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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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# The Impact of Study Clubs on Our Patients

This Spring I received a fax from Dr Fred Olsen, a member of both the Academy of Operative Dentistry and the Tucker Cast Gold Study Club of Phoenix, Arizona. He attached a letter from a Mr Robert Williams, who had been a patient at one of the study club's operating sessions. I was so impressed by the letter and the circumstance surrounding its writing, that I requested permission to publish it.

Those of us who have operated in a study club setting know the tremendous benefits derived from the close scrutiny, critiques, advice and instruction from our mentors and colleagues. It is virtually impossible not to learn and improve our professional skills with this type of input. However, we are frequently so focused on ourselves and the quality of our work that we may not consider the effect of the extra time spent and the verbal discussions on the individual in the

dental chair. This letter provides some illumination and understanding of the patient's response to the study club experience.

I would like to thank Dr Olsen for sending me this letter. But I want to express my heartfelt gratitude to Mr Williams for his time and his willingness to share his perspective and insights on the impact of study clubs on our patients. I hope that when you read his letter, if you are a member of a study club, that you will be proud of your efforts. If you have been undecided about participating in a study club environment, perhaps this will be the impetus you need to join. After all, if our patients recognize the intrinsic value of study clubs, shouldn't we?

Michael A Cochran, Editor

## My Comments on the Phoenix Dental Study Group

A few months ago when my dentist asked me to be the subject of his next study group, I found it somewhat strange that two of his office staff stated separately to me that they hoped that I would enjoy the experience. While I have always greatly appreciated quality dental care, I can honestly say that I had never actually "enjoyed" any dental procedure; until now.

As a retired career health statistician, I was sometimes involved in attempting to measure the quality of health care from afar. In a few hours in this study group, I learned more about the "hands-on" attempt by health care professionals to reach for the highest level of quality than I ever did in my career. I learned that for many, if not most, dental problems, there is not just one appropriate course of action by a dentist. There may be many possible detailed ways to approach a dental condition, and having the dentist assess and evaluate the greatest number of those possibilities before choosing to implement the most favorable of the treatment options is crucial for the best quality dental care. Obviously, the more exposure any dentist has to a variety of quality dentists' evaluations and opinions, the better that dentist will be able to assess and evaluate situations that he or she encounters later. This is exactly what the hands-on "real-time" peer evaluations and communications of this study group accomplish. Based upon my lay understanding of the conversations that I heard during my two study group sessions to replace a crown, I don't believe that I have ever encountered a more sincere, cooperative and professional attempt by any group to improve their skills and strive for perfection in their work.

Never before have I seen a group of professionals who were willing to invest so much of their valuable time, money and expertise in order to potentially improve the quality of the care that both they and their competitors are able to provide to all their patients. But, even more incredible to me, is the idea that each and every one of the fully accredited dentists in this monthly study group is willing to open him or herself up to sub-

stantial potential peer criticism on a regular basis without there being any mandate or financial advantage to do so. To me, this indicates that these dentists have a realistic level of self-confidence superimposed on an overriding fervent desire to continually reach for perfection in their profession. The fact that they keep returning to this study group, some after 20 to 30 years or more as a dentist, indicates that they fully understand that they will never quite attain that elusive perfection, although they continue to constantly strive to achieve it. One dentist even selflessly chooses to travel from his home, near Seattle, to Phoenix monthly to serve as a mentor for this Phoenix study group, while additionally, he continues to participate in his local study group.

The participating dentists in this Phoenix study group epitomize the absolute zenith of professionalism. Even though I do not have the technical knowledge to thoroughly evaluate the quality of care that my dentist regularly provides to me, I know that he is one of the best dentists simply because he actively participates in this study group. If I should ever move to a new city, I would choose my new dentist exclusively from members of that area's study group.

I thoroughly enjoyed this study group experience and am so very appreciative that I was asked to participate in it. This was far and away the best health care experience I have ever had. It is what the entire dental profession should aspire to, what every patient should hope for, and what every dentist should voluntarily participate in. I only hope that the other health fields will imitate this concept.

**Robert Wilmot Williams**  
**April 12, 2005**

# Effectiveness of Composite Cure Associated with Different Light-curing Regimes

BJ Neo • MS Soh  
JW Teo • AUJ Yap

## Clinical Relevance

Soft-start and turbo cure regimens may be more effective than the standard continuous cure mode.

## SUMMARY

This study investigated the use of various light-curing regimens with standardized light energy density on the effectiveness of cure of a visible light activated resin composite (Z100, 3M-ESPE). A light-cure unit (Variable Intensity Polymerizer (VIP), BISCO Inc) which permitted individual control over time and intensity, was used. The five light-curing modes investigated include Pulse Delay (PD), Pulse Cure (PC), Soft-start (SS), Turbo (T) and Control (C). Effectiveness of cure was established by measuring the top and bottom

Knoop hardness of 2-mm thick composite specimens using a digital microhardness tester (n=5, load=500g; dwell time=15 seconds) immediately and at one-day post-polymerization. Data obtained was analyzed using one-way ANOVA/Scheffe's post hoc test and Independent Samples *t*-tests ( $p<0.05$ ). Top KHN observed immediately after polymerization with C was significantly lower than PD. At one day post-polymerization, the top KHN obtained with C was significantly lower than PD, SS and T. No significant difference in bottom KHN was observed among the different curing modes immediately after curing. At one day post-polymerization, the bottom KHN obtained with C was significantly lower than SS and T. Regardless of curing regimens, top and bottom values at one day were significantly higher than those observed immediately after light polymerization. No significant difference in mean hardness ratio was observed among the different curing regimens immediately and one day later. Effectiveness of the cure at the bottom surfaces of composites may be increased by soft-start and turbo polymerization regimens.

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## INTRODUCTION

Light-activated resin composites, introduced in the 1970s, revolutionized clinical dentistry by maximizing



working time and decreasing setting time. In the last decade, composite restoratives and adhesive techniques have become the basis of modern dentistry. Composites set by a “chemical reaction between dimethacrylate resin monomers and results in an inflexible and heavily cross-linked polymer network, encircling the inert filler particles” (Ferracane, 1995). The extent of this reaction, commonly referred to as the degree or effectiveness of cure, is vital, as the physico-mechanical properties of the composite restoration depends on it (Asmussen, 1982a,b). Caughman and others (1991) have stated that it is critical to prevent clinical complications due to the cytotoxicity of insufficiently polymerized material. The degree of polymerization is dependent on the chemistry of the material, concentration of the initiator and the type, size and quantity of the filler particle. Furthermore, the effectiveness of the radiation sources, such as spectral distribution intensity, exposure time and position of the light-tip guide, also influences the effectiveness of polymerization (Harrington, Wilson & Shortall, 1996).

Many different light curing modes have emerged, in response to the dramatic rise in the use of composite restorations over the past few years. Most curing modes aim at decreasing polymerization shrinkage or reducing curing time (Sakaguchi & others, 1991; Peutzfeldt, Sahafi & Asmussen, 2000; Yap, Ng & Siow, 2001; Yap, Soh & Siow, 2002). The technology utilized for curing lights ranges from conventional halogen bulbs to more expensive systems using lasers, plasma arc and LEDs (light-emitting diodes). Halogen-based light curing units (LCUs) are the most widely used light-curing units used in dentistry today.

In an attempt to reduce polymerization shrinkage, soft-start and low-intensity curing regimens have been used (Uno & Asmussen, 1991; Unterbrink & Muessner, 1995; Feilzer & others, 1995; Davidson & Feilzer, 1997; Yap & others, 2001; Yap & others, 2002). However, despite the advantages of the latter, the use of high intensity lights for short durations is also employed for curing composites (St Georges & Miguez, 2001). Rueggeberg and Jordan (1993) reported that the use of high intensity light sources increases the degree of conversion, depth of cure and the physical and mechanical properties of composite restoratives. However, the use of high intensity light sources has also been reported to produce higher contraction strains during composite polymerization (Unterbrink & Muessner, 1995;

Sakaguchi & Berge, 1998; Dennison & others, 2000). One method used to minimize polymerization shrinkage without compromising the degree of conversion on light-activated composites is to allow flow during hardening by means of controlled polymerization (Davidson & Feilzer, 1997). Polymerization shrinkage may be minimized by curing with short pulses of energy or pre-polymerization followed by a final cure with high intensity (soft-start). Studies have shown that these polymerization modes resulted in restorations with smaller marginal gap; increased marginal integrity and improved material properties (Kanca & Suh, 1999; Uno & Asmussen, 1991; Mehl, Hickel & Kunzelmann, 1997).

Miyazaki and others (1996) highlighted that the polymerization process relies on total light energy (intensity x time) rather than light intensity alone and that the effectiveness of cure depends on light energy density. While previous studies reported on different light-curing profiles on the effectiveness of cure based on manufacturer’s profiles, this study compared the influence of five different curing profiles with a standardized light energy density on the effectiveness of composite cure.

METHODS AND MATERIALS

A mini-filled resin composite (Z100, 3M-ESPE, St Paul, MN, USA) of A2 shade and a commercial light-cure unit that allowed for independent command over time and intensity (Variable Intensity Polymerizer [VIP], BISCO Inc, Schaumburg, IL, USA) were selected for this study. VIP was developed to provide high intensity light in situations requiring rapid intense polymerization, such as sealants and veneers, and to deliver multiple-curing intensities for composite restorations. It has an output wavelength range of 400 to 500 nm and is programmed with preset exposure times of 2 to 5, 10, 20 and 30 seconds and a continuous mode of up to 225 seconds. The programmed intensity or power settings are 100, 200, 300, 400, 500 and 600 mW/cm². This allows clinicians to manually select any time and intensity combinations or use pre-programmed values for the curing of composites. The light intensities for each light-curing mode were checked with the in-built radiometer before use. The five light-curing modes investigated are detailed in

Table 1: The Different Light-curing Modes Examined					
Light-curing Mode		Regimen			
Control (C)		400mW/cm² (40 seconds)			
Pulse Delay (PD)		100mW/cm² (10 seconds)	→	Delay (3 minutes)	→ 500mW/cm² (30 seconds)
Pulse Cure (PC)		400mW/cm² (20 seconds)	→	Delay (20 seconds)	→ 400mW/cm² (20 seconds)
Soft-start (SS)		200mW/cm² (20 seconds)	→	600mW/cm² (20 seconds)	
Turbo (T)		600mW/cm² (27 seconds)			

Table 1. The control mode (C) involves light irradiation at 400mW/cm<sup>2</sup> for 40 seconds. Pulse Delay (PD) uses an initial low energy dose (100mW/cm<sup>2</sup> for 10 seconds) followed by a waiting time of three minutes and a final cure at high-energy dose (500mW/cm<sup>2</sup> for 30 seconds). The three-minute waiting period is to be used for finishing and polishing the composite restoration. Pulse Cure (PC) involves two 400mW/cm<sup>2</sup> 20 second pulses with a 20 second interval in-between. The soft-start (SS) mode uses an initial low-light intensity (200mW/cm<sup>2</sup> for 20 seconds) immediately followed by final cure at high light intensity (600mW/cm<sup>2</sup> for 20 seconds). Turbo mode (T) involves light irradiation at 600mW/cm<sup>2</sup> for 27 seconds.

The hardness testing methodology used to determine the effectiveness of cure was based on that used by Yap (2000). The composite was placed in black delrin molds with square cavities 2-mm deep and 4-mm wide/long, confined between two opposing acetate strips (Hawe-Neos Dental, Bioggio, Switzerland) to ensure smooth surfaces and minimize polymerization inhibition by oxygen (Finger & Dreyer Jorgensen, 1976). A glass slide (1-mm thick) was then placed on the molds and excess material was extruded by pressure application. The composite was then irradiated from the top through the glass slide and acetate strip using the different light-curing modes. Immediately after light polymerization, the acetate strips were removed and the specimens in their molds were positioned centrally beneath the indenter of a digital microhardness tester (FM7, Future-Tech Corp, Tokyo, Japan) to assess the Knoop Hardness Number (KHN) of the top and bottom surfaces. A 500g load was then applied through the indenter with a dwell time of 15 seconds. The KHN corresponding to each indentation was computed by measuring the dimensions of the indentations and using the formula  $KHN = 1.451 \times (F/d^2)$ , where F is the test load in Newtons and d is the longer diagonal length of an indentation in millimeters. Five specimens were made for each light-curing mode. Three readings were taken for each specimen and averaged to form a single value for that specimen. The mean KHN and hardness ratio of the five specimens were then calculated and tabu-

lated using the following formula: Hardness ratio = KHN of bottom surfaces/KHN of top surfaces. After immediate post-polymerization, KHN readings were taken and the specimens were stored at 37°C and 100% relative humidity for 24 hours. Hardness testing was then repeated.

Data was subjected to two-way ANOVA to determine interaction between curing modes and time. One-way ANOVA and Scheffe's post-hoc test were used to compare the different curing modes and independent samples *t*-test was used to evaluate differences in KHN between the two testing periods.

## RESULTS

Table 2 shows the mean KHN and hardness ratio for the different light curing regimes. Results of statistical analysis are reflected in Tables 3 and 4.

Immediately after polymerization, the top KHN of the composite cured with C (control) was found to be significantly lower than that of PD. At the bottom surfaces, no significant difference in KHN was observed among the different cure modes. No significant differences in hardness ratio were also observed between C and all the other curing modes. One day after polymerization, the top KHN of C was found to be significantly lower than PD, SS and T. At the bottom surfaces, the KHN of samples cured with C was found to be signifi-

Table 2: Mean KHN and Hardness Ratio Observed for the Different Curing Modes Immediately After Curing and One Day After Curing

Time	Cure Modes	Top KHN	Bottom KHN	Hardness Ratio
Immediate	PD	70.48 (0.85)	61.98 (2.11)	0.88 (0.04)
	PC	65.70 (2.46)	60.20 (1.11)	0.92 (0.04)
	SS	65.90 (0.65)	63.82 (0.83)	0.97 (0.02)
	T	67.44 (1.11)	62.50 (1.12)	0.93 (0.02)
	C	65.52 (0.85)	62.90 (1.32)	0.96 (0.02)
1 Day	PD	85.68 (2.01)	77.70 (3.67)	0.91 (0.05)
	PC	81.64 (1.45)	77.30 (1.74)	0.95 (0.01)
	SS	89.30 (3.09)	84.20 (2.56)	0.94 (0.03)
	T	90.54 (1.25)	82.18 (2.57)	0.91 (0.04)
	C	76.96 (1.58)	74.16 (1.82)	0.96 (0.01)

Standard deviations in parentheses

Table 3: Comparison of Mean KHN and Hardness Ratios of the Various Curing Modes to the Standard Cure Mode

Time	Variable	Significance
Immediate	KHN Top	C < PD
	KHN Bottom	No significance
	Hardness Ratio	No significance
1 Day	KHN Top	C < PD, SS, T
	KHN Bottom	C < SS, T
	Hardness Ratio	No significance

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

Table 4: Comparison Mean KHN and Hardness Ratios Immediately After Polymerization to That of One Day After Polymerization of Different Curing Modes

Variable	Significance
KHN Top	PD, PC, SS, T, C (immediate) < PD, PC, SS, T, C (1 day)
KHN Bottom	PD, PC, SS, T, C (immediate) < PD, PC, SS, T, C (1 day)
Hardness Ratio	No significance

Results of Independent Samples t-tests ( $p < 0.05$ ). < indicates statistical significance.

cantly lower than SS and T. No significant differences in hardness ratio were found between C and all the other curing modes.

For all curing regimes, mean KHN values for specimens tested at one day were found to be significantly higher than those tested immediately after light polymerization for both top and bottom KHN. For the mean hardness ratio, no significant difference was observed between specimens tested immediately and one day after curing.

## DISCUSSION

Techniques used to assess the effectiveness of cure of resin composites can be broadly grouped into direct and indirect methods. Direct methods include the use of infrared spectroscopy and laser Raman spectroscopy to assess the degree of conversion. However, the use of spectroscopy is relatively complex, time consuming and expensive (Rueggeberg & Craig, 1988). In contrast, indirect methods, such as visual inspection, scraping and hardness testing, are relatively easier to perform. Hardness testing is a good indicator of the degree of conversion (Asmussen, 1982a,b) and Knoop hardness reportedly exhibits a good correlation with infrared spectroscopy (DeWald & Ferracane, 1987). In this study, the effectiveness of cure of both the top and bottom surfaces of the specimens was assessed by a digital microhardness tester, due to its relative ease of use.

As light intensity decreases with increasing distance from the light cure tip (Pires & others, 1993), the distance from the light cure tip was standardized in this study. To minimize the effects of colorants on light polymerization, shade A2 was selected (Bayne, Heymann & Swift, 1994). Two-mm thick specimens were assessed, as they gave uniform and maximum polymerization (Yap, 2000). An inherent drawback of light-activated composites is that the degree of polymerization is directly proportional to the amount of light that is exposed to them (Rueggeberg, Caughman & Curtis, 1994). As the total light energy density ( $1600\text{mJ}/\text{cm}^2$ ) of the different light-curing modes investigated was generally the same, any difference in KHN (or effectiveness of cure) could be attributed to the different light-curing regimes employed. A standard continuous cure mode with an intensity of  $400\text{mW}/\text{cm}^2$  (Tate, Porter & Dosch, 1999) for an exposure time of 40

seconds (manufacturer's recommendation) was used as a control.

As there is sufficient light to activate the camphoroquinone photoinitiator, composite cure at the top surface is not as dependent on light intensity as the bottom surface (Fowler, Swartz & Moore, 1994). Studies have shown that a curing source with a relatively low intensity could cure the resin matrix to an extent almost equal to high intensity lights (Rueggeberg & Jordan, 1993). Light intensity is, however, greatly reduced as light passes through the bulk of the material. The absorption and scattering of light through the material decreases the potential for polymerization (Ruyter & Øysæd, 1982). Hence, the effectiveness of cure cannot be assessed by the top surface hardness alone. The bottom hardness, which is more critically affected by the light intensity, is thus a better gauge on the effectiveness of cure of a composite.

At the bottom surfaces, no significant difference was observed between samples polymerized with the different cure modes immediately after curing. As the degree of polymerization is directly proportional to the light energy density (Rueggeberg & others, 1994), this phenomenon can be attributed to the same light energy density used in all the different curing modes. At one-day post polymerization, significant differences in KHN were observed between C, PD, SS and T at the top surfaces of the specimen. At the bottom surfaces, significant differences were also seen between C, SS and T, which indicates that composites cured with different curing modes continued to polymerize but to different extents after the light source is removed. This may be attributed to the different setting reactions and cross-linking of the monomer units within the composite as a result of the different curing regimes used. This study suggests that post-curing of composites may occur to a greater extent with controlled polymerization techniques such as PD and SS, as well as curing with high intensity lights such as T. The exact mechanism is not known and warrants in-depth investigation. It is not known exactly when this post-cure polymerization ceases. However, from previous studies, post-cure polymerization appears to cease within 72 hours for standard continuous cure (Yap & others, 2000).

For polymerization to be effective, an ideal ratio of 1:1 between the top and bottom surface hardness values should be achieved. This means that the top surface KHN should be similar to the bottom. Studies suggest that the hardness ratio should not be less than 0.8 for composites that are adequately cured (Pilo & Cardash, 1992). The hardness ratios of all the 2-mm specimens in this study were above 0.8. All curing regimes fulfilled this criterion.



## CONCLUSIONS

Under the conditions of this *in vitro* study:

1. The effectiveness of cure associated with the various curing modes was time dependent.
2. Composite cure associated with the use of pulse cure, soft-start and turbo modes was comparable to that with standard continuous mode immediately after curing.
3. At day one, the effectiveness of cure associated with soft-start and turbo modes was significantly better than that of the standard continuous mode.

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## References

- Asmussen E (1982a) Restorative resins: Hardness and strength vs quantity of remaining double bonds *Scandinavian Journal of Dental Research* **90**(6) 484-489.
- Asmussen E (1982b) Factors affecting the quantity of remaining double bonds in restorative resin polymers *Scandinavian Journal of Dental Research* **90**(6) 490-496.
- Asmussen E & Peutzfeldt A (2001) Influence of pulse-delay curing on softening of polymer structures *Journal of Dental Research* **80**(6) 1570-1573.
- Bayne SC, Heymann HO & Swift EJ Jr (1994) Update on dental composite restorations *Journal of the American Dental Association* **125**(6) 687-701.
- Caughman WF, Caughman GB, Shiflett RA, Rueggeberg F & Schuster GS (1991) Correlation of cytotoxicity, filler loading and curing time of dental composites *Biomaterials* **12**(18) 737-740.
- Davidson CL & Feilzer AJ (1997) Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives *Journal of Dentistry* **25**(6) 435-440.
- Dennison JB, Yaman P, Seir R & Hamilton JC (2000) Effect of variable light intensity on composite shrinkage *Journal of Prosthetic Dentistry* **84**(5) 499-505.
- DeWald JP & Ferracane JL (1987) A comparison of four modes of evaluating depth of cure of light-activated composites *Journal of Dental Research* **66**(3) 727-730.
- Finger W & Dreyer Jorgensen K (1976) Inhibition of polymerization by oxygen in composite filling materials and enamel sealers *Schweizerische Monatsschrift für Zahnheilkunde* **86**(8) 812-824.
- Feilzer AJ, Dooren LH, de Gee AJ & Davidson CL (1995) Influence of light intensity on polymerization shrinkage and integrity of restoration-cavity interface *European Journal of Oral Science* **103**(5) 322-326.
- Ferracane JL (1995) Current trends in dental composites *Critical Review in Oral Biology and Medicine* **6**(4) 302-318.
- Fowler CS, Swartz ML & Moore BK (1994) Efficacy testing of visible-light-curing units *Operative Dentistry* **19**(2) 47-52.
- Harrington E, Wilson HJ & Shortall AC (1996) Light-activated restorative materials: A method of determining effective radiation times *Journal of Oral Rehabilitation* **23**(3) 210-218.
- Kanca J 3<sup>rd</sup> & Suh BI (1999) Pulse activation: Reducing resin-based composite contraction stresses at the enamel cavosurface margins *American Journal of Dentistry* **12**(3) 107-112.
- Mehl A, Hickel R & Kunzelmann KH (1997) Physical properties and gap formation of light-cured composites with and without "soft start polymerization" *Journal of Dentistry* **25**(3-4) 321-330.
- Miyazaki M, Oshida Y, Moore BK & Onose H (1996) Effect of light exposure on fracture toughness and flexural strength of light-cured composites *Dental Materials* **12**(6) 328-332.
- Peutzfeldt A, Sahafi A & Asmussen E (2000) Characterization of resin composites polymerized with plasma arc curing units *Dental Materials* **16**(5) 330-336.
- Pilo R & Cardash HS (1992) Post-irradiation polymerization of different anterior and posterior visible light-activated resin composites *Dental Materials* **8**(5) 299-304.
- Pires JA, Cvitko E, Denehy GE & Swift EF Jr (1993) Effects of curing tip distance on light intensity and composite resin microhardness *Quintessence International* **24**(7) 517-521.
- Rueggeberg FA & Craig RG (1988) Correlation of parameters used to estimate monomer conversion in a light-cured composites *Journal of Dental Research* **67**(6) 932-937.
- Rueggeberg FA & Jordan DM (1993) Effect of light-tip distance on polymerization of resin composite *International Journal of Prosthodontics* **6**(4) 364-370.
- Rueggeberg FA, Caughman WF & Curtis JW Jr (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19**(1) 26-32.
- Ruyter IE & Øysæd H (1982) Conversion in different depths of ultraviolet and visible light activated composite materials *Acta Odontologica Scandinavica* **40**(3) 179-192.
- Sakaguchi RL & Berge HX (1998) Reduced light energy density decreases post-gel contraction while maintaining degree of conversion in composites *Journal of Dentistry* **26**(8) 695-700.
- Sakaguchi RL, Sasik CT, Bunczak MA & Douglas WH (1991) Strain gauge method for measuring polymerization contraction of composite restoratives *Journal of Dentistry* **19**(5) 312-316.
- St Georges AJ & Miguez PA (2001) Visible light-curing: Part I *Journal of Esthetic and Restorative Dentistry* **13**(2) 140-143.
- Tate WH, Porter KH & Dosch RO (1999) Successful photocuring: Don't restore without it *Operative Dentistry* **24**(2) 109-114.
- Uno S & Asmussen E (1991) Marginal adaptation of a restorative resin polymerized at reduced rate *Scandinavian Journal of Dental Research* **99**(5) 440-444.
- Unterbrink GL & Muessner R (1995) Influence of light intensity on two restorative systems *Journal of Dentistry* **23**(3) 183-189.
- Yap AU (2000) Effectiveness of polymerization in composite restoratives claiming bulk placement: Impact of cavity depth and exposure time *Operative Dentistry* **25**(2) 113-120.
- Yap AU, Ng SC & Siow KS (2001) Soft-start polymerization: Influence on effectiveness of cure and post-gel shrinkage *Operative Dentistry* **26**(3) 260-266.
- Yap AU, Soh MS & Siow KS (2002) Effectiveness of composite cure with pulse activation and soft-start polymerization *Operative Dentistry* **27**(1) 44-49.
- Yap AU, Wang HB, Siow KS, Gan LM (2000) Polymerization shrinkage of visible-light-cured composites *Operative Dentistry* **25**(2) 98-103.

# Salivary Contamination and Bond Strength of Glass-ionomers to Dentin

KE Kulczyk • SK Sidhu • JF McCabe

## Clinical Relevance

Salivary contamination did not affect the mean shear bond strength of highly viscous glass ionomers to conditioned dentin; instead, it increased the probability of failure at low stresses.

## SUMMARY

This study evaluated the effect of salivary contamination on the shear bond strength of two highly viscous glass ionomer cements (Fuji IX GP Fast and Ketac-Molar Maxicap) to conditioned dentin and assessed the effect of cleaning the contaminated field prior to bonding.

The buccal surfaces of 90 human molars and premolars were ground to expose dentin and the teeth were then set in resin. The specimens were divided into two groups for each material, then further subdivided into three groups of 15 teeth each: Group 1—uncontaminated (control), Group 2—dentin contaminated with saliva, Group 3—dentin contaminated, washed and air dried. The specimens were made by bonding the test

material to dentin using a 4 mm diameter gelatin capsule. All specimens were protected with varnish and placed in distilled water at 37°C for seven days prior to measuring bond strength in shear. Fractured surfaces were examined visually and by using SEM to assess mode of failure.

There were no significant differences in mean shear bond strength among the three test groups for either material (ANOVA). However, shear bond strength of Fuji IX to dentin was significantly greater than Ketac-Molar ( $p=0.019$ ) for all groups. Weibull analysis showed that contaminated (Group 2) specimens had a greater probability of failure at low stresses. Modes of failure were mostly cohesive for Fuji IX and adhesive/cohesive for Ketac-Molar.

In conclusion, salivary contamination did not affect the mean shear bond strength of Fuji IX GP Fast and Ketac-Molar Maxicap to conditioned dentin; however, it increased the probability of failure at low stresses.

## INTRODUCTION

Wilson and Kent (1972) first reported on glass ionomer cement (GIC) in dental literature. GIC consists of a basic glass and acidic polymer and sets by an acid-base reaction (McLean, Nicholson & Wilson, 1994). It has

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achieved widespread use as a material for the restoration of Class V and Class III lesions in permanent dentition and Class II lesions in deciduous teeth. In addition, GIC may be used as a fast setting liner or base under resin composites or amalgam, as a luting cement, fissure sealant, core build-up material or retrograde root filling material (Walls, 1986).

The relatively poor physical properties (Mount, 1999) and sensitivity to water absorption or desiccation during the initial setting stages (Wilson & McLean, 1988) have led to the development of highly viscous GICs. These have been adopted for the Atraumatic Restorative Technique (ART). ART involves the hand excavation of caries and placement of the material as a restoration and sealant. It is a technique adopted in developing countries where resources may be limited. GICs are chosen for this technique due to their ease of use, hardening without special equipment, adhesion to dentin (Mount, 1991), low coefficient of thermal expansion similar to dentin (Walls, McCabe & Murray, 1988) and fluoride release (Forsten, 1998). In addition, the highly viscous GICs are stronger than traditional GICs (Guggenberger, May & Stefan, 1998; Ewoldsen, Covey & Lavin, 1997) and condensable, yet syringable with a wide bore cannula. They contain finer glass particles and in greater proportion than conventional GICs (Guggenberger & others, 1998). The glass is chemically modified to reduce the calcium content, thus limiting the production of calcium polyalkenoate chains that are more water-soluble than the equivalent aluminum salts. This allows for faster maturation of the setting reaction and produces a cement that is less soluble than the traditional GIC (Mount, 1999). Two such commercially available viscous GICs are Fuji IX GP Fast (GC Corp, Tokyo, Japan) and Ketac-Molar Maxicap (ESPE, Seefeld, Germany).

The mechanism of adhesion of GICs to tooth substance is primarily diffusion-based (Akinmade & Nicholson, 1993), with the exchange of ions and formation of an intermediate layer representing a chemical union. Bond strength measurement is a common means of assessing the adhesion of materials to tooth substance. It is calculated as the apparent load at failure, divided by the bonded surface area (Nakabayashi & Pashley, 1998). Bonds tested in tensile or shear mode tend to fail over a wide range of stresses, with standard deviations of 30% to 40% often seen. Several authors have discussed the problems with standardization of methodology in bond strength testing, which makes it difficult to compare directly the results of different studies (Van Noort & others, 1989; Øilo, 1993). Øilo (1993) found that different methods can provide differences in bond strength values for the same material from two to four times. He also stated that shear testing might, in some studies, give higher values than tensile tests but with the same ranking of materials.

Standardization of test procedures has thus been recommended (Van Noort & others, 1989) and International Organization for Standardization (ISO) specifications have been formulated (ISO/TC106/Subcommittee 1, 1994).

Nakabayashi and Pashley (1998) discuss the importance of "toughness" of the components of the bonded interphase or their ability to undergo strain without fracture. They conclude that bond toughness gives more information about its ability to absorb loading energy. This may be particularly relevant for dentin bonding agents (DBAs), though perhaps less so for GICs, which are more brittle materials.

The failure mode of a material is important in establishing its use and limitations. If failure is cohesive, an effective bond is assured. Mount (1999) suggests that the increased strength of the highly viscous GICs may make it easier to assess adhesive strength, though he emphasizes the importance of allowing sufficient time, preferably a week (as in this study), for the maturation of GICs prior to bond strength testing and identifying the mode of failure. However, ISO recommends storing specimens for 24 hours before testing (ISO/TC106/Subcommittee 1, 1994). Most of the studies that have been carried out on the bonding of conventional GICs to dentin, without contamination, have shown the failure to be cohesive in nature (Tyler & others, 1994; Tanumiharja, Burrow & Tyas, 2000; Powis & others, 1982). Sidhu, Sherriff and Watson (1999) found failure to be both adhesive and cohesive between resin-modified glass ionomer cement (RMGIC) and dentin. Burke and Lynch (1994) found adhesive and cohesive failure between GIC base materials and dentin.

One of the clinical factors that may affect adhesion, while being particularly relevant where equipment or patient cooperation is limited, is contamination of the restorative field with saliva. There have been a number of studies investigating the effect of salivary contamination on the bond strength of composites and fissure sealants to enamel and of DBAs to dentin, but very little work has been done in this field with GICs. Prodger and Symonds (1977) subjected one of the earliest GICs (ASPA, AD International Ltd, London) to bond strength testing, with a view to assessing the importance of certain clinical recommendations made in the instructions. They found that exposing dentin to saliva for 10 days significantly decreased tensile bond strengths, but applying 50% citric acid returned bond strengths to pre-exposure levels. Aboush and Jenkins (1987) immersed dentin specimens in saliva for one minute or 10 days then washed and air dried them all prior to bonding GIC to the surface. A significant reduction in bond strength was found, which returned to control levels if the surface was subsequently cleaned with 25% polyacrylic acid (PAA), 50% citric acid or pumice. Adhesive modes of failure were



observed in the specimens that had been washed and dried only and cohesive failures were found in specimens that had been subsequently cleaned. They concluded that saliva interfered with the ability of the GIC to form chemical adhesive bonds with dentin.

Dietrich and others (2000) contaminated dentin with saliva and blood. They assessed microleakage as well as marginal adaptation and found that sandwich restorations of RMGIC and resin composite showed less sensitivity to contamination than resin composite restorations alone. Microleakage and marginal adaptation was worse than in uncontaminated controls. In addition, one study looked at the effect of salivary contamination on the shear bond strength of RMGIC to dentin (Safar, Davis & Overton, 1999). A reduction in bond strength was observed whether the samples were dried; rinsed and dried; or rinsed, dried and re-etched after contamination. Contaminated specimens exhibited a mixed or adhesive mode of failure, whereas cohesive failures were observed in the uncontaminated control group. It was suggested that salivary residue precluded the thorough wetting of dentin by the RMGIC.

None of the studies have evaluated the effect on bond strength when salivary contamination occurs after conditioning of the dentin and is not subsequently removed by drying or rinsing prior to placement of GIC. This may occur clinically if contamination were to go unnoticed or the operator was unable to correct the situation, particularly under the type of operating conditions where the ART may be used. In addition, there are no studies evaluating the effect of contamination with the newer, highly viscous GICs.

The purpose of this *in vitro* study was to:

1. Compare the shear bond strength of two highly viscous GICs to conditioned dentin contaminated with saliva with that of the same materials to non-contaminated dentin.
2. Compare the bond strengths after rinsing with water and air drying the contaminated tooth surface before placement of the material.
3. Assess the mode of failure using scanning electron microscopy (SEM).

## METHODS AND MATERIALS

Two highly viscous GICs were used in this study (Table 1). Ninety extracted intact human molars and premolars stored in 0.5% aqueous chloramine T solution were used. The buccal surfaces were ground with 150-grit silicon carbide (SiC) paper and copious water irrigation until a flat dentin surface of sufficient area for bonding a 4-mm diameter gelatin capsule filled with the material was exposed. The ground teeth were set in polyester resin in styrene monomer (Resinous Chemicals, Tyne and Wear, UK), such that the flattened dentin surface was exposed and parallel to the base of the cylindrical 3-cm diameter polyethylene mold (Metallurgical Services Ltd, Surrey, UK). These specimens were stored in distilled water until ready for bonding.

