1.2 mm diameter
Yip & others, 2001	12.4 (8.6) Fuji IX GP capsulated	Chemflex 15.0 (9.3)	0.81 mm^2
Tay & others, 2001		Chemflex 14.0 (3.4) 14.0* (3.7)	0.81 mm^2
Burrow & others, 2002	12.2 (3.0) Fuji IX GP capsulated		1.2 mm diameter
Current study	9.3* (2.4) 9.3 (2.5) Fuji IX hand mixed		1.0 mm^2

The use of a dentin conditioner as a vehicle for delivering antibacterial agents may be considered appropriate, as it is a recommended stage in the restorative procedure and allows for targeting of the antibacterial to infected dentin. In addition, as polyacrylic acid is an integral component of the GIC, it may be expected to have little or no effect on the setting or bonding mechanisms of the dental material (Watson, 1999).

SEM views showed little difference in the appearance of the conditioned dentin surfaces that were either washed or unwashed; this observation was supported by the identical bond strength values of the two groups. This finding was confirmed by Tay and others (2001), who also found no significant difference in the bond strength of GIC to washed or unwashed conditioned dentin (Table 2).

A comparison of other studies that bonded Fuji IX GIC to dentin shows the bond strength values of this study to be similar to those of Tanumiharja and others (2000), however, they are slightly lower than those of Burrow, Nopnakeepong and Phrukkanon (2002) and Yip and others (2001) (Table 2). This may be attributed to minor differences in the microtensile bonding technique or the fact that capsulated versions of the GIC material were tested. The slightly larger surface area for bonding used in the current study may be expected to have a greater number and size of flaws in the test material, thereby contributing to an earlier fracture. Also, mechanically mixed encapsulated cements are thought to be stronger than hand-mixed materials, based on a faster, more predictable and homogeneous mix of the cement. Hand-mixing has been shown to entrap larger air bubbles in the setting cement compared to capsulated cements (Covey & Ewoldsen, 2001; Mitchell & Douglas, 1997); such air bubbles may act as foci for stress concentration and possible premature failure (Tanumiharja & others, 2000). This is supported by the finding that mechanically mixed GICs significantly increase the strength of Fuji IX GP (Fleming & Zala, 2003) and Fuji II LC (GC Corp) (Covey & Ewoldsen, 2001) when compared to their hand-mixed counterparts. Despite these observations, however, Nomoto and McCabe (2001), using a different GIC, have reported no difference in the compressive strength between hand-mixed and mechanically mixed GICs. Finally, it is known that the same dental materials testing procedures carried out at different test centers will produce varying results highlighting the sensitivity of some testing procedures (McCabe & others, 1990).

The 5% BC-dentin conditioner significantly reduced the bond strength of the GIC to dentin, which is attributed to the residual adherent film observed when allowed to dry *in situ*. A thinner, more discreet coating was apparent with the 1% BC-dentin conditioner, although this did not have a significant difference on the control specimens.

Other studies have reported the formation of a white precipitate between chlorhexidine and an acid; in this case, phosphoric acid etchant (Chan & Hui, 1992) most likely represented a salt formation between the chlorhexidine and phosphate groups of the acid. Therefore, if a chlorhexidine polyacrylic dentin conditioner were to be investigated further, the long-term stability of such a combination would have to be monitored.

CONCLUSIONS

Under the limitations of the conditions tested, the combination of antibacterials with a 10% polyacrylic acid dentin conditioner showed no adverse affect on the microtensile bond strength of Fuji IX to dentin when left *in situ*, apart from the 5% BC-dentin conditioner.

Previous studies have shown that the combination of cetrimide with Fuji IX GIC and with Fuji Dentin Conditioner to have significantly greater bacterial inhibitory effect *in vitro* than comparable combinations of chlorhexidine, cetylpyridinium chloride and benzalkonium chloride (Botelho, 2002, 2003). Taking this and the fact that CT-dentin conditioners do not significantly affect the bond strength of Fuji IX to dentin into account, further investigations are warranted into the potential use of CT antibacterial dentin conditioner.

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Effect of Peroxide-based Bleaching Agents on Enamel Ultimate Tensile Strength

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Clinical Relevance

According to results of this *in vitro* study, the intrinsic strength of enamel can be affected after peroxide bleaching regimens; however, its *in vivo* effect must be further evaluated, since no clinical reports about bleached enamel fractures have been described.

SUMMARY

This study evaluated the effects of peroxide bleaching regimens on the ultimate tensile strength (UTS) of human enamel. A resin composite block was built-up on the bonded occlusal surface of 14 extracted, sound, erupted third molars to enable posterior preparation for the microtensile test. The bonded teeth were serially sectioned in a buccal-lingual direction into approximately 0.7-mm thick slices. Each slice was trimmed with a fine diamond bur to reduce the area of the buccal, internal slope of the cusps to a dumb-bell shape with a cross-sectional area of less than 1 mm². The samples were randomly divided into seven groups (n=10): unbleached control group and bleached groups treated with six bleaching regimens. The specimens were tested in tension at 0.5 mm/minute and the data were analyzed by ANOVA and Tukey test.

Specimens from the control group presented 51.3 ± 8.6 MPa, while the UTS of bleached enamel ranged from 22.0 ± 5.6 to 36.3 ± 9.1 MPa. All bleaching procedures significantly reduced enamel UTS ($p<0.05$). Differences were also observed among treatments. The results suggested that bleaching regimens can significantly reduce enamel UTS.

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INTRODUCTION

According to application mode and peroxide concentration, two methods of whitening procedures are generally used in aesthetic dentistry to treat discolored and stained teeth. Bleaching agents for professional use only contain high concentrations of carbamide peroxide (35-37%) and hydrogen peroxide (30-35%) solutions, while

at-home whiteners contain low concentrations of both peroxides and are used in a custom tray under the supervision of a dentist (Li, 1996; Haywood & Robinson, 1997).

High concentrations of hydrogen peroxide have been used to whiten teeth for more than 50 years and it was the first effective technique for in-office tooth bleaching (Smith & McInnes, 1942; Corcoran & Zillich, 1974; Arens, 1989). Haywood and Heymann (1989) introduced the bleaching technique in 1989 in which a patient wore a customized tray containing bleaching agent (usually 10% carbamide peroxide) for five to eight hours a day for between two and five weeks. High-concentrated carbamide peroxide gels have been introduced for in-office procedures. These bleaching agents are an alternative to high concentrations of hydrogen peroxide and they can be used alone or in combination with at-home night-guard vital bleaching (Gultz & others, 1999; Clark & Hintz, 1998).

In an attempt to reduce the whitening treatment period and application time needed to produce whitening effects similar to conventional 10% carbamide peroxide, some bleaching products present changes in composition or increased peroxide concentrations (Haywood & Robinson, 1997; Clark & Hintz, 1998; Lyons & Ng, 1998). The widespread use of bleaching agents expresses concern regarding the safety of peroxides applied to dental hard tissues (Li, 1996; Marshall, Cancro & Fischman, 1995; Swift & Perdigão, 1998), particularly the detrimental effects on the mechanical properties of enamel (Seghi & Denry, 1992; Cavalli, Carvalho & Giannini, 2002). Thus, the null hypothesis tested was that bleaching regimens do not affect enamel's ultimate tensile strength. Dental enamel was treated with three whitening products containing high concentrations of both carbamide and hydrogen peroxides for in-office application and three bleaching agents containing low peroxide concentrations for at-home bleaching. Bleaching regimens were compared to unbleached specimens. In addition, enamel fractures were analyzed with a scanning electron microscope (SEM).

METHODS AND MATERIALS

Fourteen sound, erupted, extracted human third molars stored in 0.1% thymol solution for up to two weeks after extraction were used in this study. The teeth were obtained after receiving informed consent from the patients (20 to 25 years old) under the protocol (107/2001), which was analyzed and approved by the Ethical Research Committee of Piracicaba School of Dentistry/UNICAMP, Brazil. The teeth were cleaned of gross debris and placed in deionized water for 24 hours before the beginning the experiment.

The intact occlusal enamel surface was etched with 35% phosphoric acid for 30 seconds, air dried and bonded with Single Bond adhesive system (3M ESPE, St Paul, MN, USA). A resin composite block (6.0 mm high—TPH Spectrum—Dentsply Caulk, Milford, DE, USA) (Figure 1a) was incrementally built-up on the enamel bonded surfaces and the restored teeth were stored in water at 37°C. After 24 hours, the roots were removed and the crowns were vertically and serially sectioned into 0.7-mm thick slabs with a diamond saw (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA) under water cooling (Figure 1b). Both sides of each slab were trimmed (Figure 1c) with a fine diamond bur (KG Sorensen Ind e Com Ltda, Barueri, Brazil) (Figure 1d) under water cooling to conform the specimen to an "hour-glass" shape and to reduce the enamel cross-sectional area located at the internal slope of the buccal cusps of the teeth to approximately 0.8 mm^2 (Figure 1e) (Carvalho & others, 2000). The orientation of enamel prisms could be checked and visualized due to specimen thickness, thus, the enamel sites exhibited a similar prism orientation.

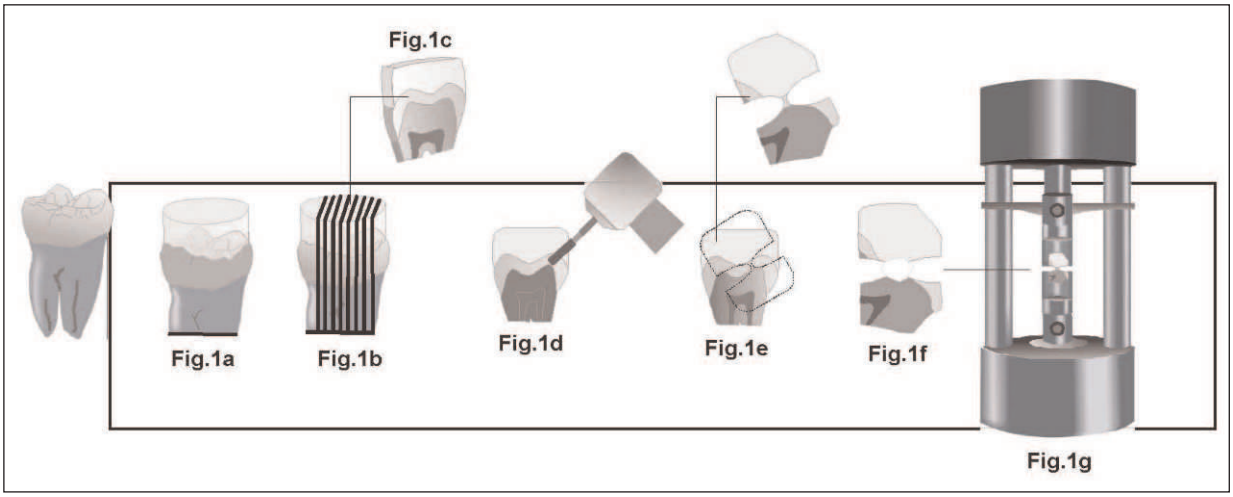

Figure 1. *Schematic representation of specimen preparation.*

Table 1: *Bleaching Agents Composition, Regimens and Batch Numbers*

Bleaching Agent/Manufacturer	Active Ingredient	Regimen	Batch #
Whiteness Perfect/Whiteness FGM Prod Odont, Joinville, SC, Brazil	10% Carbamide peroxide	1 daily application (6 hours) for 14 days	170501
Colgate Platinum Overnight ColgateOral Pharmaceutical, Canton, MA, USA	10% Carbamide peroxide	1 daily application (6 hours) for 5 days	105004
Day White 2Z Discus Dental Inc, Culver City, CA, USA	7.5% Hydrogen peroxide	1 daily application (30 minutes) for 14 days	OGJ-OGD
Whiteness Super/Whiteness FGM Prod Odont Joinville, SC, Brazil	37% Carbamide peroxide	2 applications (30 minutes each) with 5 days of interval between applications	070601
Opalescence Quick Opalescence, Ultradent Products Inc South Jordan, UT, USA	35% Carbamide peroxide	2 applications (30 minutes each) with 5 days of interval between applications	3PW1
Whiteness HP/Whiteness FGM Prod Odont Joinville, SC, Brazil	35% Hydrogen peroxide	2 applications (15 minutes each) with 7 days of interval between applications	240601

Table 2: *Ultimate Tensile Strength Mean Values (MPa) for Control and Experimental Groups (n=10)*

Group	Bleaching Agent	Mean	SD	Tukey Test
1	Control (unbleached)	51.3	8.6	a
2	Whiteness Perfect–CP 10%	31.1	8.4	bc
3	Colgate Platinum–CP 10%	32.1	13.1	bc
4	Day White 2Z–HP 7.5%	36.3	9.1	b
5	Opalescence Quick–CP 35%	34.6	9.1	b
6	Whiteness Super–CP 37%	34.4	7.4	b
7	Whiteness HP–HP 35%	22.0	5.6	c

Means followed by different letters are significantly different at p<0.05.
Abreviations: CP- Carbamide Peroxide and HP- Hydrogen Peroxide.

Seventy trimmed slabs were obtained from the 14 teeth (five each). The specimens were selected based on their integrity after trimming. Those presenting cracks or defects when examined under 40x magnification were discarded. The remaining specimens were then randomly assigned to seven groups (n=10) according to type and concentration of peroxide bleaching agents. Table 1 shows the composition, regimen and batch numbers of the bleaching agents used in this study.

Group 1 comprised the control group that received no bleaching treatment. Groups 2 through 7 were bleached with the following whitening products: Whiteness Perfect, Colgate Platinum, Day White 2Z, Whiteness Super, Opalescence Quick and Whiteness HP. In order to simulate the clinical routine and indications of each whitening product, manufacturers' application protocols were followed.

The control group was kept in artificial saliva (Cavalli & others, 2001) at 37°C for two weeks. For the G2, G3

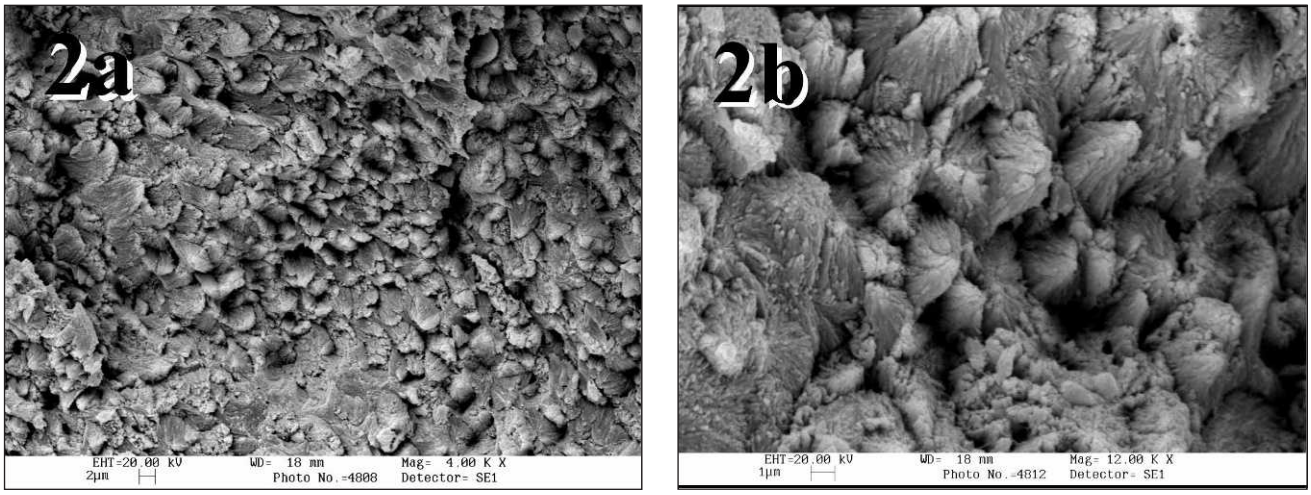

Figure 2. *Fractured surface of unbleached enamel. A typical, transversal fracture pattern when the load is applied parallel to the orientation of the prisms. Cone-like, protruding prisms are seen.*

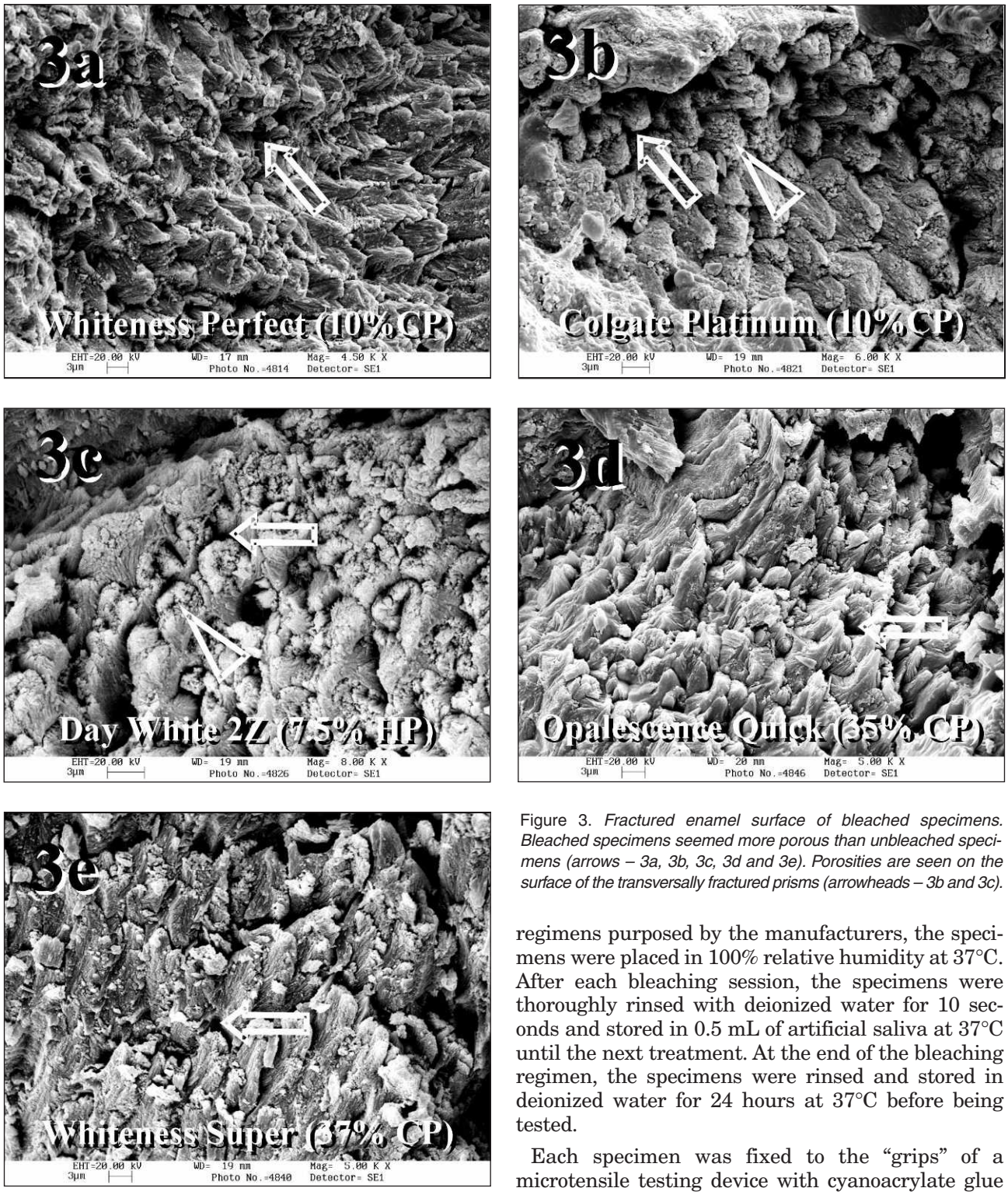

Figure 3. *Fractured enamel surface of bleached specimens. Bleached specimens seemed more porous than unbleached specimens (arrows – 3a, 3b, 3c, 3d and 3e). Porosities are seen on the surface of the transversally fractured prisms (arrowheads – 3b and 3c).*

and G4 groups, the trimmed enamel area was exposed daily to a mixture of 0.1 mL of the respective bleaching agent with 0.05 of artificial saliva according to the bleaching regimen recommended by each manufacturer (Figure 1f). For G5, G6 and G7, only 0.1 mL of bleaching agent was applied to enamel. During the bleaching regimens purposed by the manufacturers, the specimens were placed in 100% relative humidity at 37°C. After each bleaching session, the specimens were thoroughly rinsed with deionized water for 10 seconds and stored in 0.5 mL of artificial saliva at 37°C until the next treatment. At the end of the bleaching regimen, the specimens were rinsed and stored in deionized water for 24 hours at 37°C before being tested.

Each specimen was fixed to the "grips" of a microtensile testing device with cyanoacrylate glue (Zapit, DVA, Corona, CA, USA). The specimens were properly positioned to ensure that orientation of the enamel prisms was aligned parallel to the tensile force applied and tested in tension in a universal testing machine (4411 Instron Co, Canton, MA, USA) at 0.5 mm/minute until failure (Figure 1g). After testing, the fractured specimens were carefully removed from the

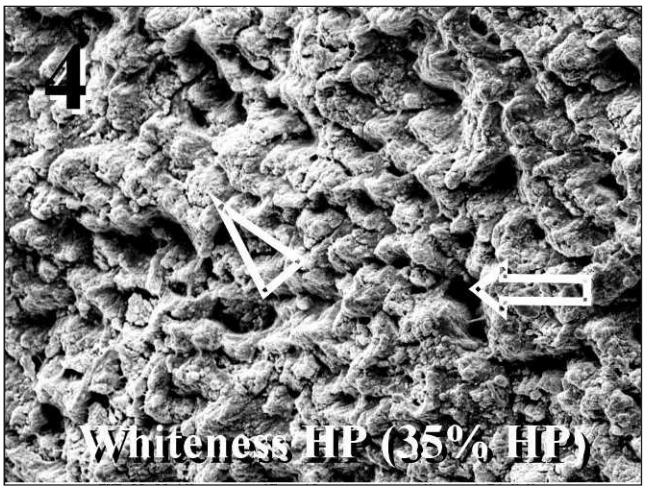

Figure 4. *Fractured enamel surface treated with Whiteness HP 35% hydrogen peroxide. Porous prism cores (arrowhead) and a possible loss of interprismatic material can be noted (arrow). General appearance is similar to acid-etched enamel.*

fixtures with a scalpel blade and the cross-sectional area at the site of fracture was measured to the nearest 0.01 mm using a digital caliper (727, Starrett Ind Com, Ltda, Itu, Brazil) to calculate the ultimate tensile strength expressed in MPa. The data were statistically analyzed by one-way analysis of variance (ANOVA) and Tukey test at the 0.05 confidence level.

The tested specimens were allowed to dry in an oven overnight and the fractured edges were sputter-coated (MED 010, Balzer, Leichtenstein) with gold and observed under a scanning electron microscope (VP 435, Leo, Cambridge CB13JS, England).

RESULTS

The mean ultimate tensile strength values for the experimental groups are shown in Table 2. ANOVA showed a statistically significant difference among groups (p=0.00022). All bleaching regimens produced a significant decrease in the ultimate tensile strength (UTS) of enamel as compared to the control, untreated substrate (p<0.05). Significant differences were also found among the bleaching regimens (p<0.05).

The UTS of untreated enamel was 51.3 ± 8.6 MPa, while the UTS of bleached enamel ranged from 22.0 ± 5.6 MPa to 36.3 ± 9.1 MPa. Bleaching procedures resulted in approximately 30% to 57% reduction of the UTS of enamel. The greatest reduction in the UTS was observed after application of Whiteness HP that, while not significantly different from Whiteness Perfect and Colgate Platinum (p>0.05), resulted in UTS values that were significantly lower than the other materials (p<0.05).

SEM observations depicted a typical fractured surface of enamel specimens that were stressed in a direction parallel to prismatic orientation (Figure 2a) (Carvalho & others, 2000). The prisms were dislodged in a cone-like shape and no porosity was observed at the fractured ends of unbleached specimens (Figure 2b). The definition of the prismatic structure appears greater in bleached teeth, which might indicate a loss of interprismatic matrix (Figures 3a, 3b, 3c, 3d and 3e). Also, the presence of porosities at the prism-fractured ends in the bleached fractured enamel might indicate that some intraprismatic material was also lost (Figures 3a, 3b, 3c, 3d and 3e). The above features were noticeably more evident in the high-concentration hydrogen peroxide group (Whiteness HP 35%), rendering the surface with an appearance similar to phosphoric acid etched enamel (Figure 4).

DISCUSSION

The available information on the safety and biological properties of peroxides for tooth whitening indicates minimal risks with appropriate use of the bleaching products (Li, 1996; Marshall & others, 1995; Kelleher & Roe, 1999). However, another concern regarding peroxide bleaching is related to the loss of dental structure strength during and after whitening treatment. Some authors have reported a reduction in enamel surface microhardness (Attin & others, 1997; Cimilli & Pameijer, 2001; Akal & others, 2001) and morphological changes after whitening treatment with different peroxide bleaching agents (Akal & others, 2001; Josey & others, 1996; Perdigão & others, 1998; Hegedüs & others, 1999). Studies have shown that such surface alterations could occur due to mineral (Cimilli & Pameijer, 2001; Rotstein & others, 1996) and organic substance removal (Arends & others, 1984; Nainar & Clarkson, 1994), which are promoted by the oxidizing process on the surface and urea formation after carbamide peroxide degradation. Significant alterations to the enamel surface, such as increased porosity or roughness, pitting and erosion were described after early vital tooth bleaching techniques that comprised the application of high concentrations of hydrogen peroxide (Titley, Torneck & Smith, 1988; McGuckin, Babin & Meyer, 1992).

Seghi and Denry (1992) evaluated the effects of 10% carbamide peroxide gel on fracture toughness, hardness and abrasion characteristics of enamel after one session of bleaching for 12 hours. The results showed that bleached enamel abrasion resistance decreased and enamel toughness was reduced in approximately 30% of cases. However, no significant changes in surface hardness were observed, indicating some alteration of the enamel organic matrix produced by urea. In this study, the specimens were treated in a similar way to the clinical application protocol (Table 1) and presented a reduction in the UTS, ranging from 30% to 57%, depending on the peroxide composition and concentration. Statistical analysis revealed that all bleaching

agents reduced enamel strength when compared to the control group.

Two of the bleaching agents contained 10% carbamide peroxide and were indicated for six-hour daily applications. Even though the relative concentration of hydrogen peroxide in these two products was low (that is, 3% after carbamide degradation), the six-hour daily application for five or 14 days was probably long enough to cause the observed reduction in the UTS of enamel. A more concentrated bleaching agent containing 7.5% hydrogen peroxide was applied for only 30 minutes daily during 14 days and also reduced enamel strength. Apparently, the effects of bleaching agents depend on the association of hydrogen peroxide concentration and application time, however, all bleaching regimens tested in this study significantly reduced enamel tensile strength.

Approximately 10% hydrogen peroxide is released after high-concentrated carbamide peroxide degradation (Gultz & others, 1999). Despite the higher amount of hydrogen peroxide released by high-concentration carbamide peroxide gels, no significant differences were found when compared to low-concentration carbamide and hydrogen peroxide bleaching agents. The daily application of low-concentration carbamide peroxide solutions produced effects similar to those observed when high-concentration carbamide peroxide was applied. The high-concentration solutions were applied for 30 minutes in four separate sessions. Similar results were also observed between 10% carbamide peroxide and 35% hydrogen peroxide whiteners applied four times every 15 minutes in a seven-day period.

Changes in enamel surface and decreased microhardness are related to superficial mineral and organic loss (Perdigão & others, 1998). However, the effects of bleaching are not solely restricted to superficial enamel. Hydrogen peroxide can penetrate through enamel and dentin due to its low molecular weight (Kelleher & Roe, 1999; Arwill, Myrberg & Söremark, 1969) and alter the inner structure of such substrates. This might explain the reduced mechanical property observed in this study, since four ground enamel surfaces were exposed to the bleaching agents. Therefore, it can be speculated that the alterations observed on bleached enamel surfaces (Cavalli & others, 2002; Akal & others, 2001; Josey & others, 1996; Perdigão & others, 1998; Hegedüs & others, 1999) might have occurred internally at the fractured sites. The oxidizing effect of peroxide bleaching agents could weaken the enamel surface and subsurface. This offers another possible explanation as to why the bond strengths of resins to bleached enamel are usually lower (Cavalli & others, 2001; Lai & others, 2002). Bleaching agents penetrate enamel deeper than adhesive agents. Although adhesive resin can reinforce the etched surface, deeper weakened areas remain unfilled by the resin and can serve as the site of fracture upon stress. Only careful examination of the fractured site in such bond strength studies can reveal the true cause of reduced bond strengths to bleached enamel.

Fractured surfaces of the unbleached control group exhibited no signs of morphological alterations, showing a compact structure and typical fracture pattern after tensile strength test (Figures 2a and 2b) (Carvalho & others, 2000). Fractured areas of the bleached groups presented increased porosity at prism ends, depending on the bleaching agent (Figure 3). It has been speculated that urea removes not only organic components, but also some mineral content associated with proteins (Nainar & Clarkson, 1994). Urea penetrates up to 50-60 µm into enamel, which may increase the adverse effects of carbamide peroxide (Arends & others, 1984). The current study did not find evidence indicating that the presence of urea would increase the detrimental effects of bleaching agents. Two hydrogen peroxide urea-free bleaching agents also resulted in similar morphological changes in enamel (Figures 3c and 4). Additionally, the resultant UTS values of urea-containing agents were not different from those that do not release urea (Table 2). The highly concentrated hydrogen peroxide agent (Whiteness HP, 35% HP) produced morphological alterations in enamel that resembled acid-etched enamel when viewed under the SEM (Figure 4). This material also caused the greatest reduction in the UTS of enamel (Table 2).

In order to approach the clinical situation, the bleaching regimens used in this *in vitro* study followed the recommendations of the respective manufacturers and the specimens were maintained in artificial saliva between each bleaching application period. It has been shown that saliva is a potent remineralizing solution and its action on enamel could recover the structural damage caused by bleaching agents or demineralizing solutions (Attin & others, 1997; Cavalli & others, 2001). However, the volume of artificial saliva used in this study was probably not able to remineralize the altered enamel and prevent or recover the structural damage that resulted in reduced UTS values. The effects of using natural saliva in simulated laboratory conditions or *in vivo* remain to be determined.

This study has shown that all bleaching agents and the respective treatment regimens used caused a significant reduction in enamel UTS. Such a reduction was accompanied by noticeable changes in the internal morphology of enamel which was characterized by increased porosity of the enamel prisms. The widespread use of bleaching agents to whiten teeth for aesthetic reasons increases concern about the safety of peroxide containing tooth whiteners on dental hard tissues. Although no clinical reports about fractures or cracks in tooth structures have been presented in dental literature, this *in vitro* study showed that a possible

reduction in enamel strength might occur after bleaching procedures. Thus, the null hypothesis of this study was rejected.

CONCLUSIONS

The results of this *in vitro* study suggest that peroxide bleaching regimens can significantly reduce enamel's intrinsic strength. Reduction in the ultimate tensile strength was accompanied by changes in the enamel internal micromorphology.

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Liner and Light Exposure: Effect on *In-Vitro* Class V Microleakage

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Clinical Relevance

The results of this study suggest that ramp and pulse-delay light curing methods did not improve marginal sealing compared to the conventional technique. The reduced microleakage of glass ionomer/resin restorations make it a positive restorative option, while high intensity light curing increased microleakage in cavities with dentin margins.

SUMMARY

This *in vitro* study evaluated the influence of different glass ionomer liners and curing methods on microleakage of resin composite restorations. Class V root preparations were made in 120 bovine incisors randomly divided into 12 groups according to liner and curing method. The resin composite system (Single Bond + Z100) was inserted and polymerized in one increment in all groups. Cavity preparations were either not lined (control), lined with a resin modified glass-ionomer cement (Vitrebond) or a conventional glass-ionomer cement (Ketac Bond). The restorations were light-cured using one of four curing methods. The teeth were thermocycled and immersed in 0.5% basic fuchsin, sectioned, and dye penetration was measured (Image Tool). No significant difference in leakage among conventional, ramp or pulse-delay methods was seen. High intensity light groups showed significantly greater penetration compared to other curing methods. No significant difference existed in marginal leakage between liners, but microleakage was significantly higher in groups restored using no liner. No relationship between lining technique and light curing method was observed. The use of glass ionomer liners reduced microleakage, while high intensity light curing produced the greatest dye penetration.

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INTRODUCTION

Polymerization shrinkage of resin composites can create contraction forces that may disrupt the bond to cavity walls, leading to gap formation and, consequently, microleakage (Carvalho & others, 1996; Ferracane & Mitchem, 2003; Hilton, 2002; Koran & Kürschner, 1998; Obici & others, 2002). A number of ways to improve the marginal integrity of resin-based

restorations have been suggested, including applying lining materials with low elastic modulus that absorb polymerization stresses (Obici & others, 2002; Tolidis, Nobecourt & Randall, 1998). Glass ionomer cements have been recommended as a low modulus liner for resin composite restorations, especially when the gingival margin extends beyond the cemento-enamel junction (Schwartz, Anderson & Pelleu, 1990). The ability of glass ionomer cements to adhere to tooth structure, their ability to release fluoride and their reported improved microleakage performance over that of resin composites, are among the qualities that suggest their use as liners for improving the marginal seal (Holtan & others, 1990; Mount, 1999).

Another approach to improving marginal integrity has been attempted through modifications of the curing method by controlling the light intensity of the lamp. These so-called "soft-start" or "two-step" polymerization techniques rely on the application of low light intensity during the initial stages of curing, followed by a final cure at high light intensity (Feilzer & others, 1995; Kanca & Suh, 1999; Rueggeberg, 1999; Uno & Asmussen, 1991; Unterbrink & Muessner, 1995). In the "ramp technique," resin composite is irradiated by a constant rate, increasing light intensity (for example, 100 to 800 mW/cm^2) during the first 15 to 20 seconds, followed by irradiation at the highest intensity for the remaining curing time (Rueggeberg, 1999; Hofmann & others, 2003). An alternative to this method is to irradiate the resin for the first 15 to 20 seconds at a light intensity that is about 30% to 40% of the maximum, followed by a "jump" to the maximum intensity that is then irradiated for the remaining curing time (Rueggeberg, 1999).

In the pulse-delay curing technique, a short (three seconds), low light intensity is applied to the resin, followed by a three-to-five minute non-irradiated period that is completed with high intensity irradiation for the remaining curing time (Rueggeberg, 1999; Davidson & de Gee, 2000; Kanca & Suh, 1999). The overall advantages of these "soft-start" polymerization techniques are that, contrary to conventional technique that initiates curing with an already high light intensity, initiating the curing with low light intensity would reduce the curing rate at the early stages, thus reducing the rate of contraction and ultimately minimizing stresses at the bonded interface (Koran & Kürschner, 1998; Goracci, Mori & Casa de Martinis, 1996; Kanca & Suh, 1999; Obici & others, 2002). This concept would result in smaller marginal gaps and increased marginal adaptation (Goracci & others, 1996; Uno & Asmussen, 1991). Conversely, there are fast curing light units on the market that are used to reduce the time taken in the polymerization of resin composite restorations, thus, speeding up the polymerization process (Peutzfeldt, Sahafi & Asmussen, 2000). These systems rely on the concept that very high light intensities (1200 – 1600 mW/cm^2) irradiated for a short period (three seconds) would be sufficient to completely cure the resin layer, thus reducing the need for longer exposures (Davidson & de Gee, 2000; Peutzfeldt & others, 2000; Brackett, Haisch & Covey, 2000; Rueggeberg, 1999; Hofmann & others, 2003). However, high light intensity at the early curing stage will result in a fast curing rate that tends to produce immediate shrinkage of the resin and increase the contraction stresses transferred to bonded interfaces (Feilzer & others, 1995; Brackett & others, 2000). The use of high light intensity, such as that produced by the plasma system, might create more contraction forces and subsequently marginal leakage (Brackett & others, 2000). Some authors, however, found that marginal adaptation was not compromised by this curing method (Hasegawa & others, 2001).

Alternative curing methods with different intensities and time exposure are available to photopolymerize resin composites. The question of whether different light intensities can improve the marginal sealing of glass ionomer lined resin composites, thereby reducing microleakage, has not been established. The aim of this study was to test the influence of liners (conventional and resin-modified glass ionomer cements) and different curing methods (conventional, ramp, pulse and high intensity) on the marginal sealing of resin composite restorations in box-shaped Class V cavities.

METHODS AND MATERIALS

One hundred and twenty bovine incisors were initially debrided and stored in a 0.1% thymol solution at room temperature. Box-shaped Class V cavity preparations were cut on the facial aspect of root dentin with a #245 bur at high speed using air/water coolant. The burs were replaced after every five preparations. The cavities were made 2 mm below the cemento-enamel junction and the dimensions were 1.5-mm deep, 4 mm in height and 3 mm in width. The teeth were randomly divided into 12 groups of 10 and restored according to liner and curing method (Table 1):

Unlined Restorations (Groups 1, 4, 7 and 10)

Dentin was etched with 35% phosphoric acid gel (3M ESPE, St Paul, MN, USA) for 15 seconds, thoroughly rinsed for 30 seconds and dried with absorbent paper to remove excess water, leaving a moist surface. Two coats of adhesive system (Single Bond, 3M ESPE, St Paul, MN, USA) were consecutively applied and light-cured (Optilux 500, Demetron/Kerr, Danbury, CT, USA) following manufacturer's instructions.

Vitrebond Liner (Groups 2, 5, 8 and 11)

A single 0.5-mm thick layer of Vitrebond (3M ESPE) was applied to the untreated axial wall using a small ball burnisher. The liner was immediately cured for 30

seconds with a visible light-curing unit (Optilux 500, Demetron/Kerr). For the unlined groups, the lateral walls were etched with phosphoric acid and bonded with Single Bond adhesive as described above.

Ketac Bond Liner (Groups 3, 6, 9 and 12)

The axial wall was conditioned with a 40% aqueous solution of polyacrylic acid (Durelon liquid, 3M ESPE) for 10 seconds and rinsed with an air-water spray for 20 seconds. Conventional glass ionomer (Ketac-Bond 3M ESPE) was prepared according to manufacturer's instructions and applied in a single 0.5-mm thick layer using a small ball burnisher to cover the axial dentin. The glass ionomer was allowed to set for five minutes before etching and bonding the cavity as described in the Vitrebond groups.

Bonded lined and unlined cavities were filled with a single increment of Z 100 resin composite (3M ESPE). The resin composite was light-cured using four curing techniques: Groups 1, 2 and 3 [conventional technique–450 mW/cm^2 for 40 seconds]; Groups 4, 5 and 6 [ramp technique - 100–800 mW/cm^2 for 15 seconds + 800 mW/cm^2 for 25 seconds]; Groups 7, 8 and 9 [pulse-delay technique–200 mW/cm^2 for 3 seconds + 3 minutes delay + 600 mW/cm^2 for 30 seconds]; and Groups 10, 11 and 12 [high light intensity 1600mW/cm^2 for 3 seconds]. The light tip was positioned as close as possible to the surface of the restoration during photo-polymerization. The light curing units and respective curing techniques are listed in Table 1. The restorations were finished with a #15 surgical blade and polished with graded discs (Sof-Lex 3M ESPE). The specimens were stored at 37°C in physiologic saline solution for one week, then were thermocycled in distilled water (500 cycles, 5°C-55°C; dwell time 60 seconds). The apices were sealed with resin-modified glass ionomer and cyanoacrylate. The teeth were coated with two coats of nail varnish, except for a 1-mm wide zone around the margins of the restorations. All the teeth were immersed in an aqueous solution of 0.5% basic fuchsin for four hours, rinsed for six hours in running water, dried and imbedded in epoxy resin. Each tooth was longitudinally sectioned into three sections, approximately 0.7-mm thick across the restorations using a low-speed water-cooled diamond saw. The section showing the maximum dye penetration of each tooth was selected for determination of the extension of marginal leakage. The extent of dye penetration was measured in millimeters along the interface from the cavosurface to the deepest point up to the axial wall by means of computer software (Image Tool–UTH-SCSA–University of Texas Health Science Center, San Antonio, TX, USA). Data were analyzed by two-way ANOVA and Tukey tests.

RESULTS

Two-way ANOVA revealed significant differences in the effects of liner (F=11.7, p=0.000025) and curing method (F=8.0, p=0.000072); however, no significant differences were found for the interaction between them (F=1.0, p=0.38). Unlined restorations presented significantly higher leakage values than lined restorations, regardless of the glass ionomer liner used (p<0.001). No differences were found between the two glass ionomers (p=0.55). For curing methods, no differences were found between pulse vs ramp (p=0.7), pulse vs conventional (p=0.8) and conventional vs ramp (p=0.9). The use of high light intensity resulted in significantly higher leakage values than all the other curing methods (p<0.01 for all comparisons). No combination of liner

Table 1: *Restorative Procedures, Curing Technique and Curing Light Used for Different Experimental Groups*

Groups	Liner	Curing Technique	Curing Unit
1	No	Conventional	Optilight Digital[a]
2	Vitrebond	Conventional	Optilight Digital
3	Ketac Bond	Conventional	Optilight Digital
4	No	Ramp	Elipar Trilight[b]
5	Vitrebond	Ramp	Elipar Trilight
6	Ketac Bond	Ramp	Elipar Trilight
7	No	Pulse-delay	VIP[c]
8	Vitrebond	Pulse-delay	VIP
9	Ketac Bond	Pulse-delay	VIP
10	No	High intensity	Apollo 95E[d]
11	Vitrebond	High intensity	Apollo 95E
12	Ketac Bond	High intensity	Apollo 95E

[a]*Gnatus, Ribeirao Preto, Brazil*
[b]*3M ESPE, St Paul, MN, USA*
[c]*BISCO, Schaumburg, IL, USA*
[d]*Dental/Medical Diagnostic Systems, Inc (DMD), Westlake Village, CA, USA*

Table 2: *Leakage Values (mm) According to the Lining and Curing Methods. Values Are: Mean ± SD*

	Conventional	Ramped	Pulse-delay	High Intensity	Row Total
No Liner	0.83 ± 0.65	0.62 ± 0.49	0.93 ± 0.39	1.13 ± 0.47	0.88[b]
Vitrebond	0.32 ± 0.26	0.28 ± 0.19	0.36 ± 0.22	0.92 ± 0.42	0.47[a]
Ketac Bond	0.40 ± 0.32	0.58 ± 0.39	0.49 ± 0.37	0.76 ± 0.32	0.56[a]
Column Total	0.52[a]	0.50[a]	0.59[a]	0.94[b]	

Same superscript letters indicate no significant difference among values for the main effects. No significant difference was found for effect interactions.

and curing method was capable of completely preventing leakage. The overall results are summarized in Table 2.

DISCUSSION

It has been shown, experimentally, that slow polymerization of resin composites at low light intensity, followed by final cure at high light intensity, results in better adaptation at the dentin/restoration interface than constant high intensity light irradiance (Feilzer & others, 1995; Uno & Asmussen, 1991; Unterbrink & Muessner, 1995). Conversely, the results of this study showed that the pulse and ramp activated groups did not exhibit less marginal leakage compared to conventional polymerization in Class V dentin cavities, which is corroborated by other studies (Amaral & others, 2002; Cavalcante & others, 2003; Friedl & others, 2000; Muangmingsuk, Senawongse & Yudhasaraprasithi, 2003; Sahafi, Peutzfeldt & Asmussen, 2001). Perhaps the lower initial light intensities used in the ramp and pulse-delay groups were not enough to activate initiator molecules to trigger polymerization reaction. The final curing step of such techniques at higher light intensities may have actually been responsible for the overall curing and corresponded to an immediate full intensity curing (Friedl & others, 2000; Mehl, Hickel & Kunzelmann, 1997), thereby, masking the effect of the initial low energy curing step. Another possibility is that the very high photo-initiator concentration in Z100 resin composite might have caused immediate polymerization upon activation by the light sources, even though at low light intensity. In this case, the gel-point would be reached within the first seconds of curing with very low doses of light energy (Ernst & others, 2000; Muangmingsuk & others, 2003). This would explain why ramp and pulse-delay light-curing methods had no influence on marginal sealing when compared to the conventional technique. Different results could be obtained with different composites that contain different initiator/accelerators.

High light intensity groups showed significantly greater dye penetration than the other curing techniques. Some studies have shown that the use of plasma arc light units seems to offer an increased risk of leakage along the dentin margins of restorations, because the resin composite polymerizes so rapidly that the flow is restricted as its stiffness rapidly increases (Brackett & others, 2000; Peutzfeldt & others, 2000). Fast conversion of monomers to polymers is accompanied by rapid shrinkage, which tends to produce excessive polymerization stresses on adhesive bonds, resulting in debonding and leading to poor marginal adaptation along dentinal margins (Brackett & others, 2000; Mehl & others, 1997; Uno & Asmussen, 1991). The short burst of high light energy produced by plasma arc light does not necessarily induce greater shrinkage stress in resin-based composites compared to halogen light sources; however, shrinkage develops more rapidly (Davidson & de Gee, 2000). There is a linear relationship between polymerization contraction and light intensity (Sakaguchi, Douglas & Peters, 1992). It has been reported that three seconds of curing time was insufficient for the optimal curing of composites when a plasma arc curing technique was applied (Park, Krejci & Lutz, 2002). If such incompletely polymerized restorations are subjected to thermal stress, it is likely that the weak adhesion to the margins could fail, resulting in increased microleakage (Brackett & others, 2000).

The placement of glass-ionomer liners produced significantly less leakage at the dentin/restoration interface when compared to groups restored using no liner. Several other studies have reported better marginal adaptation and significant reductions in microleakage in resin composite restorations lined with glass-ionomer cements (Douglas & Fundingsland, 1992; Holtan & others, 1990; Mathis & others, 1990; Schwartz & others, 1990; Tjan & Dunn, 1990; Trushkowsky & Gwinnett, 1996). Contrary to resin composites, glass-ionomer cements possess a coefficient of thermal expansion similar to dentin. This may have helped them to maintain wall adaptation during thermocycling and result in less leakage compared to unlined restorations (Mathis & others, 1990; Schwartz & others, 1990). Moreover, the effect of thermocycling and polymerization contraction of the resin composite may have been reduced in lined restorations because of the smaller volume of resin composite in the preparation (Mathis & others, 1990). The application of an intermediate layer of either a low-viscosity resin or glass ionomer cement between dentin and the restorative resin has been shown to relieve polymerization contraction stress by 20% to 50% (Kempt-Scholte & Davidson, 1990). A 41% reduction in the volumetric contraction of resin composite restorations lined with a resin modified glass ionomer cement (Vitrebond) has been reported (Tolidis & others, 1998).

In this study, the experimental protocol combined the use of a fast-rate developing, high modulus resin composite (Kanca & Suh, 1999; Ferracane & Mitchem, 2003) that was inserted in bulk increment in a high C-factor cavity configuration. This represents a challenge to the integrity of the tooth-restorative interface. It has been shown that polymerization contraction stresses at the composite-adhesive interface can be relieved by placement of a low-modulus shock-absorber lining material between the relatively rigid dentin and resin composite (Kemp-Scholte & Davidson, 1990; Davidson & Feilzer, 1997). Because glass ionomer cements have relatively low modulus of elasticity (3-6 GPa), placing a layer of these materials between the resin composite and dentin may absorb significant polymerization stresses (Choi, Condon & Ferracane, 2000).

Conventional GIC liner groups showed slightly more leakage than resin-modified GICs, but the difference was not significant. A possible explanation for the improved seal with light-cured glass ionomer liner might be related to its ability to adhere immediately to dentin, while conventional GICs develop adhesion over time (Aboushala, Kugel & Hurley, 1996; Martin & O'Rourke, 1993; Sjödin, Uusitalo & Van Dijken, 1996). This stronger, immediate adhesion may be more capable of resisting contraction forces generated by the curing of resin composite, thus minimizing the formation of contraction gaps and reducing the possibility of microleakage (Aboushala & others, 1996; Tjan & Dunn, 1990; Martin & O'Rourke, 1993).

While there was no significant interaction between the liners and curing methods (Table 2), it is interesting to note that relatively higher leakage values were found when high light intensity was used in combination with Vitrebond compared to using Ketac Bond. Vitrebond cement is said to have a dual-cure setting reaction (Bourke, Walls & McCabe, 1992). The primary setting reaction is initiated by exposure to visible light and subsequent curing is due to an acid-base reaction which commences during mixing and continues after light activation. The hardness of this material increases rapidly for up to one hour after light activation, then continues to increase gradually for up to 24 hours, after which there is no significant increase (Bourke & others, 1992). Vitrebond was lined in the cavity and cured with a halogen lamp for 30 seconds. Subsequently, the thin layer (approximately 1 mm) of Z100 was inserted to fill the cavity and was light-cured with the high light intensity curing unit (1,600 mW/cm^2). It is possible that such high intensity may have induced an additional and accelerated cure of Vitrebond underneath the thin layer of resin composite. In this case, if adhesion of the resin composite to glass ionomer cement or resin-modified glass ionomer cement was greater than the strength of the adhesion of these liners to dentin, then the immediate polymerization shrinkage stresses that develop during high-intensity light-curing of the composite might have debonded the liners from dentin, thereby increasing microleakage. Alternatively, glass ionomer cements might have failed cohesively, creating a gap that filled with dye, even thought the dentin remained sealed by the glass ionomer cement. The measurement of microleakage by means of software, as in the current study, could not differentiate between those two possibilities.

CONCLUSIONS

The results of this study indicated that ramp and pulse-delay light curing methods were not effective in improving the marginal quality of Class V resin composite restorations when compared to conventional light-curing methods. A high-intensity light curing method can compromise adaptation to the cavity wall and lead to increased leakage values, regardless of the use of glass ionomer liners. The combination of glass ionomer/resin composite restoration was effective in reducing microleakage at dentin margins and may prove to be a useful restorative option.

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Coronal Microleakage of Temporary Restorations in Previously Restored Teeth with Amalgam and Composite

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Clinical Relevance

During root canal treatment, maintaining partially removed amalgam or composite permanent restorations does not seem to cause a problem with achieving a marginal seal.

SUMMARY

Aim: This study evaluated microleakage at the interface between various temporary restorative materials and existing amalgam or composite restorations, and dental tissues in previously restored teeth after partial removal of the restoration.

Materials and Methods: The distal half of amalgam (Ag) and composite restorations (Co) in 45 teeth were removed, then filled with temporary restorative materials (IRM, Coltosol and CLIP). After thermal cycling, microleakage was measured microscopically as the penetration of basic fuchsine according to a four-unit-scale: The data were evaluated with Friedman and Kruskal-Wallis tests using Bonferroni correction ($p<0.05$).

Results: In almost all groups except the Co-IRM and Ag-CLIP interface, lower microleakage values were observed in temporary restoration-permanent restoration interfaces compared to temporary restoration-tooth interfaces. For the Ag and Co groups except for the Ag-IRM-b interfaces, the highest microleakage values were observed with IRM for b and c interfaces followed by Coltosol and CLIP. Interestingly, although CLIP was a temporary restoration, CLIP-tooth interface (Ag-CLIP-c) values were lower than amalgam-tooth interface (Ag-CLIP-a) values.

Conclusions: CLIP provided a better seal against microleakage at amalgam and especially composite interfaces. This material also provided a better seal against microleakage at the tooth tissue interface. The use of a resin based temporary restorative material over partially removed resin composite restorations could be beneficial in achieving better resistance to marginal leakage.

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Within the limitations of this study, maintaining partially removed permanent restorations does not seem to cause a problem with achieving marginal seal.

INTRODUCTION

An important consideration in endodontic therapy is the seal provided by a temporary restoration placed in the access preparation of a tooth undergoing treatment (Turner & others, 1990; Heling & others, 2002; Naoum & Chandler, 2002). The primary purpose of sealing access cavities is to prevent contamination of the root canal system (Webber & others, 1978; Cruz & others, 2002; Bobotis & others, 1989).

Many temporary restorative materials have been advocated for use in provisional restorations, but several studies evaluating the microleakage of temporary restorative materials have often shown contradictory results (Uranga & others, 1999; Lee & others, 1993; Barthel & others, 1999; Anderson, Powell & Pashley, 1990). Commonly used provisional restorative materials include zinc-oxide-eugenol reinforced with polymethyl methacrylate (IRM, Dentsply Caulk, Milford, DE, USA), zinc oxide and calcium sulfate (Coltosol-Cavit,Coltene AG, Altstätten, Switzerland) and light-polymerized composite based on urethane dimethacrylate polymer (CLIP, Vuco, Cuxhaven, Germany). A variety of other materials are also used (Chohayep & Bassiouny, 1985; Friedman & others, 1986; Blaney & others, 1981; Pai & others, 1999).

Some *in vitro* and *in vivo* studies evaluated the sealing ability of endodontic temporary restorative materials in intact teeth (Turner & others, 1990; Naoum & Chandler, 2002; Webber & others, 1978; Bobotis & others, 1989; Uranga & others, 1999). However, many teeth requiring endodontic therapy have large, permanent coronal restorations of acceptable quality and sometimes it is not necessary to remove the complete restoration to gain access to the pulp chamber, either originally or in the course of endodontic treatment. In cases where removal of the restoration is indicated, temporary restorative materials come in contact with previously restored enamel and dentin. Few studies (Turner & others, 1990; Pai & others, 1999) have tested the sealing ability of temporary restorative materials in such situations.

Recently, resin containing temporary restorative materials have been developed. There is no report on the bonding or microleakage properties of resin containing temporary restorative materials with previously treated dental tissues restored with composite. However, when restoring anterior teeth with temporary resin based material, adequate esthetics cannot be achieved. For that reason, when the primary restoration is of an acceptable quality, the access cavity can be prepared on the primary restoration. There are several studies which report that maintaining the partially removed restoration and using temporary restorative materials for filling the endodontic access cavity will not affect microleakage (Turner & others, 1990; Pai & others, 1999; Orahood & others, 1986). However, there are no studies investigating microleakage between the resin composite restorations and the resin composite based temporary restorative materials used for a certain period of time after insertion of the resin composite restoration.

In cases where endodontic therapy is completed in several appointments with longer inter-appointment time intervals and the patient carries temporary restorations, 1) if removal of the permanent restorations implies an important esthetic problem or 2) if removal of the permanent restorations at the posterior region and the making of temporary restorations with irregular form results in a lack of sufficient resistance to chewing, resulting in difficulties, it can be beneficial to maintain the partially removed permanent restorations in order to achieve the marginal seal.

This study evaluated microleakage at the interface between the various temporary restorative materials and existing amalgam or composite restorations and dental tissues.

METHODS AND MATERIALS

Ninety-four freshly extracted, non-carious and non-restored human permanent molars were used in this study. The teeth were stored in deionized water containing thymol until ready for experimentation. The occlusal surfaces of the 90 molars were reduced and their cusps flattened with cylindrical diamond burs without affecting the enamel tissues near the cavity borders. One operator performed standard box-shaped endodontic access cavity preparations 4.0 x 4.0-mm wide and 5.0 mm deep on 92 teeth using a high-speed carbide #4 round bur (SS White Burs, Inc, Lakewood, NJ, USA) under copious air-water spray, avoiding exposure of the pulp chambers. The teeth were then randomly divided into two groups (Ag and Co) of 45 teeth each.

The cavities of the Group Ag were filled with a spherical 40% silver amalgam (Amalga 48 High Copper, Non-Gamma2, Amalgam Alloy Ltd, S Africa) as the primary restorative material and polished 24 hours after placement. No cavity varnish or amalgam adhesive agent was used under the amalgam restorations. The cavities of Group Co were filled with composite (Filtek 250, 3M Dental Products, St Paul, MN, USA), with its bonding agent (Single Bond 3M Dental Products), and finished with a 12-fluted finishing bur 10 minutes after polymerization, followed by polishing with abrasive disks (Soft-Lex, 3M Dental Products).

All specimens were placed in normal saline at 37°C for 14 days. Teeth from Groups Ag and Co were randomly divided into three temporary restorative material subgroups, each containing 15 teeth: Ag-IRM (Amalgam-Intermediate Restorative Material, Dentsply Caulk, Milford, DE, USA); Ag-Colt (Amalgam-Coltosol, Coltene, Switzerland); Ag-CLIP (Amalgam-CLIP Light-curing temporary one-component filling material, VOCO, Cuxhaven, Germany); and Co-IRM (Composite-Intermediate Restorative Material, Dentsply Caulk), Co-Colt (Composite-Coltosol); Co-CLIP (Composite-CLIP Light-curing temporary one-component filling material VOCO).

The distal halves of the primary restorations were removed with carbide fissure burs, then filled with temporary restorative materials. Each material was placed according to the manufacturer's instructions. The only exception was IRM, which was mixed with a powder-to-liquid (P:L) ratio of 2g/ml. In previous studies (Lee & others, 1993; Anderson & others, 1990), it has been reported that IRM had less leakage when mixed at this P:L ratio than when it was mixed according to the manufacturer's recommendations (Powder/liquid ratio 6g/ml).

The specimens were then placed in normal saline at 37°C for two days. Two positive control teeth had access preparations without primary or secondary restorations. Two negative control teeth had intact crowns with no access opening and no restorations (Pai & others, 1999).

All specimens were subjected to 1,000 cycles of thermal stress between at 5°C and 55°C with 30 seconds dwell time. The apices of all samples were occluded with a resin composite (Concise White Sealant Systems, 3M Dental Products). The samples were then air dried and covered with two layers of nail polish except for the access areas. The specimens were then immersed in 0.5% basic fuchsine dye at 37°C and 100% humidity for seven days, washed under tap water and dried. All teeth were then sectioned just apical to the cemento-enamel junction with a high-speed diamond disc and segments were embedded in epoxy resin (Buehler Ltd, Lake Bluff, IL, USA). After polymerization, the blocks were sectioned diametrically with a low speed diamond saw (Isomet Buehler Ltd, Chicago, IL USA). The crowns were longitudinally sectioned into two parts in a mesiodistal orientation following the experimental method of Pai and others (1999).

The sectioned surfaces were examined at 25x with a stereomicroscope (Wild TVP, Heerbrugg, Switzerland). Microleakage at the enamel/dentin and restoration interfaces was scored using an ordinal scale where 0 = no evidence of dye penetration; 1 = dye penetration of less than half the cavity depth; 2 = dye penetration to the full cavity depth and 3 = dye penetration to the axial wall and beyond. Three measurements of dye penetration for each tooth were made. The first measurement "a" was the length of dye penetration along the interface between the primary restorative material and the access cavity wall. The second measurement "b" was between the primary and secondary restorative materials. The third measurement "c" was between the secondary restorative material and the access cavity wall.

Statistical evaluations of the test groups' data were performed with Friedman test, and in-groups statistical evaluations were performed with Kruskal-Wallis test using Bonferroni correction with SPSS statistical program (Version 9.01, SPSS Inc, Chicago, IL, USA).

RESULTS

The positive controls demonstrated complete dye penetration, whereas, the negative controls showed no dye penetration. Table 1 summarizes the actual numbers of teeth, means, medians and standard deviations of the three measurements of dye penetrations of the six experimental groups.

Table 1: *The Base Statistical Values for Amalgam and Composite Restored Sample Groups*

Group (n=15)	Measurement Interface	Mean	Median	SD
Ag-IRM	**a**	0.6	1	0.63
	b	0.4	0	0.50
	c	2	2	0.37
Ag-Colt	**a**	0.33	0	0.48
	b	1.13	1	0.35
	c	1.19	1	0.35
Ag-CLIP	**a**	0.8	1	0.56
	b	0.26	0	0.45
	c	0.13	0	0.35
Co-IRM	**a**	0.53	1	0.51
	b	1.93	2	0.25
	c	1.80	2	0.77
Co-Colt	**a**	0.26	0	0.45
	b	0.93	1	0.25
	c	1.20	1	0.41
Co-CLIP	**a**	0.26	0	0.45
	b	0.20	0	0.41
	c	0.26	0	0.45

Ag-IRM: Amalgam restored teeth with temporary restoration with IRM
Ag-Colt: Amalgam restored teeth with temporary restoration with Coltosol
Ag-CLIP: Amalgam restored teeth with temporary restoration with Clip
Co-IRM: Composite restored teeth with temporary restoration with IRM
Co-Colt: Composite restored teeth with temporary restoration with Coltosol
Co-CLIP: Composite restored teeth with temporary restoration with Clip
a: Permanent restoration-tooth interface
b: Permanent restoration-temporary restoration interface
c: Temporary restoration-tooth interface

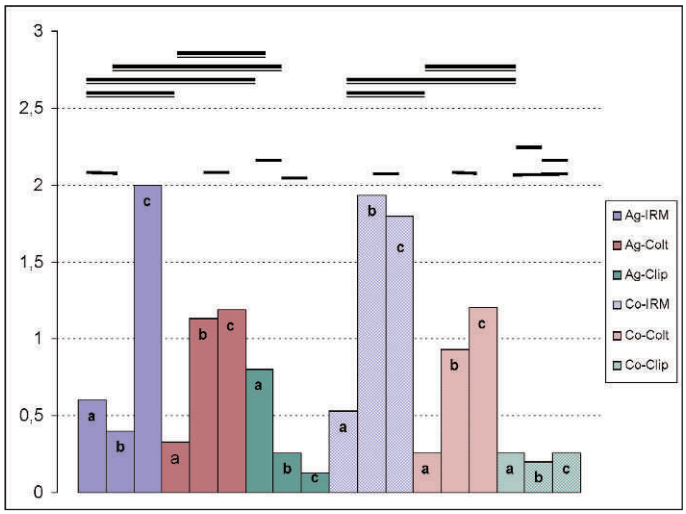

Figure 1. *Mean microleakage values of test groups and statistically significant differences between related groups.*

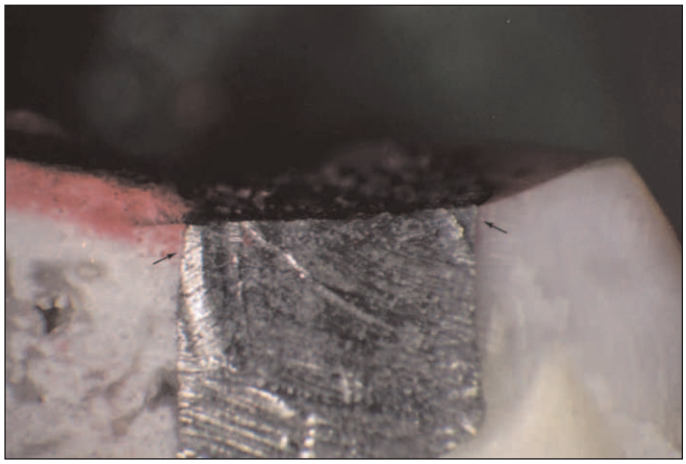

Figure 3. *Amalgam Coltosol interface (Ag-Colt b) exhibiting grade 1 dye penetration and Amalgam tooth interface (Ag IRM-a) exhibiting grade 1 dye penetration.*

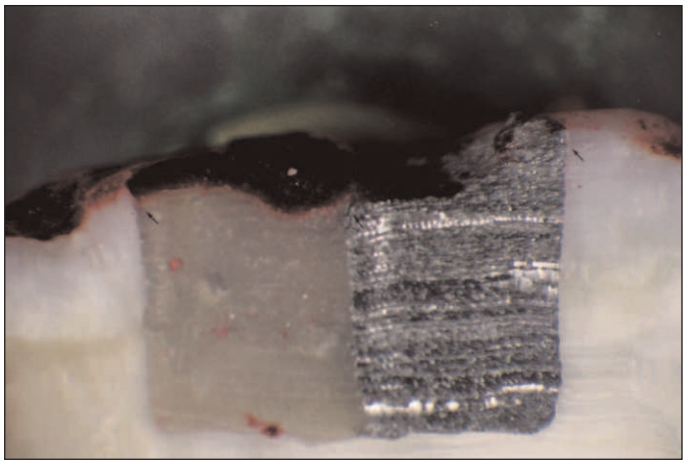

Figure 4. *Amalgam restored teeth with temporary restoration with CLIP exhibiting no microleakage.*

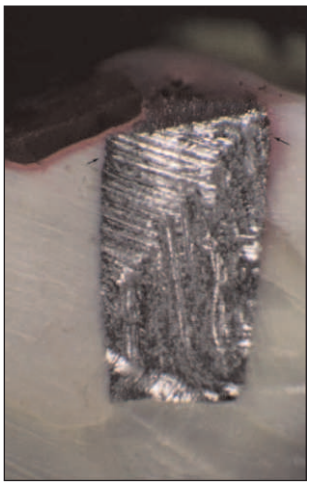

Figure 2. *Amalgam IRM interface (Ag-IRM b) exhibiting grade 1 dye penetration and Amalgam tooth interface (Ag IRM-a) exhibiting grade 2 dye penetration.*

The mean values and statistically significant differences in amalgam restored tooth groups (Ag-IRM, Ag-Colt, and Ag-CLIP) and in composite restored tooth groups (Co-IRM, Co-Colt, Co-CLIP) are shown in Figure 1.

The difference between microleakage values of amalgam-tooth and composite-tooth interfaces was not statistically significant ($p>0.05$), though higher values were observed at the amalgam tooth interfaces.

In the amalgam groups, the highest microleakage values at the temporary-permanent restorations interface (b) were observed with Coltosol (1.13) followed by IRM (0.40) and CLIP (0.26) (Figures 2, 3 and 4). Statistically significant differences were observed ($p<0.01$) except for IRM and CLIP ($p>0.05$). In the composite groups, the highest microleakage mean values at the temporary-permanent restorations interface (b) were observed with IRM (1.93) followed by Coltosol (0.93) and CLIP (0.20) (Figures 5, 6 and 7) and the differences were found to be statistically significant ($p<0.001$).

In the amalgam groups, the mean values at the previously-restored tooth tissues-temporary restorations (c) interface were respectively, from higher to lower, IRM (2)>Coltosol (1.19)>CLIP (0.13). These differences were statistically significant ($p<0.001$). In the composite groups, the mean values at the previously restored tooth tissues-temporary restorations (c) interface were respectively from higher to lower, IRM (1.80)> Coltosol (1.20)> CLIP (0.26) (Figures 5, 6 and 7). These differences were statistically significant ($p<0.05$).

DISCUSSION

Turner and others (1990) reported that Cavit, Cavit G, TERM, IRM and glass ionomer cement placed in endodontic access preparations made entirely within amalgam provided a seal that was as leak-proof as the control teeth which had Class I preparations restored with amalgam alone.

Pai and others (1999) pointed out that dye penetration between the primary and secondary restorative materials was less than the primary restorative materials and access cavity wall interface. In this study, all temporary restorative materials exhibited lower dye

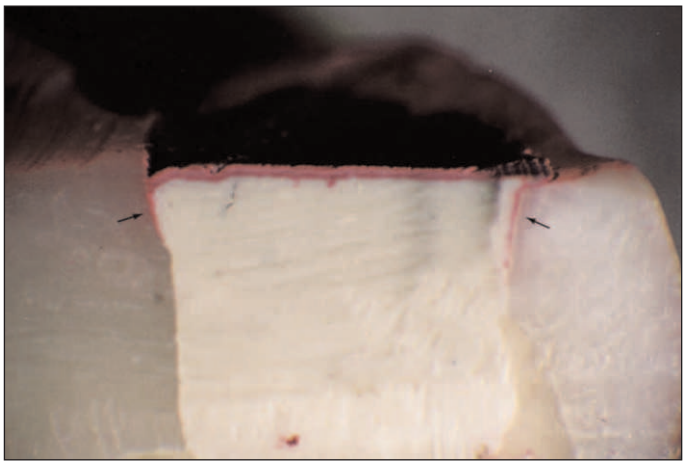

Figure 5. *Composite-IRM interface (Co-IRM-b) exhibiting grade 1 dye penetration and IRM-tooth interface (Co-IRM-c) exhibiting grade 1 dye penetration.*

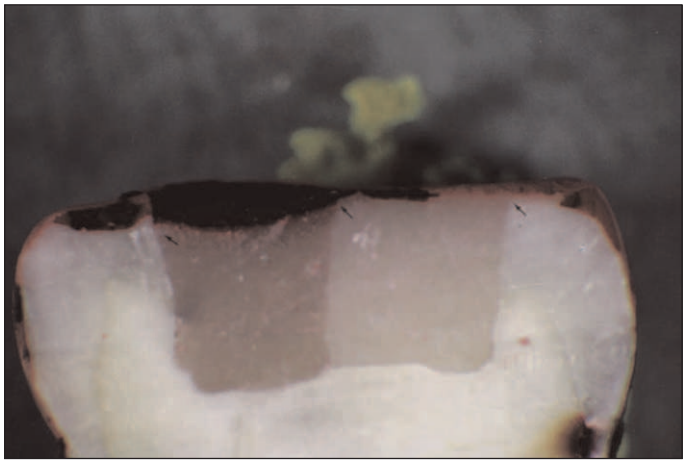

Figure 7. *Composite restored teeth with temporary restoration with CLIP. All interfaces exhibited no dye penetration.*

penetration values between the permanent and temporary restorative material compared to microleakage between the temporary restorative material and the access cavity wall except for the IRM-Composite and Amalgam-CLIP interface values. In particular, the microleakage values at the IRM-Amalgam interface were statistically lower than the IRM-tooth interface. In accordance with Pai's findings, the results of this study indicated that "there is no risk of increasing coronal microleakage without removing all the acceptable permanent restorations and only temporary restorations for access cavity are replaced between endodontic appointments."

A 5-mm total thickness for all restorations was used in this study in accordance with Webber and others (1978), who had found that a 3.5-mm thickness of zinc oxide-calcium sulphate without eugenol temporary materials was the minimum thickness necessary to prevent total leakage of the dye molecule.

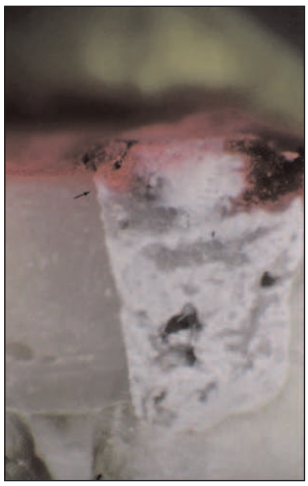

Figure 6. *Composite-Coltosol interface (Co-Colt-b) exhibiting grade 1 dye penetration and Coltosol-tooth interface (Co-Colt-c) exhibiting grade 1 dye penetration.*

The sealing ability of eugenol-free zinc oxide-calcium sulphate temporary restorative materials has been tested in many studies *in vivo* and *in vitro* with generally favorable results. Hygroscopic materials such as Cavit and Coltosol possess a high coefficient of linear expansion, which is almost two times greater than ZOE. This may explain Coltosol's excellent marginal sealing ability. Reinforced ZOE, IRM seems to demonstrate less expansion than Coltosol. These expansions enhance contact between the material and the access cavity, which will improve the seal (Anderson & others, 1990; Chohayeb & Bassiouny, 1985; Friedman & others, 1986).

Considering previous studies on the various temporary restorative materials with dye penetration (Blaney & others, 1981; Tamse, Ben-Amar & Gover, 1982; Teplitsky & Meimaris, 1988; Noguera & McDonald, 1990; Kazemi, Safavi & Spangberg, 1994; Barkhordar & Stark, 1990) or fluid penetration (Mayer & Eickholz, 1997; Anderson, Powel & Pashley, 1988; Pashley, Tao & Pashley, 1988) techniques, most of the studies suggest that Cavit has better microleakage properties except for studies by Blaney and others (1981) and Cruz and others (2002). On the other hand, these were not dye or fluid penetration, but bacterial penetration studies.

ZOE containing temporary restorative materials can give lower bacterial penetration values as a consequence of its antibacterial properties. Coltosol exhibited significantly lower microleakage values than IRM at temporary restorative material-tooth interfaces and temporary restorative material-permanent restorative material interfaces. However, it was obvious that it had absorbed dye fluid during hygroscopic expansion. This was in accordance with the findings of Lee and others (1993), who stated that water absorption on setting might explain why surface penetration of the dye was noted in Cavit. The inability of IRM to prevent microleakage has been previously reported (Anderson & others, 1990; Blaney & others, 1981). In this study, higher microleakage values were found with IRM prepared with a 2:1 powder/liquid ratio compared to Coltosol in accordance with the findings of Lee and others (1993).

CLIP has similar physical properties to other composites containing temporary restorative materials, such as TERM (Dentsply Caulk) and Fermit (Vivadent, Schaan, Liechtenstein). Teplitsky and Meimaris (1988) stated that unacceptable marginal leakage with TERM is contradictory to the claims made by the manufacturer. On the other hand, Bobotis and others (1989) demonstrated that the mean microleakage values of Cavit, Cavit-G, TERM and glass-ionomer cement did not differ significantly from intact crowns. In accordance with the findings of previous studies (Cruz & others, 2002; Noguera & McDonald, 1990; Anderson & others, 1988; Hansen & Montgomery, 1993), lower microleakage values were observed with CLIP, which has good handling properties and has demonstrated lower microleakage values than the other conventional temporary restorative materials. The manipulative aspects of TERM, which have similar properties to CLIP, were reported to be exceptional. It is easily dispensed, placed and cured. It is neither messy nor difficult to finish. In this study, although being a temporary restorative material, significantly lower microleakage values were observed at the CLIP-tooth interface when compared with the amalgam-tooth interface. Also, the values observed at the composite-CLIP interface were lower than at the composite-tooth interface. This can be explained by the fact that both materials have similar components.

Eugenol adversely affects the physical properties of resin composites (Reisbick & Brodsky, 1971), increasing their surface roughness and discoloration and inhibiting composite polymerization (Lingard, Davies & von Fraunhofer, 1981; Taira & others, 1992; Meryon, Johnson & Smith, 1988; Rutledge & Montgomery, 1990; Yap & others, 2002). In this study, consistent with the findings of these previous studies, the highest microleakage values were observed at the composite-IRM interface.

If endodontic therapy is completed in several appointments with longer inter-appointment intervals, and if the permanent restoration at the tooth requiring endodontic therapy provides better esthetics or higher resistance to chewing forces, preparing an access cavity through the partially removed permanent restoration and using temporary restorative materials for temporary obstruction of these cavities would not increase the risk of microleakage.

CONCLUSIONS

CLIP provided a better seal against microleakage at the amalgam interface and especially at the composite interface. This material also provided a better seal against microleakage at the tooth tissue's interface. The use of a resin based temporary restorative material over partially removed resin composite restorations could be beneficial in achieving better resistance to marginal leakage.

Within the limitations of this study, maintaining partially removed permanent restorations does not seem to cause a problem with achieving the marginal seal.

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Factors Affecting Microleakage of a Packable Resin Composite: An *In Vitro* Study

T Pamir • M Türkün

Clinical Relevance

Different adhesive systems may not equally affect microleakage of a packable resin composite. When the self-etching adhesive Prompt L-Pop was selected as a bonding agent, the flowable resin composite reduced microleakage. However, cavity preparation techniques had no effect on microleakage of the packable resin composite used in this study.

SUMMARY

This study was designed to determine the effects of three factors on the microleakage of a packable resin composite: different adhesive systems (single-step self-etching adhesive or total-etch and one-bottle adhesive), the use of a flowable resin composite (as a liner) and the different techniques of cavity preparation. Sixty extracted non-carious human first and second molars were selected and randomly divided into six groups. Cervical cavities were prepared using the conventional technique on the distal sides and the air-abrasive technique was used on the mesial sides of the teeth. The experimental groups were restored with PQ1 + SureFil or Prompt L-Pop + SureFil with or without PermaFlo. In the control groups, only SureFil was used on 10 teeth and PermaFlo + SureFil was applied on the remaining 10 teeth. The restored teeth were stored in 100% humidity at 37°C for 24 hours and thermocycled between 5°C and 55°C for 100 cycles. Each tooth was immersed in India ink for 48 hours, then sectioned. Dye penetration at the occlusal and gingival margins was scored by two independent operators. The data were statistically analyzed to assess the differences between the test and control groups. No significant differences among the adhesives in terms of the occlusal margins of the cavities were observed. However, PQ1 led to less microleakage compared to Prompt L-Pop at the gingival margins ($p<0.0062$). When flowable resin composite was used with Prompt L-Pop, microleakage was reduced ($p<0.0125$). However, no significant difference was observed between the two cavity preparation techniques ($p>0.0125$).

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INTRODUCTION

Even though chemically cured resin composites were first introduced in the early 1960s, they were not adequate for restoring posterior teeth (Jackson & Morgan, 2000) since they had shortcomings due to inadequate wear resistance, leakage, secondary caries formation and lack of appropriate proximal contact (Jackson & Morgan, 2000; Nash, Lowe & Leinfelder, 2001).

Recently, a new type of resin composite, termed, "condensable" or "packable," has been introduced to the market, which claims to have better physical properties, particularly in the restoration of posterior teeth (Combe & Burke, 2000; Jackson & Morgan, 2000; SureFil Technical Manual, 1998). Even though the average annual wear of these new resin composites may be equal to amalgam, microleakage still seems to be a problem (Leinfelder, 1995; Eakle & Ito, 1990; Cvitko, Denehy & Boyer, 1992). Packable resin composites of thick consistency have presented greater problems related to voids and cavity wall adaptation (Opdam & others, 1996; Opdam & others, 2002). In order to improve cavity wall adaptation and reduce microleakage, flowable resin composites may be suggested as an intermediate restorative material between dentin and packable resin composite. Due to their low moduli of elasticity, flowable resin composites may be useful in absorbing stresses caused by polymerization shrinkage (Jackson & Morgan, 2000; Unterbrink & Liebenberg, 1999). However, there is not enough data on the performance of flowable resin composites when used with packables.

Cavity preparation techniques may also be important for preventing microleakage. Recent advances in the microabrasion technique and the current ultra-conservative cavity preparation approach have resulted in a reemergence of the air abrasion technique (Black, 1955; Elderton, 1984). The surface characteristics of a cavity prepared by the air abrasion technique differ from those of the conventional technique (Banerjee, Kidd & Watson, 2000; Laurell & Hess, 1995). Differences in surface structure may affect the bonding strength of adhesives and eventually influence the microleakage of restoratives.

Another potent factor of microleakage may be the type of adhesive system used in the bonding of restorative material. In order to obtain adequate bonding, the smear layer formed during cavity preparation must be removed or treated primarily (Banerjee & others, 2000; Nakabayashi, Ashizawa & Nakamura, 1992; van Meerbeek & others, 1993), which is achieved by adhesives. However, the effects of the different adhesive systems on the smear layer and bonding quality vary widely.

Even though there is considerable research on the different adhesive systems, flowable resin composites and cavity preparation techniques, there is no single study that compares all of these factors, combined. Therefore, the aim of this study was to observe the effects of these components on the microleakage of a packable resin composite.