The teeth were randomly allocated into three groups of 15 for each of the two materials. All ground dentin surfaces were finished with 800-grit SiC paper with copious water irrigation immediately before bonding. The specimens were prepared and bonded according to the individual manufacturer's instructions. In Group 1, the specimens were bonded onto conditioned and uncontaminated dentin. In Group 2 specimens, the conditioned dentin surface was contaminated for 20 seconds with 0.05 ml (a volume sufficient to cover the entire dentin surface) of fresh, whole unstimulated human saliva collected from one healthy individual. This saliva was collected immediately prior to bonding, ensuring that two hours had elapsed since the volunteer had brushed his or her teeth, eaten or drank liquids. Group 3 specimens were similarly conditioned and contaminated but had the further treatment of being washed with water from a dental spray for five seconds and gently air-dried prior to bonding.

All bonded specimens were varnished, including the bonding interface, and placed in distilled water at 37°C for seven days prior to testing. They were then tested for shear bond strength using an ISO testing rig in an Instron universal machine at a crosshead speed of 1 mm/minute (ISO/TC106/ Subcommittee 1, 1994). The recorded load at failure and the surface area of bonding were used to calculate the bond strength in MPa. The values were analyzed by analysis of variance (ANOVA) General Linear Model. The two materials, type of tooth

Table 1: Glass-ionomer Cements Used in This Study

Material	Conditioner	Powder	Liquid	Batch #	Manufacturer
Fuji IX GP Fast capsules	25% PAA* (Cavity conditioner)	Fluoro-alumino-silicate glass, PAA*, pigment	PAA*, polybasic carboxylic acid, water	0007189	GC Corp, Tokyo, Japan
Ketac-Molar Maxicap capsules	20-30% PAA*, benzoic acid (Ketac conditioner)	Calcium lanthanum- fluorosilicate-fluorosilicate glass, acrylic acid-maleic acid copolymer, benzoic acid, iron oxides (pigments)	Acrylic acid-maleic acid copolymer, benzoic acid, tartaric acid, water	113278	ESPE, Seefeld, Germany

\*PAA = polyacrylic acid



(molar or premolar) and “surface treatment group” (1, 2 or 3), were the three variables. The shape of the graph of force *vs* displacement was examined for each group to obtain comparative information on the energy to debond. Weibull analysis was used to calculate the probability of failure ( $P_f$ ) at different stress levels for each test group.

Visual examination of all fractured surfaces was carried out under 4x magnification. A typical specimen was then selected from each group and examined using SEM for mode of failure. Additional specimens were viewed, using the SEM, for observation of the dentin

surface in the three surface treatment groups prior to bonding.

## RESULTS

Three specimens broke under stress upon setting up. It was not possible to determine the value of stress; therefore, these specimens were discounted. The mean shear bond strengths for all groups are shown in Table 2. ANOVA showed no significant difference between any of the surface treatment groups or type of tooth (molar or premolar). However, the mean bond strength for Fuji IX was significantly greater ( $p=0.019$ ) than for Ketac-Molar.

Similar shapes of graph of force *vs* displacement were observed in all groups.

The values of Weibull modulus ( $m$ ) and characteristic stress in MPa are shown in Table 3 (correlation coefficient = 0.97-0.99). For both materials, Group 2 (contaminated) specimens had the lowest Weibull modulus; characteristic stress was slightly increased in Group 2 and reduced in Group 3 specimens.

The stress at which  $P_f=5\%$  is shown in Table 4. Contaminated specimens required the least stress for  $P_f=5\%$  for both materials, though the situation was improved by washing and drying (as seen in Group 3). Weibull analysis showed that the  $P_f$  was greater for Ketac-Molar than for Fuji IX in all groups and the  $P_f$  of Group 2 (contaminated) specimens was greater than for the uncontaminated controls at lower stress levels for both materials (Figures 1 and 2).

SEM observations showed that Fuji IX displayed cohesive failure within the cement (Figure 3). This was the case in all three groups. However, it was only by using SEM that a thin layer of material retained on the dentin surface could be observed. For Ketac-Molar, the mode of failure was adhesive in some areas and cohesive in others for Group 1 and 3 specimens (Figure 4). The typical Group 2 specimen showed cohesive failure. SEM observation of a typical conditioned dentin surface showed patent dentinal tubules and the smear layer removed. The saliva contaminated surface showed the presence of residue and obliterated tubules. The contaminated, washed and

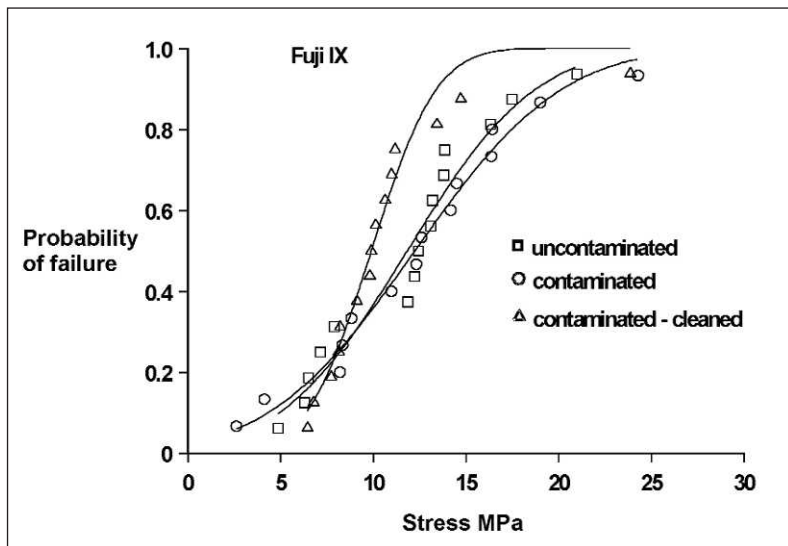


Figure 1. Probability of failure ( $P_f$ ) *vs* shear stress for Fuji IX. Lines were fitted using a Weibull distribution function.

Table 2: Mean Shear Bond Strengths  $\pm$  SD (MPa)

	Group 1 (uncontaminated)	Group 2 (contaminated)	Group 3 (contaminated/cleaned)
Fuji IX*	11.9 $\pm$ 4.6	12.4 $\pm$ 5.8	10.8 $\pm$ 4.3
Ketac-Molar*	9.5 $\pm$ 4.9	10.2 $\pm$ 5.7	7.9 $\pm$ 2.8

\*statistically significant difference ( $p=0.019$ ) between materials only.

Table 3: Values of Weibull Modulus ( $m$ ) and Characteristic Stress (MPa)

	Group 1 (uncontaminated)		Group 2 (contaminated)		Group 3 (contaminated/cleaned)	
	$m$	stress	$m$	stress	$m$	stress
Fuji IX	2.4	13.6	2.2	14.3	4.0	10.9
Ketac-Molar	2.7	10.1	2.0	11.0	2.7	8.9

Table 4: Stress at Which the Probability of Failure ( $P_f$ )=5 % (MPa)

	Group 1 (uncontaminated)	Group 2 (contaminated)	Group 3 (contaminated/cleaned)
Fuji IX	4.0	3.8	5.2
Ketac-Molar	3.4	2.5	3.0

dried surface showed mostly obliterated tubules and traces of residue.

## DISCUSSION

In this study, no attempt was made to simulate pulpal pressure, nor was the effect of blood proteins or products investigated. Fresh whole saliva has been an accepted medium for contamination in research but there may be differences in chemical composition and effectiveness of saliva samples. Every effort was made in this study to standardize the collection of saliva (Tenovuo & Lagerlof, 1994). Although teeth were divided randomly, other uncontrolled variables were depth of

dentin and storage time from extraction to dentin preparation.

The situation is further complicated by interpretation of the data and statistical analysis. ANOVA should be used when comparing the means of three or more groups. However, it may be criticized, as it assumes the bond strength data are drawn from a normally distributed population. Mean values might not demonstrate significant differences, but they may be the best indicators of performance (Fox, McCabe & Buckley, 1994). Johnson and others (1994) looked at the effect of salivary contamination on the bond strength of DBAs to dentin and found no significant differences in mean shear bond strengths between groups; however, they noted higher standard deviations in contaminated groups and thought this might indicate a less predictable bond.

Weibull analysis has been found to be an appropriate form of analysis in the mechanical testing of materials (Fleming, Marquis & Shortall, 1999; Palin & others, 2003; McCabe & Walls, 1986; Swift, Walls & McCabe, 1995). The Weibull equation relates probability of failure to stress. Graphs obtained by Weibull analysis have a shape factor, corresponding to the Weibull modulus, and a scale factor (its position along the x-axis) corresponding to the characteristic value of stress. A high value of the Weibull modulus indicates a close grouping of fracture stress values. A low value of Weibull modulus indicates a wide distribution with a long tail at low stress levels. This factor is also reflected in the high standard deviation values obtained, as in Group 2 specimens in this study.

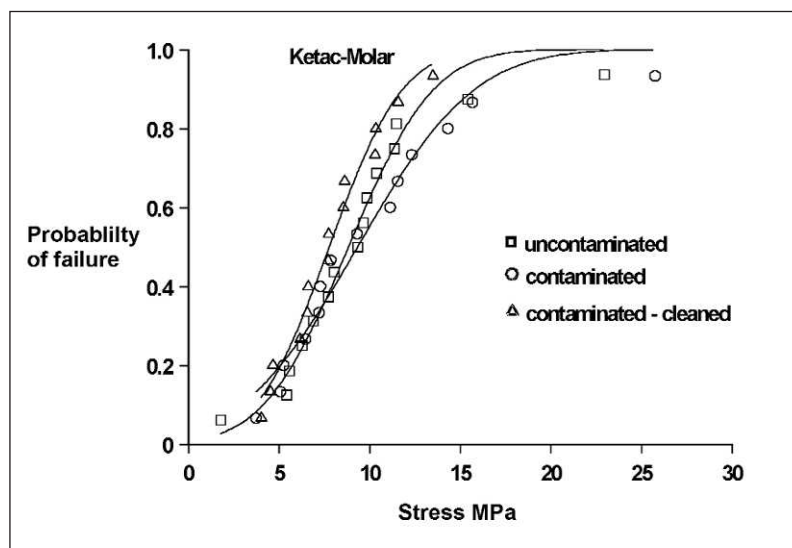


Figure 2. Probability of failure ( $P_f$ ) vs shear stress for Ketac-Molar. Lines were fitted using a Weibull distribution function.

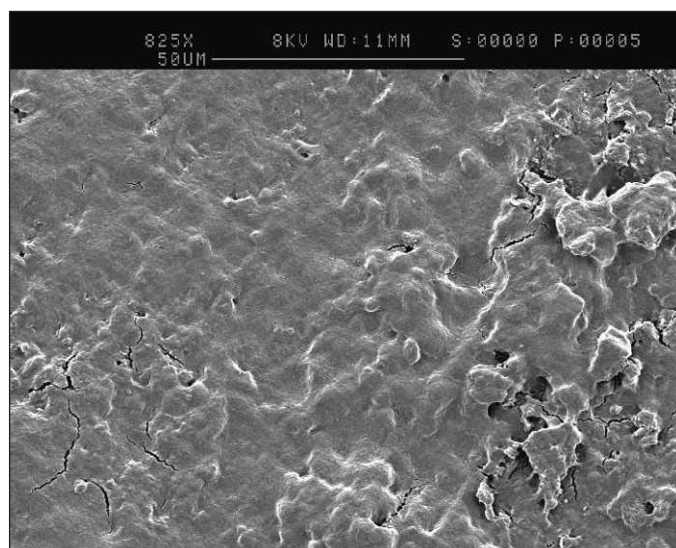


Figure 3. SEM photomicrograph of the dentin surface of a typical Fuji IX specimen, following debonding, showing cohesive failure within the cement (magnification bar=50  $\mu$ m).

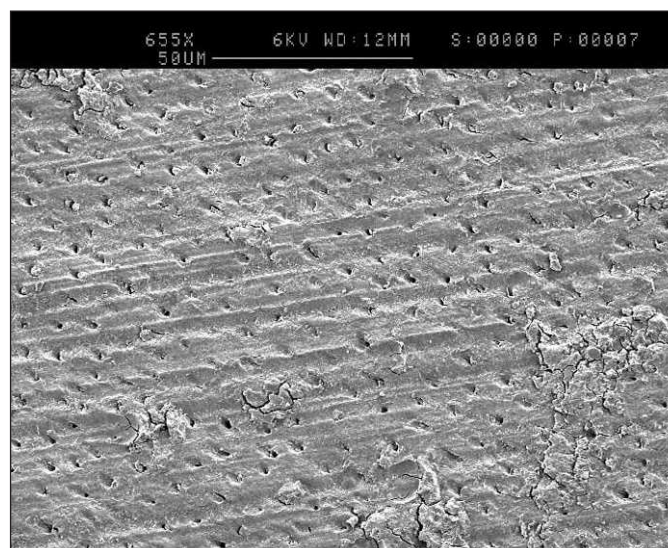


Figure 4. SEM photomicrograph of the dentin surface of a typical Ketac-Molar specimen, following debonding, showing adhesive/cohesive failure (magnification bar=50  $\mu$ m).

The Weibull modulus, therefore, gives an indication of the dependability of the test material. Relatively large sample groups need to be considered for this type of test. One or two abnormal results in a group of 10 specimens may be misleading. ISO recommendations state that a minimum of 15 specimens should be used (ISO/TC106/Subcommittee 1, 1994). One potential danger with Weibull analysis is extrapolation to the extremes of the data. Extrapolation is most valid when the data closely fits the Weibull distribution, shown by the values of correlation coefficient approaching unity (McCabe & Walls, 1986). Weibull analysis was used to calculate the  $P_f$  at different stress levels (Figures 1 and 2). The  $P_f$  was greater for Ketac-Molar than for Fuji IX in all groups and increased for both materials in Group 2 specimens at low stresses. This data will be highly meaningful when the range of stresses that are likely to be encountered clinically can be established.

In this study, salivary contamination generally did not affect the mean shear bond strength of either material to dentin. This finding is contrary to that of Prodger and Symonds (1977), Aboush and Jenkins (1987) and Safar and others (1999), who found that salivary contamination reduced bond strength. However, other investigators have looked at salivary contamination and the bond strength of DBAs to dentin and found that, in agreement with this work, it was not adversely affected (Johnson & others, 1994; Abdalla & Davidson, 1998; el-Kalla & García-Godoy, 1997). Swift and others (1995) found that saliva contamination (followed by air drying) did not significantly affect the mean shear bond strength of porcelain to resin composite. The possible explanations given were the presence of adhesive proteins in saliva or that saliva did not wet the surface and became immediately detached. It is also possible that the water content of saliva helps to maintain the structure of the collagen so as not to adversely affect bonding. Gwinnett (1994) showed that loss of moisture through simple air drying led to morphological degradation and collapse of dentinal collagen. It has been shown that, for some bonding systems, wet dentin surfaces show higher bond strengths than dry dentin (Gallo, Henderson & Burgess, 2000).

SEM showed the presence of a residue, likely to be saliva, on the dentin surface that was not washed away by water. However, this did not appear to decrease the mean bond strength. Silverstone, Hicks and Featherstone (1985) demonstrated that salivary contamination of enamel for one second or longer resulted in the formation of surface coatings that could not be removed by washing with water (for 30 seconds). Aboush and Jenkins (1987) showed salivary residue remained on the dentin surface after washing and drying but was removed by pumice, citric acid or PAA. In contrast, others found that salivary residue was still present after rinsing or rinsing and re-etching (Safar &

others, 1999). The formation of salivary pellicle in enamel has been investigated, but little is known regarding what happens when saliva comes into contact with collagen. Lendenmann, Grogan and Oppenheim (2000) state that the pellicle formed on dentures is different from that formed on enamel, that pellicle on enamel alters in its composition and structure over time and that maturation to a protective pellicle takes 18 hours *in vivo*, while transglutaminase forms covalent bonds between salivary proteins leading to cross-linking. In the studies that found a decrease in the bond strength of GICs to saliva contaminated dentin, dentin specimens were immersed in saliva for much longer than in this study. It is possible that, in 20 seconds (as used in this study), the layer of adsorbed salivary components is thin enough that either the GIC can displace it or bond through it. As the surface is exposed to saliva for longer, the nature of the residue may change. The method of contamination used in this study was felt to be more controlled and simulated the clinical situation more closely.

In this study, graphs of force *vs* displacement demonstrated fractures of a brittle nature. The slopes of the graphs were of a very similar shape in all groups; therefore, the energy to de-bond would be proportional to the force to de-bond, making an in-depth evaluation of little further use and, in this case, bond strength was probably a good indicator of toughness.

The results of this study showed mostly cohesive failures for Fuji IX regardless of group. For Ketac-Molar, adhesive/cohesive failures were observed in Groups 1 and 3 and cohesive failures in Group 2, bearing in mind that only one specimen from each test group was observed by SEM. In most of the cohesive failures, the layer of material remaining on the dentin was very thin. These findings can be used to support those of Sidhu and others (1999), who noted that the interfacial region was the weak link, irrespective of mode of failure. In microtensile bond strength tests of Fuji IX and Ketac-Molar to dentin, Yip and others (2001) found the predominant failure modes to be adhesive and mixed. Four specimens from each group, which were initially classified as adhesive failures, were examined using a transmission electron microscope (TEM). TEM analysis of the debonded dentin sides of the fractured beams revealed the presence of an intermediate layer. Several investigators have used microscopic evaluation at a much lower magnification. McInnes-Ledoux, Weinberg and Grogono (1989) found adhesive and cohesive failures between GIC base material and dentin, but used only 8x magnification. Safar and others (1999) found cohesive failure with non-contaminated specimens of RMGIC and adhesive or mixed failures with contaminated samples. However, they only used 10x magnification with a stereomicroscope.



The observations of this study are contrary to those of Hewlett, Caputo and Wrobel (1991), who found predominantly adhesive failures between GICs and dentin with or without the use of PAA. Aboush and Jenkins (1987) found predominantly adhesive failures (up to 2.3 MPa) in specimens contaminated with saliva, then washed with water and air dried. However, cohesive failure was observed in specimens that had subsequently been cleaned with PAA, citric acid or pumice. They concluded that saliva interfered with the ability of the GIC to form chemical adhesive bonds with dentin. They did state, however, that cohesive failures on dentin, as opposed to enamel, were not as readily observed and that SEM was needed.

Moisture contamination during early setting can lead to a partial loss of unreacted ions and newly formed polyacrylates, thereby weakening the cement (Suliman & others, 1989) and increasing the possibility for cohesive failure. Walls (1986) suggested that salivary contamination can cause dilution of cement at the interface, and interference by a layer of precipitated salivary proteins can alter the surface energy, thereby decreasing wetting. There is evidence that water and saliva can affect the physical properties of GICs, potentially increasing the possibility of cohesive failure within the cement (Dupuis & others, 1996; Mojon & others, 1996).

In this study, a significant difference was observed between materials. This is in agreement with Yip and others (2001) and may be attributed to differences in composition of materials (Table 1). Notwithstanding this, clinical behavior cannot be easily predicted from *in vitro* experiments. This study does not imply that one should not try to prevent salivary contamination, particularly as it appeared to show lower dependability of the material and greater probability of failure at lower stress levels. Moreover, microleakage of restorations under conditions of salivary contamination may be significant. However, it is interesting to note that salivary contamination may not have the detrimental effect on mean bond strength that might be expected. This could be particularly relevant for the ART.

## CONCLUSIONS

This *in vitro* study demonstrated that:

1. Salivary contamination did not significantly affect the mean shear bond strength of Fuji IX GP Fast and Ketac-Molar to conditioned dentin.
2. Salivary contamination increased the probability of failure at low stresses for both materials.
3. The mean shear bond strength of Fuji IX to dentin was greater than to Ketac-Molar under any of the conditions investigated.
4. The mode of failure for Fuji IX was generally cohesive within the cement and adhesive/cohesive for Ketac-Molar.

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## References

- Abdalla AI & Davidson CL (1998) Bonding efficiency and interfacial morphology of one-bottle adhesives to contaminated dentin surfaces *American Journal of Dentistry* **11**(6) 281-285.
- Aboush YE & Jenkins CB (1987) The effect of poly(acrylic acid) cleanser on the adhesion of a glass polyalkenoate cement to enamel and dentine *Journal of Dentistry* **15**(4) 147-152.
- Akinmade AO & Nicholson JW (1993) Review: Glass-ionomer cements as adhesives. Part 1. Fundamental aspects and their clinical relevance *Journal of Material Science in Medicine* **4** 95-101.
- Burke FM & Lynch E (1994) Glass polyalkenoate bond strength to dentine after chemomechanical caries removal *Journal of Dentistry* **22**(5) 283-291.
- Dietrich T, Kraemer M, Losche GM, Wernecke K-D & Roulet J-F (2000) Influence of dentin conditioning and contamination on the marginal integrity of sandwich Class II restorations *Operative Dentistry* **25**(5) 401-410.
- Dupuis V, Moya F, Payan J & Bartala M (1996) Depth microhardness of glass ionomer cements *Biomaterials* **17**(1) 71-74.
- el-Kalla IH & García-Godoy F (1997) Saliva contamination and bond strength of single-bottle adhesives to enamel and dentin *American Journal of Dentistry* **10**(2) 83-87.
- Ewoldsen N, Covey D & Lavin M (1997) The physical and adhesive properties of dental cements used for atraumatic restorative treatment *Special Care in Dentistry* **17**(1) 19-24.
- Fleming GJ, Marquis PM & Shortall AC (1999) The influence of clinically induced variability on the distribution of compressive fracture strengths of a hand-mixed zinc phosphate dental cement *Dental Materials* **15**(2) 87-97.
- Forsten L (1998) Fluoride release and uptake by glass-ionomers and related materials and its clinical effect *Biomaterials* **19**(6) 503-508.
- Fox NA, McCabe JF & Buckley JG (1994) A critique of bond strength testing in orthodontics *British Journal of Orthodontics* **21**(1) 33-43.
- Gallo JR, Henderson M & Burgess JO (2000) Shear bond strength to moist and dry dentin of four dentin bonding systems *American Journal of Dentistry* **13**(5) 267-270.
- Guggenberger R, May R & Stefan KP (1998) New trends in glass-ionomer chemistry *Biomaterials* **19**(6) 479-483.
- Gwinnett AJ (1994) Chemically conditioned dentin: A comparison of conventional and environmental scanning electron microscopy findings *Dental Materials* **10**(3) 150-155.
- Hewlett ER, Caputo AA & Wrobel DC (1991) Glass ionomer bond strength and treatment of dentin with polyacrylic acid *Journal of Prosthetic Dentistry* **66**(6) 767-772.



- ISO/TC106/Subcommittee 1 (1994) CD TR 11405—Guidance on Testing of Adhesion to Tooth Structure International Organization for Standardization, Geneva, Switzerland.
- Johnson ME, Burgess JO, Hermes CB & Buikema DJ (1994) Saliva contamination of dentin bonding agents *Operative Dentistry* **19**(6) 205-210.
- Lendenmann U, Grogan J & Oppenheim FG (2000) Saliva and dental pellicle—A review *Advances in Dental Research* **14** 22-28.
- McCabe JF & Walls AWG (1986) The treatment of results for tensile bond strength testing *Journal of Dentistry* **14**(4) 165-168.
- McInnes-Ledoux PM, Weinberg R & Grogono A (1989) Bonding glass-ionomer cements to chemomechanically-prepared dentin *Dental Materials* **5**(3) 189-193.
- McLean JW, Nicholson JW & Wilson AD (1994) Proposed nomenclature for glass-ionomer dental cements and related materials *Quintessence International* **25**(9) 587-589.
- Mojon P, Kaltio R, Feduik D, Hawbolt EB & MacEntee MI (1996) Short-term contamination of luting cements by water and saliva *Dental Materials* **12**(2) 83-87.
- Mount GJ (1991) Adhesion of glass-ionomer cement in the clinical environment *Operative Dentistry* **16**(4) 141-148.
- Mount GJ (1999) Glass ionomers: A review of their current status *Operative Dentistry* **24**(2) 115-24.
- Nakabayashi N & Pashley DH (1998) *Hybridization of Dental Hard Tissues* Tokyo, Japan Quintessence Publishing Co, Ltd.
- Øilo G (1993) Bond strength testing—what does it mean? *International Dental Journal* **43**(5) 492-498.
- Palin WM, Fleming GJ, Burke FJ, Marquis PM & Randall RC (2003) The reliability in flexural strength testing of a novel dental composite *Journal of Dentistry* **31**(8) 549-557.
- Powis DR, Folleras T, Merson SA & Wilson AD (1982) Improved adhesion of a glass ionomer cement to dentin and enamel *Journal of Dental Research* **61**(12) 1416-1422.
- Prodger TE & Symonds M (1977) ASPA Adhesion study *British Dental Journal* **143**(8) 266-270.
- Safar JA, Davis RD & Overton JD (1999) Effect of saliva contamination on the bond of dentin to resin-modified glass-ionomer cement *Operative Dentistry* **24**(6) 351-357.
- Sidhu SK, Sherriff M & Watson TF (1999) Failure of resin-modified glass-ionomers subjected to shear loading *Journal of Dentistry* **27**(5) 373-381.
- Silverstone LM, Hicks MJ & Featherstone MJ (1985) Oral fluid contamination of etched enamel surfaces: An SEM study *Journal of the American Dental Association* **110**(3) 329-332.
- Suliman AA, Schulein TM, Boyer DB & Kohout FJ (1989) Effects of etching and rinsing times and salivary contamination on etched glass-ionomer cement bonded to resin composites *Dental Materials* **5**(3) 171-175.
- Swift B, Walls AWG & McCabe JF (1995) Porcelain veneers: The effects of contaminants and cleaning regimens on the bond strength of porcelain to composite *Dental Materials* **17**(6) 203-208.
- Tanumiharja M, Burrow MF & Tyas MJ (2000) Microtensile bond strengths of glass ionomer (polyalkenoate) cements to dentine using four conditioners *Journal of Dentistry* **28**(5) 361-366.
- Tenovuo J & Lagerlof F (1994) Chapter 2: Saliva in *Textbook of Clinical Cariology* 2<sup>nd</sup> ed Munksgaard Publishing Co p 17-43.
- Tyler MW, Fitzgerald M, Dennison JB & Heys DR (1994) The effect of pulpal fluid flow on tensile bond strength of a glass-ionomer cement: An *in vivo* and *in vitro* comparison *Operative Dentistry* **19**(3) 116-120.
- Van Noort R, Noroozi S, Howard IC & Cardew G (1989) A critique of bond strength measurements *Journal of Dentistry* **17**(2) 61-67.
- Walls AW, McCabe JF & Murray JJ (1988) Factors influencing the bond strength between glass polyalkenoate (ionomer) cements and dentine *Journal of Oral Rehabilitation* **15**(6) 537-547.
- Walls AW (1986) Glass polyalkenoate (glass-ionomer) cements: A review *Journal of Dentistry* **14**(6) 231-246.
- Wilson AD & Kent BE (1972) A new translucent cement for dentistry. The glass ionomer cement *British Dental Journal* **132**(4) 133-135.
- Wilson AD & McLean JW (1988) *Glass-Ionomer Cement* Chicago, Illinois Quintessence Publishing Co.
- Yip HK, Tay FR, Ngo HC, Smales RJ & Pashley DH (2001) Bonding of contemporary glass ionomer cements to dentin *Dental Materials* **17**(5) 456-470.

# Effect of LED Curing Modes on Cusp Deflection and Hardness of Composite Restorations

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## Clinical Relevance

Using pulse or stepped curing modes with LED to cure MOD resin composite restorations decreased the polymerization-induced cusp deflection.

## SUMMARY

**This *in vitro* study measured cusp deflection associated with MOD resin composite restorations in maxillary premolars with different curing light modes. Soft-start polymerization may reduce cusp deflection by reducing polymerization shrinkage stress. Forty maxillary premolars were mounted in stone and slot MOD cavities were prepared. The teeth were randomized into four groups: Group A—cavities were etched, bonded and restored with two increments of Z-100 composite. Each increment was cured with an LED curing light (fast curing mode). Group B—similar to Group A except that the LED curing light with pulse curing mode was used. Group C—similar to Group A except that the LED curing light with stepped curing mode was used. Group D—a visible curing light was used for curing the composite. The distance between the indexed cusp tips was measured before the restorations were completed and five minutes after, 24 hours after and two**

**weeks after completion of the restorations. The mean contraction of the cusps in  $\mu\text{m}$  at five minutes, 24 hours and two weeks, respectively, for each group was A: 25.4, 16.2 and 8.2, B: 6.4, 3.4 and 2.2, C: 11.6, 7.0 and 4.4, D: 33.0, 21.6 and 15.8. Group D resulted in the highest deflection, Group A was intermediate and Groups B and C were the lowest. Ten samples of the composite for each group with 2-mm thickness were prepared for the Vickers hardness test. No difference among the samples was found.**

## INTRODUCTION

A major limitation of light-cured composites is polymerization shrinkage (Sakaguchi & others, 1991; Yap & others, 2000), which will lead to the development of mechanical stresses on the tooth structure. These stresses will either cause cusp flexure and strain on the tooth structure with subsequent tooth fracture or marginal failure with subsequent microleakage and secondary caries (Alomari, Reinhardt & Boyer, 2001).

Recently, new devices for the light-activated polymerization of composite have been introduced in addition to conventional halogen-based light activation units. These include light-emitting diode (LED), plasma arc and laser. LEDs hold several advantages over halogen-based units, including having longer lifetimes of several thousand hours, converting electricity to light more efficiently, producing less heat, not requiring filters and resistance to shock and vibration (Rueggeberg, 1999;

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Althoff & Hartung, 2000; Davidson & de Gee, 2000; Leonard & others, 2002). In addition, a higher degree of polymerization and higher stability of three-dimensional structures has been reported for resins cured using LEDs compared to halogen-based units (Nomura & others, 2002).

However, other studies have concluded that LEDs show less effectiveness of composite cure compared to halogen-based units (Dunn & Bush, 2002; Kurachi & others, 2001; Price & others, 2003a; Soh, Yap & Siow, 2003a). In addition, it was found that composite cure is device and depth dependent when different LEDs are used (Price, Felix & Andreou, 2003b; Soh & others, 2003a; Soh, Yap & Siow, 2003b; Yap & others, 2004).

Composite shrinkage can be divided into pre-gel and post-gel phases. During the pre-gel phase, composite is able to flow, causing relief of stresses within its structure (Davidson & de Gee, 1984). However, after gelation, the composite is unable to flow and cannot compensate for shrinkage, resulting in the development of stresses. The effects of post-gel polymerization can be minimized in several ways, including allowing for flow during setting by means of controlled polymerization. This can be achieved by pre-polymerization at low light intensity (soft-start polymerization), applying short pulses of light energy (pulse activation) or a combination of both. Studies have shown that these polymerization modes result in smaller marginal gaps and increased marginal integrity (Mehl, Hickel & Kunzelmann, 1997; Kanca & Suh, 1999). However, other studies have found that pulse activation and soft start regimens can affect the effectiveness of cure (Yap, Soh & Siow, 2002a; Soh & others, 2003a; Yap, Wong & Siow, 2003). The effect of pulse activation and soft-start polymerization on post-gel shrinkage of light cured composite was reported by Soh and others (2003a), who found that there was no significant difference between the control, all-pulse activation and soft-start polymerization regimens. No study was found that measured the effect of different curing regimens on the cusp flexure of teeth restored with composite.

Therefore, this study measured the effect of using an LED-curing device utilizing fast, pulse and stepped curing modes of polymerization on cusp deflection, resulting from polymerization shrinkage of resin composites. A visible curing light was used as a control group.

## METHODS AND MATERIALS

### Cusp Deflection

Forty extracted maxillary premolars, stored in 0.1% thymol, were used. The teeth were examined under 2x magnification and determined to be caries free and without fractures or cracks. The roots of the specimens were embedded in dental stone. The cemento-enamel

junctions were positioned 3 mm above the stone to prevent reinforcement of the crown by the stone. Following mounting, MOD slot cavities were prepared in the teeth using a high-speed handpiece and a #55 bur with water spray coolant. The preparations were centered between the facial and lingual cusps so as to preserve the maximum dentinal support for both cusps. The width of the cavities was 3.5 mm and the depth of the pulpal floor was 4 mm. The teeth in which pulp exposure occurred during preparation were replaced. The specimens were sorted in four categories according to size, then randomly distributed into four groups of 10 teeth each.

In Group A, the cusp tips were leveled off and a thin diamond shaped aluminum foil was bonded to each cusp tip using the Single Bond adhesive system (3M Dental Products Division, St Paul, MN, USA). The distance between the cusp tips (between the tips of the aluminum foils) was measured using a micrometer (Mitutoyo, number 2931-10, Tokyo, Japan) with an accuracy of 0.001 mm. Following measurement, the cavities were acid etched using 35% phosphoric acid for 15 seconds. The teeth were then rinsed and gently air dried to remove excess water. A dentin bonding agent (Single Bond) was applied in two layers and air dried gently, then cured with Mini LED (Satelec, ZI du phare BP 216. 33708 MERIGNAC Cedex, France, Serial #73-4105), using the fast curing mode, which provides a power of 1100mw/cm<sup>2</sup> for 10 seconds. Z100 resin composite, shade A3 (3M Dental Products, batch #247865A) was placed in two equal increments, each cured with the same curing mode for 10 seconds. No matrix band was used during the filling so that no tension would be applied to the cusps. At five minutes, the distance between the cusp tips was again measured from the same points. The teeth were then stored in water at 4°C for 24 hours. The distance between the cusp tips was then re-measured to determine the degree of stress relaxation in the cusps. The teeth were then stored under the same conditions for another measurement after two weeks.