METHODS AND MATERIALS

Sixty extracted intact human first and second molars were selected for this study. The teeth were immediately scraped of any residual tissue tags, immersed in 0.5% NaOCl for 15 minutes and rinsed under running water for 15 minutes. The molars were then stored in deionized water at 4°C until cavity preparation.

After polishing the molars with pumice, round cavities were prepared on the cervical area using two different techniques, conventional and air abrasion. The cavities were prepared by conventional technique

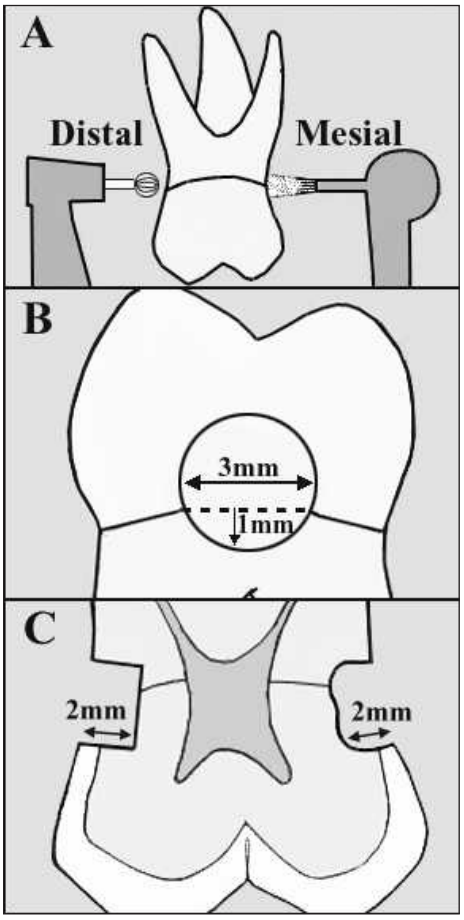

Figure 1A, B and C. *Schematic diagram of the experimental cavities.*

Table 1: *Experimental Design of This Study*

	Ultra Etch (Ultradent)	PQ1 (Ultradent)	Prompt L-POP (ESPE)	Permaflo (Ultradent)	Surefil (Dentsply/ Caulk)
Group 1	+	+			+
Group 2	+	+		+	+
Group 3			+		+
Group 4			+	+	+
Group 5					+
Group 6				+	+

Table 2: *List of Products Used in This Study*

Product	Manufacturer	Lot	Composition
Ultra etch	Ultradent	4FMV	Phosphoric acid, cetylpyridinium chloride.
PQ1	Ultradent	57PV	2-hydroxyethyl methacrylate, camphorquinone, ethyl alcohol as solvent carrier.
Prompt L-Pop	3M-ESPE	FW62210	di-HEMA-phosphate, complex fluoride, micro filler, water.
PermaFlo	Ultradent	58R2	Methacrylate monomer, alkylamino methacry late, camphorquinone.
SureFil	Dentsply/Caulk	990216	Barium boron fluoroalumino silicate glass, urethane modified Bis-GMA dimethacrylate, polymerizable dimethacrylate resin, amorphous fused silica.

using #FG6 tungsten carbide burs (SS White, Lakewood, NJ, USA) in a high-speed handpiece with water spray on the distal surfaces of the teeth (Figure 1A). The air abrasion technique (Micadent, Medidenta Inc, Woodside, NY, USA), with Al_2O_2 powder with a particle size of 27.5 microns (Kreativ Abrasive, Industrial Way, SW, Albany, NY, USA), was used for cavity preparation on the mesial surface of the same tooth (Figure 1A). All cavities were 3-mm wide and 2-mm deep, and the cervical margins of the cavities were located 1 mm below the cemento-enamel junction (Figure 1B-1C). The size of each preparation was measured using a compass.

Following the cavity preparations, the teeth were randomly divided into six groups (Table 1). The materials used in this study are listed in Table 2.

In the first group, all cavities received a total-etch with 35% phosphoric acid etching gel for 15 seconds and were rinsed with water and an air syringe for 20 seconds. PQ1 bonding agent was rubbed onto the cavity surfaces for 15 seconds using the wet bonding system. Excess bonding agent was air-thinned with a quick air-burst, and the agent was light-cured for 20 seconds using an Optilux 401 visible light-curing unit (Kerr/Demetron, Danbury, CT, USA) with an intensity output in excess of 450 mW/cm^2. The output of the device was controlled before and after the operation. SureFil packable resin composite was then placed in increments following the manufacturer's recommendations and was light-cured for 40 seconds.

In the second group, all cavities were also subjected to total etching with 35% phosphoric acid gel for 15 seconds. After application of PQ1 bonding agent, a flowable resin composite (PermaFlo), approximately 1-mm thick, was placed on the cavity floor and axial wall. Following light curing of the material for 40 seconds, SureFil was used for final restoration, as in the first group.

In the third group, a single-step self-etching adhesive Prompt L-Pop was directly applied to all preparations in a single coat with agitation for 15 seconds, without a separate etching procedure. A gentle stream of dry air was used to disperse the material into a thin film, light cured for 10 seconds, then the restorations were completed with SureFil.

In the fourth group, flowable resin composite PermaFlo was placed between Prompt L-Pop and SureFil.

In the fifth and sixth groups, no adhesive was used, since these groups were considered the control groups. In the fifth group, only SureFil was used, whereas the teeth were restored with PermaFlo and SureFil in the sixth group. All procedural steps were performed according to the manufacturers' instructions. The margins of the restorations were finished using a series of SofLex discs (3M ESPE, St Paul, MN, USA).

All samples were stored in 100% humidity at 37°C for 24 hours and thermocycled between 5°C and 55°C in a water bath for 100 cycles with a 30-second dwell time. Following the thermal cycles, the samples were air dried and a dark finger nail polish was applied to the teeth, except on the restorative material and tooth structure 1.0 mm from the restoration margins. All specimens were then immersed in India ink (Pelikan, Hanover, Germany) in separate sealable glass vials at 37°C for 48 hours. This dye was used as a tracer in previous studies, since bacterial ingress and India ink penetration provided a similar rank order for the sealing ability of the materials tested (Chong & others, 1995; Torstenson & Brannström, 1988).

The teeth were rinsed under running water and sectioned in two different directions: buccolingually along the long axis and longitudinally crossing from the center of the restoration. Dye penetration at the occlusal and gingival margins was examined using a light microscope (Olympus Co, Tokyo, Japan) under 30x magnification by two independent pre-calibrated examiners. The following scoring criteria were used to evaluate the microleakage:

0: No evidence of dye penetration.

1: Dye penetration less than half of the occlusal or gingival wall.

2: Dye penetration along the occlusal or gingival wall.

3: Dye penetration along the axial wall.

In case of any disagreement, new readings were performed until a consensus was reached. Differences among the groups were analyzed statistically by

Kruskall Wallis test, and α was set as 0.05. Then, pairwise comparisons were made using the Mann-Whitney U test with Bonferroni correction (p=0.0062). Additionally, the Friedman Test, followed by the Wilcoxon signed ranks test with Bonferroni correction, were used to analyze differences between microleakage of the occlusal and gingival margins and the effect of the cavity preparation techniques on microleakage scores (p=0.0125). The level of significance was primarily set as p=0.05 in all tests. However, after Bonferroni correction, this level was reset to p=0.0062 and p=0.0125, respectively.

Following microleakage assessment, the specimens were dehydrated by immersion in increasingly concentrated alcohol solutions (30%, 50%, 70% and 100%, respectively) for 30 minutes in each bath. Subsequently, they were sputter-coated with gold (200 A° in thickness) and viewed with a scanning electron microscope (SEM) (JEOL, JSM-5200, Tokyo, Japan) to observe the quality of bonding between the restorations and dental hard tissues.

RESULTS

Tables 3 and 4 show distribution of the microleakage scores at the occlusal and gingival margins of the cavities prepared by the conventional and air abrasion technique.

Microleakage scores of the adhesives exhibited statistically significant differences between the study groups (p<0.05). Post-hoc tests for pairwise comparisons indicated that differences between the adhesives occurred at the gingival margins of the cavities prepared by either technique and PQ1 led to significantly less microleakage than Prompt L-Pop (p<0.0062).

The use of a flowable resin composite reduced the microleakage of Prompt L-Pop at the gingival margin

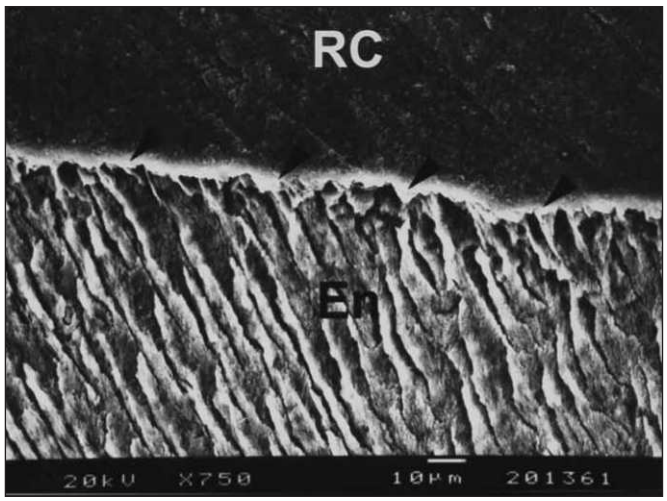

Figure 2. *Good enamel adaptation of PQ1 (RC: resin composite, En: enamel, arrows: PQ1).*

Table 3: *Microleakage Scores Obtained from the Occlusal Margins*

Scores	GROUPS											
	PQI[a]		PQI + flowable[a]		Prompt L-Pop[a]		Prompt L-Pop + flowable[a]		No Adhesive + no flowable (Control I)[b]		No Adhesive + flowable (Control II)[b]	
	Air Ab[c]	Con[c]	Air Ab[d]	Con[d]	Air Ab[e]	Con[e]	Air Ab[f]	Con[f]	Air Ab[g]	Con[g]	Air Ab[h]	Con[h]
0	10	10	9	10	8	5	10	9	0	0	0	0
1	0	0	1	0	2	5	0	1	0	0	0	0
2	0	0	0	0	0	0	0	0	0	0	0	0
3	0	0	0	0	0	0	0	0	10	10	10	10

Same superscript letters indicate no statistically significant difference.

Table 4: *Microleakage Scores Obtained from Gingival Margins*

Scores	GROUPS											
	PQI[a]		PQI + flowable[a]		Prompt L-Pop[b]		Prompt L-Pop + flowable[a]		No Adhesive + no flowable (Control I)[c]		No Adhesive + flowable (Control II)[c]	
	Air Ab[d]	Con[d]	Air Ab[e]	Con[e]	Air Ab[f]	Con[f]	Air Ab[g]	Con[g]	Air Ab[h]	Con[h]	Air Ab[i]	Con[i]
0	6	3	6	5	1	0	5	5	0	0	0	0
1	2	4	2	3	0	0	2	2	0	0	0	0
2	2	3	2	1	1	1	2	1	0	0	0	0
3	0	1	0	1	8	9	1	2	10	10	10	10

Same superscript letters indicate no statistically significant difference.

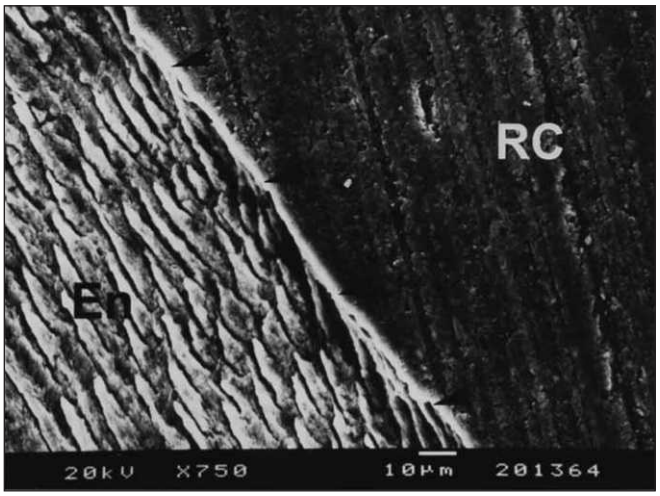

Figure 3. *Good enamel adaptation of Prompt L-Pop (RC: resin composite, En: enamel, arrows: Prompt L-Pop).*

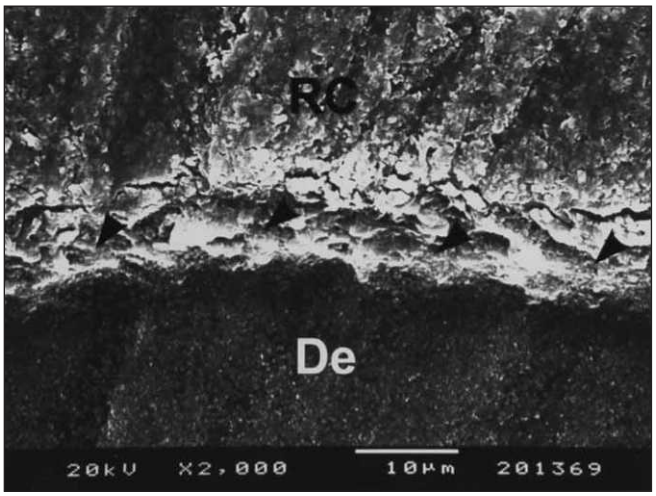

Figure 4. *Dentin adaptation of PQ1 without flowable resin composite (De: dentin, RC: resin composite, arrows: PQ1).*

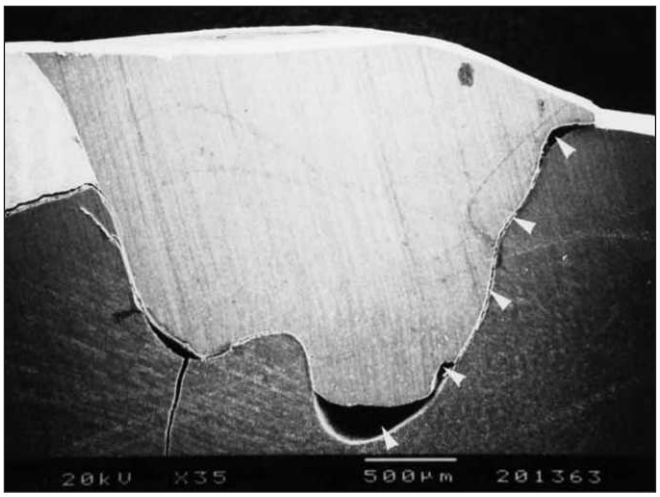

Figure 5A. *Gap formation beneath the packable resin composite bonded Prompt L-Pop without flowable resin composite (arrows: gap formation).*

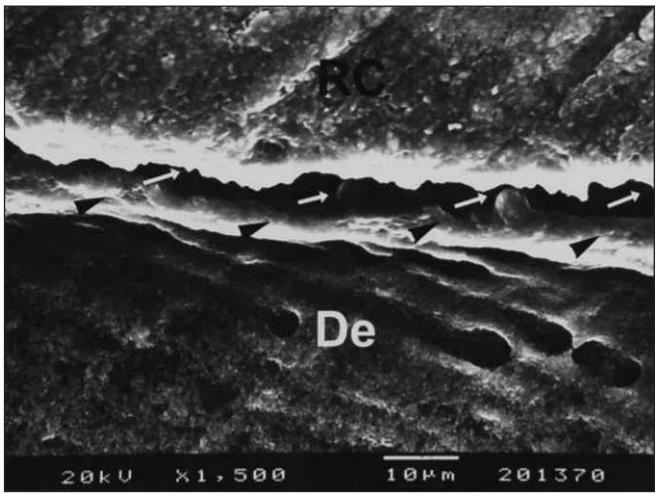

Figure 5B. *Gap formation of another sample from the same group in higher magnification (De: dentin, RC: resin composite, black arrows: Prompt L-Pop, white arrows: gap).*

($p<0.0062$). Adversely, the use of PQ1 with flowable resin composite did not significantly change microleakage compared to its single use ($p>0.0062$). No significant differences were revealed between treatment groups at the occlusal margins of the cavities prepared by either technique ($p>0.062$). Additionally, neither the conventional nor the air-abrasion technique significantly affected the microleakage values of the groups ($p>0.0125$).

In SEM evaluation, both PQ1 and Prompt L-Pop exhibited good enamel adaptation at the occlusal margins (Figure 2 and 3). PQ1 bonded not only to enamel but also to dentin at the gingival margin (Figure 4). Nevertheless, when Prompt L-Pop was used without flowable resin composite, gap formations between the restoration and dentin were observed (Figure 5A-5B). Prompt L-Pop showed better dentin adaptation when used in combination with flowable resin composite (Figure 6).

DISCUSSION

Determination of microleakage performance of a material is important, because microleakage can lead to staining, sensitivity and/or caries (Yap, Lim & Neo, 1995). In this study, potent factors likely to affect the microleakage of a packable resin composite were examined and assessed by using dye penetration into the occlusal and gingival margins of the cavities. The results of this study did not exhibit any significant differences among the experimental groups at the occlusal margins, except for the controls. With improvements in resin technology, microleakage may not be a problem at the occlusal margins of cavities. However, in this study, higher microleakage values in dentin were observed

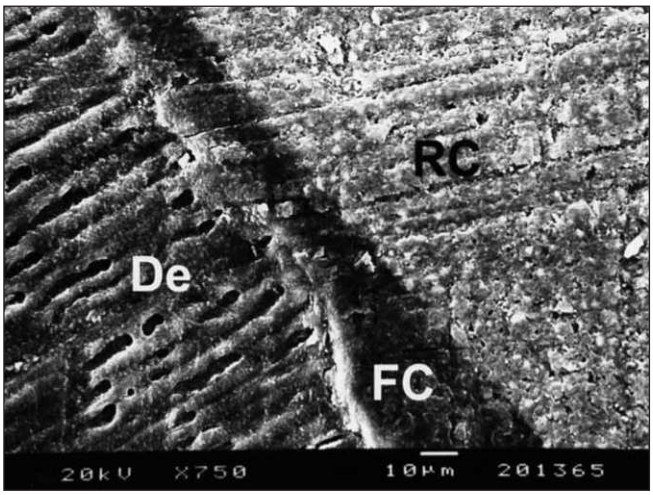

Figure 6. *Dentin adaptation of Prompt L-Pop with flowable resin composite (De: dentin, FC: flowable resin composite, RC: resin composite).*

compared to those in enamel, as observed in previous studies, (Leevailoj & others, 2001). Therefore, efforts have still concentrated on reducing the high leakage values in dentin by applying alternative methods and materials. In this respect, the development of adhesives has received enormous interest in dental practice.

In this study, the total-etch and one-bottle adhesive system PQ1 had significantly lower microleakage scores compared to the single-step, self-etching adhesive Prompt L-Pop. Similar results have been reported in various studies (Cardosa & others, 1999; Tung, Estafan & Scherer, 2000). The manufacturer of PQ1 recommends rub and scrub action to apply the agent to the treated moist dentin surface. Tung and others (2000) have suggested that this action might have pushed the filler particles within the bonding agent into the exposed dentinal tubules, resulting in deeper penetration into the tubules.

Being a single-step self-etching adhesive, Prompt L-Pop has the combination of etching, priming and adhesive potentials in a single solution. A recent approach towards the simplification of the bonding procedure has introduced this new system to the market. However, the sealing capability of Prompt L-Pop is questionable, despite its easy handling and time saving advantages. In a previous study (Pontes, de Melo & Monnerat, 2002), the least microleakage on enamel and dentin margins was reported with Prompt L-Pop. On the contrary, there are studies which state complete bond failures and the worst microleakage values of this adhesive, especially on the dentin surface (Agostini, Kaaden & Powers, 2001; Pradelle-Plasse & others, 2001; Yazici, Baseren & Dayangaç, 2002; Bouillaquet & others, 2001). Although the manufacturer of Prompt L-Pop put this adhesive on the market for both resin composites and compomers, Rosa and Perdigão (2000) mentioned that, since Prompt L-Pop is a water-based material, it is chemically more compatible with restorative materials that have enhanced hydrophilic properties, such as compomers, rather than with more hydrophobic restoratives, such as resin composites. Recently, it has been shown that single-step self-etching adhesives acted as permeable membranes that permit water to diffuse from hydrated dentin and, therefore, deteriorated the composite-adhesive adaptation (Tay & others, 2002; Tay & others, 2002). According to these explanations, it may be expected that better composite adaptation would be obtained with a total-etch, ethanol-based adhesive, such as PQ1.

It was reported that most of the restorations bonded with Prompt L-Pop showed interfacial gaps between dentin and the restorative and presented no visible hybrid layer (Da Silva Telles & others, 2001). The current study also revealed gaps between the restorations and teeth in SEM images when Prompt L-Pop was used without a flowable resin composite (Figure 5A-5B). However, these dentinal gaps might also have been observed as a result of the dehydration process that was required for preparation of the samples for SEM evaluation. Poor adaptation and voids between the resin composite and cavity walls are the most significant shortcomings of packable resin composites, as they are highly viscous materials (Opdam & others, 2002). In order to overcome these problems, the use of a flowable resin composite in the cavity floor may be suggested (Chuang & others, 2001). Due to their low moduli elastomeric properties, flowable resin composites may partially compensate the stress caused by polymerization shrinkage (Bayne & others, 1998; Unterbrink & Liebenberg, 1999). Therefore, many researchers have recommended the use of flowable resin composites beneath packables and have claimed that this application may reduce microleakage (Leevailoj & others, 2001; Tung & others, 2000; Peutzfeldt & Asmussen, 2002). It has been suggested that restorative materials, such as flowable and packable resin composites of the same manufacturer, should be used (Jackson & Morgan, 2000). However, the products of different manufacturers have been widely used in dental practice and different brands of flowable and packable resin composite were also used in this study. Nevertheless, SEM evaluation did not reveal any signs of incompatibility.

In this study, flowable resin composite, in combination with Prompt L-Pop, reduced microleakage at the gingival margin and also decreased voids in the restored interface. SEM photographs of this application displayed better dentin adaptation at cavity walls (Figure 6). We suggest that in the single-step, self-etching adhesive system Prompt L-Pop applications, flowable resin composite should be applied beneath a packable resin composite.

However, when total-etch and one bottle adhesive PQ1 was used, flowable resin composite did not significantly improve microleakage at dentin margins. This finding may be explained by better bonding of the two-step adhesive system PQ1 to tooth structure compared to single-step Prompt L-Pop. SEM evaluation indicated that, at the gingival area, the dentin bonding of PQ1 without flowable resin composite was satisfactory (Figure 4).

In a previous study (Laurell & Hess, 1995), it was suggested that the nozzle-tip diameter, air pressure and powder flow rate had no effect on the bonding of resin composite to air-abraded enamel. However, tooth structure was rapidly and efficiently cut at 120 psi without causing discomfort to the patient. Therefore, in this *in vitro* study, air pressure and nozzle-tip diameter were selected as 120 psi and 0.016 inch, respectively.

SEM examination of the samples prepared with the air abrasion technique revealed rounded cavosurface margins and internal line angels. Also, a halo of abraded enamel surrounding the outline of the cavity and microscopic roughness of the treated enamel/dentin surfaces were different from those cavities where the conventional technique was used (Banerjee & others, 2000; Laurell & Hess, 1995). These characteristics conform to currently held theories of preparation for direct composite resin restorations (Elderton, 1984). Therefore, better adaptation of a resin composite to cavity walls and, hence, lower microleakage may be expected with the air abrasion technique compared to the conventional technique. However, the results of this study showed that the cavity preparation technique had no significant effect on the microleakage of packable resin composite. In other words, microleakage values did not change significantly when the cavity was prepared using the air abrasion technique. Therefore, it was concluded that the conventional technique was still the method of choice when the higher cost, the need for a powerful aspirator and non-user friendliness of the air abrasion technique were considered.

CONCLUSIONS

In the current study, factors affecting microleakage of a packable resin composite were examined. In this respect, both total-etch and one bottle PQ1 and single-step self-etching Prompt L-Pop have performed similarly on enamel margins. However, on dentin margins, Prompt L-Pop had significantly higher microleakage scores when compared to PQ1. This adhesive performed as well as PQ1 when used with a flowable resin composite. SEM examination revealed that there were voids and gaps at the restored interface when Prompt L-Pop was used without a flowable resin composite. However, the use of PQ1 with or without a flowable resin composite did not affect the performance of the material. The preparation of cavities using the conventional or air abrasion technique did not significantly change the microleakage values of the packable resin composite used in this study.

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Measurement of Linear Polymerization Contraction Using Digital Laser Interferometry

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Clinical Relevance

Digital holographic interferometry as a new method of polymerization shrinkage measurement presents new insights into the setting of composite material during polymerization with curing lights of different light intensity. Based on this, the clinican should be able to choose an adequate curing light for resin composite polymerization that can maximally compensate its negative influence on polymerization shrinkage.

SUMMARY

Polymerization shrinkage is an unavoidable consequence of resin composite photopolymerization and is one of the most important factors in determining the clinical quality and durability of composite filling. Many different methods of measuring polymerization shrinkage are described in the literature. Digital laser interferometry is a method that enables direct observation of polymerization shrinkage in real time. This study used the digital holographic interferometry method to measure the linear polymerization contraction of composite materials: Tetric Ceram (Vivadent), Spectrum TPH (Dentsply) and Valux Plus (3M Dental Products) polymerized with three different curing modes of the Elipar Trilight (ESPE) halogen curing unit. The highest polymerization contraction was recorded by "standard mode" (ETS) (1.24±2.66% lin), and the lowest by "medium mode" (ETM) (0.40±0.41% lin) during 40 second illumination. The "exponentional mode" (ETE) showed the highest expansion during the first 10 seconds of illumination. Curing units with initial low intensity enable better inner adaptation of composite material, preventing the detachment of material from dentin during polymerization and avoiding the negative consequences of polymerization shrinkage.

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INTRODUCTION

The first units of photopolymerization emitted UV light were, because of their negative components and harmful influence on the organism, replaced with a blue light of 400-500 nm. Halogen curing units of 390-520 nm wavelength and light intensity between 150-1200 mW/cm^2

are mostly used in clinical practice today (Watts & Cash, 1994; Meniga & others, 1999).

An unavoidable consequence of resin composite photopolymerization, which greatly influences the quality and durability of composite filling, is polymerization contraction. The contraction depends on the following factors: cavity configuration, resin composite components, application technique of the composite material in the cavity, light source, light intensity and exposure time.

In general, volumetric curing shrinkage determinations are basically free shrinkage measurements and therefore offer total pre- and post-gel curing contraction. Dimensional changes in linear curing contraction determinations are more or less "hindered" and therefore should be regarded as a post-gel curing phenomenon (Davidson & Feilzer, 1997). Until today, a small number different methods for measuring photopolymerization shrinkage have been described, including: dilatometry (mercury or water dilatometry), linometer, measuring of specific density and weight of the material, the "strain-gauge" method and others (de Gee, Feilzer & Davidson, 1993; Tarle & others, 1998; Cook, Forrest & Goodwin, 1999; Watts & Cash, 1991).

Measurement using the dilatometer method includes weighting the samples with a microbalance, then the data, along with the determined density of the composite paste, is entered into the computer's program. Polymerization shrinkage was calculated using integrated software. Data were recorded continuously as a volume shrinkage measurement. The obtained values varied from 2.09±0.08% to 3.77±0.06)% (Rosin & others, 2002).

Polymerization shrinkage of the specimens was also often determined by the density bottle method, with values varying from 2.5±0.40% to 4.1±0.42 (Tarle & others, 1998).

Curing contraction is usually determined linearly. Watts and Cash (1991) used an indirect method of determination, the "deflecting disk" method, which implies reproducible measurement of polymerization shrinkage. They calculated the volumetric contraction from the "post-gel" linear displacement of a deflecting disk resting on a brass ring in which a resin composite disk is centrally located. Results varied from 1.28±0.07% to 2.05±0.05%.

Aw and Nicholls (2001) measured dimensional changes in a linear direction using a calibrated light microscope. Polymerization shrinkage values varied from 0.430±0.041% to 0.640±0.092%.

The linear curing contraction from the start of the setting reaction was determined by a linometer device using a contactless displacement transducer and the values varied from 0.52±0.02% to 0.99±0.05% (Feilzer, de Gee & Davidson, 1988).

Linear polymerization contraction was also measured by Park, Krejci and Lutz (1999) using a custom-made linometer. In this linometer, the amount of displacement of an aluminum disk, which was caused by linear shrinkage of the resin composite, was recorded by computer each second for 90 seconds. Linear shrinkage (μm) varied from 10.5 to 14.3.

Yap, Ng and Siow (2001) used a strain-monitoring device to measure linear polymerization shrinkage associated with the different cure modes and exposure times greater than 180 minutes. The rate of shrinkage for all curing parameters was greatest during the light polymerization reaction and continued after the curing light was removed.

The newest method of measuring shrinkage is digital laser interferometry. Digital laser interferometry is a procedure based on recording a sequence of interference patterns by using an optoelectronic interferometer. To record the two-dimensional fringe patterns of the interference of laser beams, a charge coupled device (CCD) camera was used. And, to process the recorded patterns, a computer program was used. Regardless of the experimental devices used, fringe distribution shows the phase relationship between the two laser beams. Usually, one beam is called a reference beam and the other is referred to as an object beam. Keeping the reference beam constant, a change in fringe distribution of the sequence indicates a deformation of the object beam.

Most of the algorithms used to extract the deformation from a series of measurements have a typical accuracy of $\lambda/10$ (λ–wavelength, for He-Ne laser λ=632.8 nm). However, to interpret deformation, fringe analysis must be performed with specific data about the beam characteristics and the type of expected change. It is not necessary to calibrate deformation values, since the absolute value of λ is known (Demoli & others, 2004).

Recent applications of digital interferometry demonstrate the effectiveness of the technique, especially in measuring dynamic processes (Lovric & others, 2003; Demoli, Vukicevic & Torzynski, 2003) or for industrial metrology with a commercial instrument (de Groot & others, 2002). A device based on the Michelson interferometer was used to demonstrate polymerization shrinkage measurements of the dental restoratives (Fogleman, Kelly & Grubbs, 2002).

In this study, digital interferometry is used to measure the thickness variations of the resin composite materials due to initial thermal expansion and polymerization shrinkage of the specimens during continuous curing. The proposed device differs from the reported one (Fogleman & others, 2002) based on the experimental approach (different optical configurations), by

Table 1: *Composite Materials Used in This Experiment*

Composite Material (shade A2)	Manufacturer	Batch #	Type of Composite	Size of Anorganic Filler Particle	Abbreviation
Tetric Ceram	Vivadent, Schaan, Liechtenstein	914760	fineparticle hybrid	0.04-3µm	TC
Spectrum TPH	Dentsply GmbH Konstanz, Germany	9912000398	fineparticle hybrid	0.04-4µm	S
Valux Plus	3M Dental Products St Paul, MN, USA	20000506	fineparticle hybrid	0.01-3.5µm	VP

detector selection (two-dimensional versus one-dimensional), by the way the information is being processed and by the sample carrier solution.

This study determined the linear polymerization shrinkage of resin composites polymerized with different curing modes using digital laser interferometry and compared the measurements obtained with this method against other methods of polymerization shrinkage measurements.

METHODS AND MATERIALS

Table 1 shows the composite materials used in this experiment.

For measuring polymerization shrinkage, resin composite samples 0.65-mm thick and 7 mm in diameter were prepared. For that purpose, an adequate amount of composite material was placed between two transparent foils, each 0.175-mm thick and placed in a stainless steel ring 1-mm thick and pressed between two round plates made of the same material. The total thickness of the sample with sheets was 1 mm (Figure 1).

Resin composite samples were polymerized with the Elipar Trilight halogen curing unit using three different illumination programs:

- "medium mode"—which emits continuous light at 450 mW/cm^2 intensity for 40 seconds.
- "exponentional mode"—illumination begins at 100 mW/cm^2 and rises exponentionally for the first 15 seconds to 800 mW/cm^2, then for the next 25 seconds, it emits a continuous light of 800 mW/cm^2 intensity.
- "standard mode"—emits continued light with an intensity of 800 mW/cm^2 for 40 seconds.

The light intensities were determined with the Curing Radiometer Model 100 (Demetron Research, Danbury, CT, USA). The spectrum wavelength for this curing unit lies between 400 and 515 nm.

Five independent measurements for each material and illumination mode were made. The measurement values obtained by digital holographic interferometry are the result of linear polymerization contraction.

For measuring thickness variations of the resin composite materials during blue light photopolymerization, a set-up schematic, shown in Figure 2, was used. The

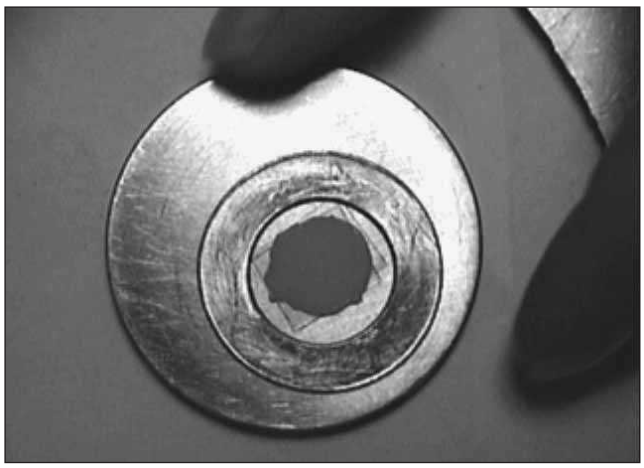

Figure 1: *Resin composite sample prepared for polymerization shrinkage measurement.*

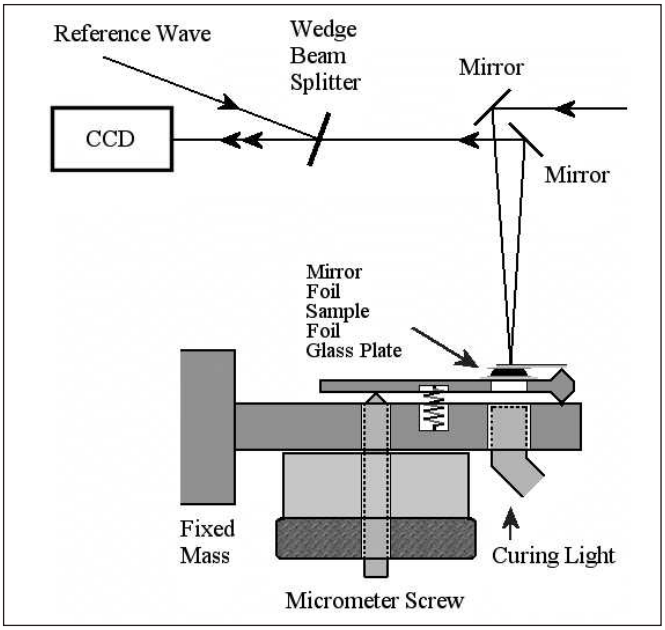

Figure 2: *Schematic set-up for measuring polymerization contraction.*

beam emerging from the He-Ne laser (Spectra Physics, model 127, power = 25 mW, wavelength = 632.8 nm) was first split into an object and a reference beam. The reference beam was expanded and steered directly onto the CCD camera. The object beam was first expanded,

collimated and steered onto the mirror situated on the upper surface of the sample, then reflected at a small angle from the mirror and finally directed to the CCD camera. In this way, the CCD camera recorded the fringe pattern formed by the interference of two beams. The samples were sandwiched between two glass plates, placing transparent foils between the glass and composite material. The device was designed to allow photopolymerization of the samples from one side (curing light direction) and simultaneous measuring of the thickness variation of the samples using the digital interferometry method from the other side (laser light direction). The micrometer was mounted to enable the manual compensation of the displacement of the upper surface of the sample. It was used at the end of each measurement to find the final linear shrinkage of the sample.

RESULTS

Table 2 gives the results of linear polymerization shrinkage as mean values and standard deviations in the 10th, 20th and 40th second of illumination. The mean values and standard deviation were gained using analyses of variance. Figures 3, 4 and 5 show the values of polymerization shrinkage in each second of illumination.

The results of polymerization shrinkage measurement show that the highest shrinkage was recorded by resin composite samples polymerized with the ETS mode for resin composite S (1.24±2.66% lin). The lowest values of polymerization shrinkage were recorded by ETM illumination mode (resin composite S 0.40±0.41% lin, and for TC 0.41±0.9% lin).

The initial expansion was recorded during the first 10 seconds for all tested materials and all illumination modes. In the tables, expansion can be seen only for the ETE illumination mode, because in this mode, illumination lasts the longest (Table 2). ETM and ETS illumination programs show rapid bending of the curve in a negative direction at the start of polymerization, which points to fast expansion that ends in five to eight seconds. On the contrary, in ETE mode, expansion is slightly weaker and lasts longer (10 to 13 seconds) (Figures 3, 4 and 5).

DISCUSSION

Light intensity is an important factor in the quality of resin composite polymerization, which is influenced by construction and execution of the curing unit. The results of some experiments have shown that light with intensities ranging from 200 to 300 mW/cm^2 will not polymerize enough resin composite during 40-second illumination. Therefore, if intensity is maintained, it would be necessary to prolong the illumination time to 120 seconds. It is possible to achieve optimal polymerization of resin composites using 30 seconds of illumi-

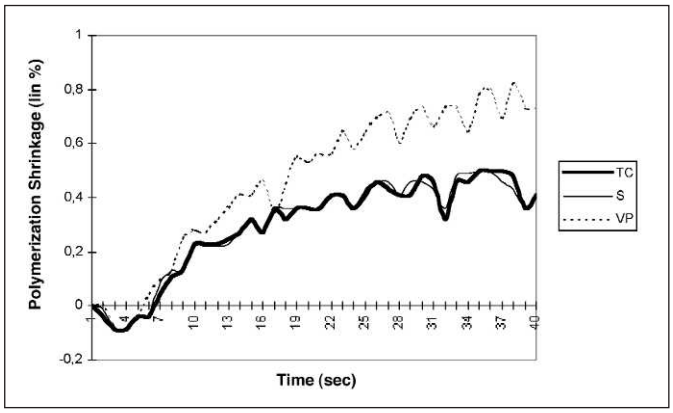

Figure 3: *Linear polymerization shrinkage of composite materials illuminated with ETM mode.*

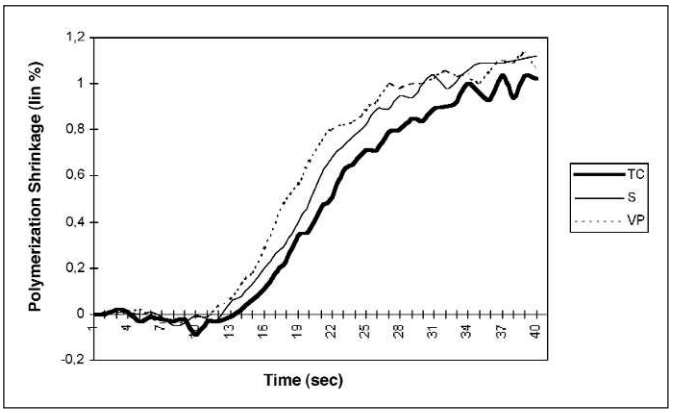

Figure 4: *Linear polymerization shrinkage of composite materials illuminated with ETE mode.*

Table 2: *Linear Polymerization Shrinkage*

Polymerization Shrinkage (LIN%)																		
Composite Material	ETM Mode						ETE Mode						ETS Mode					
	10 Seconds		20 Seconds		40 Seconds		10 Seconds		20 Seconds		40 Seconds		10 Seconds		20 Seconds		40 Seconds	
	$\bar{x}$	sd	$\bar{x}$	sd	$\bar{x}$	sd	$\bar{x}$	sd	$\bar{x}$	sd	$\bar{x}$	sd	$\bar{x}$	sd	$\bar{x}$	sd	$\bar{x}$	sd
TC	0.23	0.01	0.36	0.01	0.41	0.9	-0.09	0.45	0.36	1.03	1.02	1.21	0.24	1.08	0.69	1.35	1.04	1.63
S	0.22	0.11	0.36	0.01	0.40	0.41	-0.01	0.51	0.48	1.86	1.12	1.59	0.30	2.50	0.93	3.58	1.24	2.66
VP	0.28	0.29	0.53	0.24	0.70	0.28	-0.01	0.44	0.66	0.89	1.06	1.72	0.53	2.31	0.98	2.12	1.23	2.46

nation and light intensity from 500 to 600 mW/cm^2 (Yap & Senevirante, 2001). Studies by many authors have shown that the light intensity of polymerization units, which are used in daily clinical practice, in most cases, are not adequate. Barghi, Berry and Hatton (1994) have shown that 45.5% of 209 tested curing units had a light intensity of less than 300 mW/cm^2. In 1998, light curing units testing in clinical practice was conducted in Zagreb and showed that only 56% of the tested light curing units satisfy clinical conditions for use (Knezevic & others, 1999).

During exposition of the resin composite by low light intensity, activation of the photoinitiator could become difficult, especially in the deep parts of the filling. On the other hand, faster polymerization with high intensity leads to a quicker initiation phase but also to a more rapid termination phase of the polymerization reaction (Miyazaki, Fukuiski & Onose, 1999). Uno and Asmussen (1991) and Sakaguchi and Berge (1997) recommended the use of low light intensity for polymerization to enable slow hardening of the composite material and a decrease in polymerization shrinkage, polymerization shrinkage stress and formation of microleakage as its consequence.

The authors tried to confirm their opinion in this study by using different illumination modes with different light intensities. Research of polymerization contraction using digital laser interferometry showed that the highest polymerization contraction exhibited was in the composite materials polymerized with ETS illumination mode, whereas, the lowest contraction was recorded for resin composite samples polymerized with the ETM illumination mode. Those results were expected, because the ETS illumination mode has almost two times higher an intensity than the ETM illumination mode.

The curves of polymerization contraction by illumination of the composite materials reached with the ETS (Figure 5) and ETM mode (Figure 3) are similar in shape, but the numeric values reached by the ETM illumination mode were lower (Table 2). The similar curve shape of these two modes relates to how they are illuminated, that is, continuous intensity, whereas, numerical values are the consequence of light intensity (for the ETS mode, 800 mW/cm^2 and for ETM, 450 mW/cm^2). The polymerization contraction curve reached by the ETE illumination mode (Figure 4) is obviously different from the other two illumination modes as a consequence of the lower light intensity emitted during the first 15 seconds. It can be seen that the curve from the 15th second, when the light intensity is 800 mW/cm^2 (it stays the same for the next 25 seconds), is similar to the polymerization contraction curve from the ETS and ETM mode, because the ETE mode also emits a continuous intensity of light from

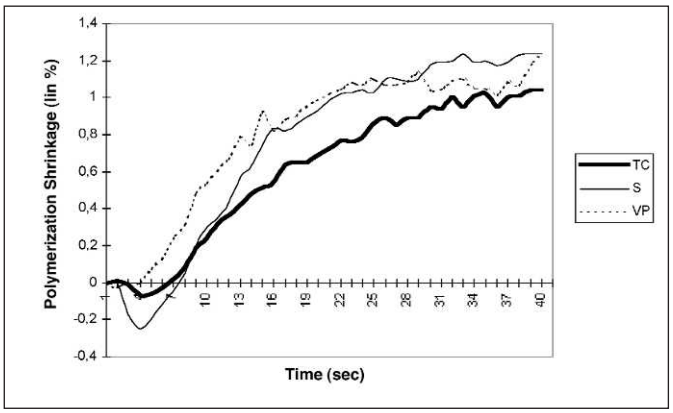

Figure 5: *Linear polymerization shrinkage of composite materials illuminated with ETS mode.*

the 15th to the 40th second. When observing the results from Table 2 only, there is not a big difference in final values of polymerization contraction after 40-second illumination for both the ETS and ETE modes. However, the difference is in the time schedule of polymerization contraction.

It is known that the polymerization process occurs in two phases: pre-gelation and post-gelation. The intermediate phase, gel point, is a moment of material hardening. Polymerization contraction occurs in both phases. Contraction in the pre-gelation phase (uncured composite) can be compensated with material expansion. From Table 2, we can see the negative values of polymerization shrinkage in the case of illumination of composite samples with the ETE illumination mode in the first 10 seconds of illumination. These negative values can be marked as "expansion" of the composite material during the first 10 to 15 seconds during the pre-gelation phase of the polymerization process. "Expansion," in the beginning of composite material hardening, can be seen in Figures 3, 4 and 5 in all tested illumination modes, but from the Table 2, it can be seen only in the case of illumination with the ETE mode. This can be a consequence of prolonged low light intensity at the beginning of illumination with this mode (100 mW/cm^2) contrary to the ETS and ETM modes, where "expansion" is shorter than 10 seconds and therefore cannot be seen as negative values in Table 2. Therefore, the aim of "soft-start" polymerization (such as the ETE mode) is to prolong the pre-gelation phase, in which contraction can be compensated with expansion of the composite material, and to shorten the post-gelation phase, in which tension on the cavity walls occurs. Expansion of the composite material during the first 10 to 15 seconds occurs in the pre-gelation phase of the polymerization process. The so-called "soft-start" polymerization technique also enables excellent inner adaptation of composite material, preventing detachment of the material from the dentin

surface during polymerization (Watts & Cash, 1991; Thormann & Lutz, 1999; Koran & Kurschner, 1998).

A number of devices for determining volumetric curing contraction have been reported in the dental literature, most of which are based on measurements in a mercury dilatometer or a water dilatometer. Dilatometry is laborious and time-consuming and is also subject to data scattering when used for low-viscosity resins. In other studies, density change determinations have been carried out (Davidson & Feilzer, 1997), whereby, the total amount of volumetric shrinkage (pre- and post-gel) was also established.

Interferometry offers an alternative method to shrinkage measurements that promises improved accuracy and precision over dilatometry and strain-gauge/transducer methods. Quality data can be collected over a computer. The process of extracting the linear shrinkage data from the interferograms is simple in comparison to the dilatometric and strain-gauge/transducer methods, where considerable temperature corrections and gypsum calibration procedures are required to achieve high accuracy and precision.

The results of polymerization contraction in this study are in correlation with the results of other authors: Meiers, Kazemi and Meier (2001) reported the values of polymerization contraction from 0.8 to 1% lin; Goldman (1983), Watts and al Hindi (1999), Feilzer and others (1988) and de Gee and others (1993) reported values from 0.52 to 0.99% lin shrinkage. The values of polymerization contraction in this experiment measured, using digital laser interferometry, lie between 0.40 and 1.24% linear shrinkage.

Yap and Senevirante (2001) tested three illumination modes: "high," "low" and "soft-start." The authors confirmed that the "soft-start" polymerization mode exhibited less polymerization contraction than the "high" mode, but that there is no statistical difference between these two polymerization modes. These results are in correlation with the results of this study.

Differences in the results are a consequence of the various material shades and light sources used. Although polymerization shrinkage is the cause, shrinkage stress is responsible for clinical problems in adhesive restorations such as separation from cavity walls and cohesive failures in one of the structures.

CONCLUSIONS

The results of this experiment show that the lowest polymerization shrinkage was reached with the ETM polymerization mode, whereas the highest shrinkage was reached with the ETS mode. The ETE mode showed the highest expansion during the first 10 seconds of illumination, which is in correlation with its emitted light intensity shape in time. Therefore, curing units with low intensity at the beginning of illumination could be recommended for decreasing polymerization contraction in the pre-gelation phase of resin composite polymerization.

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Influence of NaOCl Treatment of Etched and Dried Dentin Surface on Bond Strength and Resin Infiltration

H Sato • M Miyazaki • BK Moore

Clinical Relevance

The data suggests that the bonding mechanism of the adhesive system used in this study can be influenced by NaOCl treatment on the etched dentin surface. Besides their effect and ability to remove organic substrates from adherent dentin, the penetration ability of adhesive resin should be considered.

SUMMARY

This study evaluated the effect of NaOCl treatment of etched air-dried dentin on the bond strength and state of monomer penetration. Ten percent NaOCl was applied after rinsing the etchant and air drying the dentin surface. Wet bonded, untreated teeth were used as a control. The resin composite was bonded and stored in 37°C water for 24 hours, then shear tested. One-way ANOVA, followed by the Duncan test, was done. For Raman microscopy, bonded specimens were cut parallel to the dentinal tubules and polished. Raman spectra were successively recorded along lines perpendicular to the dentin-adhesive interface. The decreased bond strengths found with air-dried dentin increased with NaOCl treatment, but the highest bond strength was obtained with wet bonding. From Raman spectroscopy, the widths of demineralized dentin decreased with prolonged NaOCl treatment time. The patterns of gradual transition of components differed among the groups.

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INTRODUCTION

The success of dentin bonding is believed to be dependent on the infiltration of resin monomers into an acid etched dentin followed by polymerization *in situ* (Ferrari & Davidson, 1996). Hydrophilic monomers may form a complex structure with exposed collagen fibers and partially demineralized dentin that contains residual hydroxyapatite. This has been termed the hybrid layer (Nakabayashi, Kojima & Masuhara, 1982). For systems that use phosphoric acid for dentin treatment, the so-called wet bonding technique is required to maintain an open collagen meshwork for subsequent penetration of resin monomers (Kanca, 1992). The collapse of unsupported collagen after phos-

Table 1: *Reported Bond Strengths to Etched Dentin After NaOCl Treatment*

Bonding System	Reference	Control	NaOCl Treatment	
Scotchbond MP	Vargas & others (1997)	17.3 (1.9)	5%, 2 minutes	16.8 (2.2)
	Inai & others (1998)	18.2 (2.8)	13%, 2 minutes	4.2 (1.5)
	Prati & others (1999)	16.1 (10.7)	1.5%, 2 minutes	9.6 (7.4)
Single Bond	Inai & others (1998)	30.7 (2.2)	13%, 2 minutes	5.5 (2.2)
	Prati & others (1999)	14.9 (3.9)	1.5%, 2 minutes	10.6 (5.3)
	Saboia & others (2000)	15.9 (4.3)	10%, 30 seconds	15.3 (4.7)
	Phrukkaron & others (2000)	15.9 (3.7)	12.5%, 30 seconds	19.3 (3.6)
			60 seconds	20.4 (5.6)
			120 seconds	15.3 (3.6)
	Perdigão & others (2000)	25.0 (4.1)	10%, 15 seconds	21.0 (5.7)
			30 seconds	18.5 (6.8)
			60 seconds	9.7 (2.4)
Prime&Bond 2.1	Inai & others (1998)	27.4 (3.4)	13%, 2 minutes	37.5 (7.9)
	Pioch & others (1999)	4.1 (2.2)	10%, 60 seconds	7.4 (4.6)
	Saboia & others (2000)	17.6 (6.3)	10%, 30 seconds	21.8 (6.5)

phoric acid treatment and exposure to air has been shown to inhibit resin monomer penetration to the entire depth of decalcified dentin.

To improve penetration of resin monomers into demineralized, air-dried dentin, sodium hypochlorite (NaOCl) has been applied to dissolve the collapsed collagen fibrils network that prevents monomer infiltration. It has been reported that the removal of collagen fibers increased bond strengths for some bonding systems and that a hybrid layer formation with exposed collagen might not play an important role in the establishment of these higher bond strengths. In contrast, decreased bond strengths after NaOCl treatment were reported for some bonding systems (Table 1) (Vargas, Cobb & Armstrong, 1997; Inai & others, 1998; Prati, Chersoni & Pashley, 1999; Pioch & others, 1999; Saboia, Rodrigues & Pimenta, 2000; Phrukkanon & others, 2000; Perdigão & others, 2000). The bonding area between the resin composite and cavity wall represents a gradual transition of different materials, which exhibits a gradient in the physico-mechanical properties (Miyazaki, Inage & Onose, 2002a). Such an elastic layer might assist in absorbing the stress that occurs during curing of the resin composite, preventing gap formation at the cavity wall (Van Meerbeek & others, 1993). In view of these contrasting reports, application of NaOCl on etched-dried dentin needs further studies to add more information regarding the dentin bonding mechanism.

This study evaluated the effect of NaOCl treatment on the shear bond strength of phosphoric acid etched, air-dried dentin and examined the degree of penetration of resin monomers using laser Raman microscopy.

The null hypothesis was that application of NaOCl to etched, air-dried dentin does not result in bond strengths or in resin monomer penetration comparable to that obtained with etched, dried dentin without NaOCl application.

METHODS AND MATERIALS

Specimen Fabrication

A one-bottle adhesive system, Single Bond (3M ESPE, St Paul, MN, USA) with a restorative Filtek Z250 (3M ESPE), was used in this study. The adhesive system was used according to the manufacturer's instruction. The light intensity of a curing unit (Optilux 500, Demetron/Kerr, Danbury, CT, USA) was adjusted to 600 mW/cm^2, as measured with a dental radiometer (Model 100, Demetron/Kerr).

Mandibular incisors extracted from two-to-three year old cattle were used as a substitute for human teeth (Schilke & others, 1999). After removing the roots with a low-speed saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA), the pulps were removed and final finish was accomplished by grinding on wet 600-grit silicon carbide paper. After ultrasonic cleaning with distilled water for one minute to remove the excess debris, these surfaces were washed and dried with a three-way syringe. Thirty-five percent phosphoric acid (Etchant, 3M ESPE) was applied for 15 seconds and rinsed with water, followed by air drying with oil-free compressed air with an air pressure of 2 kgf/cm^2 for 10 seconds from 10 cm above the dentin surface using a three-way syringe. Following the air drying, 10% NaOCl solution was applied for 0, 30 and 120 seconds. The wet bonded group, which was not air dried after etching, served as a control. A Teflon mold, 2.0 mm high and 4.0 mm in diameter, was used to form and hold the materials to the tooth surface. The bonding agent was applied and the resin composite condensed into the mold and cured for 20 seconds. Fifteen specimens were made for each group.

Dentin Bond Strength Measurement

Bonded specimens were stored in 37°C distilled water for 24 hours after placement. After storage, the specimens were tested in shear mode using a shear knife edge testing apparatus in a universal testing machine

(Type 4204, Instron Corp, Canton, MA, USA) at a crosshead speed of 1.0 mm/minute. After testing, the specimens were examined under an optical microscope at a magnification of 10x to determine the location of the bond failure.

Laser Raman Microscopy Measurement

Three additional specimens for each group were prepared and, after 24 hours storage in 37°C water, were embedded in self-curing epoxy resin (Epon 812, Nisshin EM, Tokyo, Japan). After curing at 37°C for 12 hours, the epoxy blocks were sectioned parallel to the dentinal tubules and the sectioned surfaces of the cut halves were polished to a high gloss with an Ecomet 4/Automet 2 (Buehler Ltd) using silicon carbide papers of 600, 1200 and 4000-grit size, successively. The surfaces were polished with a special soft cloth and diamond paste to a grit size of 1 µm (Buehler Ltd). Raman spectra were obtained with a computer controlled laser Raman microscope equipped with a monochrometer and a back-thinned MPP-type CCD camera (System 2000, Renishaw, UK). A He-Ne laser (GLG-5900, NEC, Tokyo, Japan) tuned to a wavelength of 632.8 nm with an output level of 75 mW was used as an excitation source. The sample was moved perpendicular to the resin-bond agent interface in 0.2-µm steps and spectra were obtained at each position across the dentin-adhesive interface. Raman spectra of unaltered dentin and cured adhesive resins were also recorded as references.

The Raman spectra in the region of 500~1800 cm^{-1} was obtained at each measuring point. The intense peak at 960 cm^{-1} associated with the P-O stretching vibration in the mineral apatite component of dentin and the intense peak at 640 cm^{-1} assigned to the aromatic ring group of Bis-GMA were selected as measures of dentin and resin. The peak at 1450 cm^{-1} assigned to the C-H alkyl group was selected as a measure of all the organic components, including resin and collagen, in the dentin-resin interface (Miyazaki, Onose & Moore, 2002b).

The acquired spectra in the region of interest were analyzed using a curve-fitting program and Raman microscope software, and the relative amounts of hydroxyapatite, adhesive resin and organic substance in the dentin-resin interface were calculated. The measurements were done three times with different specimens for each treatment group.

Statistical Analysis

The data for each specimen were subjected to one-way ANOVA followed by Duncan multiple range test at a *p*-value of 0.05. The statistical analysis was carried out with the Sigma Stat software system (Ver 2.01, SPSS Inc, Chicago, IL, USA).

RESULTS

Mean shear bond strength in MPa and failure mode are listed in Table 2.

When the etched dentin surface was dried without application of NaOCl (0 second NaOCl), a significant decrease in bond strength to 16.5% of the control value was observed. Though application of NaOCl tended to increase bond strengths compared to the 0 second NaOCl group, significant decreases in bond strength were still observed compared to the control. Failure mode correlates well with fracture strength. Wet bonding resulted in 100% cohesive failure; air drying with 0 or 30 seconds NaOCl treatment showed 100% adhesive failure. Only 120 seconds treatment restored some cohesive failures.

Intensities of the selected Raman bands scanned across the resin-dentin interface are shown in Figure 1.

Gradual changes in Raman intensity of the peaks of mineral and organic components were observed. Phosphoric acid etching with wet bonding technique

Table 2: *Shear Bond Strength of Single Bond to Bovine Dentin*

	Control Wet Bonded	0 Seconds NaOCl Air Dried	30 Seconds NaOCl Air Dried	120 Seconds NaOCl Air Dried
Bond strength	18.2(3.2)[a]	3.0(2.1)[b]	4.2(2.0)[b]	7.9(3.9)[c]
Mode of failure*	0/12/3	15/0/0	15/0/0	2/6/7

N=15, unit: MPa
Numbers within parentheses indicate standard deviations.
Means with the same superscript letters indicate no significant difference (p>0.05).
**: Adhesive failure/cohesive failure in dentin/cohesive failure in resin.*

Table 3: *Thickness of Three Different Phases in the Resin-dentin Interface*

	Control	0 Seconds NaOCl	30 Seconds NaOCl	120 Seconds NaOCl
Phase I	3.8 (0.2)[a]	2.4 (0.2)[c]	1.0 (0.1)[e]	0 (0)[g]
Phase II	2.1 (0.1)[b]	2.3 (0.1)[c]	2.1 (0.1)[f]	1.4 (0.3)[h]
Phase III	1.9 (0.1)[b]	1.9 (0.1)[d]	1.9 (0.1)[f]	0.5 (0.1)[i]

N=3, unit: µm.
Numbers within parentheses indicate standard deviations.
Means with the same superscript letters indicate no significant difference in the thickness of each phase (p>0.05).

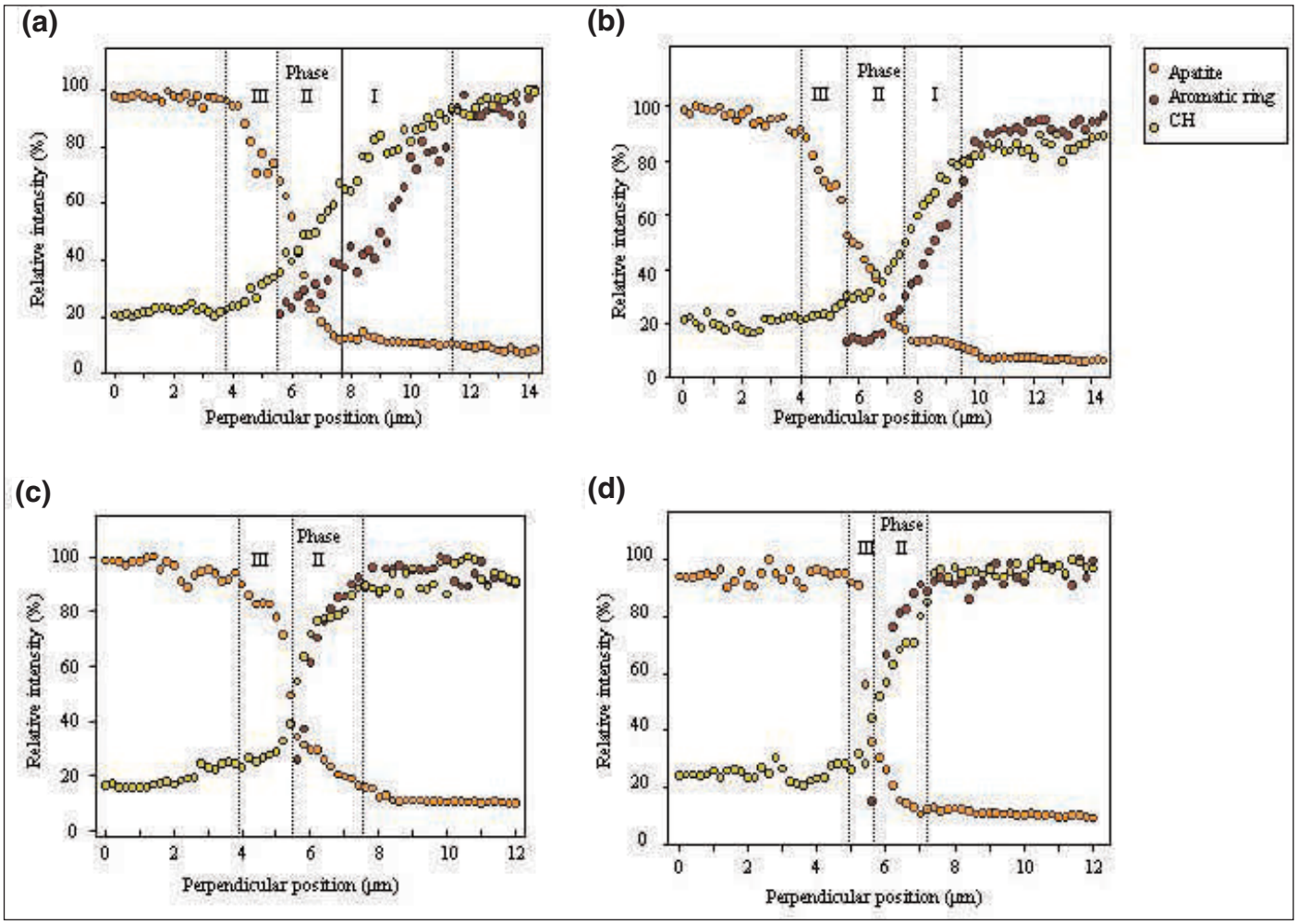

Figure 1. *Representative line scans from left to right across the dentin-resin interface of Single Bond adhesive system: a) wet-bonded according to the manufacturer's instruction, b) air-dried and treated with NaOCl for 0 seconds, c) for 30 seconds and d) for 120 seconds.*

produced a transition in dentin from complete removal of hydroxyapatite to partial demineralization with infiltration of resin monomers (Figure 1a). The resin-dentin interface (hybrid layer) of the control consisted of three different phases. The surface layer of dentin was totally demineralized (Phase I), leaving collagen fibers infiltrated with resin. Under the Phase I hybrid layer, gradually demineralized dentin exists, with penetration of the resin, including HEMA and Bis-GMA (Phase II). In the deepest portion of demineralized dentin, a layer without Bis-GMA penetration exists (Phase III).

Table 3 lists the average thickness of the three different phases inside the hybrid layer with respect to surface treatment.

The thickness of Phase I decreased with the prolonged application time of NaOCl gel. When NaOCl gel was applied for 120 seconds, etched-exposed collagen infiltrated with resin was no longer observed. Compared to the other treatment groups, the thickness of Phase II and III were significantly lower for the 120 second NaOCl group.

DISCUSSION

Though bond strengths were lower compared to those obtained with wet bonding, increased dentin bond strengths with increased NaOCl treatment duration were obtained in this study. It has been reported that the degree of resin infiltration of the exposed collagen fibrils within demineralized dentin has a profound influence on bond integrity (Hashimoto & others, 2000). Since air drying can cause the collapse of etched-exposed collagen fibrils that will prevent resin monomer penetration, the use of NaOCl for removing entangled collagen might increase bond strength.

The intrinsic strength of the bonding agent and the degree of porosity of the dentin substrate after NaOCl application are believed to be important factors influencing bond strength (Toledano & others, 2002). The organic component of dentin consists of collagen fibers that serve as a shock-absorbing base for the stiffer inorganic component. The collagen fibers of dentin provide elasticity and stress distribution properties, while hydroxyapatite adds stiffness and hardness to dentin.

When the collagen fiber is removed from the surface layer of etched dentin, the remaining materials are brittle. It is assumed that the resin infiltrated dentin layer has a lower elastic modulus than restorative resin, thus, acting as an elastic buffering layer that will minimize contraction stress during the curing of resin composites (Uno & Finger, 1995). The absence of such an elastic buffering layer might result in high stress concentration at the bonding interface of the deproteinized dentin and resin interface.

The role of collagen fibers in dentin bonding has not been proven, and some reports have revealed that collagen fiber offers no direct, quantitative contribution to interfacial bond strength (Gwinnett, 1994). It has been demonstrated that, in addition to collapse, part of the demineralized dentin collagen is in a denatured, unstable state, which is sensitive to hydrolysis and enzymatic degradation (Di Renzo & others, 2001). Also, a dense web of exposed collagen creates a low-surface energy surface that results in an increase in the contact angle of the adhesive resin. The diameter of dentin tubules increased after NaOCl treatment of acid etched dentin. Applications of NaOCl might remove the exposed collagen network, and this could cause the further removal of hydroxyapatite. Though NaOCl treatment produces a clean, porous surface that seems suitable for resin penetration, no evidence has yet been given that adhesive resin infiltrates the full depth of etched, deproteinized dentin.

A thick hybrid layer is no longer observed after longer application of NaOCl treatment (Pioch & others, 1999). The adhesive resin penetrates into the dentinal tubules and lateral branches of demineralized and deproteinized dentin. It has been reported that the treatment of collagen with NaOCl resulted in deeper penetration of adhesive resin in spite of decreasing bond strengths (Perdigão & others, 2000). These morphological studies suggest that removal of the exposed collagen allows for better resin monomer penetration into the dentin tubules and tubular anastmoses. More anastomoses could be observed in NaOCl treated specimens when compared to specimens where dentin was treated with phosphoric acid alone.

Laser Raman spectroscopy is used to analyze the structure of bonded samples and to determine their composition. Using this technique, the problems associated with the morphological analysis of the dentin-tooth interface with infrared spectroscopy can be avoided (Tsuda & Arends, 1997). Raman spectra can be acquired from moist specimens under normal conditions without ultra-thin sectioning. One of the disadvantages of Raman Microscopy is that the background from fluorescence can dominate the weaker Raman signal. This problem of fluorescence background noise was avoided by using a He-Ne laser as an excitation source. In addition, the light wavelengths from a He-Ne laser (632.8 nm) do not contribute to initiating further polymerization reaction of visible-light cured resins (Miyazaki & others, 2003a,b).

From laser Raman microscopy, there appeared to be three different phases within the bonding interface. Phase III corresponds to a porous band of partially demineralized dentin with poor resin infiltration. Harmony between the depth of demineralization and the extent of resin monomer penetration is the key to creating a high quality bonding interface according to proponents of the hybrid layer theory. Poor infiltration of adhesive resin into demineralized dentin (Phase III hybrid layer) leaves nano-spaces in the hybrid layer (Sano & others, 1995; Spencer & Swafford, 1999) and such a region may be susceptible to degradation from oral fluids (Sano & others, 1999). The same situation might occur in the resin-deproteinized dentin interface. After long-term service in the oral environment, the deficient impregnation of etched dentin with resin might allow water leaching into the bonding resin, resulting in resin swelling and plasticization (Söderholm, 1991). Water may accelerate hydrolysis of the bonding agent and extract poorly polymerized resin oligomers (Bastioli, Romano & Migliaresi, 1990). The question remains regarding the amount of resin monomer required to infiltrate etched dentin in order to prevent microleakage and reduced bond stability. It should be noted that the measurements of bond strengths at short times following bonding do not provide evidence of long-term stability of the resulting bonds. Information about the morphology of the bonded interface, as supplied by Raman techniques, may be more important in terms of predicting longevity of the dentin-resin bond.

CONCLUSIONS

Though phosphoric acid etching followed by NaOCl treatment could enhance bond strength compared to etched dried dentin, significant decreases in bond strength were observed compared to those obtained with the specimen made according to the manufacturer's instruction. The thickness of the demineralized and resin infiltrated layer were different among the different times of the NaOCl treatment time groups. The width of demineralized dentin decreased with prolonged NaOCl treatment time.

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Influence of Metal Conditioner Contamination of the Dentin Surface on Bond Strengths of Dentin Adhesive Systems Using Self-etching Primers

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Clinical Relevance

The data suggests that the dentin bond strengths of self-etching primer systems can be influenced by contamination of metal conditioners. Care should be taken when restoring secondary caries at a crown margin when self-etching primer systems, combined with metal conditioners, are used.

SUMMARY

Carious lesions around crown margins sometimes lead to failure of fixed prosthodontics. This study examined the influence of metal conditioner application on a dentin surface prior to bonding procedures, using two-step self-etching primer systems. Commercially available metal conditioners and self-etching primer dentin bonding systems were used. Bovine mandibular incisors were mounted in self-curing resin, and the facial dentin surfaces were ground wet on 600-grit SiC paper. The metal primers were applied on the dentin surface followed by bonding procedures with four different types of self-etching primer systems. The resin composites were condensed into a mold on the dentin surface and light activated. Ten specimens per test group were stored in 37°C water for 24 hours; they were then shear tested at a crosshead speed of 1.0 mm/minute. Two-way ANOVA and Tukey's HSD tests were done. When the metal conditioners were applied on dentin surfaces before bonding procedures, there was a tendency for decreased dentin bond strengths compared to those obtained with the controls. This tendency differed among the combinations of metal conditioners and self-etching primer systems used. Appropriate surface treatments are required to get optimum bond strengths with the use of technique sensitive bonding systems combined with metal conditioners.

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INTRODUCTION

The most common complication associated with conventional fixed prosthodontics is caries at the margins of restorations (Goodacre & others, 2003). Restoring

teeth with fixed prostheses represents a major activity in the dental profession, because of the high prevalence of caries and periodontitis experienced by the adult and elderly population. Compared with sound enamel surfaces, the margins of fixed prostheses are thought to be easily affected by acidic invasions from dental plaque. Untreated carious lesions, or the replacement of the fixed prostheses, will create further loss of dental hard tissue, leading to additional degenerative changes in the restored teeth (Zoellner & others, 2002; Glantz & others, 2002). There is no question that replacement of fixed restorations increases the risk of more complex and costly subsequent treatment, including root canal therapy (Ettinger, 1990). For minimal intervention dentistry, treatments, including non-invasive strategy and restoration of the affected area instead of replacement of the fixed prosthesis, are desired (Tyas & others, 2000; Mount & Ngo, 2000). Localized repair with resin composites might be a suitable procedure for increasing the longevity of the fixed restoration. Once infected dentin is removed, no further removal of the sound tooth is required if appropriate dentin bonding systems are used (Peters & McLean, 2001).

The two-step, self-etching primer system is one of the bonding systems developed to simplify and shorten bonding procedures by combining the dentin conditioning and priming steps (Van Meerbeek & others, 1998; Perdigão & Lopes, 1999). Self-etching primer contains acidic functional monomers that demineralize the tooth surface, while simultaneously allowing resin monomer penetration into the dentin substrate. A bonding agent is applied on the primed dentin surface to create a hybrid layer within the demineralized intertubular dentin (Watanabe, Nakabayashi & Pashley, 1994; Barkmeier, Los & Triolo, 1995; Gordan & others, 1997; Tay & Pashley, 2001). When using these bonding systems at a metal crown margin, additional pre-treatment of the casting alloy is required to enhance bond strength to resin composites and minimize leakage. Several metal conditioners for the pre-treatment of noble metal alloys have been introduced; all are single liquids containing polymerizable sulfur compounds in a solvent (Atsuta, Matsumura & Tanaka, 1992; Taira & Imai, 1995; Matsumura & others, 1999; Matsumura & others, 2001).

Since a crown margin consists of tooth substrate and casting alloy, different surface treatments for both adherend surfaces are required. Considering the clinical situation, a question arises whether contamination of the tooth substrate by metal conditioner prior to bonding procedures influences the dentin bond strengths of self-etching primer systems. For bonding systems that use a separate dentin conditioner that was rinsed off prior to application of the bonding agents, metal primers did not disturb the demineralizing potential of the dentin conditioners (Kajihara & others, 2003). Since, in contrast, a self-etching primer is not rinsed off, remnants of metal conditioners might adversely effect dentin bond strengths to resin composites.

This study examined the influence of metal conditioner application on the dentin surface prior to the bonding procedures of two-step self-etching primer systems on bond strengths to bovine dentin. The null hypothesis tested was that metal conditioner application prior to bonding procedures does not interfere with the bonding ability of self-etching primer systems.

METHODS AND MATERIALS

Materials

The self-etching primer systems Clearfil SE Bond (Kuraray Medical, Tokyo, Japan), Imperva Fluoro Bond (Shofu Inc, Kyoto, Japan), Mac Bond II (Tokuyama Dental, Tokyo, Japan) and Unifil Bond (GC Corp, Tokyo, Japan) were used as shown in Table 1. A visible light curing unit, Optilux 501 (Demetron/Kerr, Danbury, CT, USA), with a light intensity above 600 mW/cm^2 as measured with a dental radiometer (Model 100, Demetron/ Kerr), was used.

The four commercially available metal conditioners chosen were Alloy Primer (Kuraray Medical), Metal Primer II (GC Corp), Metaltite (Tokuyama Dental) and MI Link (Shofu Inc) and are listed in Table 2. The main components of the primers are 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine- 2,4-dithiol (VTD) and methacryloyloxydecyl dihydrogen phosphate (MDP) for the Alloy Primer, methacryloyloxyalkyl thiophosphate derivative (MEPS) for the Metal Primer II and 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate (MTU-6) for the Metaltite and 10-methacryroxydecyl-6,8- dithioctanate (10-MDDT), 6-methacryloxyhexyl phosphonoacetate (6-MHPA) for the MI Link.

Dentin Bond Strength Test

Two-hundred mandibular incisors from two-to-three year old cattle stored frozen (20°C) up to two weeks after extraction were used as a substitute for human teeth. After removing the roots with a low-speed diamond saw (Buehler Ltd, Lake Bluff, IL, USA), the pulp was removed and the pulp chamber of each tooth was filled with cotton to avoid penetration of the embedding media. The labial surfaces of the bovine incisors were ground on wet 240-grit silicon carbide (SiC) paper to a flat dentin surface. Each tooth was then mounted in cold-curing acrylic resin to expose the flattened area and was immersed in tap water to reduce the temperature rise from the exothermic polymerization of the acrylic. Final finish was accomplished by grinding on wet 600-grit SiC paper. After ultrasonic cleaning in distilled water for one minute to remove debris, the surfaces were washed and dried with oil-free compressed air.

Table 1: *Materials Tested in This Study*

Bonding System (Code)	Self-etching Primer (Lot #) Main Component	Bonding Agent (Lot #) Main Component	Resin Composite (Lot #)	Manufacturer
Clearfil SE Bond	Primer (00260A) MDP, HEMA, water, PI, ethanol	Bond (00265A) MDP, HEMA, PI, bis-GMA, micro filler	Clearfil AP-X (00652A)	Kuraray Medical (Tokyo, Japan)
Imperva Fluoro Bond	FB Primer (A: 060060, B: 060076) A: Catalyst, water B: 4-AET, 4-AETA, HEMA	FB Bond (109963) 4-AET, HEMA, filler, UDMA, TEGDMA, PI	Lite-Fil IIA (050113)	Shofu Inc (Kyoto, Japan)
Mac-Bond II Dental	Primer (A: 021, B: 011) A: MAC-10, HEMA, acetone, isopropyl alcohol, phosphate monomer B: Ethanol, water	Bonding Agent (0248) MAC-10, HEMA , PI, bis-GMA, TEGDMA	Palfique Estelite (219)	Tokuyama (Tokyo, Japan)
UniFil Bond	Self-etching primer (0302251) 4-MET, HEMA, water, ethanol	Bonding Agent (0302281) TEGDMA, HEMA, UDMA, PI	UniFil S (0211191)	GC Corp (Tokyo, Japan)

MDP: 10-methacryloxydecyl di-hydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, PI: photo initiator, bis-GMA: 2, 2bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl] propane, 4-AET: 4-acryloyloxyethyl trimellitate, 4-AETA: 4-acryloyloxyethyl trimellitate anhydride, UDMA: 1, 6-bis-methacryloxy-2-ethoxy carbonylamino)-2, 4, 4-trimethylhexane, TEGDMA: triethylene glycol di-methacrylate, MAC-10: 11-methacryloxy-1,1-undecandicarboxylic acid, 4-MET: 4-methacryloyloxyethyl trimellitate

Table 2: *Metal Conditioners Used*

Primer	Manufacturer	Lot #	Main Component
Alloy Primer	Kuraray Medical (Tokyo, Japan)	0138AA	6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol (VTD), 10-methacryloyl-oxydecyl dihydrogen phosphate (MDP), acetone
Metal Primer II	GC Corp (Tokyo, Japan)	013072	Methacryloyloxyalkyl thiophosphate derivatives (MEPS), methyl methacrylate (MMA)
Metaltite	Tokuyama Dental (Tokyo, Japan)	00403	6-methacryloyloxyhexyl-2-thiouracil-5-carboxylate (MTU-6), ethanol
MI Link	Shofu Inc, (Kyoto, Japan)	MTL001	10-methacryroxydecyl-6,8- dithioctanate (10-MDDT), 6-methacryloxyhexyl phosphonoacetate (6-MHPA), acetone

The bonding area was restricted to a 4-mm diameter by using adhesive tape. Metal conditioners were applied on the dentin surface followed by a gentle blow of air. Dentin surfaces without the application of metal conditioners were used as a control. Four different self-etching primers were applied to each of the different metal conditioners. Bonding agents of the same manufacturer as the self-etching primers were applied and irradiated with the curing unit. A Teflon mold 2.0-mm high and 4.0 mm in diameter was used to form and hold the resin restorative materials to the tooth surface. Resin composites were condensed into the mold and light activated for 40 seconds. Ten specimens were prepared for each of the 20 groups. The Teflon mold and adhesive tape were removed from the specimens 10 minutes after light irradiation. Bonded specimens from each group of materials were stored in 37°C water for 24 hours after placement.

After storage in distilled water, the specimens in each group were tested in shear mode using shear knife-edge testing apparatus in a universal testing machine (Type 4204, Instron Corp, Canton, MA, USA) at a crosshead speed of 1.0 mm/minute. Shear bond strengths in MPa were calculated from the peak load at failure divided by the specimen surface area.

After testing, the specimens were examined under an optical microscope at a magnification of 10x to determine the location of the bond failure. The test area on the tooth was divided into eight segments, and the percentage that was free of adhesive or restorative material was estimated. The types of failures were determined based on the predominant percentage of substrate free material as adhesive failure, cohesive failure in resin composite, cohesive failure in dentin and mixed failure (50% cohesive and 50% adhesive) for each test specimen.

Statistical Analysis

The data for each group of specimens were subjected to two-way ANOVA, with independent factors being the type of metal conditioner and bonding system, followed by Tukey's HSD tests at a p-value of 0.05. The statistical analysis was carried out with Sigma Stat (Ver 2.03, SPSS Inc, Chicago, IL, USA).