The teeth in Group B were prepared and restored in the same way as Group A, except that a pulse curing mode with the same curing light was used for curing. This option gives full power in a pulsation mode with an emission of 10 successive flashes, including a rest period of 250 milliseconds between flashes. The power was the same as that described for the fast curing menu.

The same procedures were followed in Group C as for Group A, except that a step curing option with the same curing light was used for curing. This option includes soft start for 10 seconds from 0 to 1100 mW/cm<sup>2</sup>, then full power during 10 seconds.

In Group D, a visible curing light (Caulk Spectrum 800, Caulk Division, Dentsply International Inc, Milford, DE, USA) was used for curing. The intensity of



the light unit was 800 mW/cm<sup>2</sup>. Each increment was cured for 40 seconds.

In all groups, the distance between cusp tips was measured four times: before the restoration, five minutes after curing, 24 hours after curing and at two weeks after restoration.

### Hardness Testing

Forty samples of the same Z100 composite material were packed into circular Teflon molds with internal dimensions of 2-mm diameter and 2-mm height. Two glass slides (1-mm thick) were then placed on both sides of the molds and excess composite was extruded by pressure application. The samples were divided into four groups of 10 each. Each group was irradiated according to the regimen for cusp deflection (Group A: continuous mode; Group B: pulse curing mode; Group C: stepped curing mode and Group D: visible light curing mode). The samples were irradiated from the top through the glass slide. A microhardness tester (MHT-1, Matsuzawa Seiki Co LTD, Tokyo, Japan) was used to measure Vickers hardness of the top and bottom surfaces. A 50-g load was applied through the indenter with a dwell time of 15 seconds. Three indentations at the surface of each material were performed. The statistical analysis employed the average of these three readings. The Vickers hardness (HV) number was computed as follows: HV=Test load (kgf)/ Surface area of indentation (mm<sup>2</sup>).

### Statistical Analysis

The means and standard deviations of the differences between the measurements at five minutes, 24 hours, two weeks and the pre-restoration measurements were calculated. The means and standard deviations of the differences between the measurements at 24 hours, two weeks and the pre-restoration measurements were also calculated. The means and standard deviations of the Vickers hardness of the groups were calculated for both the top and bottom surfaces. One way ANOVA and Duncan's multiple range tests were used to compare the groups at a significance level of  $\alpha < 0.05$ .

## RESULTS

The means and standard deviations of cuspal deflection at five minutes (difference between the intercusp distance at five minutes and before the restorations) are reported for each group in Table 1. The means, in  $\mu\text{m}$ , from the lowest cusp deflection to the highest were Group B (the pulse cure mode): 6.4; Group C (the

step curing mode): 11.6; Group A (the continuous curing mode): 25.4 and Group D (the visible curing light): 33.0. One-way analysis of variance indicated a significant difference between groups ( $\alpha < 0.05$ ). The Duncan test showed that Groups A and D were different from the other two groups and gave the highest cusp deflection. There was no statistical difference between Groups B and C, which showed the lowest cusp deflection.

Table 2 shows the means and standard deviations of cuspal deflection at 24 hours for each group. Again, the means in  $\mu\text{m}$  from the lowest cusp deflection to the highest were Group B: 3.4; Group C: 7.0; Group A: 16.2 and Group D: 21.6. One-way analysis of variance detected differences among the groups. The Duncan test showed that Group D had the highest cusp deflection, Group A was intermediate and Groups B and C had the lowest cusp deflection.

Table 3 shows the means and standard deviations of cusp deflection at two weeks for each group. The ascending order of the cusp deflection in  $\mu\text{m}$  was Group B: 2.2; Group C: 4.4; Group A: 8.2 and Group D: 15.8. Again one-way analysis of variance detected differences among the groups. The Duncan test showed that only Group D was different from the other three groups and had the highest cusp deflection.

Table 4 presents differences between values in Tables 1 and 2, while Table 5 represents differences between

Table 1: Means and Standard Deviations ( $\mu\text{m}$ ) of the Cusp Deflection at Five Minutes after Curing

Group	Mean $\pm$ SD ( $\mu\text{m}$ )	Range ( $\mu\text{m}$ )
Fast curing mode (LED) (A)	25.4 $\pm$ 11.2 a	25
Pulse curing mode (LED) (B)	6.4 $\pm$ 0.9 b	2
Step curing mode (LED) (C)	11.6 $\pm$ 0.5 b	1
Visible curing light (D)	33.0 $\pm$ 3.4 a	8

Duncan's homogeneous subsets are displayed ( $p < 0.05$ ).

Table 2: Means and Standard Deviations ( $\mu\text{m}$ ) of Cusp Deflection at 24 Hours

Group	Mean $\pm$ SD ( $\mu\text{m}$ )	Range ( $\mu\text{m}$ )
Fast curing mode (LED) (A)	16.2 $\pm$ 6.1 a	14
Pulse curing mode (LED) (B)	3.4 $\pm$ 0.5 b	1
Step curing mode (LED) (C)	7.0 $\pm$ 1.9 b	4
Visible curing light (D)	21.6 $\pm$ 2.9 c	7

Duncan's homogeneous subsets are displayed ( $p < 0.05$ ).

Table 3: Means and Standard Deviations ( $\mu\text{m}$ ) of Cusp Deflection at Two Weeks

Group	Mean $\pm$ SD ( $\mu\text{m}$ )	Range ( $\mu\text{m}$ )
Fast curing mode (LED) (A)	8.2 $\pm$ 7.3 a	15
Pulse curing mode (LED) (B)	2.2 $\pm$ 1.3 a	3
Step curing mode (LED) (C)	4.4 $\pm$ 1.8 a	4
Visible curing light (D)	15.8 $\pm$ 4.8 b	12

Duncan's homogeneous subsets are displayed ( $p < 0.05$ ).



values in Tables 1 and 3. These two tables represent the degree of cusp relaxation at 24 hours and two weeks, respectively. The means of cusp relaxation in  $\mu\text{m}$  at 24 hours from the lowest to the highest were Group B: 3.0; Group C: 4.6; Group A: 9.2 and Group D: 11.4. One-way analysis of variance showed a difference among the groups and the Duncan test showed that Groups A and D had higher cusp relaxation than the other two groups.

The means of the values of cusp relaxation in  $\mu\text{m}$  at two weeks from the lowest to the highest were Group B: 4.2; Group C: 7.2; Group A: 17.2 and Group D: 17.2. One-way analysis of variance showed a difference among the groups, and again the Duncan test showed that Groups A and D were in one statistical group, while Groups B and C were in another statistical group.

The mean hardness (top and bottom) for the groups is shown in Table 6. This table also shows the ratio between the bottom and top hardness numbers. One-way analysis of variance showed no difference among the groups in Vickers hardness.

## DISCUSSION

Measuring cusp deflection has been used to examine the effect of polymerization shrinkage stress of resin composites on tooth structure in more than one study (Causton, Miller & Sefton, 1985; McCulloch & Smith,

1986). The amount of cusp deflection has been shown to vary according to different variables, among which include the type of the resin composite, size of the cavity, type of tooth and measurement method used. The results of this study varied between 6.4  $\mu\text{m}$  and 33.0  $\mu\text{m}$ , the groups with low cusp deflection were those cured with pulse cure or stepped mode of curing, while the groups with higher cusp deflection were those with continuous curing mode. Composites cured with pulse and stepped modes of curing will have lower rates of polymerization and, consequently, lower rates of shrinkage force development compared to those cured with continuous curing mode. In agreement with this, Bouschlicher and Rueggeberg (2000) found that the maximum shrinkage force and force rate exhibited during the first 250 seconds of polymerization were significantly lower, using a ramped light intensity exposure. In a different study, Bouschlicher, Rueggeberg and Boyer (2000) found that stepped curing exhibited longer delays before force was recorded and also mode of curing was shown not to contribute to the overall cure.

On the other hand, Oberholzer and others (2003) found significantly lower bond strengths when curing was done by gradually increasing the intensity compared to curing with fixed intensities. They concluded that the advantage of initial slow polymerization (more elasticity and less tension) obtained by the so-called "soft start" method was offset by a rise in total polymerization shrinkage when final curing was completed at 1130  $\text{mW}/\text{cm}^2$ . According to Chen and others (2003), high contraction stress, early start of stress build-up and rapid contraction force development may lead to failure of bond to tooth structure. This could explain why Group A (LED continuous curing mode) had lower cusp deflection than Group D (halogen curing light), since the LED curing mode has higher light intensity. Also, the curing time for Group A was longer than the other groups.

In this study, in general, the groups with continuous, high curing light intensities showed the highest deflection, which means that the bond to tooth structure was intact and the stress of contraction was transferred through this bond to the weak cusps, deforming them. Although not statistically significant, the halogen curing light resulted in higher cusp deflection than the continuous mode of the LED curing light. This could be attributed to the temperature rise during polymerization and heating from radia-

Table 4: Means and Standard Deviations ( $\mu\text{m}$ ) of the Cusp Relaxation at 24 Hours

Group	Mean $\pm$ SD ( $\mu\text{m}$ )	Range ( $\mu\text{m}$ )
Fast curing mode (LED) (A)	9.2 $\pm$ 5.2 a	12
Pulse curing mode (LED) (B)	3.0 $\pm$ 1.2 b	3
Step curing mode (LED) (C)	4.6 $\pm$ 1.7 b	4
Visible curing light (D)	11.4 $\pm$ 1.9 a	5

Duncan's homogeneous subsets are displayed ( $p < 0.05$ ).

Table 5: Means and Standard Deviations ( $\mu\text{m}$ ) of the Cusp Relaxation at Two Weeks

Group	Mean $\pm$ SD ( $\mu\text{m}$ )	Range ( $\mu\text{m}$ )
Fast curing mode (LED) (A)	17.2 $\pm$ 6.1 a	14
Pulse curing mode (LED) (B)	4.2 $\pm$ 1.3 b	3
Step curing mode (LED) (C)	7.2 $\pm$ 1.6 b	3
Visible curing light (D)	17.2 $\pm$ 3.4 a	9

Duncan's homogeneous subsets are displayed ( $p < 0.05$ ).

Table 6: Means and Standard Deviations of Vickers Hardness

Group	Top Surface	Bottom Surface	Ratio
Fast curing mode (LED) (A)	74.5 $\pm$ 1.59a	68.7 $\pm$ 3.9a	92.1 $\pm$ 5.1 a
Pulse curing mode (LED) (B)	74.9 $\pm$ 14.1a	70.7 $\pm$ 14.6a	94.3 $\pm$ 4.7 a
Step curing mode (LED) (C)	82.8 $\pm$ 10.8a	78.4 $\pm$ 8.4a	95.0 $\pm$ 5.3 a
Visible curing light (D)	72.3 $\pm$ 2.1a	66.8 $\pm$ 1.5a	92.0 $\pm$ 4.1 a

Duncan's homogeneous subsets are displayed ( $p < 0.05$ ).

tion. Hofmann, Hugo and Klaiber (2002) observed that the temperature rise with halogen light irradiation was higher than with LED. The temperature and higher radiation heat result in a faster polymerization reaction and, consequently, an increase in cuspal deflection. On the other hand, Yap, Soh and Siow (2002b) found no statistical significance in post-gel shrinkage between a control and all-pulse activation/soft-start polymerization regimens.

Segura and Donly (1993) found that the degree of cusp relaxation after six months water immersion was about 60% from the original deflection. Others found the change in volume of the resin composite to range from 0.15% to 2.39%, based on the type of composite (Martin, Jedyakiewicz & Fisher, 2003). In this study, after 24 hours of water immersion, the degree of cusp relaxation was 34.5% to 46.9% of the immediate cusp deflection, while after two weeks of water immersion, about 52.1% to 67.7% of the immediate cusp deflection was relaxed. Cusp relaxation, in general, can occur due to hygroscopic expansion, elasticity of tooth structures, gap formation or tooth fracture.

The hardness of the resin composite was measured in this study to ensure that the difference in cusp deflection among the groups is due to the difference in curing mode and not to the degree of polymerization of the material.

Dennison and others (2000) found that sequentially increasing curing light intensity reduces polymerization shrinkage without compromising depth of cure. The results of this study also show no difference in hardness among the curing modes used. Yap and others (2002b) found that effectiveness of cure at the bottom surfaces of composites may be reduced by some pulse activation and soft-start polymerization regimens. However, Soh and others (2003b) found that the mean hardness ratio for all curing lights at a depth of 2 mm to be greater than 0.80 (the accepted minimum standard).

## CONCLUSIONS

This *in vitro* study showed that polymerization shrinkage of resin composite restorations can lead to stress on tooth structures. This stress can be measured as cusp deflection. After five minutes of curing, the maximum cusp deflection was 33.0  $\mu\text{m}$  (curing with halogen light) and the minimum was 6.4  $\mu\text{m}$  (with the pulse curing mode). After 24 hours, the maximum cusp deflection was 21.6  $\mu\text{m}$  using the halogen curing light, while the minimum was 3.4  $\mu\text{m}$  when using a pulse curing mode. After two weeks of water immersion, the maximum cusp deflection was 15.8  $\mu\text{m}$  using the halogen curing light, while the minimum was 2.2  $\mu\text{m}$  for the pulse curing mode.

The use of pulse and ramped curing modes reduced cusp deflection at five minutes after curing, after 24

hours and after two weeks. The pulse curing mode was mildly more effective in this regard than ramped curing, although there was no statistical difference between the two curing modes.

Water immersion of the samples for two weeks resulted in cusp relaxation between 4.2  $\mu\text{m}$  for Pulse curing mode and 17.2  $\mu\text{m}$  for both the fast LED curing mode and the halogen curing light.

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## References

- Alomari QD, Reinhardt JW & Boyer DB (2001) Effect of liners on cusp deflection and gap formation in composite restorations *Operative Dentistry* **26**(4) 406-411.
- Althoff O & Hartung M (2000) Advances in light curing *American Journal of Dentistry* **13**(Special Issue) 77D-81D. Review.
- Bouschlicher MR, Rueggeberg FA & Boyer DB (2000) Effect of stepped light intensity on polymerization force and conversion in a photoactivated composite *Journal of Esthetic Dentistry* **12**(1) 23-32.
- Bouschlicher MR & Rueggeberg FA (2000) Effect of ramped light intensity on polymerization force and conversion in a photoactivated composite *Journal of Esthetic Dentistry* **12**(6) 328-339.
- Causton BE, Miller B & Sefton J (1985) The deformation of cusps by bonded posterior composite restorations: An *in vitro* study *British Dental Journal* **159**(12) 397-400.
- Chen HY, Manhart J, Kunzelmann KH & Hickel R (2003) Polymerization contraction stress in light-cured compomer restorative materials *Dental Materials* **19**(7) 597-602.
- Davidson CL & de Gee AJ (2000) Light-curing units, polymerization, and clinical implications *Journal of Adhesive Dentistry* **2**(3) 167-173.
- Davidson CL & de Gee AJ (1984) Relaxation of polymerization contraction stresses by flow in dental composites *Journal of Dental Research* **63**(2) 146-148.
- Dennison JB, Yaman P, Seir R & Hamilton JC (2000) Effect of variable light intensity on composite shrinkage *Journal of Prosthetic Dentistry* **84**(5) 499-505.
- Dunn WJ & Bush AC (2002) A comparison of polymerization by light-emitting diode and halogen-based light-curing units *Journal of the American Dental Association* **133**(3) 335-341.
- Hofmann N, Hugo B & Klaiber B (2002) Effect of radiation type (LED or QTH) on photo-activated composite shrinkage strain kinetics, temperature rise, and hardness *European Journal of Oral Sciences* **110**(6) 471-479.
- Kanca J 3<sup>rd</sup> & Suh BI (1999) Pulse activation: Reducing resin-based composite contraction stresses at the enamel cavosurface margins *American Journal of Dentistry* **12**(3) 107-112.
- Kurachi C, Tuboy AM, Maalhaes DV & Bagnato VS (2001) Hardness evaluation of a dental composite polymerized with experimental LED-based devices *Dental Materials* **17**(4) 309-315.
- Leonard DL, Charlton DG, Roberts HW & Cohen ME (2002) Polymerization efficiency of LED curing lights *Journal of Esthetic and Restorative Dentistry* **14**(5) 286-295.

- Martin N, Jedynekiewicz NM & Fisher AC (2003) Hygroscopic expansion and solubility of composite restoratives *Dental Materials* **19**(2) 77-86.
- McCulloch AJ & Smith BJN (1986) *In vitro* studies of cuspal movement produced by adhesive restorative materials *British Dental Journal* **161**(11) 405-409.
- Mehl A, Hickel R & Kunzelmann KH (1997) Physical properties and gap formation of light-cured composites with and without "soft start-polymerization" *Journal of Dentistry* **25**(3-4) 321-330.
- Nomura Y, Teshima W, Tanaka N, Yoshida Y, Nahara Y & Okazaki M (2002) Thermal analysis of dental resins cured with blue light-emitting diodes (LEDs) *Journal of Biomedical Materials Research* **63**(2) 209-213.
- Oberholzer TG, Pameijer CH, Grobler SR & Rossouw RJ (2003) The effect of different power densities and method of exposure on the marginal adaptation of four light-cured dental restorative materials *Biomaterials* **24**(20) 3593-3598.
- Price RB, Ehrnford L, Andreou P & Felix CA (2003a) Comparison of quartz-tungsten-halogen, light-emitting diode, and plasma arc curing lights *Journal of Adhesive Dentistry* **5**(3) 193-207.
- Price RB, Felix CA & Andreou P (2003b) Evaluation of a second-generation LED curing light *Journal of the Canadian Dental Association* **69**(10) 666.
- Rueggeberg F (1999) Contemporary issues in photocuring *Compendium of Education of Dental Supplement* **25**(S4-15) quiz S73.
- Sakaguchi RL, Sasik CT, Bunczak MA & Douglas WH (1991) Strain gauge method for measuring polymerization contraction of composite restoratives *Journal of Dentistry* **19**(5) 312-316.
- Segura A & Donly KJ (1993) *In vitro* posterior composite polymerization recovery following hygroscopic expansion *Journal of Oral Rehabilitation* **20**(5) 495-499.
- Soh MS, Yap AUJ & Siow KS (2003a) Effectiveness of composite cure associated with different curing modes of LED lights *Operative Dentistry* **28**(4) 371-377.
- Soh MS, Yap AU & Siow KS (2003b) The effectiveness of cure of LED and halogen curing lights at varying cavity depths *Operative Dentistry* **28**(6) 707-715.
- Yap AU, Ng JJ, Yap SH & Teo CK (2004) Surface finish of resin-modified and highly viscous glass ionomer cements produced by new one-step systems *Operative Dentistry* **29**(1) 87-91.
- Yap AU, Wang HB, Siow KS & Gan LM (2000) Polymerization shrinkage of visible light-cured composites *Operative Dentistry* **25**(2) 98-103.
- Yap AU, Wong NY & Siow KS (2003) Composite cure and shrinkage associated with high intensity curing light *Operative Dentistry* **28**(4) 357-364.
- Yap AU, Soh MS & Siow KS (2002a) Post-gel shrinkage with pulse activation and soft-start polymerization *Operative Dentistry* **27**(1) 81-87.
- Yap AU, Soh MS & Siow KS (2002b) Effectiveness of composite cure with pulse activation and soft-start polymerization *Operative Dentistry* **27**(1) 44-49.

# Fluoride Release/Recharge from Restorative Materials— Effect of Fluoride Gels and Time

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## Clinical Relevance

A four-minute APF gel application led to a higher fluoride recharge by the tested materials; the resin-modified glass ionomers were the most influenced by this procedure.

## SUMMARY

**This study examined the differences in fluoride release and recharge among four restorative materials following treatment with APF or neutral fluoride gel for one or four minutes. Specimens were immersed in 2 mL of deionized water, while fluoride release was measured at 24-hour intervals for 15 days using an ion-selective electrode and analyzer. The materials were then treated with the fluoride gels. The fluoride**

**release was measured for 15 days. ANOVA ( $p < 0.05$ ) showed higher fluoride release for Ketac-Fil before fluoride application and for Vitremer and Fuji II LC after application of APF gel. APF gel yielded higher fluoride release when compared to neutral gel, regardless of the material. Fluoride recharge and release was greater after the four-minute APF gel application, with no difference between the times of application for the neutral gel ( $p > 0.05$ ), except for Ketac-Fil. The pattern of release before and after application of the gels was similar and was higher at day 16 compared to day one for the APF gel for resin materials, with higher release at day 15 compared to the initial for Fuji II LC and Vitremer. It was concluded that RM-GICs were the most effective materials with regards to fluoride release after application of APF gel for four minutes.**

## INTRODUCTION

Fluoride release is one of the advantages of glass ionomer cements. It was found that secondary caries initiation and propagation were significantly reduced when glass ionomer restorations were placed (Retief & others, 1984). Glass ionomer materials are recharged by fluoride from fluoridated solutions and gels (Takahashi, Emilson & Birkhed, 1993; Pedrini & others, 2003). This implies that these materials can serve

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as rechargeable fluoride reservoirs, delivering a low level of fluoride to the oral cavity after the ion source has been removed (Suljak & Hatibovic-Kofman, 1996). Since fluoride release varies with materials, it is reasonable that fluoride recharge will depend on the material and type of fluoride (Diaz-Arnold & others, 1995). However, it is not possible to determine how fluoride is released to the environment, whether it is adsorbed to the surface or incorporated into the material matrix, since fluoridated products are applied daily in study protocols (Bilgin & Ozalp, 1998; Yip & Smales, 1999).

Experimental studies have varied the time of topical application of fluoridated products on restorative materials from 2 to 10 minutes (Takahashi & others, 1993; Diaz-Arnold & others, 1995; Suljak & Hatibovic-Kofman, 1996; Bilgin & Ozalp, 1998). From these studies, the effect of the fluoride release based on the time of application is still controversial. Application times of one or four minutes have been indicated for the professional application of fluoridated gel, and investigations have (Delbem & Cury, 2002) or have not demonstrated (Hebling, Santos-Pinto & Cury, 1995) differences between the times of application on the fluoride recharge by enamel.

Therefore, it should be verified whether a reduction in time of the topical fluoride application would lead to reduced fluoride recharge and release by restorative materials, since the literature suggests adoption of a shorter time of application (García-Godoy & others, 1995; Delbem & Cury, 2002).

This study evaluated the ability of fluoride release and recharge of four restorative materials after treatment with acidulated phosphate fluoride (APF) and neutral sodium fluoride (NaF), with times of application of one and four minutes.

METHODS AND MATERIALS

The materials are described in Table 1. Twenty-four cylindrical specimens measuring 5 mm in diameter and

2 mm in height, for a surface area of 0.71 cm<sup>2</sup>, were made from each material (total 96 specimens) following the manufacturers' instructions.

The specimens were divided into four groups, consisting of six specimens of each material (Araujo & others, 1996). Chemically cured specimens (Ketac-Fil Plus) were kept in the mold under matrix protection for 10 minutes. Light-cured materials (Vitremer, Fuji II LC and Freedom) were polymerized for 40 seconds from the top surface of each specimen with an Ultralux light source (Dabi Atlante, Ribeirão Preto, SP, Brazil) with a light intensity of 500 mW/cm<sup>2</sup> (Martins & others, 2002). The specimens were randomly grouped and simultaneously immersed in water after four hours (length of time necessary for their preparation). Each specimen was suspended by the cap of an individual polystyrene tube with a 0.25-mm diameter stainless steel wire. Each polystyrene tube contained 2 mL of deionized water and was vibrated (Carvalho & Cury, 1999) for 24 hours at room temperature. An equal volume of TISAB II (acetate buffer 1.0 mol L<sup>-1</sup>, pH 5.2, CDTA 0.4%) was added to the tubes. The specimens were washed with a deionized water spray, dried with absorbent paper and transferred to new tubes containing 2 mL of deionized water.

Fifteen days after initial fluoride release, the specimens from each product were exposed to either APF gel, containing 1.23% fluoride ions at pH 3.6 to 3.9 (Nupro, Dentsply, Petrópolis, RJ, Brazil), for one minute and four minutes or 2% NaF gel at pH 6.6 to 6.9 (Nupro, Dentsply) for one minute or four minutes. After the time elapsed, the excess gel was vigorously washed off for 30 seconds with deionized water; the specimens were dried with absorbent paper and immersed in tubes containing 2 mL of deionized water. Thereafter, they were kept in tubes with deionized water and vibrated for 24-hour periods for 15 days, according to the aforementioned method.

The solutions were collected daily, identified and stored in polystyrene tubes at 4°C for later determination of fluoride release. The fluoride release was measured using a fluoride-specific electrode (ORION 9609-BN, Orion Research, Inc, Beverly, MA, USA) that was connected to a digital ion analyzer (ORION 720A, Orion Research,

Table 1: Materials Tested for Fluoride Release and Recharge			
Material	Type of Material (Symbol)	Manufacturer	Batch # (powder/liquid)
Ketac-Fil Plus	Conventional glass ionomer cement (C-GIC)	3M ESPE, St Paul, MN, USA	113296/114819
Vitremer	Resin-modified glass ionomer cement (RM-GIC)	3M ESPE, St Paul, MN, USA	20000427/20000331
Fuji II LC	Resin-modified glass ionomer cement (RM-GIC)	GC Corporation, 76-1 Hasunuma-Cho, Itabashi-Ku, Tokyo, Japan	0106011/0108021
Freedom	Polyacid-modified resin composite (PM-CR)	Southern Dental Industries, Bayswater, Victoria, Australia	002346

Inc). The measurements were expressed in  $\mu\text{g F/cm}^2$ . A standard curve between 0.0625 and 1.0  $\mu\text{g F/mL}$  or from 1.0 to 16.0  $\mu\text{g F/mL}$  in TISAB II was used to calibrate the electrode. Measurements were performed daily, yet interactions comprising the variable day were calculated for days 1, 7, 15, 16, 22 and 30.

Statistical analysis demonstrated the heterogeneity of data, which required cubic root transformation. Analysis of variance was performed considering material, gel, time of application and days. The difference between the mean fluoride release before and after topical application of APF or neutral gel was evaluated using the paired *t*-test. Analyses were performed using

GMC software version 2002 (Campos, 2002) at a significance level of 5%.

## RESULTS

The total mean fluoride released during the first 15 days was different ( $p < 0.05$ ) between materials, regardless of the group (neutral or APF gel), and was higher for the conventional ionomer (Table 2). There was a difference between materials after topical fluoride application ( $p < 0.05$ ); Vitremer displayed higher fluoride release after application of both neutral and APF gel. APF gel yielded higher fluoride release than the neutral gel (Table 2); RM-GICs showed a higher release ( $p < 0.05$ ).

Comparison of the materials throughout time (Table 3) revealed that the application of APF gel increased the fluoride release at day 16, except for the conventional ionomer, which displayed a similar value as observed at day one ( $p > 0.05$ ). Similar results were observed for the material Fuji II LC with neutral gel; yet, other materials presented different behaviors (Table 3).

The effect of the time of application of each gel throughout time can be assessed by individual analysis of the materials, as presented in Tables 4, 5, 6 and 7.

With regard to the resin-modified glass ionomer cement (Tables 4 and 6), there was higher fluoride release at day 16 compared to day one for each gel applied for four minutes ( $p < 0.05$ ). The APF gel applied for four minutes revealed higher release throughout time when compared to the first 15 days ( $p < 0.05$ ).

Regarding the conventional glass-ionomer cement, the time of application

Table 2: Comparison Between the Mean Total Fluoride Released ( $\mu\text{g F/cm}^2$ ) by the Restorative Materials According to the Gel Applied

Material	Before TFA *		After TFA	
	APF	Neutral	APF	Neutral
Vitremer	<sup>#A</sup> 2.70 <sup>b+</sup>	<sup>&amp;A</sup> 2.71 <sup>b</sup>	<sup>B</sup> 4.37 <sup>a</sup>	<sup>B</sup> 2.15 <sup>b,c</sup>
Ketac-Fil	<sup>A</sup> 3.45 <sup>a</sup>	<sup>A</sup> 3.61 <sup>a</sup>	<sup>B</sup> 2.58 <sup>c</sup>	<sup>B</sup> 2.10 <sup>d</sup>
Fuji II LC	<sup>A</sup> 1.30 <sup>c</sup>	<sup>A</sup> 1.30 <sup>c</sup>	<sup>B</sup> 2.42 <sup>b</sup>	<sup>A</sup> 1.53 <sup>e</sup>
Freedom	<sup>A</sup> 0.43 <sup>d</sup>	<sup>A</sup> 0.40 <sup>d</sup>	<sup>A</sup> 0.74 <sup>f</sup>	<sup>B</sup> 0.10 <sup>g</sup>

\* Topical fluoride application.

+ Lower-case letters should be interpreted in each column or row before or after TFA.

# Capital letters related to comparison before and after TFA for the APF gel.

& Capital letters related to comparison before and after TFA for the neutral gel.

Table 3: Mean Values of Fluoride Release ( $\mu\text{g F/cm}^2$ ) for the Interaction Between Days and Gel for the Restorative Materials

Days	Material							
	Vitremer		Ketac-Fil Plus		Fuji II LC		Freedom	
	Acid	Neut	Acid	Neut	Acid	Neut	Acid	Neut
1	8.56 <sup>b+</sup>	8.56 <sup>b</sup>	14.14 <sup>a</sup>	14.21 <sup>a</sup>	4.79 <sup>c</sup>	4.80 <sup>c</sup>	1.32 <sup>b</sup>	1.37 <sup>b</sup>
7	1.41 <sup>d,e</sup>	1.51 <sup>d,e</sup>	1.68 <sup>c,d</sup>	1.84 <sup>c</sup>	0.70 <sup>e,f</sup>	0.73 <sup>e,f</sup>	0.28 <sup>c,d</sup>	0.21 <sup>c,d,e</sup>
15	1.22 <sup>d,e</sup>	1.23 <sup>d,e</sup>	1.03 <sup>e</sup>	1.10 <sup>d,e</sup>	0.47 <sup>f</sup>	0.48 <sup>f</sup>	0.13 <sup>d,e,f</sup>	0.11 <sup>e,f</sup>
16	19.27 <sup>a</sup>	8.93 <sup>b</sup>	14.96 <sup>a</sup>	11.60 <sup>b</sup>	11.74 <sup>a</sup>	8.17 <sup>b</sup>	6.30 <sup>a</sup>	0.36 <sup>c</sup>
22	2.65 <sup>c</sup>	1.50 <sup>d,e</sup>	0.97 <sup>e</sup>	0.88 <sup>e</sup>	1.27 <sup>d</sup>	0.75 <sup>e,f</sup>	0.07 <sup>f</sup>	0.07 <sup>f</sup>
30	1.79 <sup>d</sup>	1.06 <sup>e</sup>	0.83 <sup>e</sup>	0.84 <sup>e</sup>	1.09 <sup>d,e</sup>	0.86 <sup>d,e</sup>	0.05 <sup>f</sup>	0.05 <sup>f</sup>

+ Letters should be interpreted in each column or row within each material.

Table 4: Mean Values of Fluoride Release ( $\mu\text{g F/cm}^2$ ) for Vitremer for the Interaction Days x Time of Application x Gel

Days	APF		Neutral	
	1 Minute	4 Minutes	1 Minute	4 Minutes
1	<sup>B</sup> 8.61 <sup>a</sup>	<sup>B</sup> 8.52 <sup>a</sup>	<sup>A</sup> 8.57 <sup>a</sup>	<sup>B</sup> 8.56 <sup>a</sup>
7	<sup>D</sup> 1.51 <sup>a</sup>	<sup>E</sup> 1.32 <sup>a</sup>	<sup>C</sup> 1.62 <sup>a</sup>	<sup>C,D</sup> 1.40 <sup>a</sup>
15	<sup>D</sup> 1.30 <sup>a</sup>	<sup>E</sup> 1.15 <sup>a</sup>	<sup>C,D</sup> 1.27 <sup>a</sup>	<sup>C,D</sup> 1.19 <sup>a</sup>
16	<sup>A</sup> 14.51 <sup>b</sup>	<sup>A</sup> 24.03 <sup>a</sup>	<sup>B</sup> 6.60 <sup>d</sup>	<sup>A</sup> 11.26 <sup>c</sup>
22	<sup>C</sup> 2.25 <sup>b</sup>	<sup>C</sup> 3.05 <sup>a</sup>	<sup>C,D</sup> 1.44 <sup>c</sup>	<sup>C</sup> 1.56 <sup>c</sup>
30	<sup>D</sup> 1.61 <sup>a</sup>	<sup>D</sup> 1.97 <sup>a</sup>	<sup>D</sup> 1.07 <sup>b</sup>	<sup>D</sup> 1.06 <sup>b</sup>
Means*	3.58 <sup>B</sup>	5.15 <sup>A</sup>	1.86 <sup>C</sup>	2.44 <sup>C</sup>

Means followed by different lower-case letters are different from each other at the level of 5% within each row.

Means preceded by different capital letters are different from each other at the level of 5% within each column.

\* Total mean values of the last 15 days after TFA and different capital letters are different from each other at the level of 5%.

Table 5: Mean Values of Fluoride Release ( $\mu\text{g F}^-/\text{cm}^2$ ) for Ketac-Fil Plus for the Interaction Days x Time of Application x Gel

Days	APF		Neutral	
	1 Minute	4 Minutes	1 Minute	4 Minutes
1	<sup>A</sup> 14.09 <sup>a</sup>	<sup>A</sup> 14.17 <sup>a</sup>	<sup>A</sup> 14.04 <sup>a</sup>	<sup>A</sup> 14.37 <sup>a</sup>
7	<sup>B</sup> 1.74 <sup>a</sup>	<sup>B</sup> 1.62 <sup>a</sup>	<sup>C</sup> 1.96 <sup>a</sup>	<sup>B</sup> 1.72 <sup>a</sup>
15	<sup>C</sup> 1.07 <sup>a</sup>	<sup>C</sup> 0.99 <sup>a</sup>	<sup>D</sup> 1.14 <sup>a</sup>	<sup>C</sup> 1.06 <sup>a</sup>
16	<sup>A</sup> 13.97 <sup>a</sup>	<sup>A</sup> 15.95 <sup>a</sup>	<sup>B</sup> 7.37 <sup>b</sup>	<sup>A</sup> 15.83 <sup>a</sup>
22	<sup>C</sup> 1.01 <sup>a</sup>	<sup>C</sup> 0.93 <sup>a</sup>	<sup>D</sup> 0.89 <sup>a</sup>	<sup>C</sup> 0.87 <sup>a</sup>
30	<sup>C</sup> 0.86 <sup>a</sup>	<sup>C</sup> 0.81 <sup>a</sup>	<sup>a</sup> 0.88 <sup>a</sup>	<sup>C</sup> 0.80 <sup>a</sup>
Means*	2.52 <sup>A</sup>	2.65 <sup>A</sup>	1.63 <sup>B</sup>	2.56 <sup>A</sup>

Means followed by different lower-case letters are different from each other at the level of 5% within each row.