SEM Observation

The treated dentin surface and restorative resin/dentin interface were observed by scanning electron

microscopy (SEM). For the treated dentin surface observation, the dentin surfaces were treated with and without metal conditioners, followed by self-etching primer application; they were then rinsed with acetone and water. For ultrastructure observation of the restorative resin/dentin interface, bonded specimens stored in 37°C distilled water for 24 hours were embedded in epoxy resin, then longitudinally sectioned with a diamond saw. The sectioned surfaces were polished with abrasive discs and diamond pastes down to a 0.1 μm-particle size. All the SEM specimens were fixed in 2.5% glutaraldehyde in cacodylate buffer solution, dehydrated in ascending concentrations of *tert*-butanol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes and 100% for two hours), then transferred to a critical-point dryer for 30 minutes. The polished surfaces were subjected to argon-ion beam etching for 30 seconds (Elionix Ltd, Tokyo, Japan) with an accelerating voltage of 1.0 kV and ion current density of 0.4 mA/cm^2 directed perpendicular to the polished surface (Inokoshi & others, 1993). The surfaces were coated in a vacuum evaporator with a thin film of Au. The specimens were observed in a scanning electron microscope (JSM-5400, JEOL Ltd, Tokyo, Japan). Observations were done with at least three specimens for each test group.

RESULTS

The results of the shear bond strength tests with various combinations of metal conditioners and self-etching primers are shown in Table 3.

After 24 hours storage in water, the dentin bond strengths of the control group (without metal conditioner application) were 21.5 MPa for Clearfil SE Bond, 18.2 MPa for Imperva Fluoro Bond, 18.5 MPa for Mac Bond II and 19.1 MPa for UniFil Bond. Two-way analysis of variance demonstrated that there was not a statistically significant interaction between the factors' bonding systems and metal conditioners (p=0.216). Since the difference in mean values among the different metal conditioners was greater than would be expected by chance after allowing for the effects of differences in the bonding systems (p<0.001), Tukey's HSD tests were performed within the bonding systems. When metal conditioners were applied to the dentin surfaces prior to self-etching, the dentin bond strengths of the self-etching primer systems decreased, ranging from 15.9 to 19.8 MPa for Clearfil SE Bond, 13.4 to 17.5 MPa for Imperva Fluoro Bond, 9.2 to 15.9 % for Mac Bond II and 14.8 to 18.8 MPa for Unifil Bond. When Metal Primer II was applied on dentin surfaces, a significant decrease in dentin bond strength was observed for all bonding systems tested.

The predominant failure mode was cohesive failure in dentin for the controls, while the adhesive failure tended to increase for the metal conditioner application groups.

The representative SEM observations of the treated dentin surfaces are shown in Figure 1. The smear layer was removed and the collagen fibrils were observed to some extent for the control groups. Less demineralization of the dentin surfaces was more pronounced for the dentin surfaces treated with metal conditioners prior to the self-etching step.

Representative SEM micrographs of the dentin/resin interface after argon-ion beam etching are shown in Figure 2. A thin layer with low resistance to argon-ion etching was identified as the hybrid layer, and the width of this layer was about 0.5-1.0 μm for the controls (specimens were made according to the manufacturers' instructions). For specimens treated with metal conditioner, a hybrid layer was observed but the width of this layer was less than that observed with the control specimens.

DISCUSSION

It has been reported that adhesion to the superficial layer of dentin showed no significant differences between human and bovine dentin, and dentin bond

Table 3: *Influence of Metal Conditioner Application on Dentin Bond Strengths and Fracture Modes of Self-etching Primer Systems*

	Metal Conditioner				
Bonding System	**Control**	**MI Link**	**Alloy Primer**	**Metaltite**	**Metal Primer II**
Clearfil SE Bond	21.5 (2.1)	19.8 (3.3)	19.6 (2.1)	19.5 (2.0)	15.9 (2.6)
	6/2/0/2	4/2/1/3	3/2/2/3	2/4/1/3	2/2/1/5
Imperva Fluoro Bond	18.2 (2.1)	17.5 (3.1)	16.1 (3.4)	14.9 (3.3)	13.4 (3.5)
	4/2/2/2	3/3/2/2	2/1/2/5	2/2/2/4	2/1/2/5
Mac Bond II	18.5 (1.7)	15.1 (3.4)	15.9 (2.1)	13.4 (2.3)	9.2 (3.9)
	4/1/3/2	2/0/2/6	2/3/2/3	0/1/2/7	0/0/2/8
UniFil Bond	19.1 (2.2)	18.8 (3.0)	16.8 (2.1)	16.5 (2.3)	14.8 (3.5)
	4/1/2/3	2/1/1/6	2/3/1/4	2/1/2/5	1/1/2/6

Unit: MPa, ( ): SD, n=10
Values connected by horizontal lines indicate no significant difference.
Failure mode: Cohesive failure in dentin/Cohesive failure in resin/Mixed failure/Adhesive failure.

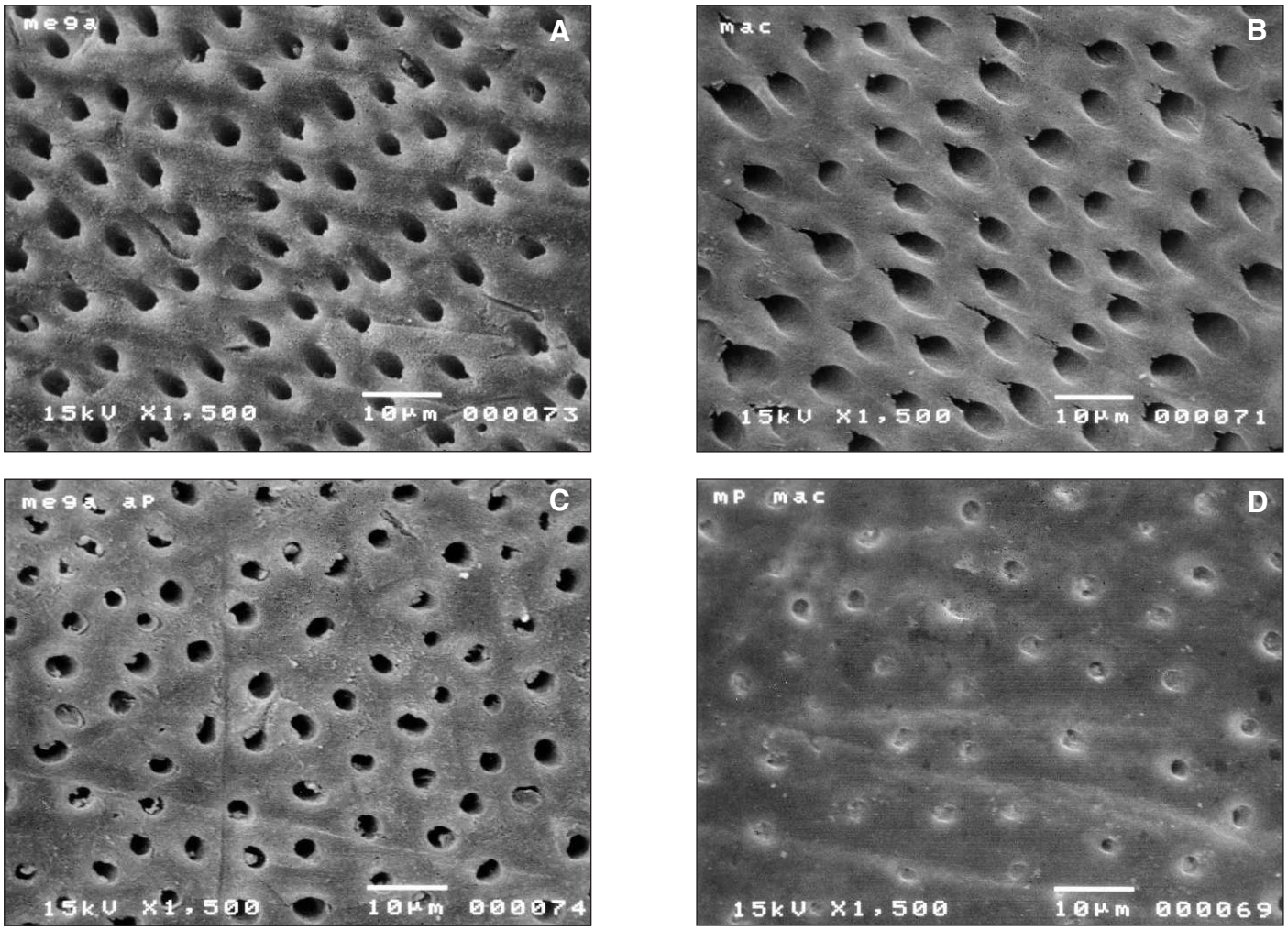

Figure 1. *SEM observations of dentin surface treated with the self-etching primers (A: Clearfil Mega Bond, B: Mac Bond II) followed by acetone and water rinsing. For both systems, the demineralization of the dentin surface and opening of the dentinal tubules were observed (Original magnification; 1,500x). When the metal conditioners were applied prior to the self-etching steps, etching effect was weakened and smear plugs were observed (C: Alloy Primer prior to Clearfil Mega Bond, D: Metal Primer II prior to Mac Bond II).*

strength decreased with the depth of dentin, because of the lower density of dentinal tubules (Schilke & others, 1999). Because the differences in tubule diameter and the number of lateral branches may have some effect on dentin bond strength (Ferrari & Davidson, 1996), bovine superficial dentin was used as a substitute for human dentin in this study, as reported in previous studies (Nakamichi, Iwaku & Fusayama, 1983; Fowler & others, 1992). Care should be taken when drawing conclusions from the bond strength data, since there are many factors that affect bond values (Miyazaki, Oshida & Xirouchaki, 1996).

Four different types of two-step bonding systems with self-etching primers were used in this study. A self-etching primer is an aqueous mixture of acidic functional monomers and other constituents. The low pH of the primers allows mineralized tissue to be conditioned and etched in a single treatment step, and all these primers are classified as mild self-etch systems based on relatively high (but still acidic) pH values (Van Meerbeek & others, 2003). The acidic polymerizable monomers used in the self-etching primers are 10-methacryloxydecyl dihydrogen phosphate (MDP) for Clearfil SE Bond, 4-acryloxyethyl trimellitate (4-AET) for Imperva Fluoro Bond, 11-methacryloxy-1,1-undecanedicarboxylic acid (MAC-10) for Mac Bond II and 4-methacryloxyethyl trimellitate (4-MET) for UniFil Bond.

The metal conditioners contain sulfur derivative monomers for targeting the direct chemical bond between noble metal alloys and resins. Metal Primer II contains MEPS, which is an athiophosphate compound that reacts with noble metal alloys (Matsumura & others, 1999). A functional monomer of VTD is utilized with MDP monomer in Alloy Primer. The mercapto groups in VTD react with noble metal alloys at the metal/resin cement interface (Kojima, Kadoma & Imai,

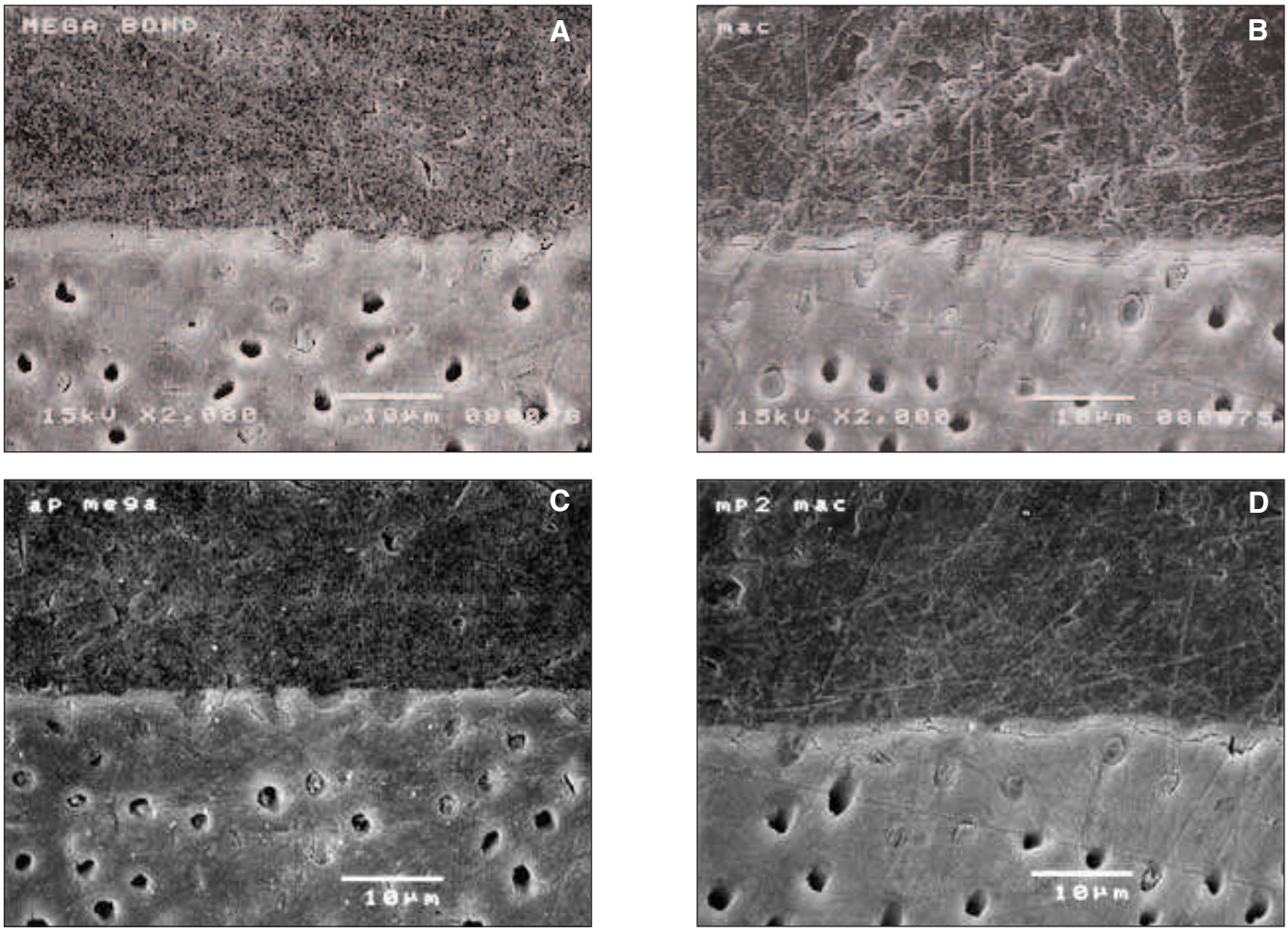

Figure 2. *SEM observations of resin-dentin interface after argon-ion etching (A: Clearfil Mega Bond, B: Mac Bond II). Perfect adaptation to the dentin with a thin hybrid layer (resin impregnated dentin) was observed (Original magnification; 2,000x). When the metal conditioners were applied on the dentin surface prior to the self-etching steps, the thickness of this layer was relatively thinner (C: Alloy Primer prior to Clearfil Mega Bond, D: Metal Primer II prior to Mac Bond II).*

1987). Metaltite contains MTU-6 monomer, which has a thiol structure similar to the thione structure in VDT monomer (Matsumura & others, 1999). The MI Link contains thioctanate monomer (10-MDDT) that has a disulfide structure for bonding to noble metal alloys and phosphonoacetate monomer (6-MHPA) for bonding to base metal alloys. An NMR study showed that VDT has a thione-type structure that is considered to be chemically stable in a solid state and in acetone solution (Mizuno & others, 1998). When VDT molecules come close to the Au surface, tautomerization occurs and chemically active mercapto groups interact with noble metal elements. This process forms a chemical bond between sulfur and Au atoms (Suzuki & others, 1999). For other monomers, additional chemical analyses are needed to determine their bonding mechanism.

It is generally accepted that the smear layer that forms on ground dentin should be removed or altered with an acidic conditioner in order to achieve good adhesion between the demineralized dentin substrate and an applied bonding system (Nakabayashi & Saimi, 1996). SEM observations of the primed dentin surface indicate that self-etching primers have the ability to dissolve the smear layer and etch the dentin surface. When metal conditioners were applied to the dentin surface prior to the self-etching step, the etching effect was somewhat weakened (Figure 1). It has been reported that dentin surface contamination with a metal conditioner did not affect dentin bond strength when a separate dentin conditioner was used (Kajihara & others, 2003). Rinsing off the metal conditioner with a solution of 10% citric acid with 3% ferric chloride, and the high diffusing potential of 4-methacryloyloxyethyl trimellitate anhydride (4-META) may have contributed to these results. In this study, a tendency towards a dentin bond strength decrease was observed when metal conditioners were applied on the dentin surface

prior to the self-etching steps. The adverse effect on dentin bond strength might relate to the non-rinsing procedures employed in the self-etching primer systems. From the SEM observations on primed dentin surfaces, the remaining smear layer with plugged dentinal tubules was observed for some combinations of metal conditioner and self-etching primer. The pH values and molecular conformation might be altered by the presence of metal conditioners, leading to differences in bond strength values and treated dentin surface appearance.

The primed dentin surface should be wetted by hydrophobic resin monomers after removal of the smear layer. Improved wetting of the dentin surface may encourage penetration of resin monomers into the dentin substrate in order to enhance adaptation. These resin monomers should infiltrate the demineralized dentin, including the exposed collagen fibril network, and establish a hybrid layer (Nakabayashi, Kojima & Masuhara, 1982). The hybrid layer thickness of the self-etching primer systems was very thin (Figure 2). The thickness of the hybrid layer depends on the bonding systems used, and variations in thickness were observed among specimens using the same bonding systems and different dentin substrates (Vargas, Cobb & Denehy, 1997; Prati & others, 1998). Though the hybrid layer and the presence of resin tags may not be the only mechanisms influencing dentin strengths, a relatively thinner hybrid layer was observed when metal conditioners were applied on the dentin surface prior to self-etching. This might relate to insufficient monomer infiltration into the etched/primed dentin.

Harmony between the degree of demineralization and the self-etching primer and the integrity of resin monomer infiltration is an important key to creating high quality dentin bonding (Miyazaki & others, 2003). It has been reported that poor infiltration of adhesive resin into demineralized dentin leaves nano-spaces in the hybrid layer (Sano & others, 1995; Spencer & Swafford, 1999). The existence of porosity at the base of the hybrid layer and within the adhesive resin one year after placement has been shown in an *in vivo* study (Sano & others, 1999). After infiltration of the resin monomers into partially demineralized dentin, subsequent polymerization of the monomers is required to create a stable bond. If polymerization of these monomers is not complete, hydrophilic monomers or small oligomers might be extracted or hydrolyzed by nanoleakage (Sano & others, 1999). Proper demineralization of dentin substrate, uniform resin impregnation and sufficient mechanical strength of cured adhesive resin are important factors in creating a high quality hybrid layer for good dentin bonding (Miyazaki & others, 2003).

After infiltration into the etched/primed dentin, the bonding resins must sufficiently polymerize to create a durable bond. If unreacted resin monomer remains, a plasticizing effect on the polymer leads to degraded mechanical properties of the bonding agent. The bonding agents used in this study were cured by a free radical polymerization reaction and the photosensitizive initiator camphorquinone (CQ) (Taira & others, 1988; Jakubiak & Rabek, 1999). CQ requires a co-initiator for an effective polymerization process to occur and a tertiary amine photoreductant is employed. The tertiary amine interacts with an activated triplet state of CQ to form an intermediate excited complex followed by the production of reactive radicals for polymerization (Cook, 1992). An explanation for decreasing dentin bond strengths could be related to the functional monomers in metal conditioners. Thiols have been employed as efficient chain transfer agents, because of weakness of the S-H bond and the higher reactivity of the thiol radicals (Okaya, Kikuchi & Morii, 1997; Henríquez & others, 2003). Additional polymerization of resin monomers in the bonding agent might be terminated by the presence of thiol radicals, leading to decreased polymer chain length. Hypothetically, stronger resins lead to stronger bonding to dentin (Pashley & others, 1995). The strength of a cured bonding agent is dependent on the composition, degree of conversion and length of polymer chain. Unreacted resin monomer remaining in adhesive resins may alter their mechanical properties, leading to inferior bond strengths.

CONCLUSIONS

From the results of this study, significant decreases in bond strengths were found for some combinations of metal conditioners and bonding systems. Thus, the null hypothesis that bond strengths of self-etching primer systems to metal conditioner-contaminated dentin do not differ from the controls can be rejected. The dentin bond strengths of some two-step self-etching primer systems were influenced by contamination with the metal conditioners used, and the etching effect of the self-etching primers was affected by the presence of metal conditioners.

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In Vitro Evaluation of the Cariostatic Action of Esthetic Restorative Materials in Bovine Teeth Under Severe Cariogenic Challenge

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Clinical Relevance

The use of fluoride-releasing restorative materials is important in inhibiting the occurrence of secondary carious lesions, especially in patients who are at high risk and/or high caries activity. Considering the commercial availability of these restorative materials, a comparative evaluation of their cariostatic action is required.

SUMMARY

Considering that caries around restorations is a serious problem in dentistry, and some restorative materials with fluoride may be important in inhibiting these lesions, this research is aimed at performing an *in vitro* evaluation of the cariostatic action of some esthetic restorative materials. Standardized cavities were prepared in the center of either intact blocks of bovine enamel or with bovine teeth containing early artificial carious lesions. The specimens were restored with a high viscosity glass ionomer cement (Molar Ketac), a resin-modified glass ionomer cement (Vitremer), a polyacid-modified resin composite (Dyract AP) and a conventional resin composite (Z-250). In addition to the restored specimens, four corresponding control groups were evaluated. All groups, except for two control groups, were subjected to a demineralization/remineralization cycling model for 14 days, simulating a situation of severe cariogenic challenge. The blocks were then longitudinally sectioned through the restorations. Mineral loss was evaluated in these specimens using the Knoop microhardness profiles in longitudinal sections at three different distances of the cavities and at eight distinct depths in relation to the external enamel surface. Statistical analysis of the results showed significant differences ($p<0.05$) among the groups, although none of the study materials completely inhibited creation of the lesions.

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Vitremer demonstrated the best cariostatic action in intact bovine enamel. Ketac Molar, in intact or demineralized enamel, and Vitremer, in demineralized enamel, presented intermediate cariostatic potential. Z-250 and Dyract AP did not demonstrate any cariostatic effect. The data suggests that glass ionomer cements demonstrated better cariostatic action compared to the other restorative materials.

INTRODUCTION

Recently, dentistry has demonstrated significant advances with regard to developing materials and restorative techniques, as well as in the field of dental caries prevention. However, further advances in restorative procedures are needed, especially concerning the replacement or repair of existing restorations as a result of secondary carious lesions (Fontana & others, 2002; Van Dijken, 1986; Mjör, 1985).

Many factors work together to determine the longevity of a restoration, such as a patient's oral hygiene, the technical skill of the professional, the inherent characteristics of each patient and the physicochemical properties of the restorative materials.

Based on the current concept that the cariostatic effect of fluoride is enhanced in lower, yet permanent, concentrations in the oral environment, incorporating fluoride into restorative materials is of special interest. These fluoridated materials are potential sources of release of this element, therefore, expanding the spectrum of prevention in restorative dentistry. The use of fluoride to demineralize and remineralize early enamel carious lesions directly interferes with caries evolution (Fontana & others, 2002; Featherstone & others, 1986). It is accepted that part of the fluoride present in restorative materials may be directly released onto the areas of risk, such as restoration margins, where secondary caries may develop (Creanor & others, 1994; Serra & Cury, 1992). Thus, the use of fluoride may be considered an additional method of preventing caries.

Glass ionomer cement, initially described in the early 1970s by Wilson and Kent (1972), is regarded as the material of choice in cases, where cavity sealing and prevention of secondary caries are desirable, due to its properties of adhesion to dental structure and its high rate of fluoride release (Creanor & others, 1994; Donly, 1994).

However, the predominance of resin composites among esthetic restorative materials is evident, especially due to its highly satisfactory esthetics and easy manipulation. The greatest difficulty with this material is the occurrence of secondary carious lesions adjacent to the restoration, which is observed less frequently in teeth restored with silicate cement or glass ionomer cement (Mjör, 1985).

In an attempt to obtain an ideal material that would combine the good properties of both resin composites and glass ionomer cements, new materials have been developed, such as resin-modified glass ionomers and polyacid-modified resin composites.

Therefore, considering that employing fluoride-releasing restorative materials is important for inhibiting the occurrence of secondary carious lesions, especially in patients at high risk (Serra & Cury, 1992) and/or those with high caries activity, knowledge of the behavior of such restorative materials in situations of high cariogenic challenge is fundamental. Considering the commercial availability of these different restorative materials, which contain fluoride, and the unquestionable predominance of conventional resin composites among practitioners, a comparative evaluation of the cariostatic action of these materials is required.

The main aspect evaluated in this study was the cariostatic action of four esthetic restorative materials with regards to their ability to reduce demineralization of bovine enamel (either intact or with an artificial early carious lesion) adjacent to restorations, taking into account the depth in relation to the external enamel surface and the distance from the cavity walls or restorations margins. Thus, the null hypothesis established for this study was that there would be no difference in cariostatic action of the four materials both in intact and demineralized enamel within the conditions of this study.

METHODS AND MATERIALS

Experimental Design

Three hundred and sixty tooth blocks (4 x 4 x 2 mm) were obtained from the central portion of the crown of bovine incisors. These blocks were embedded in resin disks and the fragments were polished in an attempt to create a smooth enamel surface. The blocks were standardized according to their initial microhardness and 144 blocks were selected.

Artificial early enamel carious lesions were created on half the samples through demineralization in acidic solution, pH 5.0 (White, 1987) for 16 hours. Thereafter, microhardness was measured again to confirm the percentage of demineralization (microhardness value after demineralization lower than 50% of the mean value of initial microhardness, with an expected depth of 50 µm by this protocol) and standardization of the blocks. The remaining samples were kept intact, and all specimens (144 blocks) were randomly assigned to the following groups:

- **Control I (WO/D)**–intact enamel with no cavity preparation, not submitted to the cariogenic challenge.

- **Control I (W/D)**–intact enamel with no cavity preparation, submitted to the cariogenic challenge.
- **Z-250 I**–intact enamel restored with the resin composite Filtek Z-250 (3M Dental Products, St Paul, MN, USA).
- **Dyract AP I**–intact enamel restored with the polyacid-modified resin composite Dyract AP (Dentsply, Industria e Comercio Ltda, Petropolis, RJ, Brazil).
- **Vitremer I**–intact enamel restored with the resin-modified glass ionomer cement Vitremer (3M Dental Products, St Paul, MN, USA).
- **Ketac Molar I**–intact enamel restored with the high viscosity glass ionomer cement Ketac Molar (ESPE Dental AG, Seefeld, Germany).
- **Control De (WO/D)**–demineralized enamel not submitted to cariogenic challenge.
- **Control De (W/D)**–demineralized enamel with no cavity preparation submitted to the cariogenic challenge.
- **Z-250 De**–demineralized enamel restored with the resin composite Filtek Z-250.
- **Dyract AP De**–demineralized enamel restored with the polyacid-modified resin composite Dyract AP.
- **Vitremer De**–demineralized enamel restored with the resin-modified glass ionomer cement Vitremer.
- **Ketac Molar De**–demineralized enamel restored with the high viscosity glass ionomer cement Ketac Molar.

Cylindrical cavities were prepared in the center of the block's enamel and restored with the previously mentioned materials, according to the manufacturers' instructions. The restored specimens were stored in plastic receptacles containing gauze pads moistened with deionized water at 37°C ±1°C for 48 hours prior to finishing and polishing.

pH Cycling

In order to simulate severe cariogenic challenge, all groups, except for two of the control groups, were submitted to demineralization/remineralization cycling for 14 days according to the methodology suggested by Featherstone and others (1986).

The specimens in each group were placed in closed plastic receptacles containing 480 ml (40 ml per block–Featherstone & others, 1986) of demineralizing solution (2.0 mM calcium, 2.0 mM phosphate, 75 mM acetate, and 0.02% azide as a preservative, at pH 4.3) for six hours in an oven at 37°C ±1°C. The specimens were then washed with deionized water and dried with absorbent paper and placed in 240 ml (20 ml per block–Featherstone & others, 1986) of remineralizing solution (1.5 mM calcium, 0.9 mM phosphate, 150 mM potassium chloride, 20 mM tris buffer and 0.02% azide as a preservative, at pH 7.0) for 18 hours at 37°C ±1°C. A total of 10 cycles were performed in 14 days (five consecutive cycles and two days for demineralization, then five cycles and two days for remineralization).

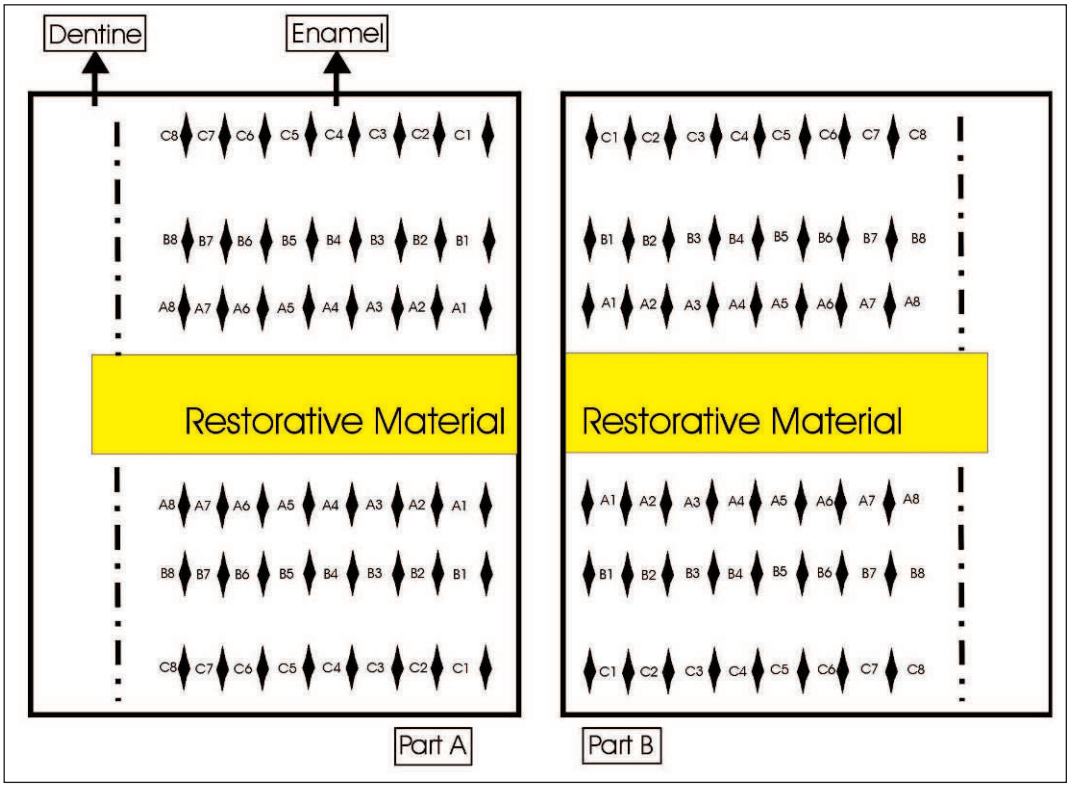

Figure 1. *Points on which the imprints for evaluation of longitudinal microhardness were made.*
- *Distance from the cavity wall (restoration margin)*
  A = 50 µm B = 150 µm C = 300 µm
- *Depth in relation to the external enamel surface*
  1 = 30 µm 2 = 60 µm 3 = 90 µm 4 = 120 µm
  5 = 150 µm 6 = 180 µm 7 = 210 µm 8 = 240 µm

Microhardness Profiles

After pH cycling, the blocks were longitudinally sectioned, embedded in resin and polished. The mineral loss was evaluated by means of Knoop microhardness tests in longitudinal sections considering two aspects: the depth in relation to the external enamel surface and distance from the cavity walls/restoration margins. The microhardness tester Shimadzu Micro Hardness Tester HMV–2000 (Shimadzu Corporation, Kyoto, Japan) was used with a Knoop indenter with a static load of 25g applied for five seconds connected to CAMS-Win software for image analysis (NewAge Industries Inc, Willow Grove, PA, USA).

Imprints were performed on both halves of each block (A and B halves) as demonstrated in Figure 1. For specimens with no restoration, the first sequence of eight imprints was performed on the center of each section. The second set of eight imprints was taken at a distance of 100 µm from the first. The last set of eight imprints was taken at a distance of 150 µm from the second set of imprints.

Statistical Analysis

Statistical analysis evaluated Knoop microhardness as the dependent variable in intact and demineralized bovine enamel with the following variation factors: restorative treatment, depth of demineralization in relation to the external enamel surface and distance from the cavity preparation. Analysis of variance (ANOVA) was employed to verify the interaction between the sources of variation. When significant differences were observed among these factors, the Tukey test for multiple comparisons was applied. The minimum significance level adopted for all tests was conventionally established at 5%. Statistical analyses were performed using the "Statistic for Windows" software, version 5.1 (StatSoft, Inc, Tulsa, OK, USA).

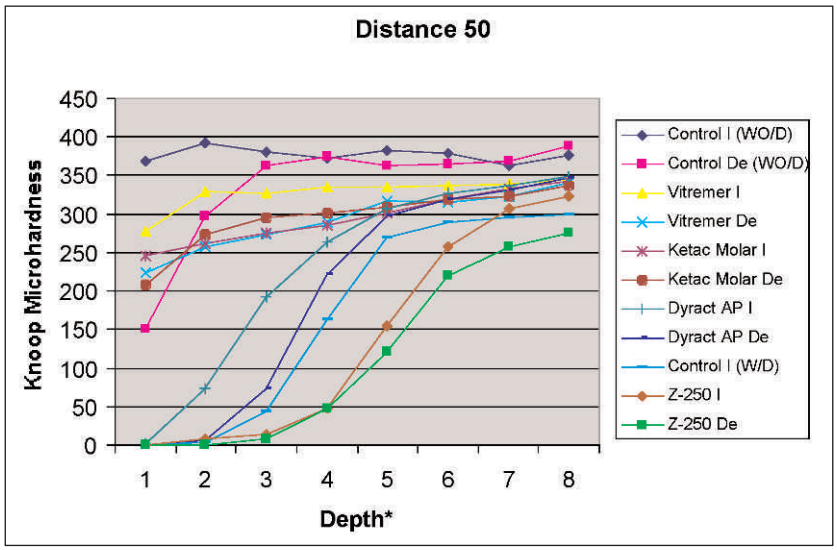

Figure 2. *Mean values of Knoop microhardness for all treatments and depths, at a distance of 50 µm from the cavity walls.*
**1 = 30 µm 2 = 60 µm 3 = 90 µm 4 = 120 µm*
5 = 150 µm 6 = 180 µm 7 = 210 µm 8 = 240 µm

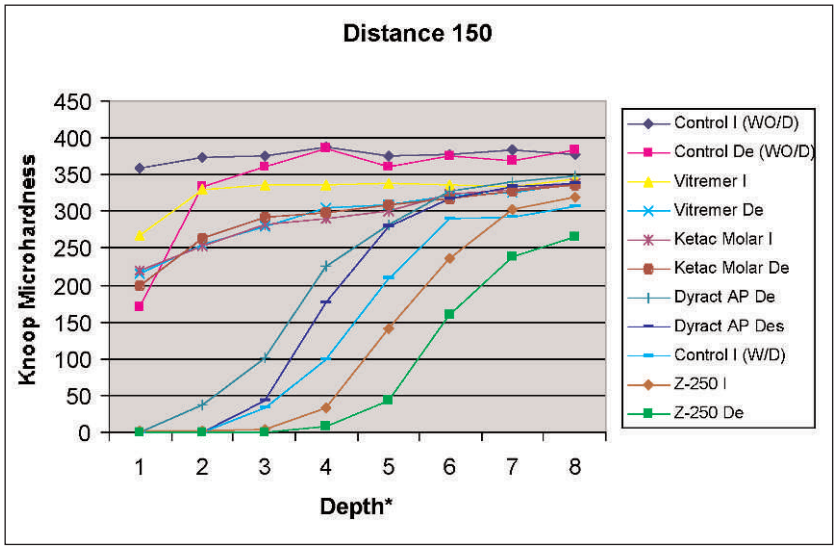

Figure 2. *Mean values of Knoop microhardness for all treatments and depths, at a distance of 150 µm from the cavity walls.*
**1 = 30 µm 2 = 60 µm 3 = 90 µm 4 = 120 µm*
5 = 150 µm 6 = 180 µm 7 = 210 µm 8 = 240 µm

RESULTS

For analysis of the results, mean values of microhardness were obtained for each block at different depths and distances (Figures 2, 3 and 4).

The ANOVA demonstrated a significant effect for all three factors (restorative treatment, distance and depth) as either being isolated or in combination ($p<0.0001$). The Tukey test for multiple comparisons, applied between the different groups, detected minimally significant differences ($\alpha=5\%$).

It should be noted that, in the corresponding control treatment, demineralized enamel submitted to the cariogenic challenge (Control De W/D) could not be submitted to the Knoop microhardness essays, since a process of erosion occurred throughout the external enamel surface and, thus, the parameter for determination of depths and test application was lost. Therefore, this group was excluded from the study.

The ANOVA demonstrated a significant effect based on the three factors (restorative treatment, distance

and depth) in isolation, as well as from the interaction between them ($p<0.0001$) (Table 1). The Tukey test for multiple comparisons applied among the different groups detected minimally significant differences ($\alpha=5\%$) (Tables 2, 3 and 4).

DISCUSSION

The results of this study demonstrate that materials displayed different behaviors in relation to the cariogenic challenge.

A comparison between Control I (WO/D) and other intact restored groups demonstrates that Control I (WO/D) presented Knoop microhardness values statistically higher than the other groups at depths of 30 µm and 60 µm for all distances evaluated. This indicates that no restorative material that was investigated was able to completely avoid demineralization adjacent to the restoration, since the restored specimens presented a reduced hardness compared to the intact enamel and they demonstrated the formation of lesions, even at different depths. These data are in agreement with other studies in the literature (Kotsanos, 2001; Smales & Gao, 2000) which demonstrated that some fluoride-releasing materials are able to interfere with progression of the lesion; yet, they are unable to completely prevent initiation of the caries process.

A comparison of the mean microhardness achieved on blocks that received the restorative materials revealed the best cariostatic properties for the resin-modified glass ionomer cement, Vitremer, applied on intact enamel. This is because the enamel area most exposed to the cariogenic challenge (30-µm and 60-µm depths) exhibited statistically significantly higher mean microhardness values for Vitremer than for the other restorative materials, just being inferior to Control I (WO/D). These findings indicate that Vitremer presented the best cariostatic effect for intact enamel, hindering progression of the lesion. The cariostatic potential of Vitremer was also demonstrated in different clinical assessments (Croll & others, 2001; Croll, Helpin & Donly, 2000), including long-term evaluations on which the material presented as being long lasting and reliable (Croll & others, 2000) and achieved a success rate of 93% (Croll & others, 2001).

The groups Vitremer De, Ketac Molar I and Ketac Molar De displayed an intermediate cariostatic potential compared to the Vitremer I group. These data further demonstrate that the glass ionomer cement Ketac Molar interfered with progression of the lesion in a similar manner in both intact and demineralized enamel.

These data agree with numerous other investigations where the cariostatic effect of glass ionomer cements in different degrees has been demonstrated (Fontana & others, 2002; Kotsanos, 2001; Smales & Gao, 2000; Featherstone & others, 1986).

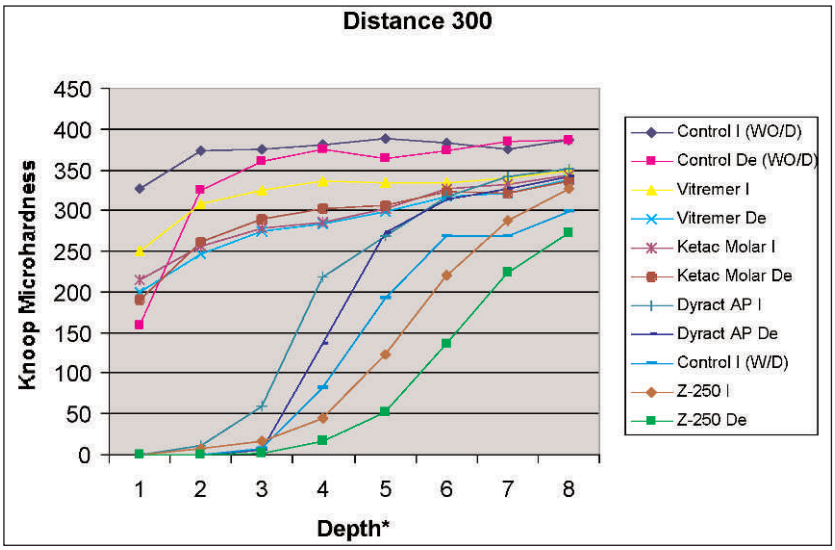

Figure 4. *Mean values of Knoop microhardness for all treatments and depths, at a distance of 300 µm from the cavity walls.*
**1 = 30 µm 2 = 60 µm 3 = 90 µm 4 =120 µm*
5 = 150 µm 6 = 180 µm 7 = 210 µm 8 = 240 µm

Table 1: *Outcome of the Analysis of Variance (ANOVA) Comparing the Following Factors: Treatments and Depths in Relation to the External Enamel Surface and Distance from Cavity Walls*

Effect	DF (Effect)	Mean Square (Effect)	DF (Error)	Mean Square (Error)	F	P
Treatment	10*	2491058*	121*	12351.51*	201.680*	0.000000*
Distance	2*	44467*	242*	1132.98*	39.248*	0.000000*
Depth	7*	2351637*	847*	1611.46*	1459.320*	0.000000*
Treat x Dist	20*	5993*	242*	1132.98*	5.289*	0.000000*
Treat x Depth	70*	137610*	847*	1611.46*	85.395*	0.000000*
Dist x Depth	14*	3060*	1694*	472.67*	6.474*	0.000000*
Treat x Dist x Depth	140*	1923*	1694*	472.67*	4.067*	0.000000*

**Statistically significant difference.*

Table 2: *Tukey Test for Comparison Among Treatments in Different Depths at a Distance of 50 µm From Cavity Walls*

	Depth (µm)							
Treatment	**30**	**60**	**90**	**120**	**150**	**180**	**210**	**240**
Control I (WO/D)	368.37	391.58	379.67#	372.21	381.54	377.62	362.17*•	376.50•
Control De (WO/D)	150.08	296.70Δ	362.62#	374.25	361.91#	363.96#	368.75•	387.87
Vitremer I	277.85	328.99	327.49#	336.09Δ	335.36Δ#	337.44Δ#	338.48Δ*•	344.39Δ
Vitremer De	223.73Δ	257.70Δ	273.51	289.84Δ	318.35Δ#	315.25Δ	324.31#Δ	341.28Δ
Ketac Molar I	244.85	261.35Δ	275.75	285.62Δ	302.34Δ	320.24Δ	333.12Δ*	342.54Δ
Ketac Molar De	207.77Δ	272.75Δ	296.14	300.67Δ	308.58Δ#	320.11Δ	324.35#Δ	336.66Δ
Dyract AP I	1.60	72.69	192.63	264.22#	307.69Δ#	326.90Δ#	337.17Δ*•	349.25Δ•
Dyract AP De	0.00	5.69	73.01Δ	221.84#	298.23Δ	320.15Δ	331.26#Δ*	345.83Δ•
Control I (W/D)	0.00	3.21	43.25Δ	163.46	268.96	290.17*	296.33#	298.71#
Z-250 I	0.00	7.02	14.11	46.99	154.65	258.44*	306.71#Δ	322.70Δ
Z-250 De	0.00	0.00	6.56	47.41	120.29	220.94	257.47	276.28#

*The vertical bars and the symbols Δ # * • indicate a lack of significant difference among the groups they join in a same column (p<0.05). Values in KHN.*

Table 3: *Tukey Test for Comparison Among the Treatments in Different Depths at a Distance of 150 µm From Cavity Walls*

	Depth (µm)							
Treatment	**30**	**60**	**90**	**120**	**150**	**180**	**210**	**240**
Control I (WO/D)	358.71	373.96	375.50	387.58#	375.17#	376.96#Δ	383.25	376.83•
Control De (WO/D)	171.46Δ	332.58	361.21	385.37#	359.67Δ#	375.83Δ	369.62•	384.46
Vitremer I	267.21	329.39	335.74Δ	334.75#	338.92Δ#	336.43#Δ	332.58#	345.08#
Vitremer De	214.74	254.97	279.93	304.77	309.88Δ#	320.67	324.53Δ#	338.31#
Ketac Molar I	220.04	252.95	283.40Δ	290.12Δ	299.59Δ	323.19	329.27#	338.23#
Ketac Molar De	198.78Δ	263.42	293.10Δ	298.25	308.69Δ#	317.85	326.99Δ#	335.88Δ#
Dyract AP I	0.00	37.51	102.24	226.87Δ	281.82	328.55#	339.60•	349.17#•
Dyract AP De	0.00	1.69	43.26	176.46	280.00	317.26	332.58#	339.43#
Control I (W/D)	0.00	0.00	34.29	99.29	209.67	289.58	292.50Δ	305.54Δ
Z-250 I	2.20	3.57	5.05	33.09	141.11	235.86	302.25Δ#	320.32Δ#
Z-250 De	0.00	0.00	0.00	7.79	44.16	159.31	238.88	264.58

*The vertical bars and the symbols Δ # * • indicate a lack of significant difference among the groups they join in a same column (p<0.05). Values in KHN.*

Table 4: *Tukey Test for Comparison Among Treatments in Different Depths at a Distance of 300 µm From Cavity Walls*

	Depth (µm)							
Treatment	**30**	**60**	**90**	**120**	**150**	**180**	**210**	**240**
Control I (WO/D)	362.41	372.79	375.29#	381.54#	388.17#	383.42#	375.00•*	387.46
Control De (WO/D)	157.67	324.83	361.42#	375.92#	364.28Δ#	373.04#	384.58*	386.62
Vitremer I	250.20	308.00	325.12Δ	336.99#	334.68Δ#	335.13#	339.27•*	350.30#
Vitremer De	199.64	247.48	274.04	283.39	298.88Δ	316.64Δ	320.58#	337.67#
Ketac Molar I	213.60	256.32	277.85	285.53	303.18Δ	327.03#	333.44#•	344.01#
Ketac Molar De	191.24	260.60	289.37Δ	302.84	307.37Δ	323.49Δ#	322.08#	335.90#
Dyract AP I	0.00	10.73	59.26	218.95	268.15	318.44Δ#	340.87•*	350.54#
Dyract AP De	0.00	0.00	5.75	135.53	272.68	314.21Δ	327.28#	342.04#
Control I (W/D)	0.00	0.00	8.12	82.29Δ	191.70	269.12Δ	269.00Δ	298.37Δ
Z-250 I	0.00	7.02	16.27	45.35Δ	123.82	219.59	288.15#	325.60#
Z-250 De	0.00	0.00	2.06	17.14	52.37	135.63	224.99Δ	272.56Δ

*The vertical bars and the symbols Δ # * • indicate a lack of significant difference among the groups they join in a same column (p<0.05). Values in KHN.*

Kotsanos (2001) conducted an *in situ* evaluation of the effect of restorative materials on intact enamel submitted to cariogenic challenge and achieved outcomes similar to those in this study. It was observed that the cariostatic potential of resin-modified glass ionomer cement was better than that of the chemically cured glass ionomer cement, which, in turn, was superior to polyacid-modified resin composite.

The materials Dyract AP and Z-250 did not exhibit any cariostatic effect either on intact enamel or incipient carious lesions, being similar to group Control I (W/D) at the lowest depths and at the three distances investigated. These materials were unable to avoid progression of the lesion at the area closest to the external enamel surface, since the enamel underwent erosion at this area in the presence of such materials. *In vivo* studies corroborate the poor performance of resin composites against the occurrence of secondary carious lesions (Tyas, 1991; Van Dijken, 1986; Mjör, 1985).

In this study, the polyacid-modified resin composite Dyract AP displayed better performance than the resin composite Z-250, as it displayed higher microhardness values from the 60-µm depth. This finding might be based on the fact that polyacid-modified resin composite releases fluoride, yet, in minimal amounts (Hatibovic-Kofman, Suljak & Koch, 1997; Cao & others, 1994).

In general, the analysis of Knoop microhardness based on eight different depths in relation to the external enamel surface for the three distances demonstrated that the lowest microhardness values were observed at the 30-µm depth, that is, at the area directly exposed to the cariogenic challenge. An increase in microhardness was observed at depths closer to the dentin-enamel junction, with a tendency towards microhardness values similar to intact enamel represented by the Control I (WO/D) group. These data are in agreement with other studies in the literature that also assessed this variable (Benelli & others, 1993; Serra & Cury, 1992). However, the depths at which the microhardness values were closer to that of the intact enamel varied according to the cariostatic property of the materials employed.

The different cariostatic effects observed in this investigation may be related to the presence or absence of fluoride release from the restorative materials (Hatibovic-Kofman & others, 1997; Donly, 1994; Benelli & others, 1993), being that glass ionomer cements release larger amounts of fluoride than polyacid-modified resin composites (Cao & others, 1994). A comparison between chemically cured and resin-modified glass ionomer cements in the literature displays conflicting outcomes. Whereas some studies state that fluoride release from both materials is similar (Burke & others, 2002; Creanor & others, 1994), other investigations have indicated higher fluoride release by resin-modified glass ionomer cements (Brooks & others, 1994; Hatibovic-Kofman & Koch, 1991).

These findings, related to the distance from the cavity wall, might be associated with the fact that fluoride release is more effective in areas closer to the restoration margins (Creanor & others, 1994; Serra & Cury, 1992), since higher microhardness values at the 30-µm depth (closer to the external enamel surface) were observed at the 50-µm distance.

The outcomes of this study demonstrated that, even though no material could completely prevent establishment of the carious lesion, the glass ionomer cements that were investigated hindered progression of the lesion both in intact enamel and in incipient carious lesions, demonstrating their cariostatic properties. However, resin composite and polyacid-modified resin composite do not display any cariostatic effect. These data may be useful in predicting the performance of such materials in the presence of severe cariogenic challenge, since they displayed significant differences in the occurrence of artificial carious lesions.

Thus, it may be concluded that, for patients at high risk and/or those with caries activity, the restorative material of choice should be either chemically cured or resin-modified glass ionomer cements, since their cariostatic properties allows them to interfere with the progression of and reduce the severity of secondary caries.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. None of the study materials were able to completely inhibit the creation of lesions.
2. Resin-modified glass ionomer cement demonstrated the best cariostatic action in intact bovine enamel.
3. High viscosity glass ionomer, in intact or demineralized enamel, and resin-modified glass ionomer cement in demineralized enamel, presented intermediate cariostatic potential.
4. Conventional and polyacid-modified resin composite did not demonstrate any cariostatic effect.
5. The data suggest that glass ionomer cements demonstrated better cariostatic action compared to other restorative materials.

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Effect of Solvent Type on Microtensile Bond Strength of a Total-etch One-bottle Adhesive System to Moist or Dry Dentin

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Clinical Relevance

In this study, the type of organic solvent and dentin moisture had an influence on bond strength to dentin. The results showed that the application of a total-etch, ethanol-based adhesive system to moist dentin results in higher bond strengths.

SUMMARY

This study evaluated the effect of organic solvent (acetone or ethanol) on the microtensile bond strengths (MTBS) of an adhesive system applied to dry and moist dentin. Sixteen extracted human third molars were ground to expose a flat occlusal dentin surface and acid etched for 20 seconds (20% phosphoric acid gel, Gluma Etch 20 Gel, Heraeus/Kulzer). After rinsing the acid etchant, an ethanol-based one-bottle adhesive system was applied to the mesial half of the occlusal dentin surface. An acetone-based, one-bottle adhesive system was applied to the distal half of the ground dentin surface. The teeth were randomly assigned to groups. In Group 1, the etched dentin was thoroughly air dried and an ethanol-based one-bottle adhesive system was applied (Gluma Comfort Bond, Heraeus/Kulzer) (GCB). In Group 2, the etched dentin was thoroughly air dried and an acetone-based one-bottle adhesive system was applied (Gluma One Bond, Heraeus/Kulzer)(GOB). In Group 3, excess moisture was removed after acid etching, leaving a moist dentin surface and a one-bottle ethanol-based adhesive was applied (Gluma Comfort Bond). In Group 4, excess moisture was removed after acid etching, leaving a moist dentin surface and an acetone-based adhesive was applied (Gluma One Bond). A hybrid resin composite (Venus, Heraeus/Kulzer) was applied to the bonded surface in four 1-mm increments and light cured according to manufacturer's directions. The specimens were then sectioned with a slow-

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speed diamond saw in two perpendicular directions to obtain sticks with a cross-section of 0.5 ± 0.05 mm². The microtensile bond strength (MTBS) test was performed with a Bencor device in an Instron machine at a crosshead speed of 0.5 mm/minute. The data were subjected to a two-way ANOVA and Scheffé *Post hoc* test ($p<0.05$). The experimental MTBS measured for dry dentin were Group 1=37.0±10.6 and Group 2=34.7±9.0 in MPa (mean ±SD); and on moist dentin, Group 3=50.7±11.0 and Group 4=38.5±10.5 in MPa (mean ±SD). The ethanol based adhesives resulted in higher MTBS than acetone-based adhesive ($p<0.008$) and bonding to moist dentin resulted in higher MTBS ($p<0.001$). GCB applied on moist dentin resulted in statistically higher bond strengths than the other groups. The highest MTBS were achieved with the use of an ethanol-based adhesive to moist dentin.

INTRODUCTION

Recent advances in aesthetic restorations have driven significant improvements in dental adhesive systems. The objectives of these advances are to establish an effective adhesion to dental structure (Bowen, Cobb & Rapson, 1982). Bonding to enamel is well known as being very clinically reliable. Since its introduction in 1955, the acid-etch-technique has provided an ideal surface morphology (Buonocore, 1955; Swift, Perdigão & Heymann, 1995; Lopes & others, 2002). Although this technique has revolutionized dentistry over the last 20 years, dentin bonding is still a challenge due to the wet tubular ultra structure and heterogeneous, partially organic composition of dentin substrate (Pashley, 1989; Pashley, 1991; Erickson, 1992; Eick & others, 1993).

For contemporary adhesive systems, dentin bonding requires the removal or modification of the smear layer and superficial demineralization through the application of an acid etchant (Gwinnett, 1994). Although chemical reactions between chemical bonding agents and dentin have been reported, it is generally accepted that dentin bonding relies primarily on micro-mechanical interaction, similar to enamel bonding, mediated by the permeation of resin monomers into acid-etched dentin (Fusayama & others, 1979; Nakabayashi, Kojima & Masuhara, 1982). The entanglement of polymerized adhesive resin with collagen fibrils and residual hydroxyapatite crystals generates an interfacial structure called the "hybrid layer" or "resin-dentin inter-diffusion zone" (Nakabayashi & others, 1982).

Exposure of the collagen fiber network by acid etching creates favorable conditions for micro-mechanical retention of an adhesive system, but the collagen network can collapse on itself due to the loss of structural support (Pashley, Horner & Brewer, 1992). Furthermore, if the exposed collagen is air dried before the bonding procedure, it may collapse over the underlying unaffected dentin (Pashley & others, 1992; Swift & others, 1999). These two phenomena create a layer of residual, denatured and collapsed collagen on top of the demineralized dentin, into which monomers may be unable to penetrate and diffuse into because of limited porosity (Pashley & others, 1992; Gwinnett, 1992). Thus, when demineralized collagen is kept moist, the fibrils are observed as being upright and separated by wide interfibrillar spaces, resulting in better opportunities for resin infiltration and higher bond strengths when compared with those that had been briefly air dried (Nakabayashi & others, 1982; Kanca, 1992a).

The efficacy of dentin adhesives seems to be improved by the addition of high vapor pressure organic solvents to the adhesives' chemical formulations. Acetone and ethanol are commonly used solvents found in the majority of current bonding systems. These chemical agents function as "water-chasers" and solubilize resin components. They increase the wettability of the dentin substrate by the bonding systems and help to replace water in the acid-etched, rinsed dentin surface with hydrophilic resin monomers (Kanca, 1992a). High bond strengths are obtained with bonding agents dissolved in high vapor-pressure solvents such as acetone or ethanol when acid-etched dentin is left visibly moist (Kanca, 1992a,b; Perdigão, Swift & Cloe, 1993). The moisture dependence of dentin is related to the type of solvent in the adhesives chemical formulations. Ethanol-based adhesive systems seem less sensitive to the amount of moisture in dentin (Jacobsen & Söderholm, 1998; Finger & Balkenhol, 1999; Reis & others, 2003). However, this reduction in sensitivity has not been tested with adhesives with the same monomer composition.

This study evaluated the effect of an organic solvent (acetone or ethanol) on the microtensile bond strengths (MTBS) of an adhesive system applied to dry and moist dentin. The null hypotheses tested were: 1) bonding strength would not be dependent on dentin moisture after acid etching and 2) the type of solvent would not have an influence on bond strength to dentin.

METHODS AND MATERIALS

The specimens utilized in this study were 16 extracted human third molars that were stored in saline solution at room temperature. The occlusal enamel and roots were removed and the occlusal surface was ground under running water to expose a flat dentin surface parallel to the occlusal surface. The specimens were inspected with magnification glasses for enamel remnants. The exposed dentin surface was polished with a series of wet silicon carbide paper (220, 360 and 600-grid). The specimens were divided in the middle using a round diamond bur (KG Sorensen, Barueri, Brazil) in a high-speed handpiece under continuous water

Table 1: *Chemical Composition of Adhesive Systems Tested*

Bonding Agent	Manufacturer	Etching Gel	Chemical Composition	Solvent Type
Gluma One Bond (GOB)	Heraeus-Kulzer	20% phosphoric acid	UDMA, HEMA, 4-META	Acetone
Gluma Comfort Bond (GCB)	Heraeus-Kulzer	20% Phosphoric acid	UDMA, HEMA, 4-META, polyacrylic and dicarboxylic acids	Ethanol/water

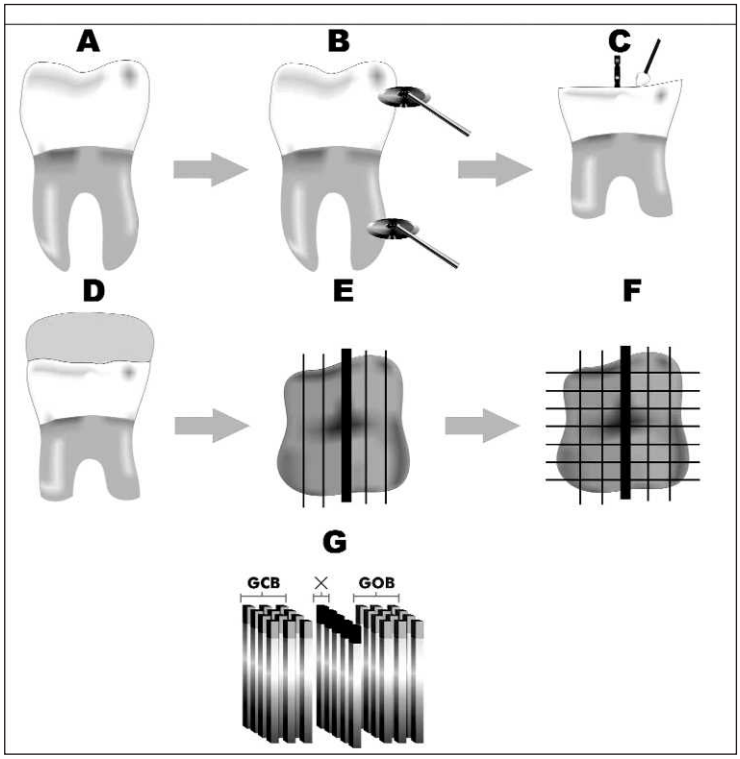

Figure 1. *Schematic illustrating the essence of the microtensile bond testing technique. A, Human third molar. B, The occlusal enamel, and roots were removed. C, Tooth was divided in two parts for a matrix, each half, corresponding to the type of adhesive system. D, Resin composite (Venus, shade A3) was applied in four layers to a height of 4 to 5 mm. E, Restored tooth was sectioned into buccal-lingual parallel slabs 0.7-mm thick. F, New sections of 0.7-mm thick were performed in each slice perpendicular to the first section and the slabs converted into sticks. G, Sticks representative of adhesive system were reserved for testing and sticks with black base were discarded.*

cooling. A metallic matrix (Microdont, São Paulo, Brazil) was positioned on the sectioned specimen and the adhesive procedures were carried out. After rinsing the acid etchant, an ethanol-based one-bottle adhesive system was applied to the mesial half of the occlusal dentin surface. On the distal half, an acetone-based one-bottle adhesive system was applied. The teeth were randomly assigned to the experimental groups. Two commercially available dentin bonding systems were used (see Table 1 for a description of materials):

Group 1–Gluma Comfort Bond (GCB), Dry: 20% phosphoric acid gel (Gluma Etch 20 Gel; Heraeus/Kulzer) was applied for 20 seconds to a dry dentin surface and rinsed thoroughly with an air-water syringe for 10 seconds. The etched dentin was dried for 10 seconds with an air syringe at a distance approximately 2 cm from the surface (dry technique). GCB, an ethanol-based one-bottle bonding agent, was applied in two consecutive coats with a saturated disposable brush lightly dried for two seconds to evaporate the solvent and light cured for 20 seconds.

Group 2–Gluma One Bond (GOB), Dry: Specimens were treated in the same manner as Group 1, except that an acetone-based one bottle bonding agent was used.

Group 3–Gluma Comfort Bond (GCB), Moist: 20% phosphoric acid gel (Gluma Etch 20 Gel; Heraeus/Kulzer) was applied for 20 seconds to a dry dentin surface and rinsed thoroughly with an air-water syringe for 10 seconds at a distance approximately 2 cm from the surface. The etched dentin was partially dried by blotting with a small piece of absorbent paper, leaving a visibly moist surface (wet technique). GCB, an ethanol-based one-bottle bonding agent, was applied in two consecutive coats with a saturated disposable brush, lightly dried for two seconds to evaporate the solvent and light cured for 20 seconds.

Group 4–Gluma One Bond (GOB), Moist: Specimens were treated in the same manner as Group 3, except that an acetone-based one-bottle bonding agent was used.

A hybrid resin composite (Venus, shade A3, Heraeus Kulzer) was applied in four layers to a height of 4 to 5 mm. Each layer was light cured for 20 seconds with a light-curing unit (Optilux 400, Kerr/Demetron, Danbury, CT, USA). The light intensity was measured with a radiometer (Demetron/Kerr) and resulted in 500 mW/cm^2.

The specimens were stored in water at room temperature for 24 hours before being sectioned. They were bonded with a cyanoacrylate-based adhesive (Zap-It, DVA, Corona, CA, USA) to acrylic cylinders for easier handling during cutting operations. The restored teeth were attached to a cutting machine (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA), where a diamond disc (South Bay Technology, San Clemente, CA, USA) running at a low speed with water coolant was used to cut the specimens into approximately 0.7-mm thick slices running in a buccal-lingual direction. The slices were then trimmed with a diamond bur to obtain a surface area of 0.5±0.05 mm^2. A total of 12 stick specimens

were prepared for each group. These were stored in water for 24 hours at room temperature (Figure 1) before taking the MTBS measurements.

In preparation for the tensile test, the beams were bonded with a quick polymerizing cianoacrylate-based adhesive (Zap-It, DVA) in a special jig for microtensile tests (Bencor Multi-T, Danville Engineering, San Ramon, CA, USA). Then, an Instron Universal testing machine (Model 4444, Instron Corp, Canton, MA, USA) operating at a speed of 0.5 mm/minute was used and the MTBS data were recorded. Before the test, the area next to the adhesive interface of the sticks specimens was computed using the Series IX Software System (Instron Corp).

The data were subjected to two-way ANOVA (Independent variable: solvent type and dentin moisture; outcome variable: MTBS) and *Post hoc* test (Scheffé test) ($p<0.05$). The statistical analysis was carried out with the SPSS 10.0 (SPSS Inc, Chicago, IL, USA) software package.

Table 2: *Microtensile Bond Strength to Dentin and SD*

Group	MTBS (Mean ± SD)
Gluma Comfort Bond, Dry	37.0±10.6[B]
Gluma One Bond, Dry	34.7±9.0[B]
Gluma Comfort Bond, Moist	50.7 ± 11[A]
Gluma One Bond, Moist	38.5±10.5[B]

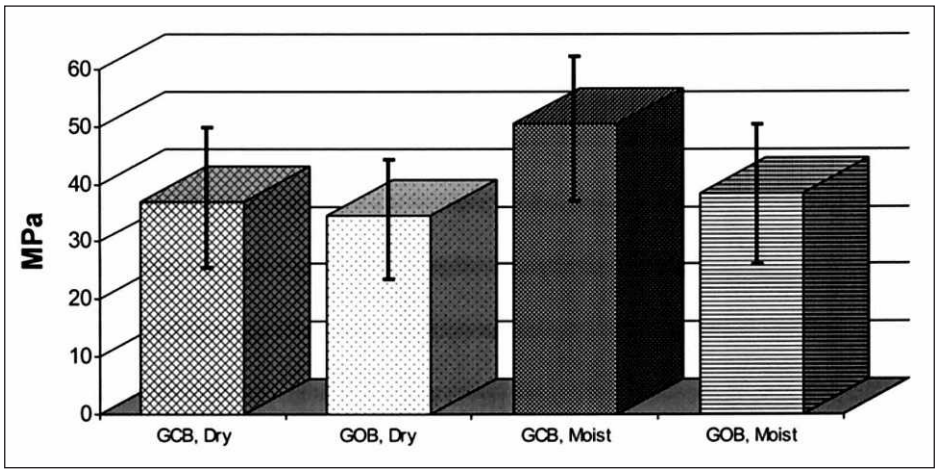

Figure 2. *Mean microtensile bond strengths to dentin (MPa).*

RESULTS

The mean MTBS in MPa and standard deviation are shown in the Table 2 and Figure 2. The analysis of variance revealed a statistically significant difference between pairs of means for the factor "solvent type," with the ethanol based adhesives higher than the acetone-based ($p<0.008$) adhesives. The *post hoc* test (Scheffé test) ranked this difference in two (Table 2). Gluma Comfort Bond, ethanol-based and applied on moist dentin, resulted in a significantly greater mean MTBS (50.7 ± 11.0 MPa) when compared with either Gluma Comfort Bond (GCB), dry (37.0 ± 10.6 MPa) or Gluma One Bond, moist (38.5 ± 10.5 MPa) and dry (34.7± 9.08 MPa). Bonding to moist dentin resulted in higher MTBS ($p<0.001$). There were no statistically significant interactions between the independent variables "solvent type" and "dentin moisture" ($p<0.069$).

DISCUSSION

The first null hypothesis was rejected. Bonding to wet dentin resulted in statistically higher bond strengths than to dry dentin. This result suggests that the moist bonding technique promotes better infiltration into demineralized dentin than the dry bonding technique. Such results are confirmed by other *in vitro* studies (Gwinnett & Kanca, 1992; Swift & Triolo, 1992; Perdigão & others, 1993) which concluded that bonding to moist dentin is more effective in terms of bond strength than techniques that air-dry the acid-etched dentin prior to priming. However, at least one researcher reported that the dry and moist techniques did not affect bond strengths (Miears, Charlton & Hermesch, 1995). It is interesting to note that the higher bond strength to wet dentin obtained in this study does not necessarily hold in microleakage tests, where at least two dye-penetration studies showed that microleakage around the dentin margins of composite restorations was similar, irrespective of the wet or dry bonding technique (Vargas & Swift, 1994; Santini & Mitchell, 1998).

The second null hypothesis was rejected; the ethanol–based bonding system (GCB) resulted in higher MTBS than the acetone-based system tested (GOB). According to Lopes and others (2004) and Ritter, Bertoli and Swift (2001), the ethanol-based adhesive had higher bond strengths when compared to all other acetone-base systems, confirming the results of this study. Moreover, other characteristics of acetone, such as its high volatilization (Van Meerbeek & others, 2001) and quick evaporation from an open bottle (Perdigão, Swift & Lopes, 1999) makes it a less favorable solvent option.

The application of a total-etch ethanol-based adhesive system to moist dentin results in higher bond strength than to dry dentin. For the acetone-based adhesive system, MTBS was not affected by the degree of dentin wetness after acid etching, resulting in similar bond strength to moist or dry dentin. These results are contradicted in the literature data by Jacobsen and Söderholm (1998), Finger and Balkenhol (1999) and Reis and others (2003), who affirm that acetone-based

adhesive systems are more dependent on an accurate wet bonding technique than ethanol-based adhesives. However, these studies compared adhesive systems with different solvent and monomer compositions. In this study, the only difference between the adhesives was the solvent type. Another possible explanation for this disagreement could be differences between the monomer composition of the Gluma systems and the other adhesives tested in these cited studies. GOB and GCB are based on a 4-META monomer. This monomer was not present in the composition of other adhesive systems tested in the cited studies, which used adhesives such as Single Bond, One-Step and Syntac Single Component. Therefore, either the difference in monomer composition or the solvent concentrations could be the reason for the results obtained.

The microtensile test (MTBS) produces a more consistent measurement of bond strengths than the conventional shear bond test (Sano & others, 1994). The microtensile test also allows for the measurement of bond strengths using bonding surfaces with a cross-sectional area in the range of 0.5 to 1.5 mm^2. Other advantages over conventional shear and tensile bond strength tests include the use of only one tooth to make several bonded interfaces, testing distinct substrates, such as sclerotic dentin and carious dentin and testing regional differences in bond strengths within the same tooth (Pashley & others, 1995). According to Pashley and others (1999), microtensile bond testing methods offer versatility that cannot be achieved by conventional methods. It is more labor-intensive than conventional testing but holds great potential for providing insight into the strength of adhesion of restorative materials used in dentistry. In conclusion, due to the use of MTBS in the current work, the variation coefficient was reduced and permitted the use of two different adhesive systems on the same dentin substrate.

Under the limitations of this *in vitro* study, further clinical studies should test the same dentin adhesives. Also, additional research, where the bonding systems are applied *in vivo* to reproduce clinical conditions such as a positive intra-pulpal pressure and the presence of fluid in the dentinal tubules, is suggested to verify the possible influence on bond strength to moist and dry dentin.

CONCLUSIONS

Based on results of this study, the use of an ethanol-based adhesive system to moist dentin resulted in higher MTBS.

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The Shear Bond Strength Between Luting Cements and Zirconia Ceramics After Two Pre-treatments

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Clinical Relevance

The luting cements tested exhibited considerable differences with regard to their shear strengths following conditioning. This fact should be considered when clinically employing these agents in order to improve the adhesive properties of dental restorations.

SUMMARY

This study evaluated the shear-bond strength of 11 luting cements from different material classes to manufactured pre-treated zirconia ceramics (Lava: 97% ZrO_2, stabilized with 3% Y_2O_3). In addition, the influence of the curing method on shear-bond strength was investigated. The cements examined were one zinc-phosphate cement (Fleck's zinc cement), two standard glass-ionomer cements (Fuji I, Ketac-Cem), three resin-modified glass-ionomer cements (Fuji Plus, Fuji Cem, RelyX Luting), four standard resin cements (RelyX ARC, Panavia F, Variolink II, Compolute) and one self-adhesive universal resin cement (RelyX Unicem). The ceramic surface was sand-blasted with 100-µm alumina or tribochemically coated with silica. After bonding procedure, one group was tested after 30 minutes (Time I), the other group was stored in distilled water at 37°C for 14 days and subsequently thermocycled 1000 times (Time II). Statistical analysis was performed by multifactorial ANOVA models with interactions. For multiple pairwise comparisons, the Tukey method was used. After sandblasting, the highest shear-bond strength was obtained for the self-adhesive universal resin cement at 9.7 MPa (Time I) and 12.7 MPa (Time II), respectively. When using the Rocatec system, the highest values were found for one of the resin cements at 15.0 MPa (Time I) and for the self-adhesive universal resin cement at 19.9 MPa (Time II).

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INTRODUCTION

Partially stabilized zirconia was integrated into indirect restorative dentistry in the past few years. The ceramic is stabilized by yttrium oxide and exists as yttria-tetragonal zirconia polycrystals (Y-TZP) at room temperature. In dentistry, zirconia ceramics have been clinically used for orthodontic brackets (Keith, Kusy & Whitley, 1994), stock post-and-core pins (Strub, Pontius & Koutayas, 2001; Kern, Simon & Strub, 1997;

Meyenberg, Lüthy & Schärer, 1995), implants (Akagawa & others, 1993), abutments for implant prosthodontics (Prestipino & Ingber, 1993) and hard-core frameworks for crowns and bridges (Sturzenegger & others, 2000; Filser & others, 1999).

The clinical success of fixed ceramic prostheses is heavily dependent on the cementation procedure. Schwartz and others (1970) found that loss of crown retention was the second most common cause of failure for traditional crowns and fixed partial dentures (FDPs). Although the establishment of optimal resistance and retention forms when preparing the tooth is of primary importance, luting cements should still provide a stable, durable connection between the natural tooth and the dental restoration, while at the same time increasing fracture resistance of the restored tooth and the restoration itself (Burke, 1995). Luting cements are also used as a barrier against microbial leakage for sealing the interface between dissimilar materials and holding them together through some form of surface attachment (Pameijer & Nilner, 1994). Additionally, dental cements should possess a range of desirable properties such as favorable compressive and tensile strengths, sufficient fracture toughness, sufficient film thickness, biocompatibility, low setting stress, adequate working and setting times and favorable handling characteristics (Rosenstiel, Land & Crispin, 1998). Currently, different types of luting cements for long-term cementation of fixed prostheses are commercially available. These include zinc-phosphate cements, standard glass-ionomer cements, resin-modified glass-ionomer cements, standard resin cements and a recently developed material described as self-adhesive universal resin cement. The goal in developing this cement was to combine the ease of handling and absence of required pre-treatment steps as found in glass-ionomer cements with the good mechanical properties (Piwowarczyk & Lauer, 2003), favorable esthetics and firm adhesion to tooth structure (Piwowarczyk, Lauer & Sorensen, 2003) offered by resin cements.

The increasing clinical use of zirconia ceramics in prosthodontics, considering the advantages of this material, such as truly esthetic, metal-free and stable restorations, requires a reliable technique for cementation in combination with adequate luting cements. The manufacturer of Lava recommends that the restorations be cemented with conventional cements or adhesive luting after tribochemical silica coating. Few studies have been published regarding the bonding methods and luting cements used to generate a stable bond to zirconia; however, these studies provide limited data and controversial results (Dérand & Dérand, 2000; Edelhoff & others, 2000; Wegner & Kern, 2000; Kern & Wegner, 1998).

This study investigated the factors that affect bonding to zirconia ceramics and, in particular, the question of which material classes and which luting cements, combined with certain pre-treatments, are suitable for a stable bond. The shear-bond strength of zirconia was evaluated after two surface and specimen treatments. The influence of the curing method on bond strength was also studied and bond failure modes were investigated by SEM characterization.

METHODS AND MATERIALS

Preparation of Specimens

This study used the Lava system (3M ESPE, Seefeld, Germany), consisting of manufactured disks made of partially yttria-stabilized zirconia ceramic disks (97% ZrO_2 stabilized with 3% Y_2O_3; Lot No R25-108-3) with a 13-mm diameter and 2-mm thickness. The ceramic samples were placed into plastic rings (inner diameter–19 mm, height–12 mm) and embedded in slow-curing epoxy resin (Buehler Epoxide; Buehler, Lake Bluff, IL, USA). After hardening at room temperature for 24 hours, the surface of the ceramic material was wet-ground sequentially to 600 grit using SiC sandpaper (Buehler; average grain size: 14 µm). All samples were ultrasonically cleaned in isopropanol for three minutes.

The surface of the zirconia ceramic disks was treated in one of two different ways. Half the samples were air-abraded with 100-µm alumina (Ivoclar Vivadent Canada, St Catherines, Ontario, Canada) at 2.8 bar pressure at a distance of 10 mm for 10 seconds. The other half was tribochemically silica-coated using the Rocatec system (3M ESPE, Seefeld, Germany). The steps in this silica coating process were: 1) cleaning by sandblasting with 110-µm alumina (Rocatec-Pre powder, Lot #FW0062286) at 2.5 bars and a distance of 10 mm for 10 seconds and 2) sandblasting the surface with a special silica particle containing 110-µm alumina (Rocatec-Plus powder, Lot #0480), forming a silica layer. After cleaning with pressurized air, the silica-coated ceramic surface was silanized using ESPE-Sil (3M ESPE, Lot #0128). The bonding surface was allowed to dry for five minutes prior to application of the luting cements.

Conducting the Experiments

Eleven luting cements from various material classes were used: one zinc-phosphate cement, two standard glass-ionomer cements, three resin-modified glass-ionomer cements, four dual-cure resin cements and one dual-cure self-adhesive universal resin cement (Table 1). The materials were handled according to the manufacturer's instructions at a temperature of 23.0 ± 1.0°C and a relative humidity of 50 ± 5%. Three cements (Ketac-Cem, Compolute and RelyX Unicem) were supplied in pre-measured capsules. Upon activation, these materials were mechanically triturated with the rotational mixing machine (RotoMix; 3M

Table 1: *Description of Luting Cements*

Material	Manufacturer	Material Group	Batch #
Fleck's zinc cement	Mizzy Inc, Cherry Hill, NJ, USA	Zinc-phosphate cement	Powder 67/081100 Liquid 52/011601
Fuji I	GC Corp, Tokyo, Japan	Glass-ionomer cement	0001251
Ketac-Cem	3M ESPE, Seefeld, Germany	Glass-ionomer cement	106292
Fuji Plus	GC Corp, Tokyo, Japan	Resin-modified glass-ionomer cement	102261
Fuji Cem	GC Corp, Tokyo, Japan	Resin-modified glass-ionomer cement	0103062
RelyX Luting	3M ESPE, Seefeld, Germany	Resin-modified glass-ionomer cement	20010716
RelyX ARC	3M ESPE, Seefeld, Germany	Dual-cure resin cement	20010718
Panavia F	Kuraray, Osaka, Japan	Dual-cure resin cement	Base 00053A Catalyst 00024A
Variolink II	Ivoclar Vivadent, Schaan, Liechtenstein	Dual-cure resin cement	Base C15032 Catalyst C33393
Compolute	3M ESPE, Seefeld, Germany	Dual-cure resin cement	101194
RelyX Unicem	3M ESPE, Seefeld, Germany	Dual-cure self-adhesive universal resin cement	CM633

ESPE) 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 on a mixing block using a hard plastic spatula at a base-to-catalyst paste ratio of 1:1 for approximately 20 and 10 seconds, respectively. Fuji Plus was mixed at a base-to-catalyst ratio of 2:1 for approximately 20 seconds. The powder-to-liquid ratio of Fleck's zinc cement was determined by weighing according to the manufacturer's instructions using an analytical balance (± 1 mg). Mixing was performed on a cool slab over a wide area to incorporate small increments of powder into the liquid for approximately 120 seconds (Fleck's zinc cement).