Means preceded by different capital letters are different from each other at the level of 5% within each column.

\* Total mean values of the last 15 days after TFA and different capital letters are different from each other at the level of 5%.

Table 6: Mean Values of Fluoride Release ( $\mu\text{g F}^-/\text{cm}^2$ ) for Fuji II LC for the Interaction Days x Time of Application x Gel

Days	APF		Neutral	
	1 Minute	4 Minutes	1 Minute	4 Minutes
1	<sup>B</sup> 4.86 <sup>a</sup>	<sup>B</sup> 4.73 <sup>a</sup>	<sup>B</sup> 4.88 <sup>a</sup>	<sup>B</sup> 4.72 <sup>a</sup>
7	<sup>D</sup> 0.75 <sup>a</sup>	<sup>D</sup> 0.65 <sup>a</sup>	<sup>C</sup> 0.73 <sup>a</sup>	<sup>C</sup> 0.73 <sup>a</sup>
15	<sup>E</sup> 0.49 <sup>a</sup>	<sup>D</sup> 0.45 <sup>a</sup>	<sup>D</sup> 0.50 <sup>a</sup>	<sup>D</sup> 0.46 <sup>a</sup>
16	<sup>A</sup> 9.10 <sup>b</sup>	<sup>A</sup> 14.38 <sup>a</sup>	<sup>A</sup> 6.60 <sup>c</sup>	<sup>A</sup> 9.73 <sup>b</sup>
22	<sup>C</sup> 1.04 <sup>b</sup>	<sup>C</sup> 1.50 <sup>a</sup>	<sup>C,D</sup> 0.69 <sup>c</sup>	<sup>C</sup> 0.80 <sup>b,c</sup>
30	<sup>C,D</sup> 0.99 <sup>a,c</sup>	<sup>C</sup> 1.18 <sup>a</sup>	<sup>C</sup> 0.85 <sup>b,c</sup>	<sup>C</sup> 0.90 <sup>b,b</sup>
Means*	1.98 <sup>B</sup>	2.85 <sup>A</sup>	1.31 <sup>B</sup>	1.76 <sup>B</sup>

Means followed by different lower-case letters are different from each other at the level of 5% within each row.

Means preceded by different capital letters are different from each other at the level of 5% within each column.

\* Total mean values of the last 15 days after TFA and different capital letters are different from each other at the level of 5%.

Table 7: Mean Values of Fluoride Release ( $\mu\text{g F}^-/\text{cm}^2$ ) for Freedom for the Interaction Days x Time of Application x Gel

Days	APF		Neutral	
	1 Minute	4 Minutes	1 Minute	4 Minutes
1	<sup>B</sup> 1.32 <sup>a</sup>	<sup>B</sup> 1.31 <sup>a</sup>	<sup>A</sup> 1.38 <sup>a</sup>	<sup>A</sup> 1.36 <sup>a</sup>
7	<sup>C</sup> 0.32 <sup>a</sup>	<sup>C</sup> 0.24 <sup>a</sup>	<sup>B,C</sup> 0.17 <sup>a</sup>	<sup>B,C</sup> 0.24 <sup>a</sup>
15	<sup>C,D</sup> 0.13 <sup>a</sup>	<sup>C,D</sup> 0.14 <sup>a</sup>	<sup>B,C,D</sup> 0.10 <sup>a</sup>	<sup>C,D</sup> 0.13 <sup>a</sup>
16	<sup>A</sup> 3.56 <sup>b</sup>	<sup>A</sup> 9.04 <sup>a</sup>	<sup>B</sup> 0.29 <sup>c</sup>	<sup>B</sup> 0.44 <sup>c</sup>
22	<sup>D</sup> 0.07 <sup>a</sup>	<sup>D</sup> 0.07 <sup>a</sup>	<sup>D</sup> 0.04 <sup>a</sup>	<sup>D</sup> 0.09 <sup>a</sup>
30	<sup>D</sup> 0.05 <sup>a</sup>	<sup>D</sup> 0.06 <sup>a</sup>	<sup>C,D</sup> 0.05 <sup>a</sup>	<sup>D</sup> 0.05 <sup>a</sup>
Means*	0.45 <sup>AB</sup>	1.03 <sup>A</sup>	0.08 <sup>B</sup>	0.11 <sup>B</sup>

Means followed by different lower-case letters are different from each other at the level of 5% within each row.

Means preceded by different capital letters are different from each other at the level of 5% within each column.

\* Total mean values of the last 15 days after TFA and different capital letters are different from each other at the level of 5%.

did not influence the fluoride release at day 16 (Table 5); it was equal to day one ( $p>0.05$ ), except for the neutral gel applied for one minute ( $p<0.05$ ). The fluoride application did not alter the level of fluoride release throughout time; it was lower or similar during the last 15 days compared to the initial period.

Regarding the compomer (Table 7), APF gel applied for one and four minutes yielded higher fluoride release at day 16 compared to day one ( $p<0.05$ ) and the four-

minute application led to a higher release when compared to the one-minute application. A different behavior was observed for the neutral gel. The application of neutral gel did not change the level of fluoride release of the compomer during the last 15 days compared to the initial period.

## DISCUSSION

The release of fluoride is a substantial advantage for a dental material, because fluoride will strengthen the neighboring enamel or dentin and prevent secondary caries (Arends, Ruben & Dijkman, 1990; Arends & van der Zee, 1990; Hattab & others, 1991).

Carvalho and Cury (1999) and Bertacchini and others (1999) observed that conventional glass ionomer cements release more fluoride than resin-modified glass ionomers, as demonstrated by the outcomes in Table 2. Momoi and McCabe (1993) noted that the type and amount

of resin employed for light curing may affect the rate of fluoride release. Possibly, HEMA absorbs enough water to allow diffusion of fluoride ions; otherwise, fluoride would be firmly embedded inside the polyacrylate matrix. For PM-CR, the presence of polyacids in their composition did not enhance the release potential of fluoride (Aboush & Torabzadeh, 1998). However, there is a change in these outcomes after the application of



fluoridated gels, highlighting the influence of the gel on these materials. According to Preston and others (1999), the exact mechanism of fluoride recharge is unknown. Some factors, such as permeability of the material and form and concentration of the fluoride, may be involved in the process, as studied in this research, on which the APF gel yielded higher fluoride release (Table 2). Composition of the material (DeSchepper & others, 1991), diffusion of fluoride through the material (Forsten, 1977) and the difference in surface energy (Cranfield, Kuhn & Winter, 1982) may also influence fluoride recharge and later release, as resin-modified glass ionomer cements displayed higher release in the last 15 days in the current research (Table 2).

The higher release during the first 24 hours and the remarkable decrease with a tendency towards stabilization throughout time has been reported in the literature (DeSchepper & others, 1991; Tenuta & others, 1997; Aboush & Torabzadeh, 1998; Vermeersch, Leloup & Vreven, 2001). However, the pattern of release after exposure of the materials to a fluoride source has not been established. Investigations have applied gels daily (Bilgin & Ozalp, 1998; Yip & Smales, 1999), which does not allow observation of what is being released to the environment; that is, whether fluoride is adsorbed to the surface or incorporated in the material matrix (Pedrini & others, 2003).

The action of APF gel on RM-GICs altered the pattern of fluoride release, which was higher during the last 15 days. Theoretically, RM-GICs may provide more interaction between the fluoride and their matrix (Takahashi & others, 1993), thus allowing a slow release. Moreover, APF gel presents low pH and high fluoride concentration (Mustafa & others, 1996) and may have affected the physical properties of the material. This physical surface modification should have been more evident for C-GIC (Yip, Peng & Smales, 2001). However, if there was surface modification, it did not increase fluoride release during the last 15 days (Table 2), with similar outcomes for days 1 and 16 and no difference as to the time of application (Table 5). Acidulated products may alter the pattern of fluoride recharge and release of RM-GICs, since fluoride release returned to the level of the first 15 days or was decreased with the application of neutral gel (Table 3). This was not observed in other investigations, which have performed frequent applications and thus could not observe the effect of one application on the material behavior throughout time (Bilgin & Ozalp, 1998; Yip & Smales, 1999). This observation may have important consequences on what is known regarding the ability of fluoride release of conventional and modified materials and their action on the caries process.

Fluoride recharge and additional release by restorative materials did not occur because of contamination

by remnants of fluoridated gel on the surface of materials, as also reported in a study by Takahashi and others (1993). The difference in fluoride recharge between restorative materials, as observed at day 16, is demonstrated to be dependent on the type and composition of a material and the gel applied (Table 3). This may be demonstrated by outcomes for PM-CR, since fluoride release after the application of neutral gel at day 16 was lower than at day one and similar to day 15 (Table 7). The time of application corroborates these results, since there was a difference in fluoride release between the times of application of one and four minutes (Tables 4, 5, 6 and 7).

The amount of absorption or adsorption varies according to the type of glass ionomer material and exogenous source; that is, fluoride concentration, vehicle (liquid or gel), frequency and time of application (Takahashi & others, 1993; Diaz-Arnold & others, 1995; Suljak & Hatibovic-Kofman, 1996; Bilgin & Ozalp, 1998; Yip & Smales, 1999). The increase in fluoride release after the application of fluoridated gel for four minutes (Tables 4, 5, 6 and 7) corroborates the reports on the time of application. This aspect should be addressed in investigations to check this effect on the process of enamel demineralization/remineralization.

## CONCLUSIONS

Even though all materials evaluated recharged fluoride after the application of fluoridated gels, there was higher fluoride release after the application of APF gel for four minutes. The RM-GICs were most effective in fluoride release after application of APF gel.

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## References

- Aboush YE & Torabzadeh H (1998) Fluoride release from tooth-colored restorative materials: A 12-month report *Journal of the Canadian Dental Association* **64**(8) 561-564, 568.
- Araujo FB, García-Godoy F, Cury JA & Conceição EN (1996) Fluoride release from fluoride-containing materials *Operative Dentistry* **21**(5) 185-190.
- Arends J, Ruben J & Dijkman AG (1990) Effect of fluoride release from a fluoride-containing composite resin on secondary caries: An *in vitro* study *Quintessence International* **21**(8) 671-674.
- Arends J & van der Zee Y (1990) Fluoride uptake in bovine enamel and dentin from a fluoride-releasing composite resin *Quintessence International* **21**(7) 541-544.
- Bertacchini SM, Abate PF, Blank A, Baglietto MF & Macchi RL (1999) Solubility and fluoride release in ionomers and comonomers *Quintessence International* **30**(3) 193-197.

- Bilgin Z & Ozalp N (1998) Fluoride release from three different types of glass ionomer cements after exposure to NaF solution and APF gel *The Journal of Clinical Pediatric Dentistry* **22**(3) 237-241.
- Campos GM (2002) GMC 2002 Ribeirão Preto: School of Dentistry, n.d. Available at: <http://www.forp.usp.br/restauradora/gmc/gmc.html#gmc>. Accessed on Nov 21, 2003.
- Carvalho AS & Cury JA (1999) Fluoride release from some dental materials in different solutions *Operative Dentistry* **24**(1) 14-19.
- Cranfield M, Kuhn AT & Winter GB (1982) Factors relating to the rate fluoride-ion release from glass-ionomer cement *Journal of Dentistry* **10**(4) 333-341.
- Delbem AC & Cury JA (2002) Effect of application time of APF and NaF gels on microhardness and fluoride uptake of *in vitro* enamel caries *American Journal of Dentistry* **15**(3) 169-172.
- DeSchepper EJ, Berry EA 3<sup>rd</sup>, Cailleteau JG & Tate WH (1991) A comparative study of fluoride release from glass-ionomer cements *Quintessence International* **22**(3) 215-219.
- Diaz-Arnold AM, Holmes DC, Wistrom DW & Swift EJ Jr (1995) Short-term fluoride release/uptake of glass ionomer restoratives *Dental Materials* **11**(2) 96-101.
- Forsten L (1977) Fluoride release from a glass ionomer cement *Scandinavian Journal of Dental Research* **85**(6) 503-504.
- García-Godoy F, Hicks MJ, Flaitz CM & Berg JH (1995) Acidulated phosphate fluoride treatment and formation of caries-like lesions in enamel: Effect of application time *Pediatric Dentistry* **19**(2) 105-110.
- Hattab FN, el-Mowafy OM, Salem NS & el-Badrawy WA (1991) An *in vivo* study on the release of fluoride from glass-ionomer cement *Quintessence International* **22**(3) 221-224.
- Hebling J, Santos-Pinto LM & Cury JA (1995) Calcium fluoride formation "*in vitro*" on the sound enamel in function of the acidulated fluoride application time *Revista Brasileira de Odontologia* **52**(4) 30-35.
- Martins F, Delbem AC, Santos LR, Soares HL & Martins Ed Ede O (2002) Microhardness of resins as a function of color and halogen light *Pesquisa Odontológica Brasileira* **16**(3) 246-250.
- Momoi Y & McCabe JF (1993) Fluoride release from light-activated glass ionomer restorative cements *Dental Materials* **9**(3) 151-154.
- Mustafa NB, Chan DCN, Titus HW & Yang Z (1996) Fluoride release from restorative materials after exposure to NaF *Journal of Dental Research* **75**(ADR) Abstract #2917 p 382.
- Pedrini D, Delbem AC, França JG & Machado Td M (2003) Fluoride release by restorative materials before and after a topical application of fluoride gel *Pesquisa Odontológica Brasileira* **17**(2) 137-141.
- Preston AJ, Higham SM, Agalamanyi EA & Mair LH (1999) Fluoride recharge of aesthetic dental materials *Journal of Oral Rehabilitation* **26**(12) 936-940.
- Retief DH, Bradley EL, Denton JC & Switzer P (1984) Enamel and cementum fluoride uptake from a glass ionomer cement *Caries Research* **18**(3) 250-257.
- Suljak JP & Hatibovic-Kofman S (1996) A fluoride release-adsorption-release system applied to fluoride-releasing restorative materials *Quintessence International* **27**(9) 635-638.
- Takahashi K, Emilson CG & Birkhed D (1993) Fluoride release *in vitro* from various glass ionomer cements and resin composites after exposure to NaF solutions *Dental Materials* **9**(6) 350-354.
- Tenuta LMA, Pascotto RC, Navarro MFL & Francischone CE (1997) Fluoride release of four restorative glass-ionomer cements *Revista de Odontologia da Universidade de São Paulo* **11**(4) 249-253.
- Vermeersch G, Leloup G & Vreven J (2001) Fluoride release from glass-ionomer cements, compomers and resin composites *Journal of Oral Rehabilitation* **28**(1) 26-32.
- Yip HK & Smales RJ (1999) Fluoride release and uptake by aged resin-modified glass ionomers and a polyacid-modified resin composite *International Dental Journal* **49**(4) 217-225.
- Yip KH, Peng D & Smales RJ (2001) Effects of APF gel on the physical structure of compomers and glass ionomer cements *Operative Dentistry* **26**(3) 231-238.

# Volumetric Polymerization Shrinkage of Resin Composites Under Simulated Intraoral Temperature and Humidity Conditions

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## Clinical Relevance

Resin composites that polymerize under the warm temperature and high humidity conditions of the oral cavity shrink more than those that polymerize under room temperature and humidity conditions. Using a rubber dam lowers the temperature and humidity in treatment areas and, therefore, may help reduce resin composite shrinkage.

## SUMMARY

This study measured the volumetric shrinkage of resin composites polymerized under temperature and humidity conditions simulating the oral cavity and compared them to those occurring under ambient room conditions. Small, semi-spherical specimens of a microhybrid (Z100), microfill (Filtek A110) and flowable microhybrid (4 Seasons Flow) resin composite were manually formed and light activated for 40 seconds using a halogen light-curing unit (Spectrum Curing

Light). The volumetric polymerization shrinkage of 10 specimens of each brand of resin composite was measured using a drop shape analysis unit (Drop Shape Analysis System, model DSA10 Mk2) under each of two temperature/relative humidity conditions: room conditions ( $22 \pm 2^\circ\text{C}$  and  $60 \pm 5\%$ ) and those simulating intraoral conditions ( $35^\circ\text{C}$  and  $92 \pm 5\%$ ). Mean volumetric shrinkage values were calculated for each resin composite and the data were analyzed using two-way analysis of variance and *t*-test ( $\alpha=0.05$ ) to determine if significant differences existed between the amount of volumetric polymerization shrinkage that occurred under ambient room conditions and that which occurred under simulated intraoral conditions. Mean volumetric shrinkage values measured for the resin composites were:  $2.26 \pm 0.04\%$  (ambient) and  $2.61 \pm 0.04\%$  (intraoral) for Z100;  $1.96 \pm 0.04\%$  (ambient) and  $2.28 \pm 0.04\%$  (intraoral) for Filtek A110 and  $4.53 \pm 0.06\%$  (ambient) and  $5.34 \pm 0.05\%$  (intraoral) for 4 Seasons Flow. For each resin composite, statistical analysis indicated that the amount of volumetric shrinkage measured under simulated intraoral conditions was significantly greater than what

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was measured under ambient room conditions ( $p < 0.0001$ ).

## INTRODUCTION

The use of light-activated resin composites in direct restorative dentistry has grown dramatically in recent years. Their excellent esthetics, acceptable strength and good wear resistance are a few of the properties that make them suitable for restoring both anterior and posterior teeth (O'Brien, 2002). In large part, greater use of resin composites has been the result of growing patient demand for mercury-free, esthetic restorations (Roulet & Degrange, 2000). Also, resin composites have become increasingly popular, because clinicians find them an appealing material to use. Well performing dentin bonding products and recently introduced light-curing units, such as those employing light-emitting diodes, have made resin composites easier and faster to place.

However, the disadvantages of resin composites limit their use for certain clinical situations. Perhaps chief among these shortcomings is polymerization shrinkage, which ranges from approximately 1.9% to 6.0% (Labella & others, 1999). Shrinkage occurs as resin composites polymerize, because monomers crosslink to form a polymer network which occupies a smaller volume than monomers (Bausch & others, 1982; Venhoven, de Gee & Davidson, 1993). A range of problems can result from this shrinkage, including gap formation at the restoration's margins, marginal staining, leakage, post-treatment sensitivity and recurrent caries (Bausch & others, 1982; Borgmeijer & others, 1991; Davidson & Feilzer, 1997; Eriksen & Leidal, 1979; Retief, 1994). Although a few low-shrinkage resin composites have been marketed, the goal of producing a non-shrinking product has yet to be accomplished.

Being able to accurately measure polymerization shrinkage is crucial for researchers in their quest to develop low-shrinkage resin composites. A number of techniques exist, including mercury and water dilatometry (de Gee, Davidson & Smith, 1981; Goldman, 1983; Penn, 1986), strain measurement (Sakaguchi & others,

1991; Yap & others, 2000), laser interferometry (Fogleman, Kelly & Grubbs, 2002), video imaging (Labella & others, 1999; Sharp & others, 2003) and the bonded disc technique (Watts & Cash, 1991; Watts & Marouf, 2000). Regardless of the specific technique used, the vast majority of researchers who have measured polymerization shrinkage have done so under ambient laboratory conditions. Of course, these temperature and humidity conditions are not reflective of those in the oral cavity where polymerization actually occurs. This study measured the volumetric polymerization shrinkage of resin composites under temperature and humidity conditions that simulate those of the oral cavity and compared them to shrinkage occurring under ambient room conditions.

## METHODS AND MATERIALS

Table 1 lists the resin composites used in this study. Each represents one of three different categories of composites: microhybrid, microfill and flowable microhybrid. Volumetric polymerization shrinkage of each resin was measured using a drop shape analysis unit (Drop Shape Analysis System, model DSA10 Mk2, Kruss America, Charlotte, NC, USA) (Figure 1). Prior to testing, an orange shield from a light-curing unit was positioned between the sample area and the Drop

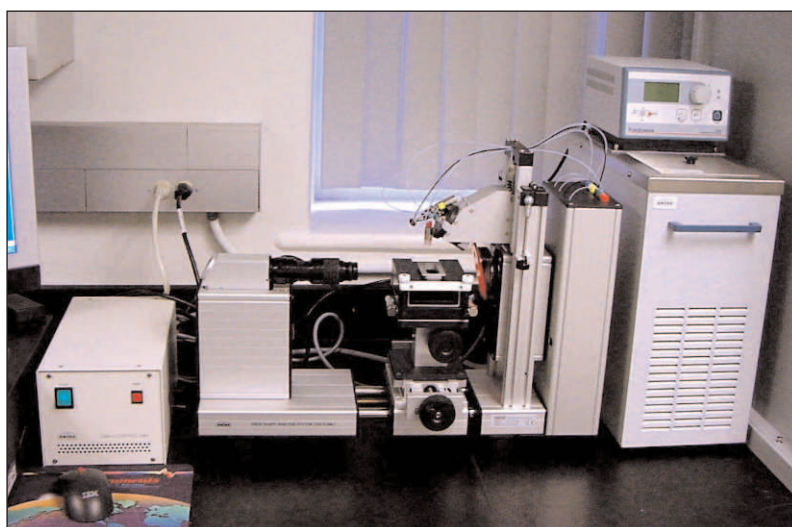


Figure 1: The Drop Shape Analysis System, model DSA10 Mk2.

Table 1: Resin Composites Used in This Study

Product	Manufacturer	Type	Lot #	Shade	Filler Content (vol%/wt%)
<b>Z100</b>	3M ESPE St Paul, MN, USA	Microhybrid	3WU	A3	66/84.5
<b>Filtek A110</b>	3M ESPE St Paul, MN, USA	Microfill	3BA	A3D	40/56
<b>4 Seasons Flow</b>	Ivoclar Vivadent Amherst, NY, USA	Flowable Microhybrid	G03122	A3	39.7/64.6

Shape Analysis System's (DSAS's) illumination source to prevent inadvertent light activation of the resin. In addition, in order to prevent overhead lighting from initiating polymerization of the resin composite specimens, testing was conducted in a laboratory equipped with gold fluorescent lamps (F40/GO Gold, Sylvania, Danvers, MA, USA) which filtered out wavelengths below 525 nm. To measure polymerization shrinkage under conditions simulating those of the oral cavity, the specimens were placed in an environmental chamber specifically made for use with DSAS (Figure 2). The chamber temperature is controlled electronically by an external heating unit, while a trough inside the chamber holds water that raises the humidity level. Unlike temperature, the chamber cannot precisely control humidity. The temperature within the chamber was monitored by its temperature controller. A digital humidity meter (Traceable Humidity Meter, Fisher Scientific, Hampton, NH, USA) was inserted into a pre-existing opening in the top of the chamber to measure its relative humidity. Measurements were made every 30 minutes over a 90-minute period during testing.

The temperature within the chamber was a constant 35°C and the relative humidity was  $92 \pm 5\%$ . The specimens were prepared by dispensing approximately 10  $\mu$ l of the resin composite, manually shaping it into a semi-sphere and placing it on a 24 mm<sup>2</sup> piece of polytetrafluoroethylene which had been positioned on the DSAS's sample stage. The stage, which can be precisely moved in three axes, was positioned so that the specimen's image appeared in the center of the display on the DSAS monitor. The specimen was allowed to rest for five minutes to eliminate the influence of slumping on the measurement and a two-dimensional visual image of it was captured by the unit's CCD camera. The specimen's volume was measured and cured for 40 seconds using a halogen light-curing unit (Spectrum Curing Light, Dentsply Caulk, Milford, DE, USA). The tip of the light wand was positioned 1 mm from the top of the resin composite during light activation. Prior to testing, the light's irradiance was measured using a laboratory-grade power meter (PM 5200, Coherent Moletron, Portland, OR, USA) and was found to be 780 mW/cm<sup>2</sup>. The specimen was allowed to rest for five minutes after light activation to allow its temperature to equilibrate to that of the chamber and its volume measured. The difference between the pre-cured and post-cured volume was used to calculate the percentage volumetric shrinkage. Ten specimens of each resin composite were tested.

To measure polymerization shrinkage under ambient room conditions, specimens were prepared as described earlier and the same measurement

process was followed. The only difference was that the environmental chamber was not used. Immediately prior to testing, the ambient room temperature and relative humidity were  $22 \pm 2^\circ\text{C}$  and  $60 \pm 5\%$ , respectively.

Volumetric polymerization shrinkage data were subjected to a two-way analysis of variance (ANOVA), with the factors being material (three levels) and test conditions (two levels—ambient room versus simulated intraoral environments). This ANOVA was followed by three one-way ANOVAs, which evaluated the effect of test conditions separately for each material.

## RESULTS

The mean volumetric polymerization shrinkage values for each resin composite under the two sets of environmental conditions are shown in Table 2 and Figure 3. Statistical analysis found that both main effects were significant ( $p=0.0001$ ), as was the interaction ( $p=0.0001$ ). The significant interaction seemed to result from the fact that simulated intraoral conditions had a greater effect on 4 Seasons Flow than on Filtek A110 and Z100. Evidence supporting this belief is that all the one-way ANOVAs indicated that polymerization

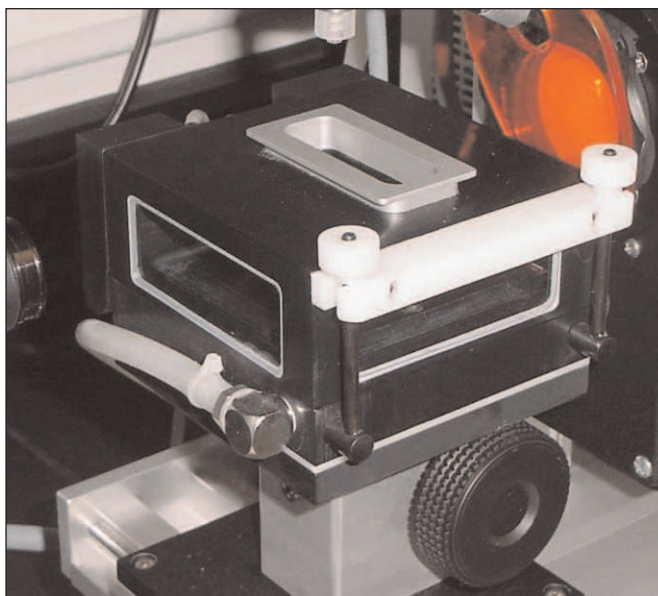


Figure 2: The DSAS environmental chamber. The light wand of the curing unit was inserted through the opening in the top surface to polymerize the resin composite specimen.

Table 2: Volumetric Polymerization Shrinkage of Resin Composites (%) (mean $\pm$ st dev)		
Material	Ambient Room Conditions ( $22 \pm 2^\circ\text{C}$ , $60 \pm 5\%\text{RH}$ )	Simulated Intraoral Conditions ( $35^\circ\text{C}$ , $92 \pm 5\%\text{RH}$ )
Z100	$2.26 \pm 0.04$	$2.61 \pm 0.04$
Filtek A110	$1.96 \pm 0.04$	$2.28 \pm 0.04$
4 Seasons Flow	$4.53 \pm 0.06$	$5.34 \pm 0.05$

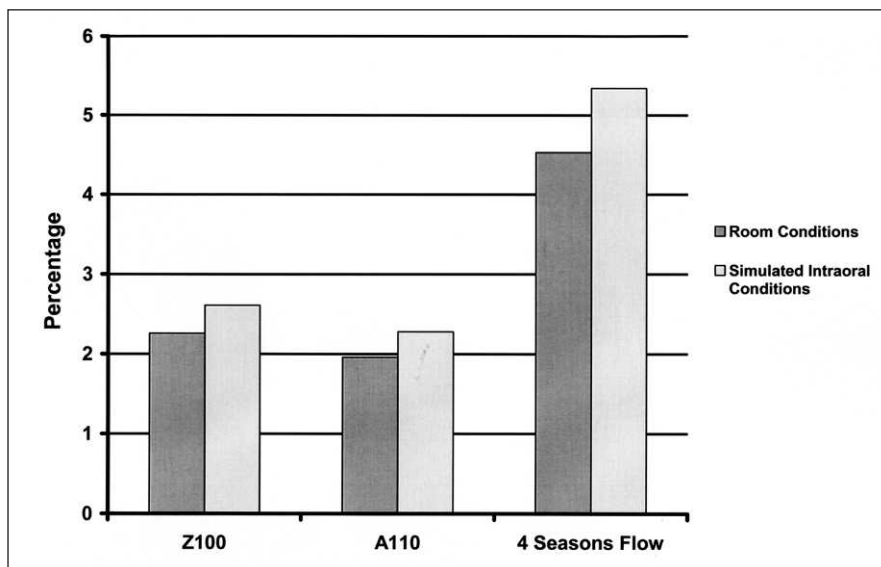


Figure 3: Volumetric polymerization shrinkage for the three resin composites.

shrinkage was greater under simulated intraoral conditions than ambient room conditions ( $p=0.0001$ ). Therefore, the conditions variable had the same general effect on all the materials.

### DISCUSSION

Shrinkage of dental resin composites occurs during polymerization as monomers covalently bond to form a polymer network. In doing so, existing Van de Waals' forces are replaced by covalent bonds. Since covalent bonds require less space between molecules, the molecules pack closer together (Alvarez-Gayosso & others, 2004; Coelho Santos & others, 2004; Rueggeberg & Tamareselvy, 1995; Yap & others, 2000), which, in turn, produces shrinkage.

In this study, the temperature and relative humidity of the testing chamber were 35°C and 92%, respectively. These values, chosen to simulate that of the oral cavity, were used based on the results of several studies in which intraoral conditions were measured. The intraoral temperature was reported to be  $35.3 \pm 0.4^\circ\text{C}$  (Spierings & others, 1987),  $35.5^\circ\text{C}$  (Michailescu & others, 1995) and "approximately" 35°C (Tibbetts & others, 1976), while the relative humidity in the center of an open mouth was  $91 \pm 4\%$  (Plasmans & others, 1994).

This study found that resin composites exhibited significantly greater shrinkage when polymerized under conditions simulating the oral cavity. The majority of studies that have measured polymerization shrinkage of dental restorative resins have been made under ambient laboratory conditions, not under conditions simulating the oral cavity. Based upon the results of this study, some laboratory studies appear to have

underreported the amount of shrinkage that actually occurs during clinical use.

There are two potential explanations for the finding that resin composites shrink more when cured at 35°C rather than at 22°C. First, it is well known that molecular motion increases with the application of heat (Dickson, 1995). If a resin-based material is warmed prior to curing, it will exhibit greater polymerization shrinkage than a cooler one, because the increased mobility of the monomer molecules results in a higher degree of double bond conversion and a greater number of covalent bonds forming. This may account, in part, for the results reported in this study and that of other researchers (Bennett & others, 1994; Lovell, Newman & Bowman, 1999). The second explanation concerns the speed with which the polymeriza-

tion reaction occurs. At higher temperatures, the viscosity of the monomer mixture decreases. This provides a longer reaction time before the propagation stage of the polymerization is limited by diffusion. As a result, a greater degree of conversion and polymerization shrinkage exists (Lovell & others, 1999).

Clinically, for temperature to have an influence on a resin composite's polymerization shrinkage, it would be necessary for the composite to be in the oral cavity long enough to equilibrate to intraoral temperature. One possible situation that might meet this requirement would be when the composite is used for direct veneering, where additional time is often used for placement and shaping of the material prior to light activation. Regardless of the clinical situation, the amount of time required would be influenced by the thickness and volume of the composite, its location in the mouth and its composition. One aspect of the clinical technique reported to have a significant effect on intraoral temperature is the use of a rubber dam. In a study of intraoral temperature under various conditions in the oral cavity, Plasmans and others (1994) reported that, when a rubber dam was used, the intraoral temperature around the treatment area was reduced to ambient room temperature.

The contribution of humidity to the greater degree of shrinkage measured in this study is unknown. A review of the literature found only one article that reported on humidity's effect on polymerization shrinkage, and an English language translation was unavailable (Reinhardt & Vahl, 1983). As with temperature, if a rubber dam is used during treatment, it may not be necessary to consider the effect of humidity, since Plasmans and others (1994) reported that intraoral



humidity in treatment areas was reduced from 91% to 33% with the use of a rubber dam. Therefore, the effect of humidity on polymerization shrinkage, if one exists, would be limited to those situations where treatment is provided in a high-humidity environment such as that experienced during some humanitarian missions in tropical areas and/or where no rubber dam is used.

It is important to note that the influence of a rubber dam in affecting polymerization shrinkage by altering intraoral temperature and humidity was not directly evaluated in this study. As discussed previously, however, rubber dams have been shown to influence intraoral conditions. Because this study found that temperature and humidity conditions had significantly affected polymerization shrinkage, it is conceivable that use of a rubber dam may also exert an effect in this regard.

It is also important to acknowledge that this study did not determine the independent contributions of high temperature and humidity to increases in polymerization shrinkage. Future research is planned to evaluate the additive and multiplicative effects of high temperature and humidity on shrinkage.

Few researchers have attempted to develop a way of cooling a resin composite intraorally as a way to reduce its polymerization shrinkage. One study used small, cooled composite inserts to chill surrounding resin composite prior to polymerization in an attempt to reduce stress associated with the composite's shrinkage (de la Torre-Moreno, Rosales-Leal & Bravo, 2003). The researchers measured microleakage at the tooth/restoration interface of Class V resin composite restorations placed in conjunction with cooled versus room-temperature inserts as an indirect measure of the effect of temperature reduction on shrinkage. The researchers found that gingival margin microleakage was significantly reduced when the cooled inserts were used compared to when room-temperature inserts or a bulk curing technique was used. They attributed the results, in part, to the effect the cooled inserts had in extending the pre-gel period of the polymerization reaction, which led to the formation of a well-sealed margin, because developing stresses could be relieved by resin composite flow.