Before the bonding procedure, plastic capsules (inner diameter: 5.5 mm; Torpac Inc, Fairfield, NJ, USA) were filled with resin composite (Herculite XRV; Kerr Co, Orange, CA, USA) and light cured with a dental curing light (HP Teklite; Belle de St Claire, Orange, CA, USA) for 20 seconds, then further cured in a light-curing device (UniXS; Heraeus Kulzer, Wehrheim, Germany) for an additional 90 seconds. These capsules were bonded by finger pressure perpendicular to the pre-treated ceramic surfaces for 10 seconds. Excess cement was removed from the bonding margin using small brushes. An oxygen-blocking gel (Oxyguard II; Kuraray, Osaka, Japan; Lot #00373A) was applied for three minutes when Panavia F was used. To evaluate the influence of the curing method, the dual-cure resin cements and the dual-cure self-adhesive universal resin cement were also light cured from four sides at 90-degree angles for 20 seconds using a dental light-curing gun (Optilux 400; Demetron Research Corp, Danbury, CT, USA). The output of the curing light was checked by a measuring device (Cure Rite; LD Caulk, Milford, DE, USA) prior to curing each group of cements. Each specimen was immersed in distilled water at a temperature of 37°C, then placed in an incubator and not removed until four minutes before testing began.

Each bonding group, containing 20 samples, was divided into two subgroups of 10 samples each. One subgroup was tested for 30 minutes after the first mixing. The other subgroup was stored in distilled water in an incubator at 37°C for 14 days and subsequently thermocycled 1000 times between 5°C and 55°C (dwell time: 20 seconds).

The bonded specimens were secured in a mounting jig. Shear force was applied by a 0.5-mm blunt-edge chisel using a universal testing machine (Instron Corp, Canton, MA, USA) at a constant crosshead speed of 0.5 mm/minute until failure occurred. The shear-bond force was recorded in newtons and the bond strength was calculated in MPa. The fractured interfaces of the ceramic samples were observed visually under a light microscope at 30x magnification to evaluate the adhesive and cohesive failure modes. Areas that presented a resin-free ceramic surface were classified as adhesively failed and areas that were covered by resin were classified as cohesively failed. Representative samples were examined under a scanning electron microscope (Leo Type 1530VP; Oberkochen, Germany) with an acceleration voltage of 1.2 keV.

Statistical analysis was performed by multifactorial ANOVA models with interactions. In a first step, the measurements after light curing were neglected, leading to a three-way ANOVA model with the main effects material, surface treatment and time. All possible interaction terms were included in the model. In a second step, the curing method was additionally considered, leading to a four-way ANOVA model. In this model, only the five materials with two curing methods were considered. Again, all possible interaction terms were included in the model. Multiple pairwise comparisons were performed using the Tukey method. A sig-

nificance level α=0.05 was chosen. SAS Version 8.02 was used for calculations.

RESULTS

The arithmetic means and standard deviations are presented in Tables 2 and 3 for all 16 bonding groups and two specimen treatments.

Examining the 11 self-polymerizing materials with the statistical three-way ANOVA model yielded significant differences (p<0.0001) between luting cements, the two pre-treatments and the two measurement times. Differences between the materials and the effects of pre-treatment differed at both measuring times. Multiple classifications and significant interactions defined several planes on which to perform pairwise multiple comparisons. Most interesting were the primary effects themselves and the triple interactions in order to find the combination that showed the highest shear-bond strength. The material with the highest mean shear-bond strength at 7.6 MPa was RelyX Unicem (Table 4). The best pre-treatment was obtained with the Rocatec system (mean of 3.8 MPa, compared to 2.0 Mpa, when air-abraded with alumina). Time II (3.2 Mpa) was the time at which the highest shear-bond strength was achieved, compared to 2.6 MPa at Time I. The combination that yielded the highest shear-bond strength (17.5 MPa) was the self-adhesive universal resin cement RelyX Unicem at Time II. This combination was significantly different from all other combinations (p<0.0001).

Looking at the five dual-cure resin cements within the four-way classification, the polymerization method had a significant influence on bond strength, in addition to material, pre-treatment and time. The magnitude of the effect of the polymerization method was dependent on material, pre-treatment and time. Pairwise multiple comparisons showed that light polymerization had advantages over self-polymerization, achieving a mean value of 8.8 MPa compared to 4.7 MPa with self-polymerization (p<0.001). The combination that yielded the highest value was RelyX Unicem in combination with the Rocatec system after 14 days of storage in distilled water and thermocycling 1000 times (19.9 MPa). This combination differed significantly from the other 39 possible combinations (p<0.001), with the exception of RelyX Unicem, with the Rocatec system and self-polymerization at Time II (17.5 MPa, p=0.507).

Fracture analysis showed that the failure modes were adhesive throughout, with failure occurring at the ceramic-cement interface.

Table 2: *Strength of Shear Bond for the Different Groups After Air-Abrading the Zirconia Surface [Mean (MPa) and Standard Deviation]*

Material	Specimen Treatment	
	30 Minutes	14 Days/1000 TC
Fleck's zinc cement	1.1 ± 0.3	0
Fuji I	1.9 ± 0.5	0
Ketac-Cem	2.4 ± 0.3	0
Fuji Plus	5.0 ± 0.8	0.3 ± 0.4
Fuji Cem	2.5 ± 0.6	0
RelyX Luting	1.9 ± 0.3	1.5 ± 1.3
RelyX ARC	2.5 ± 0.7	1.6 ± 1.4
RelyX ARC (light-cured)	4.6 ± 0.9	4.8 ± 1.8
Panavia F	2.3 ± 0.5	5.7 ± 1.6
Panavia F (light-cured)	6.6 ± 1.7	8.3 ± 2.4
Variolink II	2.1 ± 0.6	1.0 ± 1.0
Variolink II (light-cured)	6.9 ± 1.6	2.8 ± 0.9
Compolute	2.3 ± 0.5	0
Compolute (light-cured)	6.3 ± 1.4	0
RelyX Unicem	2.8 ± 0.9	7.1 ± 2.1
RelyX Unicem (light-cured)	9.7 ± 2.1	12.7 ± 2.3

Table 3: *Strength of Shear Bond for the Different Groups After Tribochemical Silica-Coating the Zirconia Surface [Mean (MPa) and Standard Deviation]*

Material	Specimen Treatment	
	30 Minutes	14 Days/1000 TC
Fleck's zinc cement	0.8 ± 0.2	0
Fuji I	1.8 ± 0.4	0
Ketac-Cem	1.6 ± 0.4	0
Fuji Plus	4.7 ± 0.9	1.3 ± 1.1
Fuji Cem	2.9 ± 0.3	0.5 ± 0.7
RelyX Luting	2.4 ± 0.6	0.6 ± 0.6
RelyX ARC	3.5 ± 0.9	6.8 ± 2.4
RelyX ARC (light-cured)	7.7 ± 1.3	9.2 ± 2.0
Panavia F	2.4 ± 0.4	8.2 ± 1.8
Panavia F (light-cured)	6.3 ± 0.8	15.2 ± 2.5
Variolink II	3.6 ± 0.8	9.7 ± 2.6
Variolink II (light-cured)	15.0 ± 2.3	12.7 ± 3.3
Compolute	4.7 ± 1.1	8.0 ± 2.2
Compolute (light-cured)	9.2 ± 2.0	8.8 ± 3.3
RelyX Unicem	2.9 ± 0.9	17.5 ± 2.9
RelyX Unicem (light-cured)	10.4 ± 1.8	19.9 ± 2.6

Table 4: *Least-Squares Means (MPa) Calculated from Three-way ANOVA Model*

Material	
Fleck's zinc cement	0.5
Fuji I	0.9
Ketac-Cem	1.0
Fuji Plus	2.8
Fuji Cem	1.5
RelyX Luting	1.6
RelyX ARC	3.6
Panavia F	4.6
Variolink II	4.1
Compolute	3.7
RelyX Unicem	7.6

DISCUSSION

Considerable differences in shear-bond strength were seen among the tested luting cements (Tables 2 and 3). Under the conditions of this study, the results showed that it was not possible to achieve a stable bond to high-density zirconia ceramic using zinc-phosphate, standard glass-ionomer or resin-modified glass-ionomer cements. These luting cements initially offered very low shear-bond strengths after air abrading and tribochemically silica-coating the ceramic surface. The samples in these groups decreased in bond strength or debonded spontaneously within 14 days of storage in water plus thermocycling, indicating insufficient adhesion to ceramics. This indicates that the shear-bond of conventional luting cements to zirconia ceramics is unstable over the storage time.

For standard resin cements and the self-adhesive universal resin cement, the results of this study also varied widely after the two different types of surface treatment. RelyX Unicem, representing a new class of luting cements, was significantly superior to all other cements. This cement exhibited the highest shear-bond strength after 14 days of storage in water plus thermocycling, regardless of whether the ceramic surface was air-abraded or pre-treated with the Rocatec system. The results of this study also demonstrate that phosphoric acid (meth)acrylates in the RelyX Unicem formulation seems to contribute to an enhanced strength of the bond with zirconia ceramics. The bond strengths of Panavia F, a phosphate monomer (MDP) containing resin cement, with a sandblasted and tribochemically silica-coated zirconia surface, was significantly lower than that of RelyX Unicem.

Previous studies had tested durability of the bond to zirconia ceramics (94.9% ZrO_2 stabilized by 5.1% Y_2O_3; BCE Special Ceramics, Karlsruhe, Germany) using the tensile bond strength test (Wegner & Kern, 2000; Kern & Wegner, 1998). These data had shown that only the use of a resin cement containing a special adhesive monomer (Panavia) achieved a durable bond with air-abraded zirconia, compared to the dual-curing conventional BisGMA luting composite (Estiseal LC/Twinlook, Heraeus Kulzer, Wehrheim, Germany) and a chemically cured polyacid-modified resin composite (Dyract Cem, DeTrey Dentsply, Konstanz, Germany).

Given the results of this study, tribochemical silica coating significantly enhanced shear-bond strengths compared to air-abrading the ceramic surface with alumina. This confirms an earlier study in which tribochemically conditioning and silanizing resulted in an increase in bond strength. Edelhoff and others (2000) investigated the influence of different surface treatments to a zirconia ceramic (TKT Metoxit, Thayngen, Switzerland) using the three-point bending test. The highest bond strength was found after 150 days in a corrosive medium with the tribochemically silanized ceramic surface and Variolink II resin cement. In contrast to these findings, Kern and Wegner (1998) found that the Rocatec procedure did not improve the bond to zirconia ceramics and deteriorated over two years of storage and 37,500 thermal cycles with a conventional BisGMA resin composite (Estiseal LC/Twinlook). The current study demonstrated that after 14 days storage in water plus thermocycling, the establishment of a stable bond to zirconia ceramics seemed to be demonstrable in combination with the Rocatec system and the use of RelyX Unicem, Panavia F and Variolink II luting cements. Long-term tensile bond strength results with zirconia ceramics were available only for Panavia 21 (Wegner & Kern, 2000). However, a comparison of the relevant articles (Dérand & Dérand, 2000; Edelhoff & others, 2000; Wegner & Kern, 2000; Kern & Wegner, 1998) shows that the testing methods used diverged widely, making a comparison of the results obtained difficult. Differences in storage conditions for artificial aging also affect the results (Wegner, Gerdes & Kern, 2002).

Light curing of Panavia F and RelyX Unicem enhanced shear-bond strength after 14 days of storage in water plus thermocycling. This process should be used clinically for these luting cements. In particular, supragingival and accessible crown margins need to be cured with a curing light. Using this kind of treatment, the adhesive bond to the restoration can be enhanced, as full ceramic restorations offer very little frictional retention compared to metal-ceramic restorations.

This study indicated that *in vitro* environmental aging, including thermocycling, erodes the advantages claimed for conventional cements. It does not seem to be efficient to use conventional cements with high-strength zirconia ceramics to achieve durable bond strengths, regardless of the pre-treatment procedure. A number of authors (Fradeani & Redemagni, 2002;

Malament & Socransky, 2001; Sorensen & others, 1998; Groten & Pröbster, 1997) compared, in laboratory and clinical studies, the success rates of restorations inserted using conventional luting materials and restorations inserted using resin-based luting materials and adhesive procedures. However, there are lamentably few clinical long-term evaluations of the success rates of ceramic restorations regarding high-strength ceramics. Ten-year results by Ödman and Andersson (2001) have shown that 4 out of 87 conventionally cemented Procera AllCeram alumina crowns exhibited failures (fractions) within the observation period. There are no comparable long-term studies for indirect restorations with zirconia frameworks. In a study by Sturzenegger and others (2000), 22 posterior zirconia bridges were cemented using a chemically cured resin cement. During the average 385-day observation period, no framework fracture and no chipping of the veneer were noted.

The results of this study may have important clinical consequences. One must appreciate, however, that current thermocycling methods may not be an accurate predictor of *in vivo* performance. The standardized procedure in this *in vitro* investigation, that is, vertical sandblasting of the ceramic surface, is not possible within the scope of the clinical pre-treatment of full ceramic restorations. The need for controlled clinical trials with pre-treated zirconia ceramics for comparing the effect of using conventional versus resin cements is therefore evident.

CONCLUSIONS

1. Zinc-phosphate, glass-ionomer and resin-modified glass ionomer cements exhibit no durable bond with zirconia ceramics, independent of the nature of pre-treatment and the time of measurement.
2. Within the group of standard resin cements and the self-adhesive universal resin cement, shear-bond values varied greatly.
3. Using the Rocatec system, the self-adhesive universal resin cement RelyX Unicem demonstrated the highest measured data after 14 days of storage in water and thermocycling.

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Mechanical Properties of Light-cured Composites Polymerized with Several Additional Post-curing Methods

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Clinical Relevance

Employing post-curing methods on conventional composites resulted in better mechanical properties that were comparable or superior to laboratory resin, enabling them to be used in indirect restorations and resulting in a significant reduction in final treatment costs.

SUMMARY

This study determined the microhardness and diametral tensile strength of two hybrid resin composites submitted to conventional light curing, which were post-cured with different methods, and compared these data with the same data collected from one indirect resin composite. Two hybrid composites (TPH Spectrum and Filtek P60) and an indirect one (Solidex) were used. Conventional composites were polymerized with 1) conventional light curing for 40 seconds. Additional curing methods were applied with 2) laboratory multi-focal light curing for seven minutes, 3) microwave curing for five minutes at 500W, 4) oven curing for 15 minutes at 100°C, 5) autoclave curing for 15 minutes at 100°C and (6) were polymerized only with a laboratory light curing unit in three increments for three minutes and post-polymerized for seven minutes. The Solidex group was done following the manufacturers' instructions only. Diametral tensile strength and Knoop hardness tests were applied for all groups of five samples. Data were compared using ANOVA, Tukey and Student *t*-tests ($p<0.05$). Post-curing methods increased the Knoop hardness and diametral tensile strength of conventional composites. In general, Filtek P60 showed higher hardness and diametral tensile strength values than TPH Spectrum resin. The Indirect resin composite showed poorer mechanical properties than conventional composites.

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INTRODUCTION

Composites have been one of the more popular filling materials for anterior and posterior teeth. However,

when composites are used directly in posterior teeth, several problems have been identified: loss of anatomic details (Ekfeldt & Oilo, 1988), inadequate proximal contacts, high polymerization shrinkage (Ruyter & Oysaed, 1987) and lack of uniformity in the matrix conversion (Tanoue, Matsumura & Atsuta, 1999). The adequate polymerization of composites is an important factor to ensure good clinical performance. The degree to which these materials are cured is proportional to the amount of light to which they are exposed (Rueggeberg & Craig, 1988; Yearn, 1985). Under-polymerization is usually associated with inferior physical and mechanical properties, higher solubility, susceptibility to abrasion (Ekfeldt & Oilo, 1988), color instability (Ruyter, Nilner & Moller, 1987) and post-operative sensitivity from unpolymerized monomer.

Table 1. *Restorative Materials—Composition and Manufacturers*

Composite	Composition	Manufacturer
Filtek P60	**Organic Matrix**: BisEMA and UDMA **Inorganic Filler**: rounded zirconia; silica (75,9% weight or 61% volume)	3M-ESPE, St Paul, MN, USA
TPH Spectrum	**Organic Matrix**: Urethane modified and Bis-GMA **Inorganic Filler**: Barium glass and silica (74,72 weight or 57% volume)	Dentsply, Milford, DE, USA
Solidex	**Organic Matrix**: Co-polymers with multifunctional resin and UDMA **Inorganic Matrix**: Inorganic micro-fillers and organic particles (53% weight or 47% volume)	Shofu, San Marcos, CA, USA

Indirect inlay and onlay techniques have been introduced to enhance the physical properties and improve the quality of restorations in posterior teeth (Asmussen & Peutzfeldt, 1990, 1991). A growing number of photocurable prosthetic composites have been introduced. The manufacturers of these composites report that they have high mechanical and esthetical properties, suggesting their use for replacing direct composite restorations. However, the most important aspect of indirect composite systems is the possibility of using high light intensity with laboratory sources (Tanoue, Matsumura & Atsuta, 1998, 1999; Matsumura, Tanoue & Atsuta, 1999) and other post-polymerization mechanisms that associate heat, pressure or high light intensity (Tanoue & others, 1998; Rueggeberg, Ergle & Lockwood, 1997), since the composition of indirect composites is mainly the same as that of direct resin composite filling materials (Kildal & Ruyter, 1994; Tanoue & others, 1998). A number of additional polymerization procedures have been proposed, including post-curing with light radiation, secondary heating and pressure application during the polymerization process. These hypotheses suggest the use of direct filling composites in the manufacturing of indirect restorations, using alternative post-polymerization systems that produce the same or even better physical properties.

This study determined the hardness and diametral tensile strength (DTS) of two direct hybrid resin composites submitted to conventional light curing and post-polymerized with different additional curing methods and compared the behavior of these resins with the hardness and diametral tensile strength of one indirect resin composite.

METHODS AND MATERIALS

Two commercially available hybrid composites, TPH Spectrum (Dentsply, USA) and Filtek P60 (3M-ESPE, St Paul, MN, USA) and an indirect resin composite Solidex (Shofu, San Marcos, CA, USA) were used in this study (Table 1). Two tests were performed, Knoop hardness test (KHN) and diametral tensile test, in order to analyze the post-polymerization effect on the mechanical properties of the composites. Five samples were made for each combination of restorative material and polymerization system, for both tests.

Diametral Tensile Strength Test

Cylindrical specimens were prepared for the diametral tensile strength test according to ADA specification n.27. A 3.0-mm high and a 6.0-mm diameter aluminum mold was used. TPH Spectrum and Filtek P60 resin composites were inserted in three increments and polymerized in one direction only (top of the sample) using the following methods:

Group 1 and 2—conventional polymerization (CP) with XL 3000 (3M/ESPE) at 850mW/cm^2 for 40 seconds.

Group 3 and 4—CP + post-polymerization with multi-focal laboratory light source, EDGLux (EDG, São Paulo, Brazil) for seven minutes.

Group 5 and 6—CP + post-polymerization in a microwave oven (Panasonic) for five minutes at 500 Watts.

Group 7 and 8—CP + post-polymerization in an autoclave (Cristália, São Paulo, Brazil) at 100°C for 15 minutes.

Group 9 and 10—CP + post-polymerization in an oven (Quimis, São Paulo, Brazil) at 100°C for 15 minutes.

Group 11 and 12—three minutes with multi-focal laboratory light source, EDGLux, then, the final polymerization for seven minutes in the same unit.

Group 13—in this group, Solidex indirect composite was inserted in three increments, polymerized for three minutes with a multi-focal laboratory light source, EDGLux, and submitted to a final polymerization for seven minutes in the same unit.

Prior to any post-curing method, the samples were removed from the metallic mold and allowed to post-cure free of metals. The specimens were then stored in a dark container in distilled water at 37°C for 24 hours prior to testing. A compressive load was applied on the diametral surface of the samples to obtain the DTS at a crosshead speed of 0.5 mm/minute (Khan & others, 1993) in a universal testing machine, Instron 4411 (Instron Testing Instruments, Canton, MA, USA).

Knoop Hardness Test

For the Knoop hardness test, five cylindrical specimens were made as described. The samples were placed in a 3/4-inch diameter PVC ring, with their long axis perpendicular to the surface. The rings were filled with acrylic resin in order to embed the samples. This cylinder was ground with a series of silicon-carbide papers (Sof-Lex, 3M-ESPE) and polished with diamond paste to produce a smooth, uniform surface and expose the top and bottom of each sample. Knoop hardness was determined with a universal indenter by applying 50g for 30 seconds. Five indentations were made on each sample (top and bottom) and the means were calculated for each sample on the top or bottom surface.

The results of the DTS tests were analyzed initially by one-way ANOVA and Tukey test ($p<0.05$) to detect differences among indirect and direct resins. Then, two-way ANOVA (2x6) and the Tukey test were used to detect differences among the post-curing methods. The results of the Knoop hardness test on the top and bottom surfaces of each group were analyzed by student *t*-test ($p<0.05$). The means of the top and bottom, together, which represent the general values of Knoop hardness for each sample, were submitted to ANOVA and the Tukey test ($p<0.05$) in order to verify the difference among the groups.

RESULTS

Tables 2 and 3 present the means and standard deviation (SD) values of the diametral tensile strength (DTS) and Knoop hardness (KHN) as a function of the post-curing method.

The DTS one-way analysis showed differences among the groups analyzed in this study ($p=0.001$). In an overall view, the Tukey test showed that Solidex had lower DTS than TPH and Filtek P60. Two-way ANOVA was applied for DTS between the TPH and Filtek P60 resin composite groups. The Solidex group was excluded from the ANOVA (2x6) analysis to verify the difference between the effect of each post-curing method and the possible interaction between the composites and treat-

Table 2. *Mean DTS Values (SD) for the Different Curing Methods (MPa) Analyzed by Tukey Test (p<0.05)*

Curing Methods	P60	TPH Spectrum	Solidex
Conventional Light Curing	95.86 (1.85)[b]	79.54 (4.11)[b]	----------
Laboratory Light Post-curing	107.56 (6.50)[ab]	88.34 (11.75)[ab]	----------
Autoclave Post-curing	108.68 (8.11)[a]	103.12 (17.49)[a]	----------
Oven Post-curing	104.54 (6.85)[ab]	101.24 (5.43)[a]	----------
Microwave Post-curing	101.48 (12.20)[ab]	89.36 (10.57)[ab]	----------
Laboratory Light Curing	100.16 (12.81)[ab]	90.32 (4.76)[ab]	57.71 (12.66)
Means (Irrespective of the curing methods)	**103.05[A]**	**91.99[B]**	**57.71[C]**

Different capital letters denote significant differences among resin composites, irrespective of the curing method (horizontal comparison only). Different lower case letters denote significant differences among curing of each resin composite alone (vertical comparison only).

Table 3. *Mean KHN Values (SD) for the Different Curing Methods Analyzed by Tukey Test (p<0.05)*

Curing Methods	P60		TPH Spectrum		Solidex	
	Top	Bottom	Top	Bottom	Top	Bottom
Conventional Light Curing	93.94 (10.0)[b]	96.78 (8.7)[b]	60.02 (8.3)[b]	61.87 (7.6)[b]	--------	--------
Laboratory Light Post-curing	117.17 (6.0)[a]	109.68 (5.6)[a]	74.94 (8.2)[ab]	72.52 (11.3)[ab]	--------	-------
Autoclave Post-curing	118.54 (5.1)[a]	119.70 (9.8)[a]	74.82 (6.5)[ab]	68.47 (7.9)[ab]	--------	--------
Oven Post-curing	100.77 (8.4)[b]	104.70 (2.9)[b]	68.36 (6.8)[ab]	69.33 (7.3)[ab]	--------	--------
Microwave Post-curing	121.48 (11.9)[a]	115.09 (11.7)[a]	78.71 (4.7)[a]	75.82 (9.1)[a]	--------	--------
Laboratory Light Curing (alone)	92.90 (6.3)[b]	100.35 (6.6)[b]	66.54 (8.5)[ab]	65.30 (4.8)[ab]	58.75 (5.98)	55.25 (6.7)
Means—irrespective of the curing methods	**107.47[A]**	**107.72[A]**	**70.57[B]**	**68.89[B]**	**58.75[C]**	**55.25[C]**

Different capital letters denote significant differences among resin composite surfaces, irrespective of the curing method (horizontal comparison only). Different lower case letters denote significant differences among each resin composite surface (vertical comparison only).

ments. The interaction between both resin composite and polymerization methods influenced the DTS significantly. The Tukey test was applied to DTS data for the interaction between these factors, showing that, for Filtek P60, additional curing produced higher diametral tensile strength, but only autoclave post-curing resulted in a significant increase (Table 2 and Figure 1). On the other hand, for TPH Spectrum, the autoclave and oven curing methods resulted in a significant increase in DTS when compared to conventional polymerization (Table 2 and Figure 1).

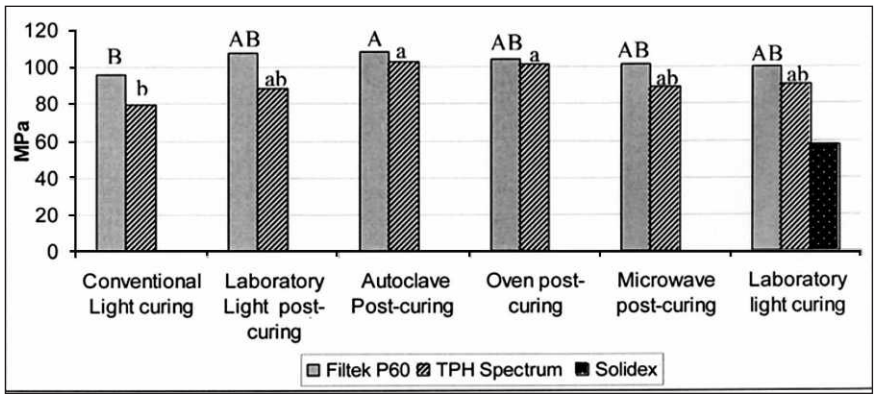

Figure 1. *Means of Diametral Tensile Strength vs curing methods (Capital letters represented the Tukey categories of Filtek P60 and lower case letters compare differences among TPH Spectrum values).*

A similar analysis was performed for KHN. In an overall analysis, the Tukey test showed that Solidex had a lower KHN than TPH Spectrum and Filtek P60. When polymerized with the conventional light-curing method, TPH spectrum produced a similar KNH (top: 60.02±8.3 and bottom: 61.87±7.6) to Solidex (top: 58.75±5.98 and bottom: 55.25±6.7), but post-polymerization resulted in higher values of KHN. The microhardness verified on the bottom and top of the samples showed no significant differences in all groups (Table 3). The Tukey test demonstrated higher values of KHN for Filtek P60 compared to TPH Spectrum, irrespective of the curing method (Figure 2).

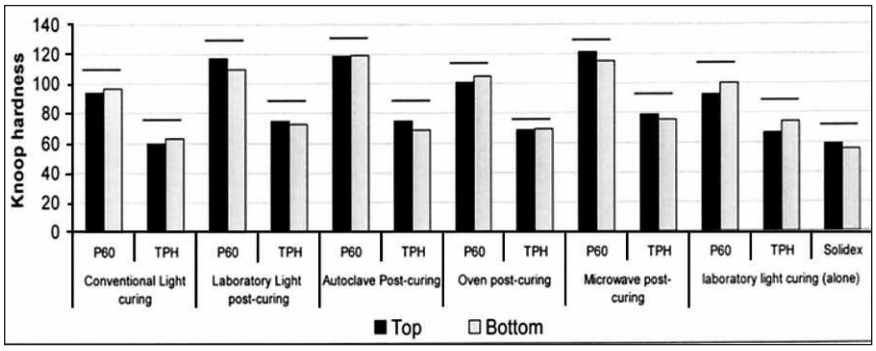

Figure 2. *Means of Knoop hardness vs curing methods for top and bottom of each restorative material analyzed by t-Student test (horizontal bars mean no significant differences).*

Table 3 represents the KHN means of Filtek P60, showing that, irrespective of the top or bottom surface, additional laboratory polymerization, autoclave and microwave post-curing methods resulted in significantly higher microhardness in relation to the conventional polymerization method. For TPH Spectrum, the microwave post-curing method produced higher microhardness than conventional curing methods with no significant differences among the remaining groups (Table 3).

DISCUSSION

Resin composites can be described as the association of inorganic particles within an organic matrix joined together by a silane-coupling agent. The composition of conventional direct hybrid composites used in this study presents similar basic components to the indirect resin composite. The use of an indirect resin should be justified by clinical benefits provided by the indirect technique, such as easier definition of a correct anatomy, better surface polishing and reduced polymerization shrinkage, for increased clinical success (Touati & Aidan, 1997).

Knoop hardness is indicative of strength against compressive loading. The indentation, according to Xu and others (2000), offers information that may be more relevant to applications that involve localized, non-uniform deformation or contact points. Such occlusal contacts occur on surface asperities or third bodies during chewing and wear. DTS provides information about bulk properties, while microhardness aims to measure the material's surface properties (Brosh & others, 1997). Both KHN and DTS are important properties of restorative materials used in posterior teeth.

Filtek P60 resin, which has a monomer phase based on Bis-GMA associated with Bis-GMA and UDMA and inorganic particles composed of zirconium glass, showed generally better values of Knoop hardness and diametral tensile strength than TPH Spectrum, which presents an organic matrix based on UDMA and inorganic particles. These results may be explained by the higher volume of inorganic fillers and the difference in monomer composition that resulted in a lower conversion degree.

Both the application of a high-intensity light source and secondary heating after irradiation are known to be effective in improving certain properties of com-

posites (Matsumura & others, 1999; Tanoue & others, 1999). Knoop hardness and DTS for Filtek P60 and TPH Spectrum increased with post-curing treatments in accordance with the findings of Khan and others (1993), Park and Lee (1996), Park (1996) and Yamaga and others (1995). However, this fact was not the same for each resin composite and all polymerization methods.

Several mechanisms have been proposed to explain the further polymerization of post-cured heated composites. The increase in the conversion degree may be an important factor that enhances the mechanical properties of composites (Park & Lee, 1996). Post-curing treatment results in increased segmental chain vibrational amplitude, allowing near radicals and methacrylate groups to collide, thus increasing monomer conversion (Rueggeberg & others, 1997). The use of a post-curing treatment immediately after the initial polymerization light is necessary to obtain this improvement (Baharav & others, 1997). The time interval between initial polymerization and post-cure application of heat has a significantly greater influence on the volume of free radicals than had already been generated during initial light-curing (Burtscher, 1993; Loza-Herrero & others, 1998).

Another factor affecting DTS may be the slow homogeneous internal stress as a result of polymerization shrinkage. Additional heat subjects the cured resin composite to an annealing process that can relieve internal stresses through increased molecular segmental movement (de Gee & others, 1990; Loza-Herrero & others, 1998).

Various post-curing treatments showed no significant difference in DTS. An important factor may have been the increase in temperature produced by post-curing methods. The four halogen lamps emit both photo and heat energy (Tanoue, Matsumura & Atsuta, 2000). Conventional light curing, laboratory post-curing heat methods and the laboratory light curing method used, alone, produced similar results, indicating that the light intensity was at least sufficient for the necessary conversion energy. Laboratory heat and light post-cure treatments were superior to conventional light curing alone.

The autoclave post-curing method tends to show higher DTS values, and this hypothesis may be explained by the loading process present in this procedure. According to Brosh and others (1997), in indirect composite techniques, pressure may be applied by a hyperbaric atmosphere. Wilson and Norman (1991) reported that the application of 6 bar pressure before light curing produced significantly fewer voids than layering or bulk packing techniques.

The oven treatment and autoclave methods were performed at 100°C for 15 minutes, because the temperature at 100°C for 10 to 60 minutes seems to be the most promising post-curing method (Peutzfeldt & Asmussen, 2000), and the laboratory unit protocol uses temperatures ranging between 95°C and 120°C, with time variations between 6 and 15 minutes (Xu & others, 2000; Khan & others, 1993; Park, 1996; Park & Lee, 1996). The microwave method, however, is indicated for composites that have a benzoyl peroxide initiator. The use of the microwave post-curing method in this study demonstrated similar behavior to other treatments, probably because the microwave energy irradiation was absorbed by monomer molecules, resulting in an increase in the conversion degree of composites (Urabe & others, 1999). At the sample surfaces, the influence of the microwave post-curing method was more easily observed, showing the highest Knoop hardness values for both direct composites. The probable and speculative hypothesis to support this factor is that the heat produced was not able to penetrate into the entire sample interior. The bulk properties of the samples were analyzed by diametral tensile strength test, which demonstrated some increase with microwave curing; however, this increase was not significant in relation to conventional curing values.

The development of techniques and materials leads to a successful indication of indirect composite restorations. When new, easy and inexpensive techniques can result in better materials' properties, dental treatment reaches the best opportunity to perform excellent health care. This work showed higher similarity among the behavior of the composites submitted to post-curing treatments, and this gives dental professionals an important clue, because the use of some equipment normally present in their offices can provide indirect composite restorations with better mechanical properties. Thus, the recommended treatment fulfills not only the esthetic requirements but also the functional necessities.

CONCLUSIONS

In accordance with the methodology developed in this study, it is possible to conclude that:

- The direct resin composites, Filtek P60 and TPH Spectrum, presented higher mechanical properties (Knoop hardness and diametral tensile strength) than the laboratory resin composite, Solidex.
- The post-curing method produced higher values of DTS and KNH for both conventional composites, but this significant increase is dependent on the interaction between the post-curing method and the composites.
- The Knoop hardness verified on the top and bottom of the samples presented similar values.

- DTS and KHN varied differently for each post-curing method. The autoclave curing method produced a greater influence on DTS, and the microwave post-curing method was the complementary polymerization that had more influence on KHN.

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Clinical Technique/Case Report

Restoring Erosion Associated with Gastroesophageal Reflux Using Direct Resins: Case Report

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Clinical Relevance

Gastroesophageal reflux disease is a systemic condition that can have an impact on the oral health by compromising tooth integrity, function and esthetics. Appropriate diagnostic and clinical management, using a conservative direct technique with resin composite is described in this article.

SUMMARY

Gastroesophageal reflux disease (GERD) is a condition where stomach acids are chronically regurgitated into the esophagus and oral cavity, resulting in pathology, such as esophagitis, varices or ulcers. Continual exposure of the teeth to these acids can also cause severe dental erosion. This condition frequently is asymptomatic, and the only evident sign may be the irreversible erosion of tooth structure. The dentist often is the first health care professional to identify the affected dentition. Knowledge of this cause and effect relationship between GERD and dental erosion will better prepare the practitioner to refer patients for appropriate diagnosis and treatment of the underlying medical condition and provide treatment for the affected teeth. This article presents a case report where dental erosion was present due to GERD. After management of the disease with medication, dental treatment of the eroded dentition is described, including diagnosis, treatment planning and restorative reconstruction.

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INTRODUCTION

Many systemic illnesses have an impact on the oral cavity. The dentist may be the first health care professional to observe the oral manifestations of early systemic disorders during routine clinical examination. Therefore, it is important that the dentist be aware of the relationship between oral findings and systemic disease in order to provide early diagnosis and intervention for the patient.

Dental erosion is an oral manifestation associated with the systemic condition Gastroesophageal Reflux (GER). GER, often called reflux, is a common gastroin-

testinal disorder characterized by transient relaxation of the lower esophageal sphincter and passive regurgitation of stomach acids into the esophagus and oral cavity (Eisen, 2001). Typical symptoms include heartburn, epigastric pain, regurgitation and dysphagia. GER has been reported to occur after heavily spiced meals, alcohol and meals high in fat content (Schonfeld, Chong & Evans, 1993). A Gallup poll estimated that most people experience reflux sometime in their lifetime, and up to 50% of adults in the United States have experienced regular episodes of reflux with only 15% of them seeking treatment (a Gallup Organization national survey, 1988). Many people have minimal symptoms with GER, this is known as "silent reflux." In this group, GER may go undiagnosed and the only sign may be erosion of the teeth.

If reflux goes untreated, it can lead to gastroesophageal reflux disease (GERD), which is a medical condition resulting from repeated assault to the esophagus by stomach acids. Manifestations of GERD can include esophageal erosion and ulcers with resultant blood loss, asthma-like symptoms and chronic cough if refluxed material is spilled into the larynx and tracheobronchial tree (Meurman & others, 1994; Kjellen & Tibbling, 1978; Bartlett, Evans & Smith, 1997). Once diagnosis is established, the purpose of medical intervention is to reduce the production of stomach acid. This can be accomplished by the use of antacids (over the counter) for mild GERD or H-2 blockers. Hydrogen ion pump inhibitors are also effective in treating GERD by blocking the production of stomach acid.

As previously noted, GERD can also have an impact on the oral health of the patient, since regurgitation into the oral cavity can be strongly detrimental to the teeth by causing severe dental erosion due acidity of the gastric content associated with a pH below 1 (Meurman & others, 1994). In dentistry, erosion normally describes the effects of nonbacterial, acid-induced tooth loss as opposed to mechanical wear. Any acid with a pH below the critical pH of dental enamel (5.5) can dissolve the hydroxyapatite crystals in enamel. Erosion interacts with mechanical wear, in that surface molecules of hydroxyapatite weakened by acid can be rubbed away more readily by mechanical factors such as occlusal wear, abrasive tongue action and toothbrushing. The destructive nature of the acid is thus potentiated (Mair, 1999).

Causes of dental erosion are classified as extrinsic or intrinsic. Extrinsic sources of erosion causing acid are mainly dietary and include carbonated beverages, citrus fruits, sport drinks, white wine and vinegar and some medicines, such as effervescent or chewable Vitamin C (Järvinen, Rytömaa & Heinonen, 1991).

Intrinsic causes of erosion are primarily associated with gastric acid regurgitation (Järvinen & others, 1991). Gastric refluxate has a pH of less than 2.0 and thus can readily cause dental erosion (Lazarchik & Filler, 2000). This acid regurgitation can result from recurrent vomiting as a result of psychological disorder, anorexia and bulimia nervosa (Milosevic & Slade, 1989), in addition to gastroesophageal reflux disease (Bartlett, Evans & Smith, 1996).

CLINICAL MANIFESTATION AND DIFFERENTIAL DIAGNOSIS

Patients exposed to intrinsic acids show more tooth wear on the lingual surfaces of maxillary teeth. It has been suggested there is a threefold reason for this pattern. First, there is the force of the gastric juice itself as it passes into the mouth. Second, the maxillary teeth are further away from the major salivary glands than other teeth, thus, the acidity is not buffered as readily. Finally, the tongue may propel and maintain the regurgitated acid against the palatal surfaces of maxillary teeth (Bartlett & others, 1996). Erosion of the occlusal and buccal surfaces of mandibular teeth occurs in more severe cases. Barron and others (2003) described the unesthetic effect that thinned enamel produces on eroded teeth with a concurrent "yellowish hue" and the appearance of a chamfer margin of a full coverage restoration. Hypersensitivity to hot, cold, sweet and tactile stimuli due to exposure of the dentinal tubules can also be found in patients with eroded teeth. Consequently, the authors stated, "Additional sequeale of dental erosion include compensatory eruption, tipping and drifting of teeth, formation of diastemae, loss of vertical dimension, overclosure and bite collapse, all of which result in auto-rotation of the mandible and a reduction of overjet towards or beyond an edge-to-edge incisal relationship. This sequelae can be exacerbated if attrition from bruxism is superimposed upon erosion or if either the acidic oral environment or pre-existing or continuing erosion increases susceptibility to caries."