While not a direct study of the effect of temperature on the amount of shrinkage, temperature has been shown to have an influence on the polymerization process. Additional research should be done to determine if other methods could be used to reduce the temperature of resin composites immediately prior to polymerization as a means of reducing shrinkage and improving clinical performance.

## CONCLUSIONS

Under the conditions of this study, the polymerization shrinkage of resin composites was significantly greater

under intraoral temperature and humidity conditions than under ambient room conditions.

## Disclaimer

The opinions expressed in this paper are those of the authors and do not necessarily reflect the official policy or position of the Departments of the Navy, Air Force, Defense or the US Government. The use of commercially available products does not imply endorsement.

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## References

- Alvarez-Gayosso C, Barcelo-Santana F, Guerrero-Ibarra J, Saez-Espinola G & Canseco-Martinez MA (2004) Calculation of contraction rates due to shrinkage in light-cured composites *Dental Materials* **20**(3) 228-235.
- Bausch JR, de Lange K, Davidson CL, Peters A & de Gee AJ (1982) Clinical significance of polymerization shrinkage of composite resins *Journal of Prosthetic Dentistry* **48**(1) 59-67.
- Bennett B, Puckett A, Pettey D & Roberts B (1994) Light source distance and temperature effects on composite polymerization *Journal of Dental Research* **73** Abstract #1002 p 227.
- Borgmeijer PJ, Kreulen CM, van Amerongen WE, Akerboom HB & Gruythuysen RJ (1991) The prevalence of postoperative sensitivity in teeth restored with Class II composite resin restorations *ASDC Journal of Dentistry for Children* **58**(5) 378-383.
- Coelho Santos MJ, Santos GC Jr, Nagem Filho H, Mondelli RF & El-Mowafy O (2004) Effect of light curing method on volumetric polymerization shrinkage of resin composites *Operative Dentistry* **29**(2) 157-161.
- Davidson CL & Feilzer AJ (1997) Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives *Journal of Dentistry* **25**(6) 435-440.
- de Gee AJ, Davidson CL & Smith A (1981) A modified dilatometer for continuous recording of volumetric polymerization shrinkage of composite restorative materials *Journal of Dentistry* **9**(1) 36-42.
- de la Torre-Moreno FJ, Rosales-Leal JI & Bravo M (2003) Effect of cooled composite inserts in the sealing ability of resin composite restorations placed at intraoral temperatures: An *in vitro* study *Operative Dentistry* **28**(3) 297-302.
- Dickson TR (1995) *Introduction to Chemistry* 7<sup>th</sup> ed New York John Wiley & Sons p 255.
- Eriksen HM & Leidal TI (1979) Monkey pulpal response to composite resin restorations in cavities treated with various cleansing agents *Scandinavian Journal of Dental Research* **87**(4) 309-317.
- Fogleman EA, Kelly MT & Grubbs WT (2002) Laser interferometric method for measuring linear polymerization shrinkage in light cured dental restoratives *Dental Materials* **18**(4) 324-330.
- Goldman M (1983) Polymerization shrinkage of resin-based restorative materials *Australian Dental Journal* **28**(3) 156-161.
- Labella R, Lambrechts P, Van Meerbeek B & Vanherle G (1999) Polymerization shrinkage and elasticity of flowable composites and filled adhesives *Dental Materials* **15**(2) 128-137.

- Lovell LG, Newman SM & Bowman CN (1999) The effects of light intensity, temperature, and comonomer composition on the polymerization behavior of dimethacrylate dental resins *Journal of Dental Research* **78**(8) 1469-1476.
- Michailescu PM, Marciano J, Grieve AR & Abadie MJ (1995) An *in vivo* recording of variations in oral temperature during meals: A pilot study *Journal of Prosthetic Dentistry* **73**(2) 214-218.
- O'Brien WJ (2002) *Dental Materials and Their Selection* 3<sup>rd</sup> ed Chicago Quintessence p 123.
- Penn RW (1986) A recording dilatometer for measuring polymerization shrinkage *Dental Materials* **2**(2) 78-79.
- Plasmans PJ, Creugers NH, Hermesen RJ & Vrijhoef MM (1994) Intraoral humidity during operative procedures *Journal of Dentistry* **22**(2) 89-91.
- Reinhardt KJ & Vahl J (1983) Does humidity affect the polymerization reaction of composites? *Deutsche zahnärztliche Zeitschrift* **38**(12) 1070-1072 [in German].
- Retief DH (1994) Do adhesives prevent microleakage? *International Dental Journal* **44**(1) 19-26.
- Roulet J & Degrange M (2000) Chapter 15—Composite resin restorations on posterior teeth, in *Adhesion: The Silent Revolution in Dentistry* Chicago Quintessence p 253.
- Rueggeberg F & Tamareselvy K (1995) Resin cure determination by polymerization shrinkage *Dental Materials* **11**(4) 265-268.
- Sakaguchi RL, Sasik CT, Bunczak MA & Douglas WH (1991) Strain gauge method for measuring polymerization contraction of composite restoratives *Journal of Dentistry* **19**(5) 312-316.
- Sharp LJ, Choi IB, Lee TE, Sy A & Suh BI (2003) Volumetric shrinkage of composites using video-imaging *Journal of Dentistry* **31**(2) 97-103.
- Spierings TA, Peters MC, Bosman F & Plasschaert AJ (1987) Verification of theoretical modeling of heat transmission in teeth by *in vivo* experiments *Journal of Dental Research* **66**(8) 1336-1339.
- Tibbetts VR, Schnell RJ, Swartz ML & Phillips RW (1976) Thermal diffusion through amalgam and cement base: Comparison of *in vitro* and *in vivo* measurements *Journal of Dental Research* **55**(3) 441-451.
- Venhoven BA, de Gee AJ & Davidson CL (1993) Polymerization contraction and conversion of light-curing BisGMA-based methacrylate resins *Biomaterials* **14**(11) 871-875.
- Watts DC & Cash AJ (1991) Determination of polymerization shrinkage kinetics in visible-light-cured materials: Methods development *Dental Materials* **7**(4) 281-287.
- Watts DC & Marouf AS (2000) Optimal specimen geometry in bonded-disk shrinkage-strain measurements on light-cured biomaterials *Dental Materials* **16**(6) 447-451.
- Yap AU, Wang HB, Siow KS & Gan LM (2000) Polymerization shrinkage of visible-light-cured composites *Operative Dentistry* **25**(2) 98-103.

# Effects of an Experimentally Developed Adhesive Resin System and CO<sub>2</sub> Laser Irradiation on Direct Pulp Capping

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M Shirono • Y Katoh

## Clinical Relevance

An experimentally developed adhesive resin system induced exposed pulp to produce reparative dentin formation earlier than commercially available adhesive resin systems. More research is required to determine the CO<sub>2</sub> laser conditions that can be used successfully for direct pulp capping.

## SUMMARY

**This study examined the wound healing process of rat pulp directly capped with various experimentally developed adhesive resin systems and treated with CO<sub>2</sub> laser irradiation. The experimental adhesive resins used in this study were**

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made from Clearfil Mega Bond (MB). The adhesive resin groups were capped with a combination of the following primers and bonding agents: commercially available MB primer (MBP), experimental MB primer containing 2wt% N-methacryloyl 5-aminosalicylic acid (5-NMSA: MP3) and 5wt% 12-methacryloyloxydodecylpyridinium bromide (MDPB: ABP); and commercially available MB bonding agent (MBB), experimental MB bonding agent containing 5wt% and 10wt% hydroxyl-calcium phosphate (hydroxyapatite: MB1, MB2) and 5wt% dicalcium phosphate dihydrate (brushite: MB3) as a reparative dentin-promoter. The combination of the three primers and four bonding agents yielded the 12 adhesive resin groups used in this study. The CO<sub>2</sub> laser group was irradiated with a laser and directly capped with MB. The CO<sub>2</sub> laser used was an Opelaser 03S II SP, and irradiation conditions were as follows: a power output of 0.5 W, super-pulse mode 1, repeat pulse mode (a cycle of 10 msec irradiation and 10 msec interval), defocused beam (approximately distance 20 mm from pulp exposure surface) and an irradiation time of



three seconds, with air cooling. The control group was capped with Dycal (DY) and MB. After the direct pulp capping procedures were undertaken, all cavities were restored with Clearfil AP-X resin composite. The rats were sacrificed on the 14<sup>th</sup> post-operative day. The specimens were alternately stained with Mayer's H & E, Hucker-Conn bacterial stain and the ABC method on TGF-beta1. These stained sections were observed using light microscopy and the following parameters were evaluated: pulp tissue disorganization, inflammatory cell infiltration, reparative dentin formation and bacterial penetration. The results of this study include the following: all experimentally developed bonding agent groups showed reparative dentin formation; whereas, the MBB-capped groups showed very little reparative dentin formation. The descending order regarding the amount of reparative dentin formation was MB2 > MB3 > MB1 >>> MBB, which tended to be dependent on the concentration of the blended reparative dentin-promoter. In terms of the quality of the formed dentin, it was observed that MB1-capped teeth tended to form tubular dentin; whereas, MB2- and MB3-capped teeth formed irregular and osteodentin types of dentin. Among the primers used, the descending order regarding the amount of reparative dentin and tubular type dentin formation was MP3 > MBP > ABP. The descending order of migration of macrophages and leukocytes was ABP > MBP > MP3. The CO<sub>2</sub> laser group showed a very irregular fibrous dentin matrix in the vicinity of the denatured and carbonized tissue but definite reparative dentin formation was not observed. The control group showed reparative dentin, which was very thick, compared with the other groups. In all the groups, pulp tissue showed almost normal morphology. Positive staining of TGF-beta1 was only observed slightly in some specimens of all groups. There was no difference in the staining of each group. Based on the results of this study, it was concluded that the combination of MP3 (containing 2wt% 5-NMSA) and MB1 (containing 5wt% hydroxyapatite) was effective in initiating an early repair process after direct pulp capping. CO<sub>2</sub> laser irradiation is effective for field control, but a longer observation time will be required to determine findings concerning dentin bridge formation.

## INTRODUCTION

Accidental pulp exposure may be encountered during the removal of carious dentin or while undertaking cavity and crown preparation. When this occurs, calcium hydroxide and calcium hydroxide agent are often placed as a direct pulp capping agent, because of its high pH,

antibacterial effect (Cox & others, 1985) and ability to induce reparative dentin formation (Akimoto & others, 1998; Medina & others, 2002). However, calcium hydroxide also has some disadvantages, such as a wide necrotic layer at the pulp exposure site by virtue of its strong alkalinity (Ebihara & Katoh, 1996; Jin, Shinkai & Katoh, 2000), reparative dentin formation, which is often of an osteodentin type with tunnel defects (Cox & others, 1996), and lack of adhesive properties and inadequate mechanical strength. Numerous researchers have focused on alternative materials. Adhesive resins have been reported to be a good alternative to calcium hydroxide, because they require only simplified procedures and bond well, thereby preventing the ingress of bacteria. The authors' laboratory has conducted several studies on human teeth (Katoh, 1993; Katoh, 1997; Katoh, Kimura & Inaba, 1997a; Katoh, Kimura & Inaba, 1999) and monkey teeth (Ebihara & Katoh, 1996; Suzuki & Katoh, 1997; Katoh & others, 1997b; Ohara & Katoh, 1999; Kitamura & Katoh, 1999; Jin & others, 2000; Medina & others, 2002; Shirono, Ebihara & Katoh, 2003) to test the pulp response to adhesive resins. They have observed that several materials are able to elicit favorable responses. The results of their long-term studies suggest that adhesive resins are almost equal to calcium hydroxide with regard to dentin bridge formation and wound healing. However, dentin bridge formation is initiated earlier in calcium hydroxide capped teeth. There are also researchers who question the biocompatibility of adhesive resins (Hebling, Giro & Costa, 1999; Hörsted-Bindslev, Vilkinis & Sidlauskas, 2003). Currently, there is no universally accepted technique or consensus on the use of adhesive resins as direct pulp capping material.

The concept of minimal intervention dentistry (MI) has been advocated in the treatment of caries, and adhesive resin systems are considered indispensable for achieving MI (Tyas & others, 2000). Modern adhesive resins have produced nearly similar bond strength values to dentin and enamel (Haller, 2000; Agostini, Kaaden & Powers, 2001); but it is not easy to maintain adhesive strength over long periods in the severe oral environment due to stress, such as thermal cycling and occlusal loading (Bedran-de-Castro & others, 2004). Moreover, it is very difficult to completely remove bacteria, because it is often lodged inside dentinal tubules and within the smear layer. These contaminants of the bonding substrate could cause pulpitis and deterioration of bond strength. The relevance of pulpitis and bacteria has been noted by previous researchers (Brännström & Nyborg, 1972; Brännström & Nordenvall, 1978; Fujitani, Inokoshi & Hosoda, 1992a). There is, therefore, a need for the development of adhesive resin systems containing components which can address this problem of bacterial contamination. A quaternary ammonium, 12-methacryloyloxydodecylpyri-

dinium bromide (MDPB), has been incorporated in a resin composite to impart an antibacterial property to it (Imazato, 1992). Adhesive systems incorporating MDPB monomer have been reported to exhibit an antibacterial effect against a wide spectrum of oral microorganisms (Imazato & others, 1997; Muto, Aoki & Ushiki, 1999; Imazato & others, 2001; Takatsuka & others, 2001). This antibacterial effect is expected to continue over the long-term, because MDPB has shown an inhibitory effect even after curing (Hiraguri & others, 1993; Imazato & others, 1998a). On the other hand, N-methacryloyl 5-aminosalicylic acid (5-NMSA) is a monomer ligand that was developed as an adhesion promoter by Kojima and others (1974) that improved the adhesion of ivory to acrylic resins. Clinical research has shown that this monomer has a desensitizing effect on hypersensitive dentin (Tagami & others, 1987). These monomers may have other beneficial effects such as prevention of microleakage and pulp sedation, which are important factors in the prognosis of direct pulp capping.

A number of research studies have been conducted on the dental use of lasers since Goldman and others (1964) reported use of the ruby laser for caries removal. The profession has explored the use of lasers in various ways. The demand for laser treatment has increased because of the reduction in price, miniaturization and the multi-functionalization of laser equipment in the past few years. The CO<sub>2</sub> laser has a wavelength of 10.6 µm, which is efficiently absorbed by water. It was observed that most of the energy of the CO<sub>2</sub> laser is absorbed within the top 0.05 mm of the soft tissue surface (Morioka & others, 1986). The CO<sub>2</sub> laser has a higher effect on hemostasis, vaporization and wound healing. In addition, the super-pulse mode of the CO<sub>2</sub> laser makes the peak output increase without changing the total energy by repeating pulse duration of less than 1/1000 seconds. Therefore, the super-pulse mode has been observed to produce less heat and better vaporization than the continuous wave (CW) mode. There are reports on the high success rate of direct pulp capping using the pulsed CO<sub>2</sub> laser in addition to conventional calcium hydroxide pulp capping (Moritz & others, 1998b).

This study examined the effects of various experimentally developed adhesive resin systems and treat-

Table 1: *Experimental Groups*

Group 1	MBP + MB1 (5wt% hydroxyapatite)	Adhesive Resin System Groups
Group 2	MBP + MB2 (10wt% hydroxyapatite)	
Group 3	MBP + MB3 (5wt% brushite)	
Group 4	MP3 (2wt% 5-NMSA) + MBB	
Group 5	MP3 (2wt% 5-NMSA) + MB1 (5wt% hydroxyapatite)	
Group 6	MP3 (2wt% 5-NMSA) + MB2 (10wt% hydroxyapatite)	
Group 7	MP3 (2wt% 5-NMSA) + MB3 (5wt% brushite)	
Group 8	ABP (5wt% MDPB) + MBB	
Group 9	ABP (5wt% MDPB) + MB1 (5wt% hydroxyapatite)	
Group 10	ABP (5wt% MDPB) + MB2 (10wt% hydroxyapatite)	
Group 11	ABP (5wt% MDPB) + MB3 (5wt% brushite)	
Group 12	MBP + MBB	
Group 13	CO <sub>2</sub> laser irradiation + MBP + MBB	CO <sub>2</sub> Laser Group
Group 14	Dycal + MBP + MBB	Control Group

ment using super-pulsed CO<sub>2</sub> laser irradiation on the wound healing process of exposed rat pulp.

## METHODS AND MATERIALS

### Experimental Animal

Before animal experiments were carried out, consent was obtained from the Laboratory Animal Committee of the School of Dentistry at Niigata, The Nippon Dental University, Niigata, Japan. The rats used in this study were Sprague-Dawley male rats (eight or nine weeks old), which were fed with water and solid feed for two weeks. Five samples were assigned to each experimental group. The following teeth were excluded: those with large cavities and pulp exposures, teeth with no bleeding and teeth with suspected fractures. A total of 50 rats were used, including those that died during the experiment proper.

### Experimental Materials and Methods Used

The summary of the experimental groups is shown in Table 1.

### Adhesive Resin System

The experimental adhesive agents used in this study were made from Clearfil Mega Bond (MB), a self-etching primer system in cooperation with the manufacturer, Kuraray Dental Co, Okayama, Japan. The following experimental adhesive resin agents were prepared: two experimental MB primers (MBP) containing 2wt% 5-NMSA (MP3) and 5wt% MDPB (ABP), and three experimental MB bonding agents (MBB) containing 5wt% and 10wt% hydroxyl-calcium phosphate (hydroxyapatite: MB1, MB2) and 5wt% dicalcium phosphate dihydrate (brushite: MB3) as a reparative dentin-promoter (Table 2). In addition to MBP and MBB, combinations of the different groups yielded 12 adhesive resin groups. These adhesive resin agents were used as follows: apply primer, wait 20 seconds, gently air dry primer, apply bonding agent and light cure for 10 seconds.

Table 2: Materials Used in the Study

Materials		Lot #	Composition	Manufacturer
Mega Bond (MB) Primer	MBP	0299AA	2-Hydroxyethyl Methacrylate Hydrophilic Dimethacrylate 10-Methacryloyloxydecyl Dihydrogen Phosphate N, N-Diethanol-p-Toluidine d, l-Camphorquinone Water	Kuraray
Mega Bond (MB) Bonding Agent	MBB	0373AA	Silanated Colloidal Silica Bisphenol A Diglycidylmethacrylate 2-Hydroxyethyl Methacrylate Hydrophobic Aliphatic Dimethacrylate 10-Methacryloyloxydecyl Dihydrogen Phosphate N, N-Diethanol-p-Toluidine d, l-Camphorquinone	Kuraray
Experimental Primers	MP3 ABP	021009 020625	MBP containing 2wt% 5-NMSA MBP containing 5wt% MDPB	Kuraray
Experimental Bonding Agents	MB1 MB2 MB3	021008 021008 021008	MBB containing 5wt% hydroxyapatite MBB containing 10wt% hydroxyapatite MBB containing 5wt% brushite	Kuraray
5-NMSA: N-methacryloyl 5-aminosalicylic acid MDPB: 12-methacryloyloxydodecylpyridinium bromide				

## CO<sub>2</sub> Laser

An Opelaser 03S II SP (Lot #MD-036, Yoshida Dental Mfg Co, Tokyo, Japan) was used in the CO<sub>2</sub> laser group. The specifications of this laser are: wavelength of 10.6  $\mu$ m, a power output of 0.5-5W (changeable per 0.1W), focus beam diameter of 0.4 mm and a green guide beam with a wavelength of 532 nm. Different power modes were also available: continuous wave mode and super-pulse mode 1 and 2. The exposure modes were as follows: continuous irradiation, repeat pulse and single pulse. The irradiation conditions used in this study were: a power output of 0.5W, super-pulse mode 1, repeat pulse mode (a cycle of 10msec irradiation and 10msec interval), defocused beam (approximate distance 20 mm from pulp exposure surface) and irradiation time of three seconds with air cooling. The CO<sub>2</sub> laser was used to achieve hemostasis, disinfection and coagulation of the exposed pulp.

## Specimen Preparation

The rats were deeply anesthetized by intraperitoneal injection of 5% pentobarbital sodium (Nembutal, Lot #73709Z721, Dainippon Pharmaceutical Co, Osaka, Japan) at a dose of 40mg/kg. After the rats were placed in an experiment stand, the teeth were cleaned with 3% hydrogen peroxide and rinsed with normal saline, then disinfected with dilute iodine tincture. The cavities were prepared on the mesial tubercle of the right and left maxillary first molars using a 440SS diamond point (Shofu Inc, Kyoto, Japan) in a high-speed handpiece under copious water spray. The pulps were then exposed with a #1/2 steel round bur (Hager & Meisinger GmbH, D40018 Dusseldorf, Germany) in a low-speed handpiece under copious distilled water spray. Hemostasis was achieved by flooding the cavity with

10% NaClO Gel (AD Gel, Lot #00417A, Kuraray Dental Co, Okayama, Japan) for five minutes. Reapplication of AD Gel was undertaken in the pulp that continued to bleed. This was followed by alternate irrigation with 3% hydrogen peroxide (Oxydol, Lot #802602, Yoshida Pharmaceutical Co, Tokyo, Japan) and 6% sodium hypochlorite (Purelox, Lot #3384, Oyalox Co, Tokyo, Japan) three times to remove dentin chips and AD Gel. The cavity was then rinsed twice with normal saline and gently air dried, then capped with the appropriate direct capping materials. The CO<sub>2</sub> laser group was irradiated using the laser conditions stated earlier and capped with MB. The control group was capped with Dycal (Lot #020121, Dentsply Caulk, Milford, DE, USA) and MB.

After direct capping and bonding procedures, all the cavities were restored with a hybrid restorative resin composite (Clearfil AP-X, Lot #0898AA, Kuraray Dental Co) and light cured for 40 seconds. All the materials were applied according to the manufacturer's instructions. The adhesive resins and composite restorative materials were light cured with a Griplight II light-curing unit (Shofu Inc). The rats were sacrificed by intraperitoneal injection of an overdose of Nembutal 14 days after the pulp capping procedures. Each pulp was fixed by transcardial vital perfusion with 4% paraformaldehyde phosphate buffer solution (4% PFA: pH 7.4). The maxillae were carefully removed, including first molars, and immersed in 4% PFA at 4°C overnight for further fixation. The specimens were then trimmed of excess tissue and decalcified with 10% EDTA-2Na solution (pH 7.4) at room temperature for four weeks. They were then dehydrated in ascending grades of ethanol and embedded in paraffin. Serial sections, 6- $\mu$ m



thick, were cut using a sliding microtome and alternately stained with Mayer's H & E, Hucker-Conn bacterial stain and the ABC method on TGF-beta1. These stained sections were observed with light microscopy (Eclipse E1000, Nikon Co, Tokyo, Japan) and the following parameters were evaluated: pulp tissue disorganization, inflammatory cell infiltration, reparative dentin formation and bacterial penetration (Medina & others, 2002). The findings were graded using the following criteria.

### Evaluation Criteria

#### *Pulp Tissue Disorganization*

1. Normal or of almost normal tissue morphology.
2. Odontoblast layer disorganization, but the deep part of the pulp was normal.
3. Loss of general tissue morphology.
4. Necrosis in the coronal one-third or more of the pulp.

#### *Inflammatory Cell Infiltration*

1. No or few scattered inflammatory cells present in the pulp.
2. Mild acute/chronic cell lesion.
3. Moderate inflammatory cell lesion seen as an abscess or dense stained infiltrate of polymorphonuclear leucocytes, histiocytes and lymphocytes in one-third or more of the coronal pulp and/or the mid-pulp.
4. Severely infected to necrotic pulp or lack of tissue in half or more of the pulp.

#### *Reparative Dentin Formation*

1. No dentin bridge formation.
2. Initial dentin bridge formation extending to not more than half of the exposure site.
3. Partial/incomplete dentin bridge formation extending to more than half but not completely closing the exposure site.
4. Complete dentin bridge formation.

#### *Bacterial Penetration*

1. Absence of stained bacterial profiles in any of the sections.
2. Positive bacterial staining profiles along the coronal or apical walls of the cavity.
3. Positive stained bacterial profiles within the cut dentinal tubules or axial wall of the cavity.
4. Positive stained bacterial profiles within the dental pulp.

In addition, the following histologic features were recorded: hemorrhaging, dentin chips (location, size

and number) and reactionary (irritation) dentin formation.

### Immunohistochemical Staining

The sections were deparaffinized and hydrated through use of xylene and a graded alcohol series, then rinsed briefly with tap water and phosphate buffered saline (PBS: pH 7.4). They were incubated in 0.3% H<sub>2</sub>O<sub>2</sub> in methanol for 30 minutes. The nonspecific protein binding was blocked with normal serum for 10 minutes. The sections were incubated with primary rabbit anti-TGF-beta1 antibodies (Lot #D1803T, Santa Cruz Biotechnology Inc, Santa Cruz, CA, USA), working dilution, 1:1000 for 12 hours at 4°C, then rinsed with PBS three times for five minutes. They were then incubated with biotinylated secondary antibodies for 10 minutes and streptavidin/peroxidase complex working solution for five minutes (Vectastain Universal Quick Kit, Vector Laboratories Inc, Burlingame, CA, USA). The antibody localized antigen was then detected by peroxidase activation of 3,3'-diaminobenzidine, DAB (DAB substrate kit, Lot #4052749, Vector Laboratories Inc) for 10 minutes. Finally, the sections were counterstained lightly with Mayer's hematoxylin.

### RESULTS

The summary of the results of the histopathologic evaluation is shown in Figure 1. The representative histologic images of each group are shown in Figures 2 through 15.

#### **The Diameters of Pulp Exposure Area**

The diameters of the exposed areas were measured with a stereomicroscope (Nikon Measurescope Model II, Nikon Co) and the widest dimension was recorded as the pulp exposure size of the specimen. The mean values for the pulp exposure sizes of each group ranged from 0.15±0.056 mm to 0.194±0.091 mm. There was no significant difference among the pulp exposure sizes of the groups (one-way ANOVA,  $p>0.01$ ).

#### **Pulp Tissue Disorganization**

The Kruskal-Wallis Test for pulp tissue disorganization data revealed no significant difference among the groups ( $p>0.05$ ). One specimen each from Groups 1, 4, 6, 8 and 9 exhibited mild pulp tissue disorganization. There were two specimens each from Groups 10 and 14 that exhibited mild pulp tissue disorganization. Specimens from Groups 2, 3, 5, 7, 11, 12 and 13 showed no pulp tissue disorganization. Normal pulp morphology was observed in almost all specimens. In the specimens that formed reparative dentin, it was observed that there was a reorganized odontoblastic layer just beneath the dentin bridge.

#### **Inflammatory Cell Infiltration**

The Kruskal-Wallis Test for inflammatory cell infiltration data revealed no significant difference among the

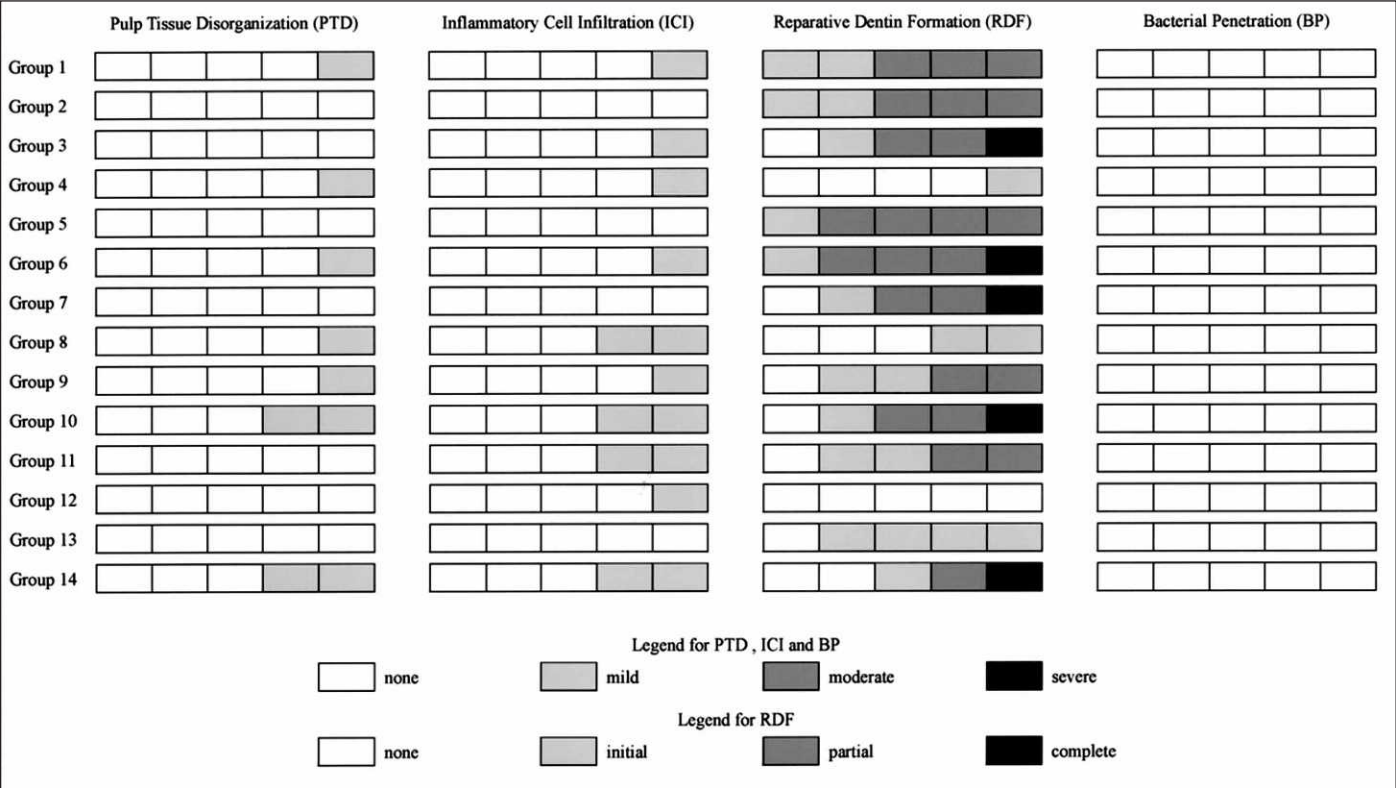


Figure 1: Results of the histopathologic evaluation.

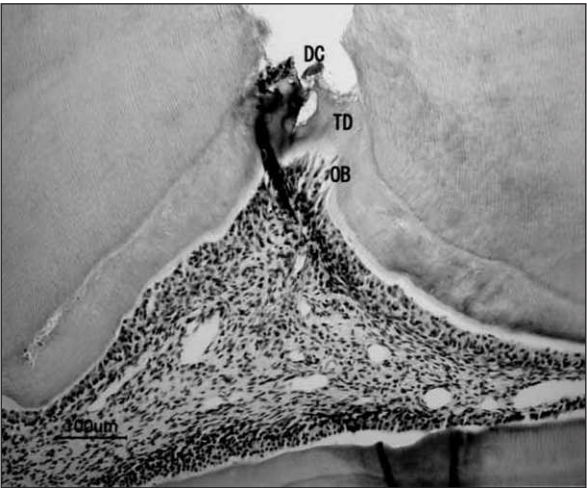


Figure 2. Representative histologic image of Group 1 (MBP + MB1). Although the dentin bridge is incomplete, it is mainly constituted of a tubular type structure (TD). Some dentin chips (DC) remain in the reparative dentin. There are reorganized odontoblast-like cells (OB) just beneath the dentin bridge. Pulpal morphology is normal (H & E, 100x).

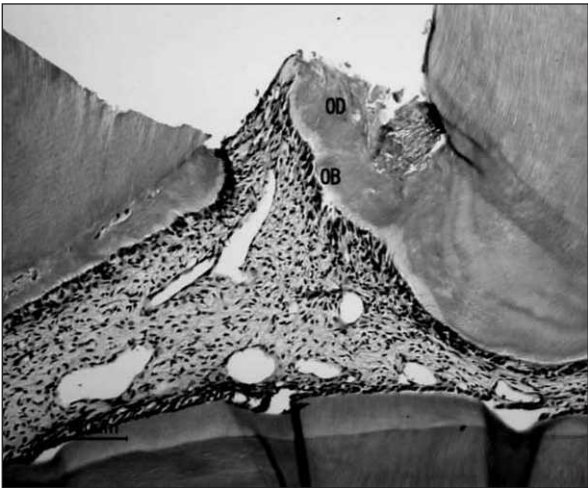


Figure 3. Representative histologic image of Group 2 (MBP + MB2). There is a partial dentin bridge that is composed of the osteodentin type (OD). There are reorganized odontoblast-like cells (OB) just beneath the reparative dentin. Pulpal morphology is normal (H & E, 100x).

groups ( $p>0.05$ ). One specimen each from Groups 1, 3, 4, 6, 9 and 12 exhibited mild inflammatory cell infiltration. There were two specimens each from Groups 8, 10, 11 and 14 that exhibited mild inflammatory cell infiltration. Specimens from Groups 2, 5, 7 and 13 showed

no inflammatory cell infiltration. Histopathological evaluation of all groups showed that no specimen exhibited severe inflammation of the pulp. Among the primers used, the descending order of migration of macrophages and leukocytes was ABP > MBP > MP3.



Figure 4. Representative histologic image of Group 3 (MBP + MB3). The complete dentin bridge is of the very irregular osteodentin type (OD). There are many dentin chips (DC) at the exposure site. There are reorganized odontoblast-like cells (OB) just beneath the dentin bridge. Pulpal morphology is normal (H & E, 100x).

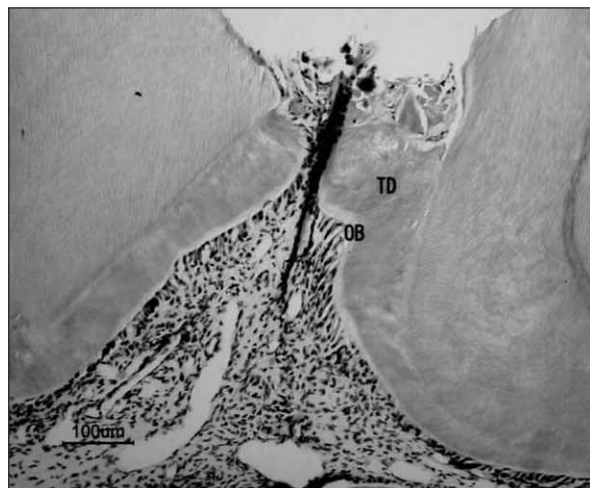


Figure 6. Representative histologic image of Group 5 (MP3 + MB1). Although the dentin bridge is incomplete, it is mainly constituted of a tubular type (TD) structure. There are reorganized odontoblast-like cells (OB) just beneath the dentin bridge. Pulpal morphology is normal (H & E, 100x).

### Reparative Dentin Formation

The Kruskal-Wallis Test for reparative dentin formation data revealed a significant difference among the groups ( $p < 0.05$ ). Mann-Whitney U-test post-hoc testing showed Group 8 had significantly lower reparative dentin formation than Groups 1, 2, 5 and 6 ( $p < 0.05$ ). Group 4 had significantly lower reparative dentin formation than Groups 1, 2, 3, 5, 6, 7 and 10 ( $p < 0.05$ ). Group 12 had significantly lower reparative dentin formation than Groups 1, 2, 3, 5, 6, 7, 9, 10, 11 and 13

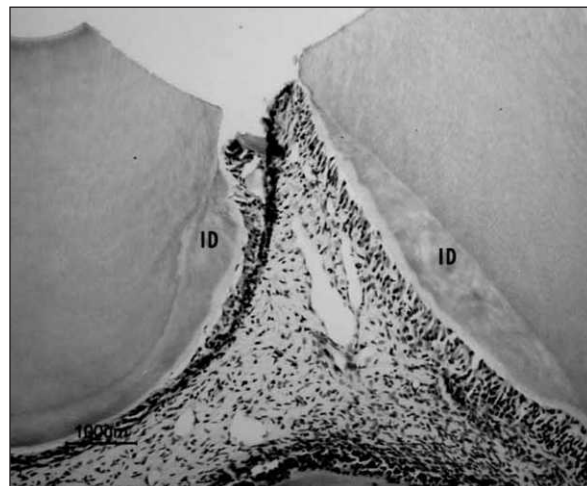


Figure 5. Representative histologic image of Group 4 (MP3 + MBB). Although irritation dentin (ID) formation was induced at the pulpal dentin wall of the periphery of the exposed site, there is no evidence of reparative dentin formation at the exposed surface. Pulpal morphology is normal (H & E, 100x).

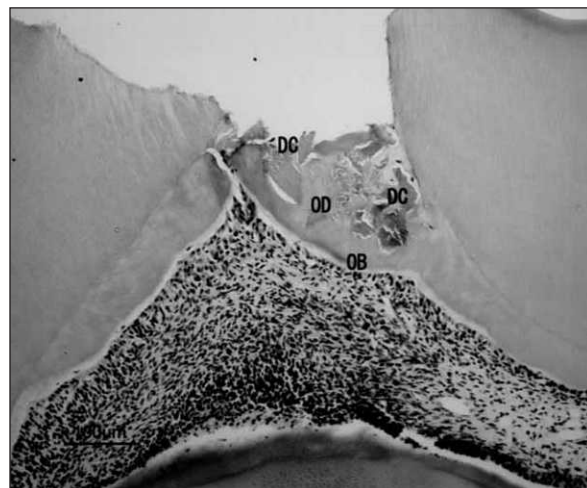


Figure 7. Representative histologic image of Group 6 (MP3 + MB2). There is a complete dentin bridge that is composed of the osteodentin type (OD) including many dentin chips (DC). A small amount of tubular dentin can be seen at the pulpal side of the dentin bridge. There are reorganized odontoblast-like cells (OB) just beneath the dentin bridge. Pulpal morphology is normal (H & E, 100x).

( $p < 0.05$ ). Comparing the bonding agents used, the groups with the experimental bonding agent showed reparative dentin formation, whereas the groups with MBB (Groups 4, 8, 12) showed very little reparative dentin formation. The groups with MB2 (Groups 2, 6, 10) tended to form slightly thicker reparative dentin than the groups with MB1 (Groups 1, 5, 9). However, reparative dentin formed by the MB2-containing groups tended to be irregular and of the osteodentin type, while the reparative dentin formed by the MB1-



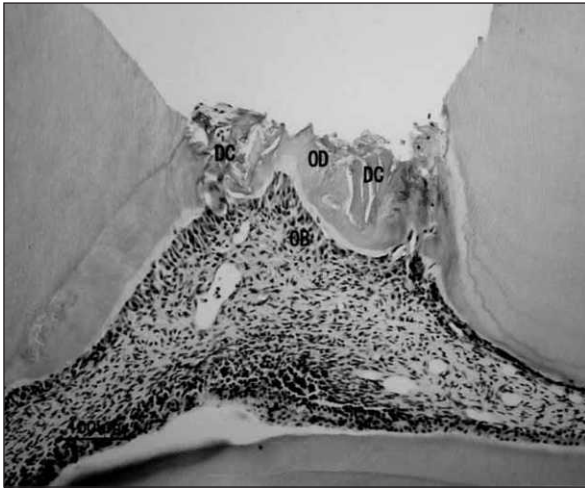


Figure 8. Representative histologic image of Group 7 (MP3 + MB3). There is a complete dentin bridge that is composed of the osteodentin type (OD). A small amount of tubular dentin can be seen at the pulpal side of the dentin bridge. Many dentin chips (DC) remain in the reparative dentin. There are reorganized odontoblast-like cells (OB) just beneath the dentin bridge. Pulpal morphology is normal (H & E, 100x).

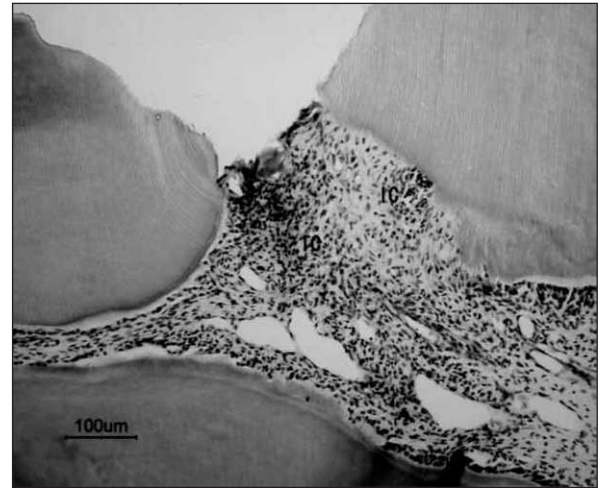


Figure 9. Representative histologic image of Group 8 (ABP + MBB). There is no evidence of reparative dentin formation at the exposed surface. There is mild pulp disorganization, and infiltrations of inflammatory cells (IC) surrounding the exposed site and in mid-pulp tissue (H & E, 100x).

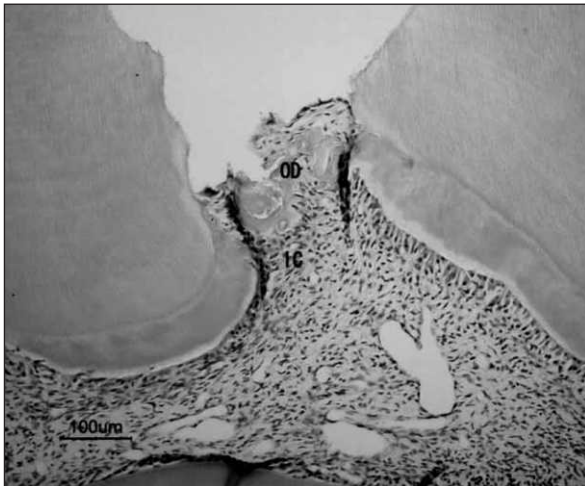


Figure 10. Representative histologic image of Group 9 (ABP + MB1). There is an irregular and incomplete thin dentin bridge that is composed of the osteodentin type (OD). Mid-pulp tissue is almost normal, but there is slight migration of macrophages and leukocytes (IC) surrounding the exposed site (H & E, 100x).

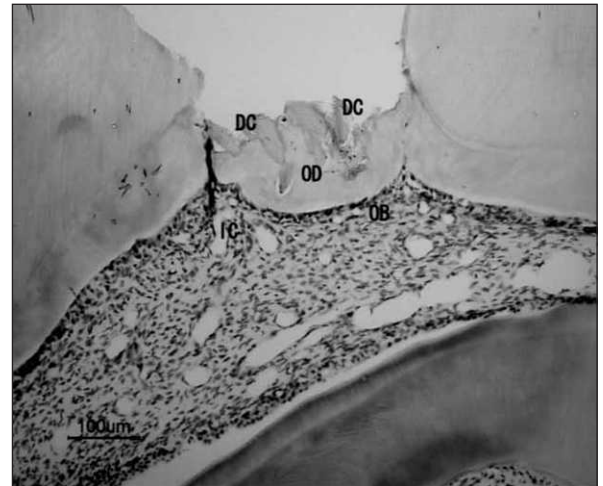


Figure 11. Representative histologic image of Group 10 (ABP + MB2). There is a complete dentin bridge that is composed of the osteodentin type (OD) including many dentin chips (DC). A small amount of tubular dentin can be seen at the pulpal side of the dentin bridge. There are reorganized odontoblast-like cells (OB) just beneath the dentin bridge. Mid-pulp tissue is almost normal, but there is slight migration of macrophages and leukocytes (IC) surrounding the exposed site (H & E, 100x).

containing groups was of the mature or tubular type. The quantity of reparative dentin formed in the MB3-containing groups (Groups 3, 7, 11) was intermediate between the MB1- and MB2-containing groups. They were also irregular and of the osteodentin type. Among the primers used, regarding the amount of reparative dentin and tubular type dentin formation, the descending order was MP3 > MBP > ABP. There were four specimens in the CO<sub>2</sub> laser group (Group 13) that showed very irregular fibrous dentin matrix adjacent to areas of heat denaturation and carbonized tissue. There was

also no evidence of dentin bridge formation. The control group (Group 14) had one specimen each of initial, partial and complete dentin bridge formation. Two specimens had no reparative dentin. The complete dentin bridge was composed of 50% osteodentin type adjacent to the pulp capping material. The reparative dentin in the control group was relatively thicker compared with the other groups.

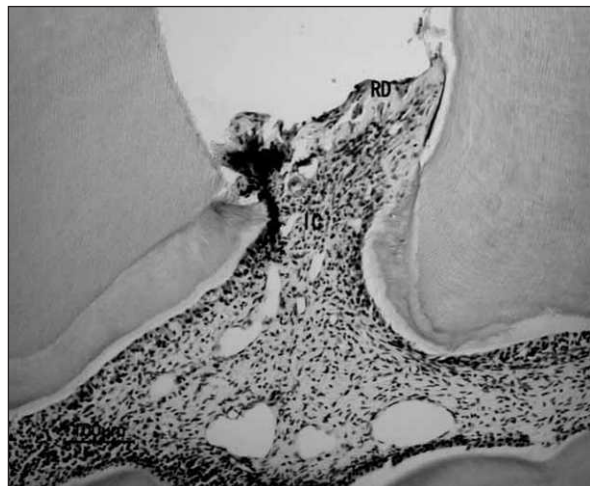


Figure 12. Representative histologic image of Group 11 (ABP + MB3). Very thin and irregular reparative dentin (RD) can be seen in superficial exposed pulp tissue. Mid-pulp tissue is almost normal, but there is slight migration of macrophages and leukocytes (IC) surrounding the exposed site (H & E, 100x).



Figure 14. Representative histologic image of Group 13 ( $\text{CO}_2$  laser Group). The denaturation layer (DL) in the pulp tissue was formed at approximately 100-200 micrometers from the exposed surface. Mid-pulp tissue immediately under the denaturation layer is normal. An irregular fibrous dentin matrix can be observed adjacent to heat denaturation tissue. Definite reparative dentin was not formed. The irradiated area on the cavity dentin wall had the following layers: a carbonization layer (black), a necrotic layer (dyed well with hematoxylin), and a protein denaturation layer (eosinophilic layer). Pulpal morphology is normal. Since the laser beam was irradiated in the cavity from the direction of the upper left, carbonization may be stronger on the right side of the cavity (H & E, 100x).

### Bacterial Penetration

No specimens stained positive for bacteria.

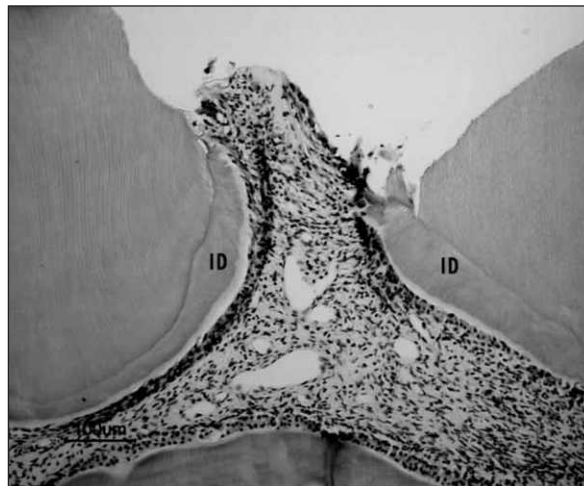


Figure 13. Representative histologic image of Group 12 (MBP + MBB). Although irritation dentin formation (ID) was induced at the pulpal dentin wall of the periphery of the exposed site, there is no evidence of reparative dentin formation at the exposed surface. Pulpal morphology is normal (H & E, 100x).

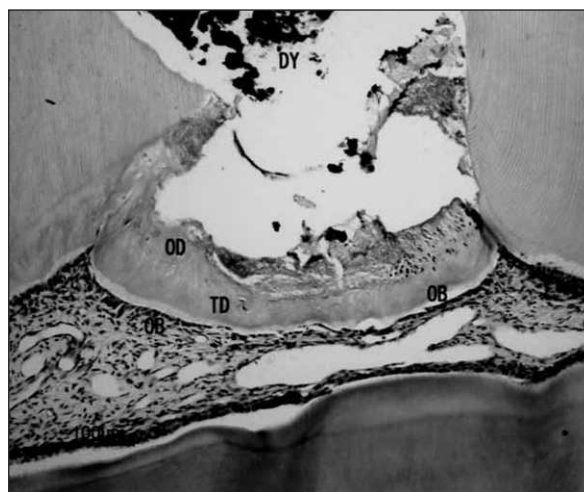


Figure 15. Representative histologic image of Group 14 (Control Group). This is a specimen in which a complete dentin bridge was formed. The dentin bridge is thick and is composed of the 50% osteodentin type (OD) including pulp cells adjacent to the pulp capping material. Tubular dentin (TD) at the pulpal side of the dentin bridge can be seen. There are new reorganized odontoblastic layers (OB) just beneath the dentin bridge. Pulpal morphology is normal. Dycal (DY) (H & E, 100x).

### Immunohistochemical Staining of TGF-beta1

The representative images of the immunohistochemical staining of TGF-beta1 are shown in Figures 16 and 17. Positive staining was only slightly noted in some of the specimens in all groups. There was no difference in the staining of each group.



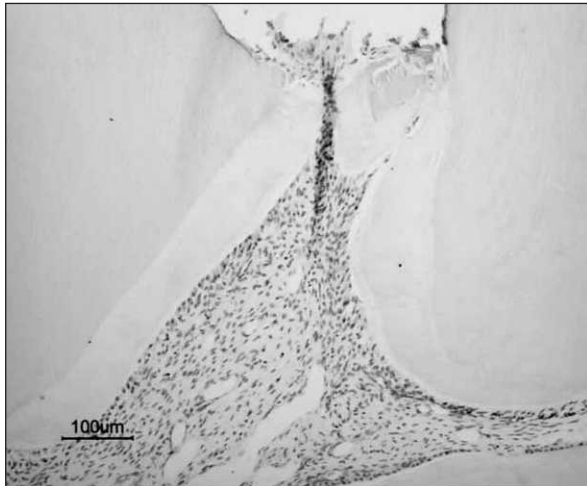


Figure 16. Representative image of staining of TGF-beta1 (Group 5). There was no positive staining (100x).

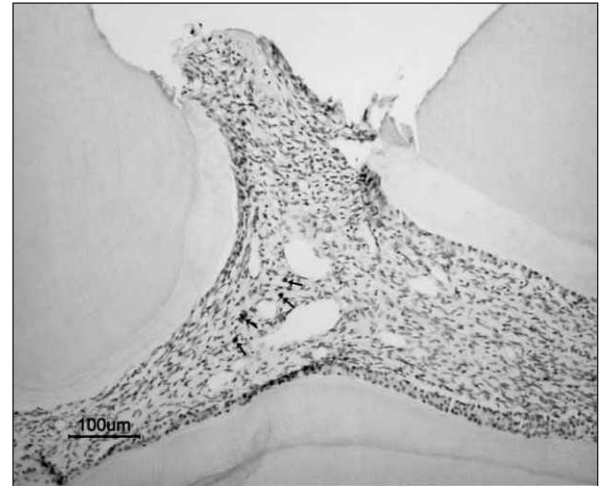


Figure 17. Representative image of staining of TGF-beta1 (Group 12). Positive staining (arrows) can only be slightly seen at the area of mid-pulp tissue (100x).

## DISCUSSION

Irrespective of the direct pulp capping material, the important factors for the success of direct pulp capping are diagnosis, degree of trauma, control of bleeding and exudation and infection control to exclude bacteria and saliva in the exposed area (Katoh, 1997; Cox & others, 2001). The prevention of contamination to produce a stable bond and eliminate microleakage is an important requirement when using adhesive resins as a direct capping material. This view is supported by many studies that have reported on the relationship between bacteria and microleakage and the failure of direct pulp capping treatment (Cox & others, 1987; Tsuneda & others, 1995; Medina & others, 2002). It has been reported that sodium hypochlorite is an effective surface-acting solvent of vital pulp tissue (Senia, Marshall & Rosen, 1971; Rosenfeld, James & Burch, 1978; Akimoto & others, 1998; Hafez & others, 2002). Alternate irrigation with 6% NaClO and 3% H<sub>2</sub>O<sub>2</sub> was used to control bleeding and remove debris. Furthermore, before undertaking alternate irrigation, the exposed area was flooded with AD Gel for five minutes. Flooding was undertaken to improve dentin bond strength, achieve hemostasis, disinfection and debridement of the exposure site, and lavage dentinal tubules inside (Katoh, 1993; Ohara & Katoh, 1999). However, in 67% of the specimens, dentin chips were still observed around the exposure site, excluding the laser group. It was suggested that AD Gel did not reach the deepest portion of the preparation since the cavities were too small and the dentin chips were forced into the pulp tissue when cavity preparation was carried out. Some believe that the dentin chips that remained in the pulp tissue could induce reparative dentin formation (Gottlieb, Orban & Stein, 1933; Katoh, 1997a). This was also observed in many specimens from this study, and the chips appeared to contribute to reparative dentin formation. However, it has

been advocated that the dentin chips should be completely removed before placement of the capping agent, because they may include bacteria.

In many specimens, hemostasis was achieved with a single application of AD Gel. However, some specimens showed a gradual exudation of tissue fluid even after achieving hemostasis. This could prevent hybridization of the substrate and could result in the persistence of protruded pulpal tissue observed in some specimens (Kitasako, Inokoshi & Tagami, 1999). It is believed that the protruded pulp tissue complicates the configuration of a direct capping area and hinders the formation of the dentin bridge, causing microleakage. Better field control methods need to be found to solve the problems mentioned above.

Compared with the conventional system, a self-etching bonding system (Mega Bond: MB) shows excellent adhesive ability to both enamel and dentin (Agostini & others, 2001). Moreover, cytotoxicity of MB is relatively lower than that of other adhesive systems (Huang & Chang, 2002). Shirono and others (2003) reported that MB is useful as a direct pulp capping material because of its satisfactory biocompatibility. It is conceivable that this system contains the characteristics necessary for use as a direct pulp capping material. There were no specimens that stained positive for bacteria or showed debonding of the filling material.

MDPB was incorporated to add an antibacterial effect to the resin composite, as reported by Imazato (1992). A significant feature of MDPB is that the antibacterial effect was demonstrated even after polymerization and there was no elution of any antibacterial component. It was therefore concluded that there is no reduction in the antibacterial effect, even after polymerization (Imazato & others, 1998a,b). The ABP-containing



groups (Groups 8, 9, 10, 11) showed slightly more migration of inflammatory cells compared with the other primer groups, but this was not statistically significant. Additionally, the reparative dentin formed was thin and had an irregular structure. One possible reason for this result is the chemistry of the ingredient of the resin system mentioned. Many studies on direct pulp capping with adhesive resins have shown that slight or moderate inflammation continues over the short term. Hydrophilic resinous monomers, such as HEMA and TEGDMA, penetrate deeply into the organic tissue to obtain firm dentin adhesion (Gerzina & Hume, 1996; Hamid & Hume, 1997). This degree of penetration is also believed to occur in pulp tissue. For instance, the adhesive materials (4-META/TBB, MMA resin) that permeated the pulp tissue produced a special layer, the “soft-tissue hybrid layer” or “pulp-tissue hybrid layer,” which is difficult to dissolve in water or organic solvent. Directly beneath this layer, some resinous monomers exist in an unpolymerized state (Kato, 1997a; Inoue, Miyakoshi & Shimono, 2001). They attract the migration of inflammatory cells, including macrophages. The primer containing MDPB is not especially cytotoxic compared to the other primers (Imazato & others, 1999; Imazato & others, 2000). The cytotoxicity level of MDPB is the same as TEGDMA (Imazato & others, 1993). However, it is possible that unpolymerized resin monomers can induce various influences on pulp cells (About & others, 2002). The cytotoxic effects of resinous monomers would depend on the concentration (Hanks & others, 1991), structure (Yoshii, 1997) and interaction (such as synergism, additivism and antagonism) (Ratanasathien & others, 1995).

It is not clear whether the results of this study were due to the influence of interaction with other components or whether it was only the influence of MDPB. This influence is considered minimal since formation of reparative dentin was not completely inhibited. However, it would be difficult to consider it to be a direct pulp-capping agent. Polymerized MDPB does not have an antibacterial effect when it is not in direct contact with microorganisms since there is no elution of any antibacterial component. However, MDPB before polymerization can kill bacteria in the dentinal tubules, because they are able to penetrate upon application to the time when the bonding agent is polymerized (Muto & others, 1999; Imazato & others, 2002). Therefore, MDPB, if used with other direct capping materials, may be able to seal the cavity and prevent bacterial invasion in the long-term.

To improve adhesion between dentin and acrylic resins, 5-NMSA was added to the primer (Kojima & others, 1974). In a study on the pulpal response of monkey teeth, adhesive liners containing salicylic acid showed a milder pulp response than the other resin

systems (Hosoda & others, 1989). When the primer that included 5-NMSA was used in vital teeth, it acted as a desensitizer of hypersensitive dentin (Tagami & others, 1987). Although the mechanism of this effect by salicylic acid is not clearly understood, one possible reason is the denaturation of protein. It is suggested that the desensitization effect on hypersensitive dentin is due to the denaturation of protein and the reduction of fluid movement through the *canaliculus dentalis* (Fujitani & others, 1992b; Tagami & others, 1993). The fixed influence of the primer containing 5-NMSA on protein is hypothesized to participate in this result. The MP3-containing groups (Groups 4, 5, 6, 7) showed the best histopathological findings, although there was a slight difference compared with other primer groups. The 5-NMSA of MP3 may have formed a more reliable protective layer at the pulp surface by protein fixation more significantly than the other primers. It is believed that the denatured layer of protein prevented exudation of tissue fluid and the diffusion of resin components into the pulp tissue (Kitasako & others, 1999; Fujitani & others, 2002). Regarding the influence 5-NMSA on reparative dentin formation, it was surmised that there was an interaction between the synthetic calcium phosphate of the bonding agent and 5-NMSA, which has outstanding chelate formation ability (Kojima & others, 1974).

Hydroxyapatite produces a very thin necrotic layer at the pulp tissue surface and is capable of inducing reparative dentin formation (Furusawa, Nakagawa & Asai, 1991; Hayashi & others, 1999). However, there have been several reports that teeth capped with hydroxyapatite had persistent acute or moderate inflammation (Jaber, Mascrès & Donohue, 1992; Sübay & Asci, 1993). These reports hypothesized that the cause of the inflammation may be due to the pH of materials. The alkalinity of calcium hydroxide (Dycal) offsets the acidity of the inflammatory exudate, whereas hydroxyapatite does not. Also, Jaber and others (1992) concluded that hydroxyapatite should not be used as a pulp capping agent, because of its tendency to cause scattered dystrophic calcification in the pulp. In this study, this phenomenon was not seen. In this study, it is believed that these events were avoided because the hydroxyapatite particles were fixed by the polymerization of the bonding agent. Teeth capped with MB2 tend to form slightly more reparative dentin than MB1-capped teeth. This shows that the quantity of the formed reparative dentin is dependent on the concentration of the reparative dentin-promoter combined with the bonding agent. The reparative dentin formed by MB2 was irregular and of the osteodentin type. In contrast, dentin formed against MB1 were tubular in structure. It was suggested that the mild reaction elicited by MB1 produced this favorable result. The reparative dentin of MB3-capped teeth was interme-

diate in quantity, irregular and mostly of the osteodentin type.

Brushite is considered to be the precursor phase of hydroxyapatite (Francis & Webb, 1971). It has been confirmed that epitaxy mutually occurs between brushite and hydroxyapatite (Aoba & others, 1974). Brushite is stable at low pH (pH 5-6) and has a higher solubility than hydroxyapatite or other calcium phosphates (Hagen, 1973). The increase in pH (Francis & Webb, 1971) and the existence of very few fluoride ions (Aoba & others, 1974) easily and promptly converts brushite into hydroxyapatite through hydrolysis. It is believed that some brushite, in contact with exposed pulp tissue, is transformed into hydroxyapatite or other forms of calcium phosphate by the buffering action of the tissue fluid and some brushite is resorbed swiftly into the pulp tissue. Apelt and others (2004) compared the influence of the different calcium phosphate cements on a cylindrical bone defect in sheep. DCPD (dicalcium phosphate dihydrate) cement showed the highest new bone formation and the least cement remnants at six months; whereas apatite was almost unchanged over all time periods.

MBB-capped teeth showed no inflammation but very little reparative dentin formation. Regarding the capability of untreated experimental pulp exposure recovering in germ-free rats, Kakehashi, Stanley and Fitzgerald (1965) observed formation of a dentin bridge beginning at 14 days, which was completed by 21 and 28 days. Inoue and Shimono (1992) reported the differentiation into odontoblasts-like cells and the appearance of osteodentin-like tissues on the third day and a new tubular dentin on the fifth day. These results demonstrate that adhesive resins, when used as direct pulp capping agents, produce a delay in dentin bridge formation. These findings revealed that the reparative dentin-promoter, mixed into the bonding agent, was effective in promoting reparative dentin formation.

The thickest reparative dentin was observed in the control group. The reparative dentin formed was of the osteodentin type adjacent to the pulp capping material. A reorganized layer of new odontoblasts was observed directly beneath the dentin bridge. However, the formation of reparative dentin was not observed in two specimens. It is possible that Dycal did not come directly in contact with pulp tissue since these two specimens had the smallest pulp exposure size.

The CO<sub>2</sub> laser was used to achieve sterilization, scar formation and minimize the formation of a hematoma in the exposed area via the thermal effects of laser irradiation (Moritz & others, 1998a; Moritz & others, 1998b). All were accomplished without contact with the pulp tissue, which is considered an advantage of the CO<sub>2</sub> laser. When irradiating pulp, short-lived discomfort is believed to be the influence of heat. It is well

known that the pulp chamber is weakened by heat (Zach & Cohen, 1965; Nyborg & Brännström, 1968). Although the surface temperature of living soft body tissue irradiated by the CO<sub>2</sub> laser can increase from about 100°C to 1500°C, there is a decrease of about 35°C on the side directly opposite the irradiated area when there is 0.9 mm of intervening tissue (Morioka & others, 1986). Since 70% to 80% of pulp tissue is made up of water, it is believed that the same phenomenon occurs in pulp tissue (Melcer & others, 1985). It is thought that the damage to pulp is somewhat avoidable by using air-cooling. Furthermore, the super-pulsed irradiation mode can make vaporization capability greater without increasing the accumulation of heat compared with the continuous wave (CW) and the normal pulse mode (Serebro & others, 1987; Kawabata & others, 1998). Moritz and others (1998b) reported that the reduced thermal stress on pulp exerted by the super-pulsed CO<sub>2</sub> laser seems to be responsible for the higher success rate with direct pulp capping compared to the CW CO<sub>2</sub> laser. In addition, it is thought that heat accumulation can be prevented by combining a repeat pulse mode (a cycle of 10msec irradiation and 10msec interval).

Within a short time (three seconds), the irradiation conditions in this study produced a layer of coagulation and carbonization. The laser irradiated exposed surface turned black from carbonization. The denaturation layer in the pulp tissue was observed at approximately 100-200 µm from the surface. The tissue immediately under the denaturation layer was normal. It can be said that the effect of heat produced by CO<sub>2</sub> laser irradiation was very local. This denaturation layer may act as a mild stimulus, such as the necrotic layer produced by the calcium hydroxide pulp capping material. In addition, dentin chips not removed by the chemical irrigation procedure were believed to vaporize or carbonize, since none were observed. The denaturation layer completely controlled the re-bleeding and exudations. Energy density is one of the important factors for attaining hemostasis. Irradiation using a defocused beam is recommended, because a focused beam can possibly perforate pulp tissue and cause re-bleeding (Shoji, Nakamura & Horiuchi, 1985). In direct pulp capping, this ability to quickly produce hemostasis may pose a problem. The clinical condition of persistent bleeding is one sign that should be considered when determining diagnosis of the exact pulp pathology. If hemostasis is achieved efficiently with laser irradiation, it may mask the real pulp condition, leading to a faulty diagnosis and problems may subsequently arise.

Although an irregular fibrous dentin matrix was observed adjacent to the heat denaturation tissue, definite reparative dentin was not formed. It is probable that new odontoblasts and reparative dentin could gradually appear from the surrounding tissue that was

damaged by the laser, since there is no sign of inflammation (Melcer, Chaumette & Melcer, 1987; McKee, 1993; Shirono & others, 2003). Except for direct heat stimulation, the laser has the effect of "low reactive level laser therapy" (such as photoactivation effects) which may contribute to dentin formation, recovery and reduction of inflammation (Utsunomiya, 1998). However, a few effects in this study may be due to the short irradiation time.

The surface of dentin irradiated by laser shows various morphological and structural changes, such as recrystallization, melting, charring, crater formation, cracking and fracturing (González & others, 1999; Malmström & others, 2001). The irradiated area on the cavity dentin wall was observed to have the following layers: a carbonization layer (black), a necrotic layer (dyed well with hematoxylin) and a protein denaturation layer (eosinophilic layer). These deteriorated layers may prevent the formation of a dentin-resin hybrid layer and result in a decrease in bond strength (Rowela & others, 2001). In order to uniformly form the protection layer at the exposed area, a certain amount of training is required.

The TGF-beta superfamily consists of multifunctional cytokines with biologic effects on various cells (Massagué, 1990). Specifically, many previous studies have reported TGF-beta1 as being related to collagen synthesis, odontoblast differentiation and primary dentinogenesis during tooth development (Cam, Neumann & Ruch, 1990; Pelton & others, 1991) and as being a potent pulpal immunosuppressant (D'Souza & others, 1998). They have also been considered to be factors that play inductive and regulatory roles during the reparative processes in the dentin-pulp complex after tissue injury (Sloan & Smith, 1999; Melin & others, 2000). *In vivo* and *in vitro* studies have shown that the adaptation of exogenous TGF-beta1 induces wound healing processes such as cell proliferation and migration, dentin extracellular matrix production, cytodifferentiation of odontoblast-like cells and reparative dentin formation (Nakashima & others, 1994; Hu & others, 1998; Tziafas & others, 1998; Tziafas & Papadimitriou, 1998; Sloan & Smith, 1999; Melin & others, 2000). In this study, the expression of TGF-beta1 was investigated as an index for comparing the difference in the recovery process induced by the various direct pulp capping materials. However, positive staining was only slightly noted in some specimens of all groups. There was no difference in the staining of each group. The length of the observation period in this study was comparatively long (14 days), and normal pulp morphology was observed in almost all the specimens of all groups during histopathological evaluation. Considering the speed and capacity of recovery of rat pulp, it is suggested that inflammation and other reactions due to injury may have decreased considerably at an early stage in all groups.

Until a completely reliable restorative material is developed, a dentin bridge made of tubular dentin is still the ideal repair form and barrier. Various factors contribute to the results of direct pulp capping treatment as mentioned earlier. Furthermore, the reactivity and repair capacity of pulp tissue would vary among animal species. Therefore, caution is advised when directly extrapolating these results to human clinical conditions. However, the authors of this study believe that a pattern has been established that can be used as a starting point for future research.

Currently, the authors are designing an additional experiment with short- and long-term observation periods regarding the use of bonding agents with lower concentrations of brushite in order to investigate, in still more detail, the wound healing process of exposed pulp with various experimentally developed adhesive resin systems and CO<sub>2</sub> laser irradiation.