The dentist should recognize the clinical manifestations of erosion as compared to mechanical wear (abrasion) to assist in the differential diagnosis, with consideration of GERD as a possible cause of erosion. This is particularly important in cases of "silent reflux." Erosion is different from abrasion in both appearance and etiology. Tooth surfaces affected by erosion have a smooth, spoon-shaped appearance, while lesions caused by abrasion appear sharp, flat and angular. Moreover, since erosion does not affect metal or plastic dental restorations as does abrasion, these remain as prominent, elevated plateaus (Bouquot & Seime, 1997). Erosion associated with reflux occurs on both the facial and lingual surfaces of teeth. The pattern of erosion caused by intrinsic acid may be modulated by the protective influence of the tongue, which forces regurgitated acid over the tongue, along the palate and into the buccal vestibule (Lazarchik & Filler, 2000).

Damage caused to the dentition by GERD depends on the severity of the case (the presence and frequency of regurgitation and the duration of the reflux disease). Bartlett and others (1996) found that, in the majority of cases, the occurrence of pathological reflux takes place during the day. Often, these patients have associated symptoms. These findings are consistent with many other studies. If the regurgitation of gastric juice occurs at night, however, when salivary flow is at its lowest, the potential for damage to the teeth increases greatly (Smith & Robb, 1989). Often, the erosion is more pronounced in the buccal vestibule of the side the patient sleeps on. In addition, the patient is less likely to be symptomatic or aware of having reflux.

TREATMENT

The immediate goal in the treatment of dental erosion resulting from GERD is formulation of the correct differential diagnosis and prompt referral to a gastroenterologist for definitive diagnosis and treatment to prevent further dental erosion and other pathology. In the meantime, it is important to provide symptomatic relief from dental pain, protect the remaining tooth structure and promote remineralization. Daily use of a neutral fluoride gel or paste (PreviDent 5000 Plus, Colgate Oral Pharmaceuticals, Inc, Canton, MA, USA) is very effective in reducing sensitivity and preventing demineralization.

Once GERD has been diagnosed and the condition brought under control, a minimally invasive approach is taken in the restoration of eroded teeth (Magne & Belser, 2002). Not all lesions resulting from erosion require restoration (Dahl, Carlsson & Ekfeldt, 1993). The dentist should consider restorative treatment when, first, the structural integrity of the tooth is threatened, second, the tooth (dentin) is hypersensitive, third, there is significant loss of tooth structure, vertical dimension and/or function, fourth, the defect is esthetically unacceptable to the patient, and finally, a pulpal exposure is likely (Lambrechts & others, 1996).

The material selection and restorative approach should preserve the natural tooth whenever possible. Many anterior and posterior teeth can be treated conservatively with directly applied resin composites, especially if enamel margins can be maintained (Barron & others, 2003). With the extensive loss of tooth structure and a vertical dimension of partial or complete coverage, indirect restorations are indicated. This may include more conservative resin or ceramic onlays or veneers or full coverage crowns may be required (Magne & Belser, 2002).

The following clinical case describes a conservative approach to restoring dental erosion resulting from GERD using direct resin composite. After management of the disease with medication, dental treatment, including diagnosis, treatment planning and restorative reconstruction of the eroded dentition was completed.

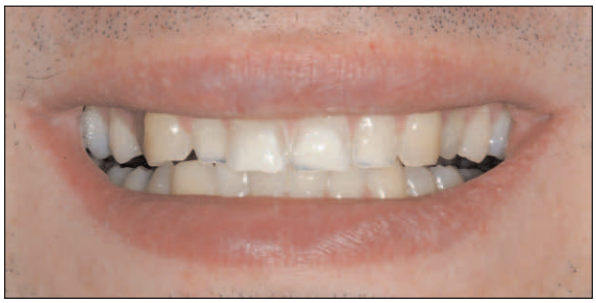

Figure 1. *Preoperative smile view. Based on the gastrointestinal symptoms and denial by the patient of bulemia, and frequent intake of acidic food or beverages, gastroesophageal reflux was included in the differential diagnosis.*

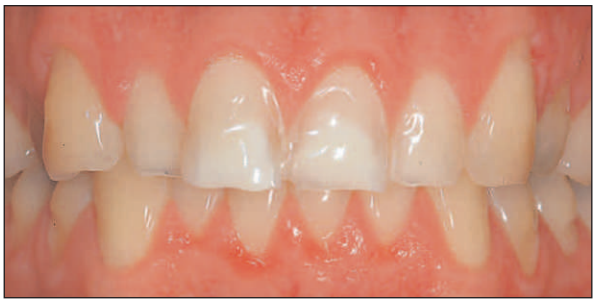

Figure 2. *Preoperative facial view of severe enamel and dental erosion with thin and chipped incisal edges of maxillary anterior teeth and wear of the cervical third of premolars.*

CASE PRESENTATION

A 30-year old Caucasian male presented to the clinic with the chief complaint, "My front teeth are chipping; I want to get them fixed." The patient was alert and normally developed and denied any systemic condition. The patient had no history of smoking and was currently taking no medications; however, the patient reported recent GI problems (heartburn, stomach pain). The patient's past dental history involved intermittent dental care, including root canal therapy, crown, amalgam restorations and extraction of the third molars. Clinical examination revealed severe generalized loss of enamel and dentin (Figures 1 and 2). The patient denied bulimia or the frequent intake of acidic foods or beverages. Based upon the patient's GI symptoms, clinical and subjective findings, gastroesophageal reflux was included in the differential diagnosis.

In order to establish a definitive diagnosis of reflux (GER) as the cause of the dental erosion, the patient was referred to a gastroenterologist. The best predictor for an extra-esophageal problem due to reflux disease is the subjective symptom of regurgitation, which the patient did not have. However, the patient did report

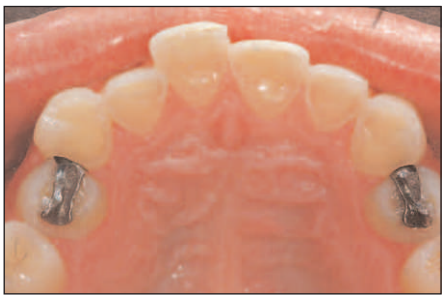

Figure 3. *Maxillary incisal-occlusal view. The pattern of erosion caused by intrinsic acid has a smooth spoon-shaped appearance on the lingual surfaces of the teeth.*

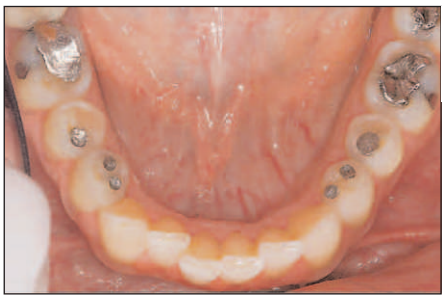

Figure 4. *Occlusal view of mandibular arch revealed generalized dental wear of enamel on occlusal premolars and molars.*

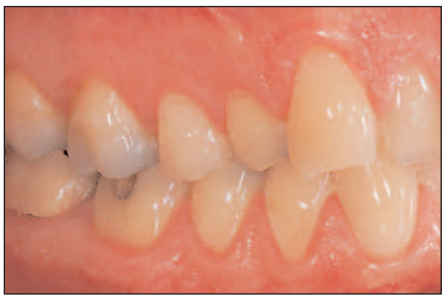

Figure 5. *Lateral right view in occlusion showing a Class I occlusal relationship with slight rotation of the maxillary and mandibular teeth. Spoon-shaped and polished surface lesions were found on maxillary and mandibular premolars and molars.*

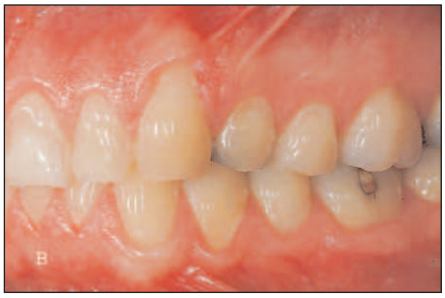

Figure 6. *Lateral left view in occlusion. Notice the spoon-shaped lesions on the cervical margin of the maxillary and mandibular premolars.*

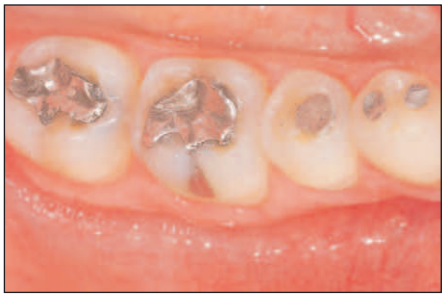

Figure 7. *Occlusal view of the mandibular left quadrant showing the diagnostic "proud" occlusal amalgam in the premolars and molars, maintaining the patient's vertical dimension.*

heartburn with intestinal discomfort, which is indicative of GER. Many patients are either asymptomatic or the reflux occurs at night when they are unaware of symptoms. Since the subjective findings were inconclusive, the patient underwent esophageal manometry, followed by a dual channel 24-hour pH study to confirm the diagnosis of reflux (GER). The manometry did not reveal inflammation or ulceration of the esophagus, which would have clearly demonstrated excessive reflux. In the absence of mucosal changes, 24-hour monitoring of esophageal pH is the most useful tool currently available for diagnosing GERD (Sontag & others, 1990). A catheter is passed through the nares to a point 5-cm above the lower esophageal sphincter. The condition is considered pathologic when pH in the distal esophagus remains below 4.0 for more than four percent of the time (Bartlett & Smith, 1996).

Clinical examination revealed generalized dental wear of enamel on the lingual and facial maxillary anterior teeth (Figures 3 and 4) and on the occlusal, buccal and lingual surface of maxillary and mandibular premolars and molars (Figures 5 and 6). The patient had a Class I occlusal relationship, with slight rotation of the maxillary and mandibular teeth. In spite of the erosion and generalized dentin exposure, no sensitivity was reported. The erosion affected both sides of the maxillary and mandibular arches with the same severity. In addition, there was excessive wear of the incisor edge of the maxillary canines, suggesting a bruxism habit.

The loss of tooth structure was primarily observed on the buccal and occlusal surfaces of both premolars and molars. The patient's vertical dimension was maintained by previously placed posterior occlusal amalgam restorations (Figure 7) and a crown on the right second mandibular molar. The shade of the teeth ranged from A2 to A3.5 (Vitapan System, H Rauter GmH & Co KG, Bad Säckingen, Germany), with the maxillary central incisors exhibiting the most variation in shade. There was no discrepancy in the gingival margin of anterior maxillary incisors.

Diagnostic data that consisted of preoperative photographs, a complete radiographic survey, a detailed clinical examination, alginate impressions for diagnostic models, a face-bow measurement and interocclusal records were obtained.

TREATMENT PLAN

The models were articulated on a semi-adjustable articulator (Hanau Articulator, Teledyne, Buffalo, NY, USA) and a diagnostic wax-up was made to determine optimum treatment. Direct resin composite veneers using two types of resin composites, a hybrid and a microfill, were proposed for the anterior maxillary teeth to re-establish esthetics and function (#4 to 13). Restorations for the cervical, saucer-shaped lesions in premolars and molars were proposed using a microfill resin composite. All-ceramic onlays were treatment planned as the final restoration for maxillary and mandibular posterior

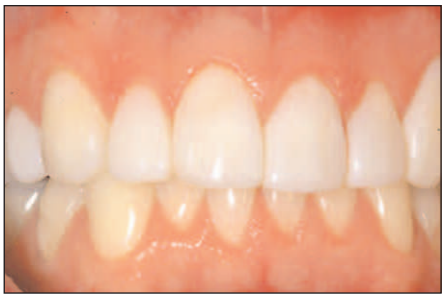

Figure 8. *Facial view in intercuspal-centric occlusion of the labial veneers.*

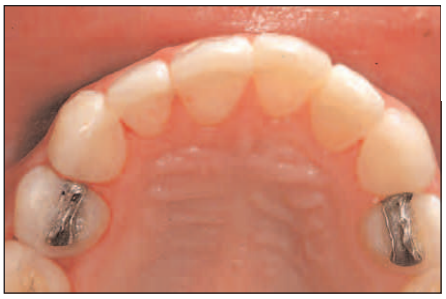

Figure 9. *Maxillary-incisal-occlusal view showing veneers from the lingual perspective.*

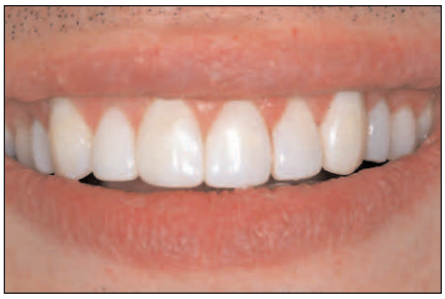

Figure 10. *Smile view of the maxillary direct-facial veneers.*

teeth; however, the patient declined this treatment option due to limited finances. A Centric relation splint was prescribed in order to protect the final restorations from posterior wear due to the patient's bruxism habit.

TREATMENT SEQUENCE

Treatment was divided into the following phases: 1) at-home bleaching; 2) restoration of the maxillary anterior teeth with direct resin composite veneers and 3) restoration of the cervical lesions using direct resin composite. At-home bleaching was carried out for 14 days. The maxillary and mandibular teeth lightened to a lighter shade than B1 (The Vitapan System). Two weeks after completion of the bleaching, #7, 8, 9 and 10 teeth were conservatively prepared with flame-shaped diamond burs (#7901, Brasseler USA, Savannah, GA, USA) and a football-shaped diamond bur (#7004, Brasseler USA). Sufficient enamel remained to justify a conservative approach and ensure adequate adhesion. A dentin adhesive was then placed (SingleBond, 3M/ESPE, St Paul, MN, USA) according to manufacturer's instructions. In terms of the restoration of the maxillary central and lateral incisors and canines, a hybrid resin composite (Herculite XRV, Kerr Manufacturing Co, Orange, CA, USA, Shade B1) was utilized for dentin replacement. All layers of the resin composite were light cured with an LED curing light (LEDemetron, Ultradent, South Jordan, UT, USA) for a total of 20 seconds. In order to mask a significant color discrepancy on the facial surface of the central incisors, an opaquer (Creative Color, Cosmodent, Chicago, IL, USA) was then placed. Next, translucent incisal resin composite was placed at the incisal edges of the maxillary central incisors (Herculite XRV, Kerr Manufacturing Co, Orange, CA, USA). For enamel replacement, a microfilled resin composite (Durafill VS, Heraeus Kulzer Inc, Armonk, NY, USA, Shade "Superlite") was placed on the buccal surface, while a hybrid resin composite (Herculite XRV, Kerr Manufacturing Co, Shade B1) approximately 0.75-mm thick was placed on the lingual surface (Figures 8, 9 and 10). Anterior guidance was not modified. The cervical lesions on the premolars were restored with microfill resin composite (Durafill VS, Heraeus Kulzer Inc). New alginate impressions were taken of the restored dentition and mounted in centric relation in a semi-adjustable articulator (Hanau Articulator, Teledyne) and an occlusal splint was made.

MEDICAL TREATMENT

Once the diagnosis of GER was established by the gastroenterologist, the patient was treated with hydrogen ion pump inhibitors (PPI)—lansoprazole once a day (Prevacid, TAP Pharmaceutical Products Inc, Lake Forest, IL, USA) and over-the-counter antacid-H_2 receptor antagonists—twice a day (Zantac 150 mg, GlaxoSmithKline, Research Triangle Park, NC, USA). Lansoprazole decreases gastric acid secretion by inhibiting H^+/ K^+ -adenosine triphosphatase (commonly called the proton pump), thereby, blocking the final common step in the secretion of gastric acid. H_2 antagonists decrease histamine-stimulated acid secretion, which renders the refluxate less damaging to the gastroesophageal mucosa. Four H_2 antagonists (cimetidine, ranitidine, famotidine and nizatidine) are currently approved by the Food and Drug Administration for symptomatic treatment of GERD. Each is available without a prescription for relief of heartburn and reduces acid secretion at half the dose approved for symptomatic treatment of GERD. The H2 antagonists are generally safe and well tolerated, with the overall incidence of drug-related adverse events low and their severity mild (Fendrick, 2001).

DISCUSSION

GERD is under-diagnosed according to the available data from population studies, due to a lack of a universally accepted definition of GERD and an awareness of the symptoms (Björkman, 2001). Most patients with GERD never seek care, because of the perception that their symptoms are the result of their lifestyle choices, which can be easily remedied by numerous over-the-counter medications. For that reason, dentists can be the first health providers to identify GERD through erosion of tooth structure and clinical manifestations of pathological reflux. Once identified, it is necessary to

have a medical follow-up to monitor the recurrence of GERD so that the successive erosion of healthy tooth structure and the risk undermining the restorations already placed can be prevented. Dental treatment in the affected teeth improves patient oral hygiene maintenance and reduces the thermal sensitivity that many patients experience due loss of tooth structure. It also helps to prevent pulpal involvement, dentifrice abrasion, acid erosion, food impaction and discomfort of the tongue and cheeks. In addition, esthetics are improved and the teeth can be strengthened (Grippo, 1992). Two factors must be considered when choosing a dental material for restoration: the wear resistance of the material and the potential for the material to cause further iatrogenic damage to the opposing dentition (Mair, 1999). Metals are the best materials for repairing worn surfaces, because they maintain a smooth surface that reduces friction, and they are unlikely to damage the opposing teeth. Other materials include resin composite, glass ionomer, amalgam and porcelain. Of the esthetic materials, porcelain provides the best wear resistance. The current generation of small particle composites have relatively good wear resistance, but this resistance is not as good as metal surfaces. Glass ionomer should not be used to restore worn surfaces, because the matrix is too soft and the glass particles easily dissolve in an acidic environment.

Persistent regurgitation greatly increases the potential for damage to teeth. Therefore, it is important to diagnose and treat the underlying etiology in order to stop dental erosion and for the long-term success of any dental restoration. The provision of definitive restorative procedures in the continuing presence of oral acid will lead to premature failure of treatment and will likely leave the patient in worse condition. However, even when the cause of dental erosion cannot be ascertained, some form of reconstruction may be necessary.

It is the practitioner's challenge to identify a history of reflux symptoms in dental patients who have erosion when no other intrinsic or extrinsic etiology can be identified. The most likely group for the dentist to diagnose is those patients with GERD who remain undiagnosed or who self-medicate. Some patients may have other symptoms of GER that were not severe enough to seek medical treatment. These could include periodic symptoms of heartburn, epigastric pain or regurgitation with or without recurrent hoarseness and laryngitis. Dental erosion should signal the dental practitioner to the possibility that a systemic pathological process may be occurring. Other possible causes of dental erosion, such as bulimia, anorexia nervosa, alcoholism or rumination, need to be investigated and ruled out prior to initiating definitive therapy. It is essential that the dentist thoroughly interview patients and provide early referral to a gastroenterologist for definitive diagnosis and treatment when GER is suspected.

CONCLUSIONS

A systemic illness, such as GERD, can cause significant destruction of tooth structure over a relatively short period of time. The patient may not even realize the connection between the systemic illness and oral manifestations. These proposed guidelines will help practitioners provide proper diagnosis of this medical condition, which is essential to ensuring the long-term viability of dental treatment and approaching a conservative restorative technique.

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Immediate Esthetic Management of a Catastrophically Fractured Anterior

RJ Eckert Jr • WJ Dunn • JS Lindemuth

Clinical Relevance

This technique presents a method for the expeditious management of a catastrophically fractured anterior tooth.

INTRODUCTION

The patient who arrives at the end of the workday with a catastrophic fracture of a tooth presents both an esthetic and time management challenge to the practitioner. The great majority of these cases will be cuspal fractures of posterior teeth that can usually be addressed by the simple application of glass ionomer cement, since treatment would not involve an esthetically sensitive area (Carroll, 1999). When an anterior tooth is involved, there is often sufficient tooth structure remaining to reattach the fractured segment (Farik & others, 2002; Garcia-Ballesta & others, 2001; Small, 1996; Maia & others, 2003; Vissichelli, 1996). However, a patient will occasionally present with a significant portion or all of the clinical crown missing. Traditional temporary treatment options can be time consuming and unpredictable (Maia & others, 2003; John, Prabhu & Munshi, 1998). Techniques that have been previously described include fabrication of a composite post and core/crown (Howell, 2003), a modification of Croll and Helpin's (2002) technique of using orthodontic wire and compomer to act as a temporary splint (or, in this case, a fixed partial denture), fabrication of an interim removable partial denture, bonding of a natural tooth pontic and the use of a thermoplastic retainer with composite to replace the missing tooth structure (Blake, Garvey & Fleming, 1998). This paper presents a technique for the immediate interim restoration of a catastrophically fractured maxillary anterior tooth that is fast, non-invasive, esthetic and allows for some limited function without fear of aspiration of tenuously bonded temporary restorative materials.

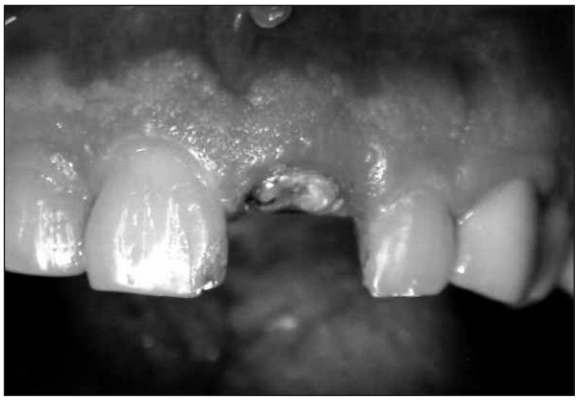

Figure 1. *Complete crown fracture at the level of the gingival margin.*

TECHNIQUE

A 47 year-old patient presented for treatment on a Friday at the end of the workday with a complete frac-

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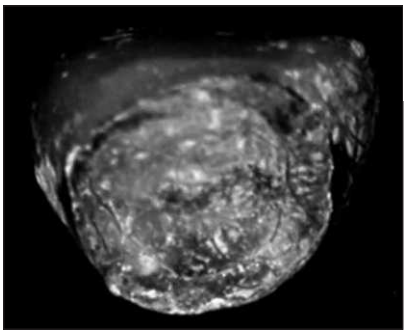

Figure 2. *Internal aspect of the failed crown reveals fractured dentin.*

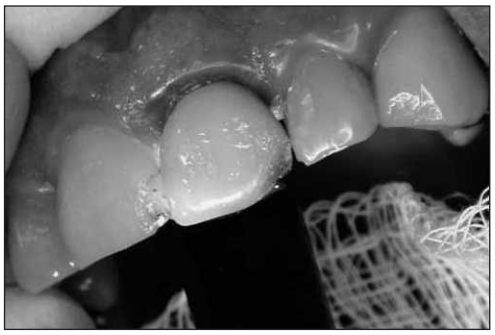

Figure 3. *Temporarily bonding the segments together.*

ture of the clinical crown on tooth #9 (Figure 1). The patient had unsuccessfully attempted to repair the tooth with an over-the-counter temporary dental filling material. The patient stated that he was not in pain. The tooth had a porcelain fused-to-metal (PFM) crown that was placed more than 30 years ago. The patient stated that he had been hit in the mouth approximately 10 years ago, fracturing the disto-incisal angle of the crown.

Examination of the patient's health history revealed no contraindications to dental treatment. Intraoral examination revealed complete fracture of the clinical crown on tooth #9 at the level of the gingival margin. All other clinical findings were unremarkable. The residual material within the crown appeared to be dentin at the level of the fracture (Figure 2). The exposed surface of the root had evidence of the formation of secondary dentin in the previous canal space. The tooth was not sensitive to manipulation by dental instruments or to rinsing with the air/water syringe. A periapical radiograph of tooth #9 revealed a 2-mm diameter apical radiolucency, no evidence of radicular fractures and sufficient root length and attachment apparatus for restoration. A diagnosis of the coronal fracture and pulpal necrosis with chronic apical periodontitis was made. A pulpal exposure was not observed. The patient was informed of the diagnosis and treatment options were presented. The patient wanted to save his tooth and chose to pursue non-surgical root canal therapy, custom dowel and core, crown lengthening and a full coronal restoration.

The patient's immediate treatment concern was the appearance of missing tooth #9. A plan for the comprehensive restoration of tooth #9 had been accomplished, but the plan for the immediate replacement of the missing central incisor prior to the start of the weekend had not yet been resolved. There are several treatment options available for restoring esthetics and function in this case, as described in the introduction. In selecting the appropriate option, the prudent practitioner should also evaluate the patient's ability to comprehend and follow instructions that are specific to each treatment technique. In some applications, a fixed interim restoration may be most appropriate. In this application, the authors determined to proceed with a removable appliance. The patient was still overwhelmed by the catastrophic failure of his tooth just hours earlier and confided that the condition was humiliating. We wanted to provide the patient with an approach that would allow some latitude in deciding on various treatment options once he recovered from the initial esthetic shock of losing his crown. We also wanted to restore the patient's appearance and confidence in the least invasive way in the shortest amount of time, without putting the patient at risk with any short-term procedure that might involve aspiration of tooth fragments.

In this particular case, the fractured segment was secured, but the prognosis and reliability of bonding the segment to the root was unfavorable, because it was too large. Similarly, the prognosis of splinting the PFM crown to the adjacent teeth would be unpredictable, because only a limited amount of composite would be available for splinting in the interproximal areas without compromising esthetics. Splinting would require an adequate bulk of heavy filled composite to provide enough resistance to overcome the forces of mastication. Furthermore, both of these treatment options would have placed the patient at risk for aspiration of the tooth segment. A provisional dowel-crown could also be performed since the comprehensive plan called for endodontic therapy, post and core and a crown, but this option would only be available after a considerable amount of chair time to initiate endodontic therapy. Fabrication of an immediate transitional, removable partial denture replacing tooth #9 would require a significant amount of time in the laboratory. The treatment option chosen was a modification of the technique described by Blake and others (1998), which involves a diagnostic wax-up and use of an Essix retainer.

Treatment required obtaining an impression with the fractured crown in place. Both the retained root segment and the displaced segment were etched with 34% phosphoric acid gel (Dentsply Caulk, Milford, DE, USA) for 20 seconds, followed by water rinse for 20 seconds. A light blast of air was applied to the etched and rinsed tooth surfaces to remove excess water without desiccating the dentin. A combination primer/nanofilled adhesive (Optibond Solo, Kerr, Orange, CA, USA) was applied to the moist dentin surfaces and thinned with a gentle stream of air. A 4x4-inch gauze was placed as a safety net posterior to the operating field. The displaced segment was carefully oriented into position and

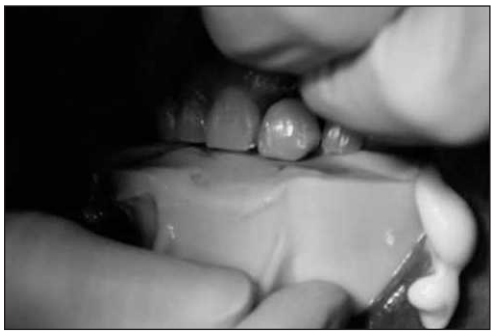

Figure 4. *Alginate impression of maxillary arch with fractured segment in place.*

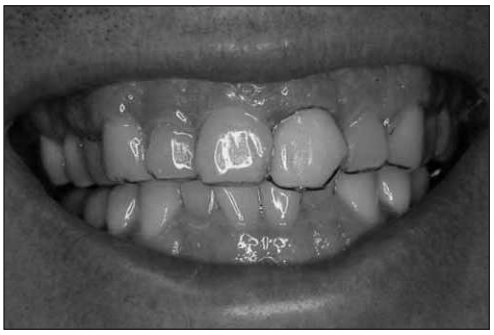

Figure 5. *Modified Essix appliance 18 minutes after initial patient assessment.*

photopolymerized (Optilux 501, Kerr) at the gingival margin from the buccal and lingual direction for 40 seconds each (Figure 3). The patient verified that the tooth was in the original location. Alginate impression material (Jeltrate regular set, Dentsply Caulk) was mixed according to manufacturer's recommendations and an impression of the patient's maxillary arch was made (Figure 4). Removal of the impression did not dislodge the crown. The fractured crown segment was easily removed by hand by applying a twisting force to the temporarily bonded segment. No composite was applied to the segments before polymerization, so the bond was easily overcome. The impression was disinfected and poured with quick setting stone and slurry water. An Essix vacuum-formed retainer was constructed from teeth #5 through #12 using Splint Biocryl material (Great Lakes Orthodontics, Tonawanda, NY, USA) on a Biostar machine (Scheu Dental, Iserlohn, Germany). The Essix retainer was disinfected, rinsed, then placed in the patient's mouth without the crown segment in place to determine fit and comfort. The patient was then instructed in the removal and replacement of the retainer. The crown was placed in the retainer and the crown segment snapped into place due to undercuts on the crown and excellent adaptation of the thermoplastic matrix. The completed appliance was inserted to evaluate final fit and comfort (Figure 5). The patient was reminded that the appliance was for esthetic and phonetic purposes only and was not to be considered a permanent treatment option. Total treatment time for this procedure, including laboratory time, was 18 minutes. A further advantage to this procedure is that the retainer can be used as a matrix for fabricating the interim dowel/crown at a subsequent appointment.

SUMMARY

A quick, failsafe method for the esthetic replacement of a catastrophically fractured anterior tooth was presented. This method required minimal armamentarium and no anesthesia. In addition, it employed a technique that is not technique sensitive. The patient and the dental team both benefit by resolving the esthetic crisis using simple methods and excellent time resource management.

(Received 1 June 2004)

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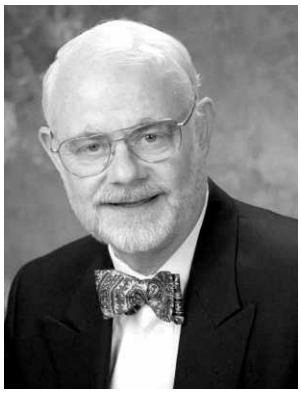
Academy of Operative Dentistry
Award of Excellence
Dr James B Summitt



James B Summitt

Dr Summitt received his DDS degree from the University of Tennessee in 1971. He completed a two-year General Dentistry Residency at Wilford Hall Medical Center and received his MS degree from the University of Texas Dental Branch in Houston. In 1984, after three years of teaching in the general dentistry graduate program, he became chairman of the Department of General Dentistry and director of the two-year General Dentistry Residency at Wilford Hall. In 1988, he became director of Dental Services at Wilford Hall. He retired from the Air Force in 1989 and joined the Department of Restorative Dentistry at the University of Texas Health Science Center at San Antonio. In 1991, he was named head of the Division of Operative Dentistry and subsequently was named chairman of the Department. Dr Summitt is actively involved in research in the areas of current restorative dentistry techniques and materials, including adhesive systems and resin composites and has published more than 130 scientific articles and abstracts.

Dr Summitt serves on the editorial boards of *Operative Dentistry*, *The American Journal of Dentistry* and the *Journal of Evidence Based Practice*. He is the lead author of a textbook, *Fundamentals of Operative Dentistry–A Contemporary Approach*. He is a diplomat of both the Federal Services Board of General Dentistry and the American Board of General Dentistry and is past president of the American Board of General Dentistry. He is a fellow in the International College of Dentists and a Master in the Academy of General Dentistry.

During his many years of teaching, Jim has received numerous awards. He has been honored with several teaching awards from dental students, including numerous selections as an assistant marshal for graduation ceremonies. In 1993, he was awarded the most prestigious honor at the Health Sciences Center at San Antonio, The Presidential Award for Teaching Excellence.

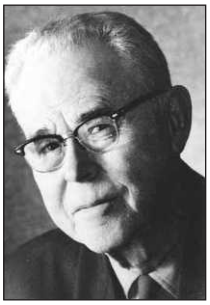
Dr Jim Summitt has served as teacher, colleague, mentor, role model and friend to many members of our Academy. It is therefore an honor for the Academy to present the Award of Excellence in 2005 to our friend and consummate teacher, Dr Jim Summitt.

Dr Fred Eichmiller

Academy of Operative Dentistry

Hollenback Memorial Prize

Dr Stephen C Bayne



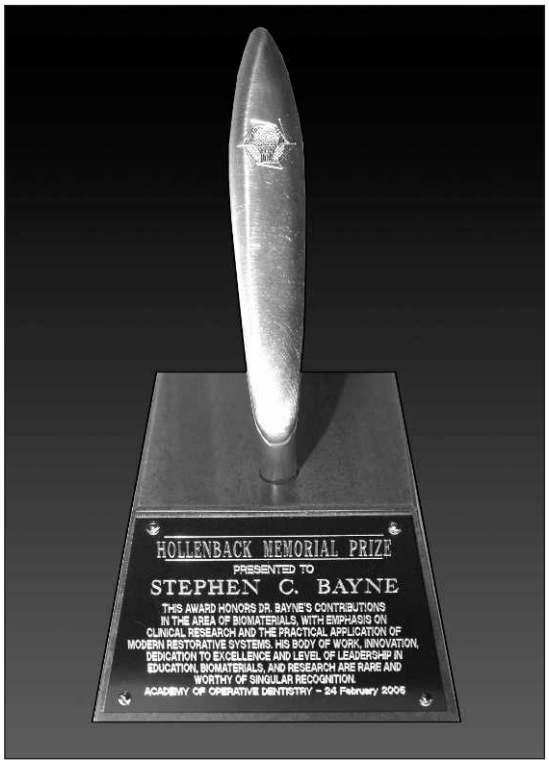
George Hollenback

Stephen C Bayne

Dr Stephen C Bayne is professor and section head of Biomaterials in the Department of Operative Dentistry at the University of North Carolina School of Dentistry. Dr Bayne has been an international figure in the field of dental biomaterials and clinical research, with special emphasis on operative dentistry, over a period of more than 20 years. His work has had a profound impact on the understanding, knowledge and clinical application of, in particular, modern restorative systems.

Stephen Bayne graduated from NU Graduate School in 1978 with the goal of becoming an entrepreneur in the field of dental materials. Resisting the offer of a position as R&D director in an established company and determined to go it alone, Stephen set about the difficult task of raising capital to start his own company. A visit, based on a whim, to the fledging University of Mississippi resulted in a change of direction. The temptation of an exciting, once-in-a-lifetime opportunity to plan biomaterials teaching and research from the ground up was too great. Seven years later, in 1984, and not withstanding an entrepreneurial venture into the emerging field of computers, came Stephen's move to UNC—a great opportunity to enter full-time clinical research. Concurrently, the national scene was calling and part of Stephen Bayne became the dental politician par excellence.

In all his subsequent work, Stephen Bayne has been innovative, meticulous and, possibly most important, inspirational. Concurrent with his success in both laboratory and clinical research, Dr Bayne has held a succession of important national and international positions of ever increasing significance, including chair, Biomaterials Section, American Dental Education Association; president, Dental Materials Group of the International Association for Dental Research (IADR); president, American Association for Dental Research and, most recently, vice president of the IADR, which he will lead as president in 2006. Such distinction is rare, but richly deserved by Stephen Bayne for his outstanding qualities in research leadership, tempered by his considerable political acumen. Notwithstanding these qualities, Dr Bayne is a teacher of the highest caliber, with many awards of excellence, notably the University of North Carolina 2004 Professor of Excellence Teaching Award.



Throughout his career, Stephen Bayne has been eminently successful in creating user-friendly research systems and environments to promote dental biomaterials, advance clinical research and have education and research joined at the hip. But Stephen Baynes' life is not limited to his work. He loves a good time, is passionate about technology, draws constantly as a closet cartoonist and always cherishes people more than things, in particular, those nearest and dearest to him in his family.

International standing at the level achieved by Stephen Bayne is hard-won and most worthy of singular recognition. The Academy of Operative Dentistry is most pleased to recognize such outstanding achievement, with the award of its prestigious Hollenback Memorial Prize. With this award, Stephen Bayne, who continues to enthuse and inspire as a great leader in his field, joins the elite group of individuals who individually and collectively have expanded the frontiers of operative dentistry. To use Stephen's own catch phrase, if we "keep the faith," operative dentistry will continue to be a cornerstone of the everyday clinical practice of dentistry and, in turn, a key element of dental education and modern research.

The Academy of Operative Dentistry salutes and congratulates Stephen Bayne on his outstanding achievements and continuing contributions to new knowledge and understanding in the advancement of restorative dentistry.

Nairn HF Wilson

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Classifieds: Faculty Positions

Tenure Track Faculty Position
in Operative Dentistry
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University of Saskatchewan

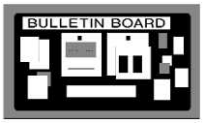
The College of Dentistry is seeking applications for a full-time tenure-track position at the Assistant/Associate Professor level in the Division of Operative Dentistry. Candidates should have a strong commitment to teaching and research. Opportunities for dental materials research exist in the college and on campus, including the Canadian Light Source (synchrotron). A new clinical simulation and six-chair senior student/graduate practice clinic are in place. Candidates with practice experience in operative dentistry and graduate qualifications at the Masters or PhD level are preferred. Responsibilities will include didactic and clinical instruction in operative dentistry for undergraduate students. On site private practice privileges are available. Start date is July 1, 2005 or when a suitable candidate is found. Rank and salary are commensurate with experience and qualifications. The University is committed to Employment Equity. Members of designated groups (women, Aboriginal people, people with disabilities and visible minorities) are encouraged to self-identify on their applications. All qualified candidates are encouraged to apply; however, Canadians and permanent residents will be given priority. Further information about our college and its programs are available at www.usask.ca/dentistry.

A letter of application, accompanied by a curriculum vitae, professional credentials, a statement of teaching and research interests and the names of three references should be sent to:

Dr James E Stakiw
College of Dentistry
105 Wiggins Road
University of Saskatchewan
Saskatoon, Saskatchewan S7N 5E4
Telephone: (306) 966-5122
Fax: (306) 966-5132
e-mail: james.stakiw@usask.ca

Applications with complete documentation will be accepted until May 31, 2005 or until a suitable candidate is found.

Announcements



Ralph Phillips Student Research Award
2006

Applications are invited for the 2006 Ralph Phillips Student Research Award, sponsored by the Founders Fund of the Academy of Operative Dentistry. The Award is open to students at all levels wishing to undertake novel research relevant to contemporary operative dentistry.

The application should take the form of a protocol outlining the background, aims and hypothesis of the proposed research, the methodology to be employed, the anticipated work schedule and the expected impact of the work on the clinical practice of operative dentistry. The protocol should not exceed three double-spaced, typewritten pages and a budget sheet, including details for payment of the award. The budget for the proposed research may not exceed $6,000.

The recipient of the award will be required to present a table clinic reporting the findings of the research supported by the Award at the 2007 annual meeting of the Academy of Operative Dentistry to be held in Chicago, 22-23 February, 2007. Additional funds not to exceed $1,000 will be made available to offset the cost of attending the meeting.

A research mentor, who is a member of the Academy of Operative Dentistry, should be named and be a co-signatory to the application.

If the research supported by the Award leads to a research report intended for publication, it is expected that the report be submitted in the first instance to *Operative Dentistry*.

Applications for the award must be submitted electronically to Dr Nairn Wilson (nairn.wilson@kcl.ac.uk), Chairman of the Research Committee of the Academy of Operative Dentistry no later than 31st December 2005.

The award recipient will be announced during the 2006 Annual Meeting of the Academy of Operative Dentistry, which will be held in Chicago, 23-24 February, 2006.

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