## CONCLUSIONS

Based on the results of this study, it was concluded that:

1. Reparative dentin-promoter mixed into a bonding agent was effective in promoting reparative dentin formation. The amount of reparative dentin depended on the concentration of the blended reparative dentin-promoter. Specifically, it was the tendency for MB1 to form tubular type dentin.
2. Among the primers used, the descending order of the amount of reparative dentin and tubular type dentin formation was MP3 > MBP > ABP. The descending order of the migration of macrophages and leukocytes was ABP > MBP > MP3. Therefore, MP3 was concluded to be the most suitable as a direct capping material.
3. In all the groups, pulp tissue showed almost normal morphology. Positive staining of TGF-beta1 was only slightly noted in some specimens of all groups. There was no difference in the staining of each group. Experiments with short- and long-term observation periods are required to investigate in greater detail the wound healing process of exposed pulp.
4. As a result, the combination of MP3 (containing 2wt% 5-NMSA) and MB1 (containing 5wt% hydroxyapatite) is effective in initiating an early repair process after direct pulp capping.
5. The CO<sub>2</sub> laser group showed very irregular fibrous dentin matrix in the vicinity of denatured and carbonized tissue, but definite reparative dentin was not formed. CO<sub>2</sub> laser irradiation is effective for field control, but a longer observation time will be required to determine findings concerning dentin bridge formation.



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## References

- About I, Camps J, Mitsiadis TA, Bottero MJ, Butler W & Franquin JC (2002) Influence of resinous monomers on the differentiation *in vitro* of human pulp cells into odontoblasts *Journal of Biomedical Materials Research* **63**(4) 418-423.
- Agostini FG, Kaaden C & Powers JM (2001) Bond strength of self-etching primers to enamel and dentin of primary teeth *Pediatric Dentistry* **23**(6) 481-486.
- Akimoto N, Momoi Y, Kohno A, Suzuki S, Otsuki M, Suzuki S & Cox CF (1998) Biocompatibility of Clearfil Liner Bond 2 and Clearfil AP-X system on nonexposed and exposed primate teeth *Quintessence International* **29**(3) 177-188.
- Aoba T, Ishida T, Hasegawa K, Moriwaki Y & Yamauchi T (1974) Epitaxial growth of brushite and whitlockite on the biological apatite *Japanese Journal of Oral Biology* **16**(2) 252-259.
- Apelt D, Theiss F, El-Warrak AO, Zlinszky K, Bettschart-Wolfisberger R, Bohner M, Matter S, Auer JA & von Rechenberg B (2004) *In vivo* behavior of three different injectable hydraulic calcium phosphate cements *Biomaterials* **25**(7-8) 1439-1451.
- Bedran-de-Castro AK, Pereira PN, Pimenta LA & Thompson JY (2004) Effect of thermal and mechanical load cycling on microtensile bond strength of a total-etch adhesive system *Operative Dentistry* **29**(2) 150-156.
- Brännström M & Nyborg H (1972) Pulpal reaction to composite resin restorations *The Journal of Prosthetic Dentistry* **27**(2) 181-189.
- Brännström M & Nordenvall KJ (1978) Bacterial penetration, pulpal reaction and the inner surface of concise enamel bond. Composite fillings in etched and unetched cavities *Journal of Dental Research* **57**(1) 3-10.
- Cam Y, Neumann MR & Ruch JV (1990) Immunolocalization of transforming growth factor beta 1 and epidermal growth factor receptor epitopes in mouse incisors and molars with a demonstration of *in vitro* production of transforming activity *Archives of Oral Biology* **35**(10) 813-822.
- Cox CF, Bergenholtz G, Heys DR, Syed SA, Fitzgerald M & Heys RJ (1985) Pulp capping of dental pulp mechanically exposed to oral microflora: A 1-2 year observation of wound healing in the monkey *Journal of Oral Pathology* **14**(2) 156-168.
- Cox CF, Keall CL, Keall HJ, Ostro E & Bergenholtz G (1987) Biocompatibility of surface-sealed dental materials against exposed pulps *The Journal of Prosthetic Dentistry* **57**(1) 1-8.
- Cox CF, Sübay RK, Ostro E, Suzuki S & Suzuki SH (1996) Tunnel defects in dentin bridges: Their formation following direct pulp capping *Operative Dentistry* **21**(1) 4-11.
- Cox CF, Tarim B, Kopel H, Gürel G & Hafez A (2001) Technique sensitivity: Biological factors contributing to clinical success with various restorative materials *Advances in Dental Research* **15** 85-90.
- D'Souza RN, Cavender A, Dickinson D, Roberts A & Letterio J (1998) TGF-beta1 is essential for the homeostasis of the dentin-pulp complex *European Journal of Oral Sciences* **106**(Supplement 1) 185-191.
- Ebihara T & Katoh Y (1996) Histopathological study on development of adhesive resinous material containing calcium hydroxide as direct pulp capping agent *The Japanese Journal of Conservative Dentistry* **39**(6) 1288-1315.
- Francis MD & Webb NC (1971) Hydroxyapatite formation from a hydrated calcium monohydrogen phosphate precursor *Calcified Tissue Research* **6**(4) 335-342.
- Fujitani M, Inokoshi S & Hosoda H (1992a) Effect of acid etching on the dental pulp in adhesive composite restorations *International Dental Journal* **42**(1) 3-11.
- Fujitani M, Morigami M, Hirano T, Sugizaki J & Hosoda H (1992b) The effectiveness of salicylic acid derivatives as adhesion promoting agents *Journal of Dental Research* **71**(Special Issue) Abstract #514 p 170.
- Fujitani M, Shibata S, Van Meerbeek B, Yoshida Y & Shintani H (2002) Direct adhesive pulp capping: Pulpal healing and ultra-morphology of the resin-pulp interface *American Journal of Dentistry* **15**(6) 395-402.
- Furusawa M, Nakagawa K & Asai Y (1991) Clinico-pathological studies on the tissue reactions of human pulp treated with various kinds of calcium phosphate ceramics *The Bulletin of Tokyo Dental College* **32**(3) 111-120.
- Gerzina TM & Hume WR (1996) Diffusion of monomers from bonding resin-resin composite combinations through dentine *in vitro* *Journal of Dentistry* **24**(1-2) 125-128.
- Goldman L, Hornby P, Meyer R & Goldman B (1964) Impact of the laser on dental caries *Nature* **203** 417.
- González M, Banderas JA, Rodríguez V & Castaño VM (1999) Particle-induced X-ray emission and scanning electron microscopic analyses of the effects of CO<sub>2</sub> laser irradiation on dental structure *Journal of Dentistry* **27**(8) 595-600.
- Gottlieb B, Orban B & Stein G (1933) The root treatment in case of a vital pulp *Australian Dental Journal* **37** 422-427.
- Hafez AA, Cox CF, Tarim B, Otsuki M & Akimoto N (2002) An *in vivo* evaluation of hemorrhage control using sodium hypochlorite and direct capping with a one- or two-component adhesive system in exposed nonhuman primate pulps *Quintessence International* **33**(4) 261-272.
- Hagen AR (1973) Structural features of biologically involved phosphates *Acta Odontologica Scandinavica* **31**(3) 149-173.
- Haller B (2000) Recent developments in dentin bonding *American Journal of Dentistry* **13**(1) 44-50.

- Hamid A & Hume WR (1997) The effect of dentine thickness on diffusion of resin monomers *in vitro* *Journal of Oral Rehabilitation* **24**(1) 20-25.
- Hanks CT, Strawn SE, Wataha JC & Craig RG (1991) Cytotoxic effects of resin components on cultured mammalian fibroblasts *Journal of Dental Research* **70**(11) 1450-1455.
- Hayashi Y, Imai M, Yanagiguchi K, Vilorio IL & Ikeda T (1999) Hydroxyapatite applied as direct pulp capping medicine substitutes for osteodentin *Journal of Endodontics* **25**(4) 225-229.
- Hebling J, Giro EM & Costa CA (1999) Biocompatibility of an adhesive system applied to exposed human dental pulp *Journal of Endodontics* **25**(10) 676-682.
- Hiraguri H, Imazato S, Kobayashi K, Tarumi H, Sakakura M, Torii M & Tsuchitani Y (1993) Studies on composites containing chemically bound non-releasing antibacterial component, Part 4. Long term antibacterial effect of composites containing MDPB *The Japanese Journal of Conservative Dentistry* **36**(6) 1609-1614.
- Hörsted-Bindslev P, Vilkinis V & Sidlauskas A (2003) Direct capping of human pulps with a dentin bonding system or with calcium hydroxide cement *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics* **96**(5) 591-600.
- Hosoda H, Inokoshi S, Fujitani M, Otsuki M & Shimada Y (1989) Pulpal response to a new bonding agent and recently designed adhesive liners containing a salicylic acid derivative *The Japanese Journal of Conservative Dentistry* **32**(2) 398-410.
- Huang FM & Chang YC (2002) Cytotoxicity of dentine-bonding agents on human pulp cells *in vitro* *International Endodontic Journal* **35**(11) 905-909.
- Hu CC, Zhang C, Qian Q & Tatum NB (1998) Reparative dentin formation in rat molars after direct pulp capping with growth factors *Journal of Endodontics* **24**(11) 744-751.
- Imazato S (1992) Studies on composites containing chemically bound non-releasing antibacterial component *The Japanese Journal of Conservative Dentistry* **35**(5) 1109-1129.
- Imazato S, Yokota W, Hiraguri H, Kobayashi K, Sakakura M, Tarumi H, Torii M & Tsuchitani Y (1993) Studies on composites containing chemically bound non-releasing antibacterial component, Part 3. Cytotoxicity of composites containing MDPB *The Japanese Journal of Conservative Dentistry* **36**(2) 436-440.
- Imazato S, Kinomoto Y, Tarumi H, Torii M, Russell RR & McCabe JF (1997) Incorporation of antibacterial monomer MDPB into dentin primer *Journal of Dental Research* **76**(3) 768-772.
- Imazato S, Ehara A, Torii M & Ebisu S (1998a) Antibacterial activity of dentine primer containing MDPB after curing *Journal of Dentistry* **26**(3) 267-271.
- Imazato S, Imai T, Russell RR, Torii M & Ebisu S (1998b) Antibacterial activity of cured dental resin incorporating the antibacterial monomer MDPB and an adhesion-promoting monomer *Journal of Biomedical Materials Research* **39**(4) 511-515.
- Imazato S, Ebi N, Tarumi H, Russell RR, Kaneko T & Ebisu S (1999) Bactericidal activity and cytotoxicity of antibacterial monomer MDPB *Biomaterials* **20**(9) 899-903.
- Imazato S, Tarumi H, Ebi N & Ebisu S (2000) Cytotoxic effects of composite restorations employing self-etching primers or experimental antibacterial primers *Journal of Dentistry* **28**(1) 61-67.
- Imazato S, Torii Y, Takatsuka T, Inoue K, Ebi N & Ebisu S (2001) Bactericidal effect of dentin primer containing antibacterial monomer methacryloyloxydodecylpyridinium bromide (MDPB) against bacteria in human carious dentin *Journal of Oral Rehabilitation* **28**(4) 314-319.
- Imazato S, Walls AW, Kuramoto A & Ebisu S (2002) Penetration of an antibacterial dentine-bonding system into demineralized human root dentin *in vitro* *European Journal of Oral Sciences* **110**(2) 168-174.
- Inoue T & Shimono M (1992) Repair dentinogenesis following transplantation into normal and germ-free animals *Proceedings of the Finnish Dental Society* **88**(Supplement 1) 183-194.
- Inoue T, Miyakoshi S & Shimono M (2001) The *in vitro* and *in vivo* influence of 4-META/MMA-TBB resin components on dental pulp tissues *Advances in Dental Research* **15** 101-104.
- Jaber L, Mascrès C & Donohue WB (1992) Reaction of the dental pulp to hydroxyapatite *Oral Surgery, Oral Medicine, and Oral Pathology* **73**(1) 92-98.
- Jin C, Shinkai K & Katoh Y (2000) New developments in histopathological studies on adhesive resinous materials having a calcification promoting function as direct pulp capping agent—The effect of Ca(OH)<sub>2</sub> on direct capping with self-etching primer and the wound healing process over time *The Japanese Journal of Conservative Dentistry* **43**(1) 85-108.
- Takehashi S, Stanley HR & Fitzgerald RJ (1965) The effects of surgical exposures of dental pulps in germ-free and conventional laboratory rats *Oral Surgery, Oral Medicine, and Oral Pathology* **20**(3) 340-349.
- Katoh Y (1993) Clinico-pathological study on pulp-irritation of adhesive resinous material, Report 1- Histopathological change of the pulp tissue in direct capping *Japan Society for Adhesive Dentistry* **11**(4) 119-211.
- Katoh Y (1997) Microscopic observation of the wound healing process of pulp directly capped with adhesive resins *Japan Society for Adhesive Dentistry* **15**(3) 229-239.
- Katoh Y, Kimura T & Inaba T (1997a) Clinicopathological study on pulp-irritation of adhesive resinous materials, Report 2- Clinical prognosis of the pulp tissue directly capped with resinous materials *The Japanese Journal of Conservative Dentistry* **40**(1) 152-162.
- Katoh Y, Yamaguchi R, Shinkai K, Hasegawa K, Kimura T, Ebihara T, Suzuki T, Ohara A, Kitamura Y, Tanaka N & Jin C (1997b) Clinicopathological study on pulp-irritation of adhesive resinous materials, Report 3- Direct capping effects on exposed pulp of *Macaca fascicularis* *The Japanese Journal of Conservative Dentistry* **40**(1) 163-176.
- Katoh Y, Kimura T & Inaba T (1999) Clinicopathological study on pulp-irritation of adhesive resinous materials, Report 4- Long-term clinical prognosis of pulp tissue directly capped with resinous materials *The Japanese Journal of Conservative Dentistry* **42**(5) 989-995.
- Kawabata A, Kawabata H, Yagasaki A, Iwasaki H & Miyazawa H (1998) The effects of heat a carbon dioxide laser on dentin—A comparison of normal pulse laser and superpulse laser *The Japanese Journal of Pediatric Dentistry* **36**(5) 823-830.
- Kitamura Y & Katoh Y (1999) Histopathological study on healing properties of exposed pulp irradiated by laser and capped directly with adhesive resin *The Japanese Journal of Conservative Dentistry* **42**(3) 461-477.

- Kitasako Y, Inokoshi S & Tagami J (1999) Effects of direct resin pulp capping techniques on short-term response of mechanically exposed pulps *Journal of Dentistry* **27**(4) 257-263.
- Kojima K, Iwabuchi S, Kunagi M, Kikuchi J & Iida K (1974) Studies of the polymer ligands (III): Preparations and copolymerizations of N-methacryloyl aminobenzoic acids and aminos- alicyclic acids *Journal of Faculty of Engineering, Chiba University* **25**(48) 65-72.
- Malmström HS, McCormack SM, Fried D & Featherstone JD (2001) Effect of CO<sub>2</sub> laser on pulpal temperature and surface morphology: An *in vitro* study *Journal of Dentistry* **29**(8) 521-529.
- Massagué J (1990) The transforming growth factor-beta family *Annual Review of Cell Biology* **6** 597-641.
- McKee MD (1993) Effects of CO<sub>2</sub> laser irradiation *in vivo* on rat alveolar bone and incisor enamel, dentin, and pulp *Journal of Dental Research* **72**(10) 1406-1417.
- Medina VO 3<sup>rd</sup>, Shinkai K, Shirono M, Tanaka N & Katoh Y (2002) Histopathologic study on pulp response to single-bottle and self-etching adhesive systems *Operative Dentistry* **27**(4) 330-342.
- Melcer J, Chaumette MT, Zeboulon S, Melcer F, Hasson R, Merard R, Pinaudeau Y, Dejardin J & Weill R (1985) Preliminary report on the effect of the CO<sub>2</sub> laser beam on the dental pulp of the Macaca mulatta primate and the beagle dog *Journal of Endodontics* **11**(1) 1-5.
- Melcer J, Chaumette MT & Melcer F (1987) Dental pulp exposed to the CO<sub>2</sub> laser beam *Lasers in Surgery and Medicine* **7**(4) 347-352.
- Melin M, Joffre-Romeas A, Farges JC, Couble ML, Magloire H & Bleicher F (2000) Effects of TGF beta1 on dental pulp cells in cultured human tooth slices *Journal of Dental Research* **79**(9) 1689-1696.
- Morioka T, Atsumi K, Komiyama K, Sato T, Shouji S, Narita Y, Furumoto K, Maeda M & Minamizato T (1986) *Laser in Dental Medicine* Ishiyaku Publishers 7-10, Honkomagome 1-chome, Bunkyo-ku, Tokyo, 113-8612 Japan 39-52.
- Moritz A, Schoop U, Goharkhay K & Sperr W (1998a) The CO<sub>2</sub> laser as an aid in direct pulp capping *Journal of Endodontics* **24**(4) 248-251.
- Moritz A, Schoop U, Goharkhay K & Sperr W (1998b) Advantages of a pulsed CO<sub>2</sub> laser in direct pulp capping: A long-term *in vivo* study *Lasers in Surgery and Medicine* **22**(5) 288-293.
- Muto Y, Aoki S & Ushiki T (1999) Antibacterial effect and dental pulp response to added tentative primers *The Japanese Journal of Conservative Dentistry* **42**(2) 395-409.
- Nakashima M, Nagasawa H, Yamada Y & Reddi AH (1994) Regulatory role of transforming growth factor-beta, bone morphogenetic protein-2, and protein-4 on gene expression of extracellular matrix proteins and differentiation of dental pulp cells *Developmental Biology* **162**(1) 18-28.
- Nyborg H & Brännström M (1968) Pulp reaction to heat *The Journal of Prosthetic Dentistry* **19**(6) 605-612.
- Ohara A & Katoh Y (1999) Histopathological study on healing properties of exposed pulp alternately treated by different chemical cleansing and direct capping agents *The Japanese Journal of Conservative Dentistry* **42**(2) 435-458.
- Pelton RW, Saxena B, Jones M, Moses HL & Gold LI (1991) Immunohistochemical localization of TGF beta 1, TGF beta 2, and TGF beta 3 in the mouse embryo: Expression patterns suggest multiple roles during embryonic development *The Journal of Cell Biology* **115**(4) 1091-1105.
- Ratanasathien S, Wataha JC, Hanks CT & Dennison JB (1995) Cytotoxic interactive effects of dentin bonding components on mouse fibroblasts *Journal of Dental Research* **74**(9) 1602-1606.
- Rosenfeld EF, James GA & Burch BS (1978) Vital pulp tissue response to sodium hypochlorite *Journal of Endodontics* **4**(5) 140-146.
- Rowela SS, Suzuki A, Tomoda S, Yamada M, Matsui O & Senda A (2001) A study on microtensile bond strength of CO<sub>2</sub> lased dentin surface *Aichi-Gakuin Dental Science* **14** 9-14.
- Senia ES, Marshall FJ & Rosen S (1971) The solvent action of sodium hypochlorite on pulp tissue of extracted teeth *Oral Surgery, Oral Medicine, and Oral Pathology* **31**(1) 96-103.
- Serebro L, Segal T, Nordenberg D, Gorfil C & Bar-Lev M (1987) Examination of tooth pulp following laser beam irradiation *Lasers in Surgery and Medicine* **7**(3) 236-239.
- Shirono M, Ebihara T & Katoh Y (2003) Effect of CO<sub>2</sub> laser irradiation and direct capping with two-step bonding system on exposed pulp of Macaca fascicularis *The Japanese Journal of Conservative Dentistry* **46**(5) 761-781.
- Shoji S, Nakamura M & Horiuchi H (1985) Histopathological changes in dental pulps irradiated by CO<sub>2</sub> laser: A preliminary report on laser pulpotomy *Journal of Endodontics* **11**(9) 379-384.
- Sloan AJ & Smith AJ (1999) Stimulation of the dentine-pulp complex of rat incisor teeth by transforming growth factor-beta isoforms 1-3 *in vitro* *Archives of Oral Biology* **44**(2) 149-156.
- Sübay RK & Asci S (1993) Human pulpal response to hydroxyapatite and a calcium hydroxide material as direct capping agents *Oral Surgery, Oral Medicine, and Oral Pathology* **76**(4) 485-492.
- Suzuki T & Katoh Y (1997) Histopathological study on 4-META/MMA-TBB resin containing calcium hydroxide as a direct pulp capping agent *The Japanese Journal of Conservative Dentistry* **40**(1) 49-77.
- Tagami J, Hosoda H, Imai Y & Masuhara E (1987) Evaluation of a new adhesive liner as an adhesive promoter and a desensitizer on hypersensitive dentin *Dental Materials Journal* **6**(2) 201-208.
- Tagami J, Nakajima M, Takatsu T & Hosoda H (1993) Influence of dentin primers on fluid flow of bovine serum through dentin *Japan Society for Adhesive Dentistry* **11**(2) Abstract #II-6 p 99-100.
- Takatsuka T, Ishio F, Kurosaki A, Takabatake Y, Konishi N, Sato K, Torii Y & Yoshiyama M (2001) Antibacterial effect of an experimental adhesive system containing an antibacterial monomer: MDPB, on anaerobic bacteria in carious dentin *The Japanese Journal of Conservative Dentistry* **44**(4) 684-691.
- Tsunedo Y, Hayakawa T, Yamamoto H, Ikemi T & Nemoto K (1995) A histopathological study of direct pulp capping with adhesive resins *Operative Dentistry* **20**(6) 223-239.
- Tyas MJ, Anusavice KJ, Frencken JE & Mount GJ (2000) Minimal intervention dentistry—a review. FDI Commission project 1-97 *International Dental Journal* **50**(1) 1-12.



- Tziafas D, Alvanou A, Papadimitriou S, Gasic J & Komnenou A (1998) Effects of recombinant basic fibroblast growth factor, insulin-like growth factor-II and transforming growth factor-beta 1 on dog dental pulp cells *in vivo* *Archives of Oral Biology* **43**(6) 431-444.
- Tziafas D & Papadimitriou S (1998) Role of exogenous TGF-beta in induction of reparative dentinogenesis *in vivo* *European Journal of Oral Sciences* **106**(Supplement 1) 192-196.
- Utsunomiya T (1998) A histopathological study of the effects of low-power laser irradiation on wound healing of exposed dental pulp tissues in dogs, with special reference to lectins and collagens *Journal of Endodontics* **24**(3) 187-193.
- Yoshii E (1997) Cytotoxic effects of acrylates and methacrylates: Relationships of monomer structures and cytotoxicity *Journal of Biomedical Materials Research* **37**(4) 517-524.
- Zach L & Cohen G (1965) Pulp response to externally applied heat *Oral Surgery, Oral Medicine, and Oral Pathology* **19**(4) 515-530.

# Radiographic Versus Clinical Extension of Class II Carious Lesions Using An F-speed Film

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## Clinical Relevance

When using F-speed film (Insight–Kodak), the radiographic extent of Class II carious lesions underestimated the true clinical extent for both “aggressive” and “conservative” diagnoses.

## SUMMARY

This study investigated the difference in the apparent radiographic and true clinical extension of Class II carious lesions. Sixty-two lesions in both maxillary and mandibular premolars and molars were radiographed using Insight bitewing film. Class II lesions were scored independently by two masked examiners using an 8-point lesion severity scale. During the restoration process the lesions were dissected in a stepwise fashion from the occlusal aspect. Intraoperative photographs

(2x) of the lesions were made, utilizing a novel measurement device in the field as a point of reference. Subsequently, the lesions were all given clinical scores using the same 8-point scale. Statistical analysis showed a significant difference between the true clinical extension of the lesions compared to the radiographic score. “Aggressive” and “Conservative” radiographic diagnoses underestimated the true clinical extent by 0.66 mm and 0.91 mm, respectively. No statistical difference was found between premolars and molars or maxillary and mandibular arches. The results of this study help to define the parameters for making restorative treatment decisions involving Class II carious lesions.

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## INTRODUCTION

Since its development in the 1920s, the intraoral bitewing radiograph has proven to be a valuable aid in the diagnosis of interproximal carious lesions in posterior teeth. Overwhelming evidence has shown that bitewing radiographs consistently reveal a high percentage of additional Class II carious lesions over and above those seen clinically (Kidd & Pitts, 1990). At the same time, the known health hazards of excessive ionizing radiation demand that patients be exposed to as little radiation as possible. The benefits of early diagnosis, gained by

the judicious use of bitewing radiography, should not be ignored (Serman, 1990).

Radiographic film manufacturers continually attempt to produce faster films without sacrificing image quality, so that the radiation exposure to patients is reduced (Thunthy & Ireland, 2001). For many years, D-speed film (Ultra-speed, Eastman Kodak Co) has been considered the "gold standard" for analyzing images. Newer generations of faster E-speed films were introduced (Ektaspeed, Ektaspeed Plus, Eastman Kodak Co), with several studies showing statistically similar diagnostic capability to D-speed film (Ludlow & others, 1997; Hintze, Christoffersen & Wenzel, 1996). Subjective preference by many practitioners, however, has propagated the continued use of the slower D-speed film, with one study showing using E-speed film in only 18% of facilities (Button, Moore & Goren, 1999).

The latest generation of radiographic film, F-speed (Insight), was introduced in the summer of 2000. With a speed 60% faster than Ultra-speed and at least 20% faster than Ektaspeed Plus, Insight has been recommended as the film of choice by some researchers (Thunthy & Ireland, 2001). Sensitometric laboratory evaluations of Insight have shown that the film is 20% to 25% faster than Ektaspeed Plus, with no evident detriment to diagnostic efficacy (Syriopoulos & others, 2001; Price, 2001). Two other studies found Insight to be less affected when processed in used and depleted developing solutions (Geist & Brand, 2001; Ludlow, Platin & Mol, 2001). Several *in vitro* evaluations, using extracted teeth, have compared Insight to Ektaspeed Plus for interproximal caries diagnosis (Ludlow, Abreu & Mol, 2001; Nair & Nair, 2001; Landis & Koch, 1977). Two of these studies utilized the same five-point confidence scale for diagnosing the presence of caries and concluded there was no difference in diagnostic capabilities between the films (Nair & Nair, 2001; Landis & Koch, 1977). The third study claimed Insight was of a poorer quality than Ektaspeed Plus in terms of graininess (Ludlow & others, 2001). Thunthy and Ireland (2001) noted mottling, and Nair and Nair (2001) indicated that all of their examiners subjectively preferred Ektaspeed Plus to Insight, which appeared grainier. Clinical studies evaluating the ability of Insight to accurately depict the depth of interproximal carious lesions have not been found in the literature.

This study evaluated the clinical performance of Insight dental film by comparing the radiographic appearance of Class II carious lesions to the clinical extension, as verified at the time of operative treatment. This study also determined whether any differences existed between teeth (molars vs premolars) or location in the mouth (maxillary vs mandibular arches) in the clinical extension of Class II lesions, given similar radiographic appearances.

## METHODS AND MATERIALS

Sixty-two Class II carious lesions in 28 patients were included in the study. Prior to initiation, approval for the project was obtained from the local institutional review board (IRB). The scope of the study was explained to each patient and informed consent was obtained. Inclusion criteria were: (1) the caries must represent a primary interproximal lesion, (2) there must be a proximal contact with the adjacent tooth and (3) the lesion should have progressed to the point that it was deemed necessary to restore by both the referring dentist and the primary investigator based on existing radiographs and/or clinical findings. Exclusion criteria included the presence of frank cavitation or pulpal symptomatology. Primary teeth and severely rotated teeth that precluded the making of a quality bitewing radiograph were not utilized. Table 1 shows the location of the 62 lesions included in the study.

On the day of treatment, a quality bitewing radiograph was made using Insight double pack film (IP-22 Size 2, Lot #2103659, Exp 10/03, Eastman Kodak Company, Rochester, NY, USA). One of the films was used for the study and the duplicate was placed in the patient record. In an effort to standardize, all films were exposed by the primary investigator using the same unit (Gendex GX-770, Gendex Corporation, Des Plaines, IL, USA), which was calibrated at regular intervals. Recommended guidelines were used when exposing the films (70KVp, 7 mA, 11 pulses). The unit utilized an 8-inch round cone that was placed in contact with the patient's cheek during exposure, and bitewing radiographs were made with the aid of self-adhesive paper bitewing tabs affixed to the film packets. A film was not considered acceptable for the study if there were overlapping proximal contacts on the test teeth. For patients in the study who had multiple qualified

Table 1: *Distribution of Lesions*

	Maxillary	Mandibular	Total
<b>Premolars</b>	24	8	32
<b>Molars</b>	15	15	30
	39	23	62

Table 2: *8-Point Lesion Severity Scale*

Category	Description
<b>0</b>	No lesion detected
<b>1</b>	Lesion in outer half of enamel
<b>2</b>	Lesion in inner half of enamel
<b>3</b>	DEJ involved/<0.5 mm into dentin
<b>4</b>	0.5 - 1.0 mm into dentin
<b>5</b>	>1.0-1.5 mm into dentin
<b>6</b>	>1.5-2.0 mm into dentin
<b>7</b>	> 2.0 mm into dentin



lesions restored on different days, additional radiographs were exposed only if the first one did not adequately depict all lesions. Each of the films was developed on the day it was exposed in an automatic roller-type processor (Gendex GXP Model 110-0096 G1, Gendex Corp) with self-replenishing solutions (Supermax GX solutions, Gendex Corp).

### Radiographic Scoring

The full set of radiographs comprising the 62 carious lesions was scored by two experienced clinicians according to an 8-point scale (Table 2). A calibration exercise utilizing a subset of 12 of the experimental lesions was performed to standardize radiographic scoring procedures prior to reading the full test set. Final radiographic scoring was performed 10 days following the calibration exercise to avoid recall bias, and each examiner independently scored the films in a darkened room. The radiographs were placed on a standard fluorescent view box (8 in x 10 in), with all areas peripheral to the film mounts blocked out with black paper. Each examiner was provided with a clear plastic millimeter ruler to aid in scoring and asked to read the films using their usual technique. All 62 lesions were scored at the same sitting, and the examiners were blinded in regard to the corresponding clinical data for each lesion.

### Operative Procedure

Validation of the radiographic findings was attained by direct visualization of the carious lesions *in situ* during the operative procedure. The primary investigator (SK) provided all operative treatment under local anesthesia and rubber dam isolation. A pre-operative photograph (2x) of the occlusal surface of the tooth was made using slide film (Kodak Elite Chrome, 100 speed-35 mm). The lesion was then accessed and the proximal surface was carefully dissected in a step-wise manner down through the carious tissue. To capture cross-sectional views of the lesions at their deepest axial extent, occlusal photographs were made (2x) at various depths during the dissection. The number of photographs taken ranged from 4 to 9, depending on the size of the tooth and the occlusal-cervical location of the lesion.

A novel measurement reference instrument was placed in the photographic field for each exposure, usually on the tooth being treated (Figure 1). The instrument was a 2-mm segment sectioned from the tip of a periodontal probe (Hu-Friedy, PCPUNCH 15, Chicago, IL, USA) that had individual millimeter markings demarcated by black bands. Following the removal of all carious tissue, the preparation was completed and the tooth restored with an appropriate restorative material per standard guidelines.

### Clinical Scoring

Clinical scoring of the lesions was done from the intra-operative photographs. The one photograph for each



Figure 1: Measurement utilizing the tip of a periodontal probe.

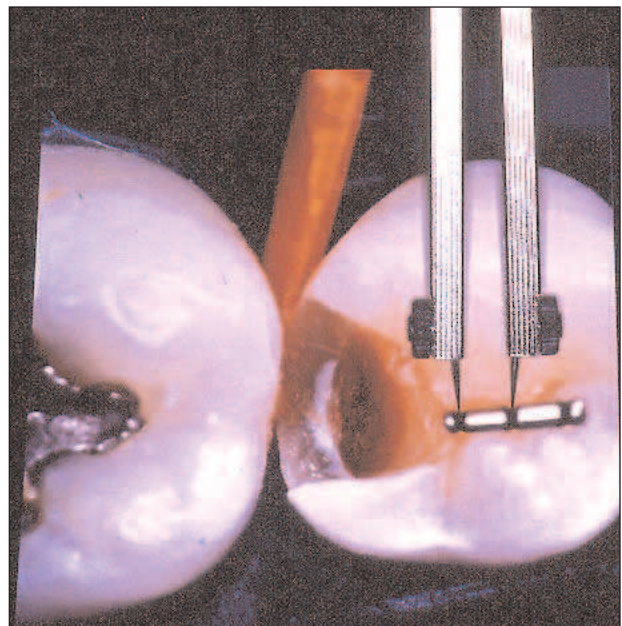


Figure 2: Measuring the reference probe with calipers.

case that depicted the carious lesion at the deepest axial point was selected. Measurements of the lesions were made directly from the photographs using a 6-inch caliper (6-inch SpringBow Divider, Miltex Instrument Company, Bethpage, NY, USA). In each case, the reference device was measured first, as this distance was known to equal 1.0 mm (Figure 2). The tips of the calipers were placed on the centers of the black marking bands of the reference device and the value recorded to the nearest 0.1 mm. Next, the carious lesion itself was

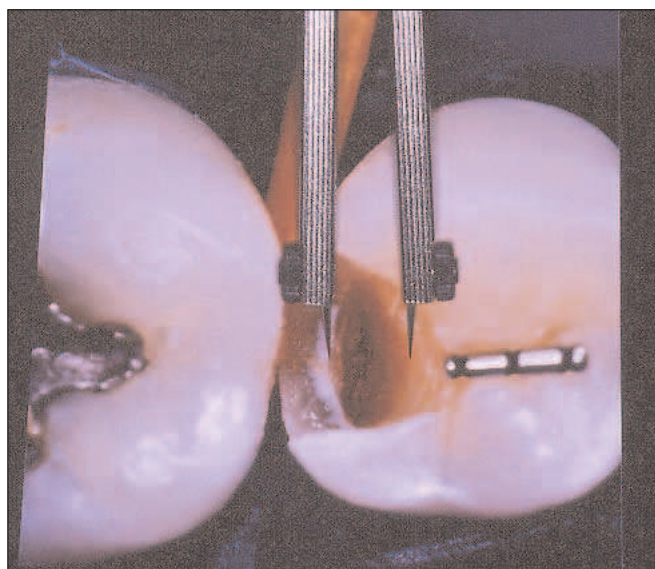


Figure 3: Caliper measurement from DEJ to the deepest axial boundary of the lesion.

Table 3: *Conservative Radiographic Set*

Category	Premolars	Molars	Total
0	1	4	5
1	5	6	11
2	12	7	19
3*	7	8	15
4	5	4	9
5	1	0	1
6	1	1	2
7	0	0	0
Mean Score	2.53	2.2	2.37

\* Shaded area represents lesions in dentin.

Table 4: *Aggressive Radiographic Set*

Category	Premolars	Molars	Total
0	1	0	1
1	1	7	8
2	6	5	11
3*	16	10	26
4	6	7	13
5	0	0	0
6	2	1	3
7	0	0	0
Mean Score	3.03	2.70	2.87

\* Shaded area represents lesions in dentin.

measured, as evidenced by visual changes in dentin. The boundaries of the lesion were demarcated by the point at which the dentin was similar in color and texture to the surrounding healthy dentin. All 62 lesions in this study extended into the dentin clinically, so one tip

of the calipers was placed at the DEJ, while the other was placed at the deepest axial boundary of the lesion (Figure 3). The true clinical value of lesion depth was obtained by multiplying the measured value of the lesion by 1.0 mm, then dividing it by the measured value of the reference device. This method eliminated any possible errors arising from slight magnification differences in individual photographs, since each print depicted the measurement reference device that had a known value of 1.0 mm.

The primary investigator made all of the clinical measurements from the photographs. To assess the repeatability of the procedure, the measurements were made twice 10 days apart to avoid recall bias. The repeatability of the measurement technique was very high (Pearson Correlation Coefficient = 0.975), and the final measured value for each lesion was obtained by taking the average of the two measurements. These values were then converted to categorical scores using the same lesion severity scale used for scoring the radiographs (Table 2).

## RESULTS

Results of the radiographic scoring of the full set of films showed an exact agreement between the two examiners (both scored the lesion as a “3,” etc) in 40% (25/62) of the cases and a score within one category of each other in an additional 50% (31/62) of the cases. The remaining six cases showed a difference between examiners of more than one scoring category. This agreement level produced a weighted Kappa value of 0.64. In cases where the examiners disagreed, there was no systematic bias involved (one of the examiners did not consistently score higher than the other), as each accounted for approximately one half of the higher scores. Given the large number of relatively small categories in the scale, a one-category difference was considered within the normal limits of variation among dentists in radiographic interpretation. The six cases in which the difference was more than one category were reviewed jointly by the two examiners and a consensus score was reached for each tooth. Accordingly, two radiographic data sets were constructed, a “conservative” set (Table 3) in which the lower score of the 31 cases was used, and an “aggressive” set (Table 4), in which the higher scores were used. For the conservative set, the mean score for all teeth was 2.37, and for the aggressive set, it was 2.87 or approximately 0.5-mm deeper. By comparing both sets to the clinical findings, differences between conservative and aggressive radiographic diagnoses could be assessed.

Results of the clinical scoring showed that all 62 lesions included in the study had penetrated the dentino-enamel junction (DEJ) clinically. Mean measured lesion depths into dentin can be seen in Table 5, with all measurements from the DEJ. These depths were

subsequently converted into categorical scores. Although this conversion is from interval data to ordinal, it was necessary in order to make a direct correlation with the ordinal values obtained from the two radiographic scales. Tables 6 and 7 show a cross comparison of clinical scores to radiographic scores for both aggressive and conservative radiographic sets. The shaded areas in the tables indicate the "zone" for expected scores if the radiographic and clinical scores correlated perfectly. Lesions that were rated above the zone were

deeper radiographically than clinically, those rated below the zone were deeper clinically. The aggressive radiographic set included 29% (18/62) of lesions in zone and 69% (43/62) below zone. The conservative set contained only 13% (8/62) of lesions in zone and 85% (53/62) below zone. In each radiographic set, only one lesion was rated above the zone.

Table 8 shows the average clinical score for all teeth was 4.18, with no difference between molars and premolars. The difference between the radiographic score and the clinical score was calculated for each tooth using both radiographic sets. The average difference in scores between the radiographic and clinical ratings was 0.37 scale units higher for molars than premolars in both radiographic sets, 1.5 vs 1.13 for the aggressive and 2.0 vs 1.63 for the conservative. The average difference in scores was also 0.5 scale units higher for the conservative set than for the aggressive set for both premolars and molars. The mean overall differences for all teeth were 1.31 for the aggressive set and 1.81 for the conservative set.

Statistical analysis of the data was performed using SAS 8.0 software. One of the primary objectives of the project was to study the differences in caries progression between what was seen on F-speed radiographs and what was found clinically. Therefore, differences between each of the radiographic data sets (aggressive and conservative) and the clinical scores were analyzed (Table 9). The average differences between the clinical scores and aggressive radiographic scores were 1.13, 1.50 and 1.31 units for premolars, molars and overall. The clinical scores were significantly greater than the radiographic scores for both the conservative and aggressive sets ( $p < 0.05$ ).

	Premolars	Molars	Overall
Depth	0.84 mm	0.91 mm	0.87 mm
*Measurements from DEJ			

Clinical Scores	Aggressive Radiographic Scores									
	Cat	0	1	2	3	4	5	6	7	
	0									0
	1									0
	2									0
	3	1	3	4	9	1				18
	4		2	5	12	6				25
	5		2	1	3	4				10
	6		1	1	1	2		3		8
	7				1					1
		1	8	11	26	13	0	3	0	62
1 above zone: 18 in zone: 43 below zone										

Clinical Scores	Conservative Radiographic Scores									
	Cat	0	1	2	3	4	5	6	7	
	0									0
	1									0
	2									0
	3	2	3	9	3	1				18
	4	2	4	8	8	3				25
	5	1	2	2	2	3				10
	6		2		1	2	1	2		8
	7				1					1
		5	11	19	15	9	1	2	0	62
1 above zone: 8 in zone: 53 below zone										

	Clinical Score	Aggressive Radiographic Score	Difference in Scores	Conservative Radiographic Score	Difference in Scores
Premolars	4.16	3.03	1.13	2.53	1.63
Molars	4.20	2.70	1.50	2.20	2.00
Overall	4.18	2.87	1.31	2.37	1.81



Another primary objective of this study was to determine if there were any differences between molars and premolars in the clinical extension of caries. In other words, given similar radiographic appearances, were lesions further extended clinically (verified intra-operatively) in one tooth type versus another. In both radiographic sets, the average delta values were 0.375 categories higher for molars than for premolars. *T*-tests determined that these differences were not statistically significant ( $p=0.294$  conservative set,  $p=0.261$  aggressive set). Comparisons were also made between maxillary and mandibular teeth. In both radiographic sets, maxillary lesions appeared to be deeper than mandibular lesions with the same radiographic appearance. However, the differences were not statistically significant using a *t*-test ( $p=0.172$  for both conservative and aggressive sets).

In evaluating each tooth clinically during preparation for cavitation of the enamel proximal surface, only 16 of the 62 lesions (26%) were cavitated (Table 10). Approximately 50% of lesions evaluated as 0.5-1.0 mm into dentin (rating of 4) were cavitated, but all lesions evaluated as >1.0 mm into dentin (rating of 5 or above) were cavitated.

## DISCUSSION

When comparing radiographic and clinical categorical scores, it was seen that both the aggressive and conservative radiographic scores underestimated the true depth of the lesions by 1.31 and 1.81 units, respectively, with differences for both radiographic sets highly significant. Jessee, Makins and Bretz (1999) found similar results using both Ultraspeed and Ektaspeed Plus films when comparing radiographic caries diagnosis to histologic findings. Measuring the clinical depth of a proximal carious lesion is a difficult task. In a recent textbook by Kidd, Fejerskov and Mjör (2003), discolored dentin is considered "a rather unreliable guide to gauge the level of dentin infection." The current accepted clinical procedure in the United States is to remove all softened dentin until the base is firm to tactile examination with an explorer. In this study, no attempt was made to determine whether the assessed caries depth, measured from the photographs as a color change in axial extension, correlated with the progression of bacterial invasion. Although the visual criterion used was somewhat subjective, it represented

Table 9: Differences Between Tooth Type and Arch for Both Radiographic Sets

	Aggressive Radiographic Set	Conservative Radiographic Set
Clinical Score	4.18	4.18
Radiographic Score	2.87	2.37
<i>p</i> -value	<0.001	<0.001
Difference-Molars	1.5	2
Difference-Premolars	1.125	1.625
<i>p</i> -value	0.2607	0.2935
Difference-Maxillary	1.4615	1.974
Difference-Mandibular	1.0435	1.5217
<i>p</i> -value	0.1715	0.1716

Table 10: Cavitated Lesions per Scoring Category for Each Radiographic Set

Category	Aggressive Radiographic Set (% cavitated)	Conservative Radiographic Set (% cavitated)
0	0	0
1	0	18
2	27	11
3	15	27
4	46	56
5	-	100
6	100	100
7	-	-
Total Found	16/62	26%

the best clinical representation of a proximal carious lesion, as seen on a bitewing radiograph. In previous similar *in vitro* studies, the gold standard for comparisons was the histological section of the extracted teeth (Jessee & others, 1999; Hintze & others, 1996). However, caries depth was evaluated in these sections based solely on the microscopic evaluation of a color change between involved and uninvolved dentin. The extent to which carious tissue should be removed is a separate issue and is not the subject of this study.

There were eight scoring categories in the lesion severity scale used in this study. Category 0 indicated no lesion, categories 1 and 2 were lesions in the outer and inner halves of enamel, respectively, and categories 3 through 7 corresponded to 0.5-mm increments in the dentin. Since the interproximal enamel just below the contact points of molars and premolars was close to 1.0-mm thick, each category in the scale closely approximated 0.5 mm. By converting the measured true clinical depths to scale ratings, a more meaningful interpretation of the data could be made. The average difference in rating scale values of 1.31 and 1.81 units for the aggressive and conservative radiographic sets translated to clinical differences of 0.66

mm (1.31 X 0.5 mm) and 0.91 mm (1.81 cat x 0.5 mm), respectively. The "Aggressive" interpretation of the radiographs actually underestimated the extension by 0.66 mm, but the "Conservative" interpretation underestimated it by 0.91 mm. This information is extremely valuable when planning restorative intervention.

Radiographic and clinical scores correlated poorly in this study. Only 13% and 29% of lesions fell "in zone" (correlated perfectly) for conservative and aggressive sets, respectively. In both sets, only one lesion was further extended radiographically than clinically. On the other hand, 69% and 85% of the lesions were further extended clinically than radiographically for the aggressive and conservative sets. Jessee and others (1999) reported levels of agreement of 23.3% for Ultraspeed film and 22.0% for Ektaspeed Plus film. Their study utilized extracted teeth and few categories for lesion depth, but the results were similar. Likewise, most of the misdiagnoses in that study involved radiographic underestimation of true lesion depths.

Higher levels of agreement between radiographic and clinical findings have been reported using much broader scoring categories. Thylstrup, Bille and Qvist (1986) found an 82% level of agreement between radiographic and clinical scores. That study utilized no standardization or masking of operators, which may have led to elevated levels of agreement. Espelid and Tveit (1986) found a level of agreement of approximately 60% in their study on extracted teeth. They utilized much broader scoring categories and histologic validation of lesion depth. In both of these studies, lesions that did not correlate well were mostly radiographic underestimations of true clinical extent.

Utilization of a greater number of categories (eight) in this study, each representing smaller differences in carious extension, undoubtedly makes correlation between radiographic and clinical scoring more difficult. However, as remineralization of shallow and non-cavitated lesions has become a more highly accepted treatment option for dental providers, the accurate monitoring of lesion size over time becomes more critical. Therefore, 0.5-mm categories were considered appropriate in this project. Pitts and Rimmer (1992) reported a 60% level of agreement when grouping all enamel and dentinal lesions together. This grouping may cause misleading results. For instance, the level of agreement in this study would have increased by more than 30% (67% aggressive, 44% conservative) if lesions were lumped into only two groups, enamel and dentin.

Another primary objective of this study was to determine if any clinical differences existed between molars and premolars given similar radiographic appearances. The alternative hypothesis was that the clinical extent would be farther in molars versus premolars due to the facial-lingual thickness disparity between

these tooth types. Other studies have found no difference between molars and premolars in the likelihood of cavitation of the proximal surface when the radiographic depth was the same. There was no statistically significant difference between tooth type in this study. Additionally, no significant differences were found between maxillary and mandibular teeth.

## CONCLUSIONS

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

1. Both aggressive and conservative radiographic diagnoses using an F-speed film (Insight—Kodak Co) significantly underestimated the true clinical extent of Class II carious lesions by 0.66 and 0.91 mm, respectively. Therefore, the aggressive interpretation of the F-speed bitewing radiograph was more accurate than the conservative interpretation.
2. There were no statistically significant differences between teeth (molar vs premolar) or location in the mouth (maxillary vs mandibular arch) in the clinical extension of Class II carious lesions when the radiographic extension was the same.
3. Cavitation was found in 16 of 62 preparations (26%) evaluated in this study; 27% for lesions that were radiographically in enamel, 50% for lesions that appeared to be into dentin less than 1.0 mm and 100% for lesions that appeared to be greater than 1.0 mm into dentin.

## Disclaimer

The views expressed in this article are those of the authors and do not necessarily reflect the official policy or position of the Department of the Navy, Department of Defense or the US Government.

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## References

- Button TM, Moore WC & Goren AD (1999) Causes of excessive bitewing exposure: Results of a survey regarding radiographic equipment in New York *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiologic Endodontology* **87**(4) 513-517.
- Espelid I & Tveit AB (1986) Clinical and radiographic assessment of approximal carious lesions *Acta Odontologica Scandinavica* **44**(1) 31-37.
- Geist JR & Brand JW (2001) Sensitometric comparison of speed group E and F dental radiographic films *Dentomaxillofacial Radiology* **30**(3) 147-152.
- Hintze H, Christoffersen L & Wenzel A (1996) *In vitro* comparison of Kodak Ultra-speed, Ektaspeed, and Ektaspeed Plus,

- and Agfa M2 Comfort dental x-ray films for the detection of caries *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiologic Endontology* **81(2)** 240-244.
- Jessee SA, Makins SR & Bretz WA (1999) Accuracy of proximal caries depth determination using two intraoral film speeds *General Dentistry* **47(1)** 88-93.
- Kidd EA & Pitts NB (1990) A reappraisal of the value of the bitewing radiograph in the diagnosis of posterior approximal caries *British Dental Journal* **169(73)** 195-200.
- Kidd EA, Fejerskov O & Mjör JA (2003) (eds) Caries removal and the pulpo-dentinal complex in Fejerskov O & Kidd EAM (eds) *Dental Caries: The Disease and Its Clinical Management* Oxford, UK Blackwell Munksgaard p 267-274.
- Landis JR & Koch GG (1977) The measurement of observer agreement for categorical data *Biometrics* **33(1)** 159-174.
- Ludlow JB, Platin E, Delano EO & Clifton L (1997) The efficacy of caries detection using three intraoral films under different processing conditions *Journal of the American Dental Association* **128(10)** 1401-1408.
- Ludlow JB, Platin E & Mol A (2001) Characteristics of Kodak Insight, an F-speed intraoral film *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiologic Endodontology* **91(1)** 120-129.
- Ludlow JB, Abreu M Jr & Mol A (2001) Performance of a new F-speed film for caries detection *Dentomaxillofacillo Radiology* **30(2)** 110-113.
- Nair MK & Nair UP (2001) An *in-vitro* evaluation of Kodak Insight and Ektaspeed Plus film with a CMOS detector for natural proximal caries: ROC analysis *Caries Research* **35(5)** 354-359.
- Pitts NB & Rimmer PA (1992) An *in vivo* comparison of radiographic and directly assessed clinical caries status of posterior approximal surfaces in primary and permanent teeth *Caries Research* **26(2)** 146-152.
- Price C (2001) Sensitometric evaluation of a new F-speed dental radiographic film *Dentomaxillofacillo Radiology* **30(1)** 29-34.
- Serman NJ (1990) Exposure to dental radiation—a perspective *Quintessence International* **21(4)** 331-333.
- Syriopoulos K, Velders XL, Sanderink GC & van der Stelt PF (2001) Sensitometric and clinical evaluation of a new F-speed dental X-ray film *Dentomaxillofacillo Radiology* **30(1)** 40-44.
- Thunthy KH & Ireland EJ (2001) A comparison of the visibility of caries on Kodak F-speed (insight) and D-speed (Ultra-speed) films *LDA Journal* **60(2)** 31-32.
- Thylstrup A, Bille J & Qvist V (1986) Radiographic and observed tissue changes in approximal carious lesions at the time of operative treatment *Caries Research* **20(1)** 75-84.



# Occlusal Loading Evaluation in the Cervical Integrity of Class II Cavities Filled with Composite

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## Clinical Relevance

The periodical supervision of condensable composite proximal restorations is essential with respect to the clinical success of such restorations, as the microleakage quality greatly increases after axial mechanical load incidence.

## SUMMARY

There are many doubts about the clinical behavior of condensable composite restorations in Class II cavities, particularly when they are submitted to axial mechanical loads. This study evaluated cervical microleakage in Class II direct fillings in composite, whether or not they were submitted to an occlusal load cycling. Twenty-three human molars with standardized cavities (proximal vertical "slot") were treated with enamel and cement endings. After completion of the filling process with condensable composite

(Surefil), they were separated into two groups: control (without occlusal loading) and test, where 4,000 one-second cycles of 150 N occlusal loading were applied. Twenty teeth were submitted to a microleakage test and then evaluated according to dye penetration. Significant statistical differences (Wilcoxon test,  $p=0.005<0.05$ ) of leakage degree in enamel and cement were found in the control group. Significant statistical differences at  $<0.05$  were also found in the test group, with  $p=0.045$ .

After paired comparison of the control and test groups, a significant statistical difference was found at the enamel level (Mann-Whitney test,  $p=0.03$ ). However, no significant statistical differences were found at the cement level ( $p=0.28$ ). Therefore, it could be concluded that there was greater microleakage in cement compared to enamel, and occlusal loading has a decisive influence, as it increases the rate of microleakage.

## INTRODUCTION

In the past, amalgam and direct gold fillings were the options dental practitioners used as posterior teeth fill-

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ings. Patients and practitioners were not concerned about esthetical cavity restorations, as materials used at that time were not suitable for this purpose. This scenario lasted for several years until a series of events, such as the introduction of enamel acid etching (Buonocore, 1955), the development of dental composites (Bowen, 1962) and the evolution of the hybrid layer (Nakabayashi, Kojima & Masuhara, 1982), changed this perspective. New conservative possibilities were created, and the dental filling process became less destructive as additional retention was no longer necessary.

Since then, a different restorative approach to anterior and posterior teeth has been considered, giving rise to more research on this issue. Great improvements in dental composites and dental adhesives, patients' increasing interest in aesthetic restorations and other concerns, such as mercury usage, made aesthetic posterior fillings a reality in dentistry. Research conducted by Barnes and others (1991) has shown significant improvement in composite fillings, which lead to other studies (Leinfelder, 1995) that have demonstrated relative clinical success. However, other studies, including that by Abdalla and Davidson (1993), emphasize the clinical limitation of this material, mainly regarding microleakage, which, besides causing post-operative sensitivity, promotes recurrent caries.

A composite, known as condensable, has been released into the market, offering the possible joining of conventional composite esthetics with the properties of amalgam condensation. It is a well-known fact that no restorative system, up to now, can hermetically seal a cavity when considering the behavior of this material in Class II cavities, particularly when submitted to axial mechanical loads. When a new material is presented to the professionals, there are many doubts regarding its clinical success. Some of these doubts relate to its clinical performance after occlusal load cycling and how it would affect the microleakage of this material.

This study evaluated microleakage *in vitro* in walls located in the enamel and cement of proximal vertical "slot" type cavities filled with condensable composite after being submitted to occlusal load cycling.

## METHODS AND MATERIALS

Twenty-three human molars with no fissures or cracks recently extracted due to surgical needs, were immersed in thymol solution at 0.5% until starting the experiment. All the molars were prepared with two standardized Class II proximal vertical "slot" cavities, one on the mesial face and the other on the distal face. They were prepared with 2143 (KG Sorensen) diamond points, which were changed after three cavity preparations, in a high speed turbine (Kavo America Corporation, Lake Zurich, IL, USA) with oil free water irrigation. All the molars had a 3-mm distance between

the vestibular and lingual walls and were 2-mm deep from the axial wall. The limit in the cervical walls was established at a proximal, which was located 1 mm above the enamel-cement junction (wall in enamel) and the other was 1 mm below the enamel-cement junction (wall in cement or dentin). None of the cavo-surface angles of these preparations had beveled edges; nonetheless, all the walls were trimmed with a low rotation multi-fluted bur.

After cavity preparation, the teeth were cleaned with fine flour of pumice using a rubber cup in a low speed handpiece for 30 seconds. The teeth were then conditioned with 37% phosphoric acid (3M-ESPE, St Paul, MN, USA) for 30 seconds in enamel and for 15 seconds in dentin. After application of the conditioning gel, the surfaces were rinsed with distilled water for 30 seconds and gently dried with oil and dust-free air for two seconds. Prime & Bond NT (Dentsply/Caulk, Milford, DE, USA) adhesive system was then applied according to the manufacturer's instructions. A thin layer of the product was applied with a brush and was left undisturbed for 30 seconds. The solvent was then removed with oil and dust-free air jets for two seconds and the surface was light-cured for 10 seconds with an Optilux 401 light source (Demetron/Kerr, Danbury, CT, USA) (intensity = 400 mW/cm<sup>2</sup>, evaluated every 10 uses by means of a radiometer). Another layer of the adhesive was then applied and immediately dried and light cured, similar to the first layer. The cavities were then restored with three portions of Surefil (Dentsply/Caulk) condensable resin according to the Lutz and others (1986) restorative technique (one cervical portion, one vestibular portion and one lingual portion) and each increment was cured for 60 seconds. A pre-contoured matrix system (Palodent, Dentsply International, York, PA, USA) was used to create the lost proximal wall.

After restoration, the samples were kept in distilled water at 37°C for seven days. The teeth were then separated according to the type of treatment they would undergo: Group 1 (control), without occlusal load; Group 2 (test), with occlusal load. The Group 2 teeth were then prepared for the mechanical test as follows: the roots were coated with melted wax up to 2 mm below the enamel-cement junction. Then, a PVC cylindrical tube (Tigre) 21 mm in diameter by 25-mm high was used for teeth inclusion in an autopolymerizing acrylic resin (Classico) up to the wax level mark (two millimeters below the amelo-cement junction). To achieve this, a deliner (Bioart) was used, with cusps that remained rigorously parallel to the base. Thus, the load coming from the test equipment (EMIC MF dl 500) was equally distributed among the cusps, according to Davidson and Abdalla (1994) and Raadal (1979). The teeth were then removed from the acrylic resin and wax was substituted by an additional silicone

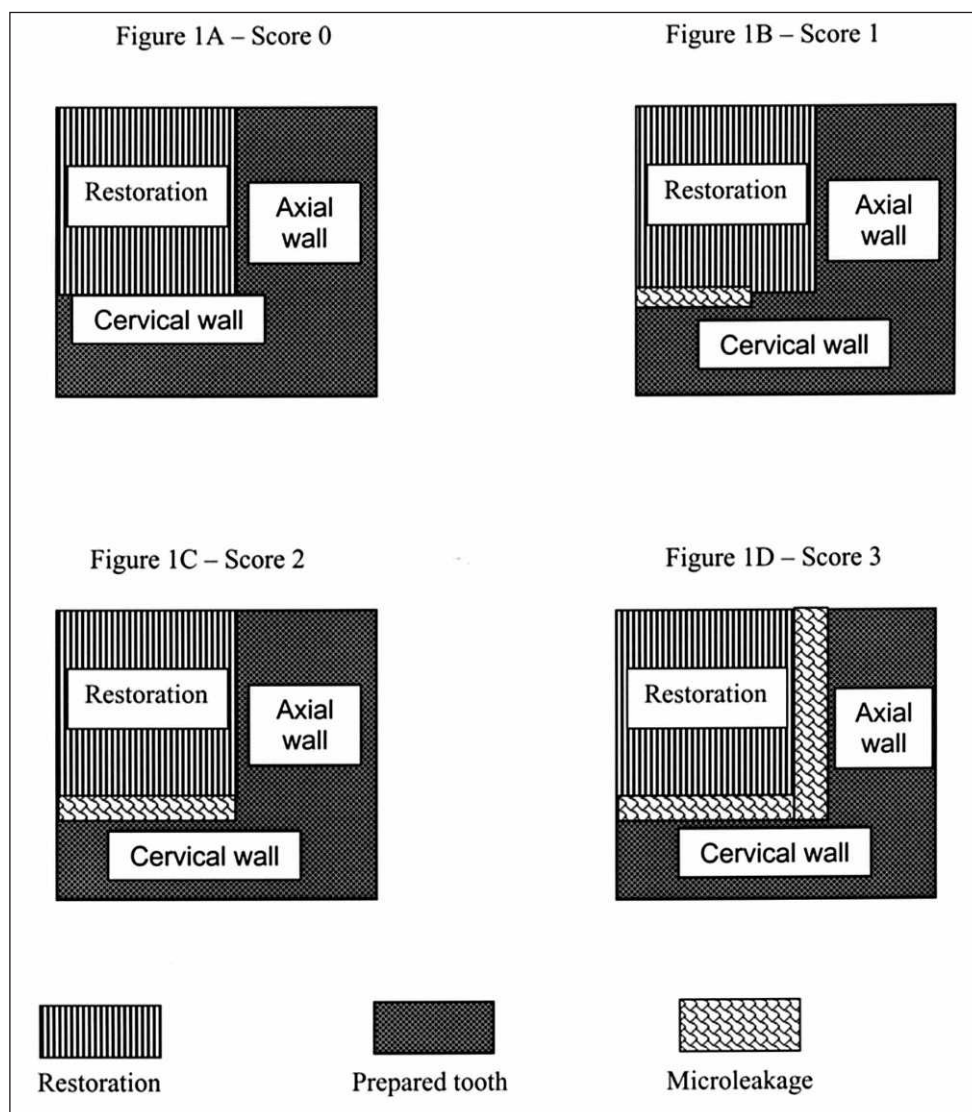


Figure 1.

(Presidente-Coltène/Whaledent, Cuyahoga Falls, OH, USA), the excess was removed by means of a Le Cron spatula at the level of the area previously marked as fulcrum. Ten teeth then underwent 4,000 loads of 150 N, which lasted one second according to Croll (1992), with the help of universal equipment controlled by “software” (TESC version 1.08).

All the teeth were then immersed in a silver nitrate dyeing solution at 50% for four hours (Taylor & Lynch, 1992). At the end of this period, all the teeth were longitudinally sectioned by means of a mesio-distal cut using a slow-speed diamond saw (Komet) under a coolant water flow. As a follow-up procedure, two professionals evaluated the pre-calibrated tooth halves according to dye microleakage degree using a 40x magnifying glass. The results of the analysis presented the following score: 0 = no leakage; 1= leakage up to half of

the cervical wall; 2 = leakage in the entire cervical wall without reaching the axial wall; 3=leakage in the entire cervical wall, even reaching the axial wall. (Figure 1A,B,C,D).

## RESULTS

The results, submitted to a non-parametrical statistical analysis, were: in the control group, a significant statistical difference (Wilcoxon test,  $p=0.005$ ) of the degree of leakage was found in the enamel and cement, as demonstrated in Figure 2. A significant statistical difference was also found in the test group, with a value of  $p=0.045$ , as shown in Figure 3. After paired comparison of both the control and test groups, a significant statistical difference at the enamel level (Mann-Whitney test,  $p=0.03$ ) was found, as shown in Figure 4. However, no significant difference at the cement level ( $p=0.280$ ) was registered and is demonstrated in Figure 5.

## DISCUSSION

The results that were found agree with the world literature; that is, the margins located in cement allow for a greater microleakage rate when compared to the margins in enamel. However, the values found in

enamel are surprising, as many studies have demonstrated excellent results (Wendt, McInnes & Dickinson, 1992; Tung, Estafan & Scherer, 2000; Duncalf & Wilson, 2001). The conjunction of some parameters may lead to as high a microleakage percentage as 60%, as shown in Figure 3. The factors of polymerization contraction, photopolymerization speed and intensity, cavitory configuration factor (factor-C), the fact that the “condensable” composite has an elasticity auto-module and the enamel structure in the cervical region is aprismatic or anisotropic, are probably responsible for this high microleakage percentage.

Studies done by Takamizu and others (1988), Sakaguchi, Douglas and Peters (1992) and Feilzer and others (1955) report that polymerization speed is directly related to the composite visco-elasticity property and, because of this polymerization contraction, a greater



light intensity will result in a lesser viscosity, which, in turn, leads to greater composite contraction. In order to reduce the composite contraction effect, it may be stated that, if light intensity could be reduced, the contraction effect will diminish. However, according to Peutzfeldt (1994), the composite conversion degree will also be diminished, thus reducing the material's mechanical properties. Although Hansen and Asmussen (1993) do not consider the direct relation between light intensity and composite mechanical properties, authors such as Rueggeberg, Caughman and Curtis (1994) and Rueggeberg and Jordan (1993) have suggested that, due to this composite resistance reduction and considering the intensity decrease, polymerization time should be increased. Additionally, according to studies by Goracci, Mori and Casa De Martinis (1996) and Koran and Kurschner (1998), equipment producing gradual photopolymerization should be used, alternating low frequency with high frequency. However, Yearn (1985) considers that, as layers nearer the surface acquire maximum polymerization, it would be very difficult to extend photopolymerization to the deeper layers by increasing the time of exposure, as it is very difficult to have light reach those layers. The same author states that the light source should be located as near as possible to the material to which it is being applied.

In their studies, Davidson and de Gee (1984), Feilzer, de Gee and Davidson (1987) and Carvalho and others (1996) observed the direct relation between polymerization contraction and the cavitory configuration factor. They determine that the greater the number of walls related to the composite, the greater the stress generated during the reaction, thus producing a greater polymerization contraction, which would create an opening of the tooth restoration interface.

Research conducted by Bouschlicher, Vargas and Boyer (1997) has demonstrated that "condensable" composites produce considerable stress during polymerization contraction due to the reduction of the viscoelasticity property during polymerization. A relevant fact for a probable improvement in the automodule elasticity of the composites' performance is observed in research conducted by Francci and others (1999) and Tung and others (2000). When improvements in cervical adaptation are observed and when low-module elasticity resin materials are associated with "condensable" compos-

ites, with the interpositioning of these materials as lining between the hybrid layer and the more rigid restorative composite, there will likely be a reduction of stress generated during polymerization.

In his studies, Gwinnett (1973) demonstrated that, in the case of an aprismatic or anisotropic enamel structure, both the number and the resin "tags" size formation is smaller and, as a consequence, damages the enamel/filling interface sealing, thus allowing for greater microleakage to occur.

The occlusal loading effect acted negatively, altering the degree of microleakage according to studies by Mandras, Retief and Russel (1991) and Lundin and Noren (1991), which showed that samples submitted to

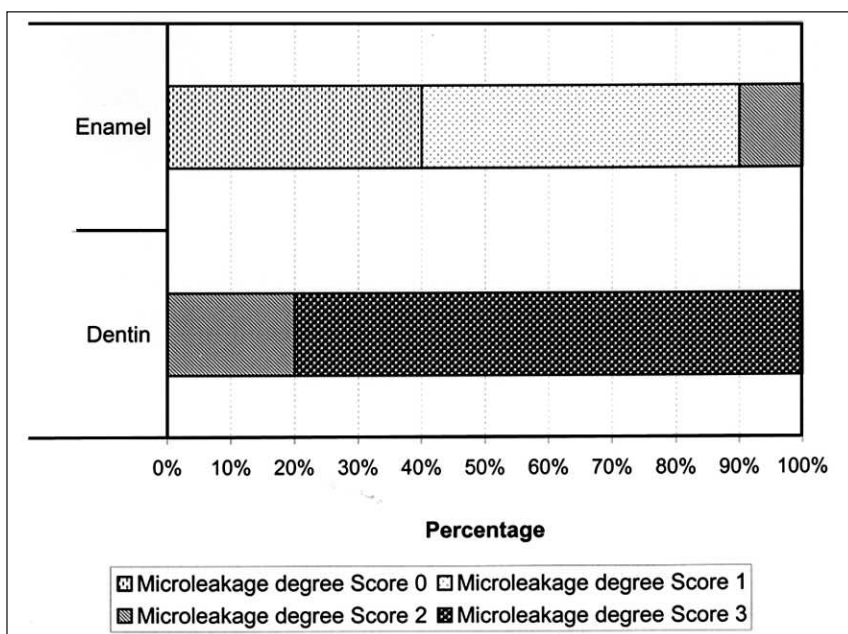


Figure 2. Leakage percentage degree by locality in control group.

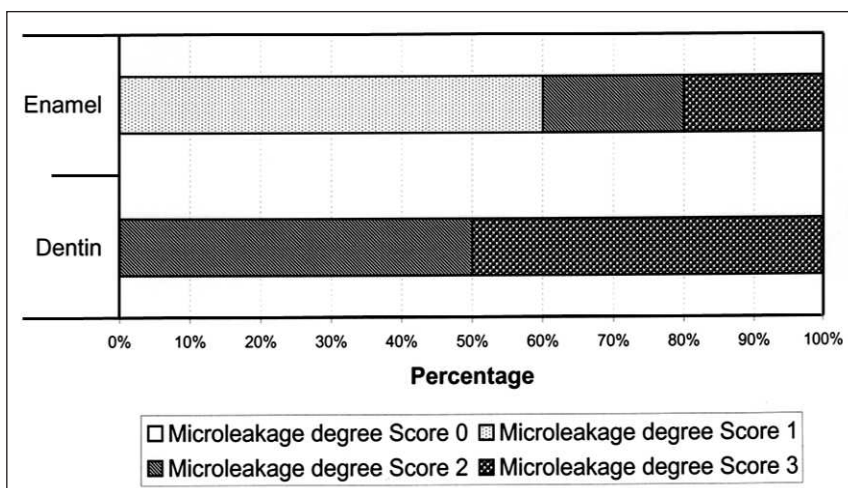


Figure 3. Leakage percentage degree by locality in test group.

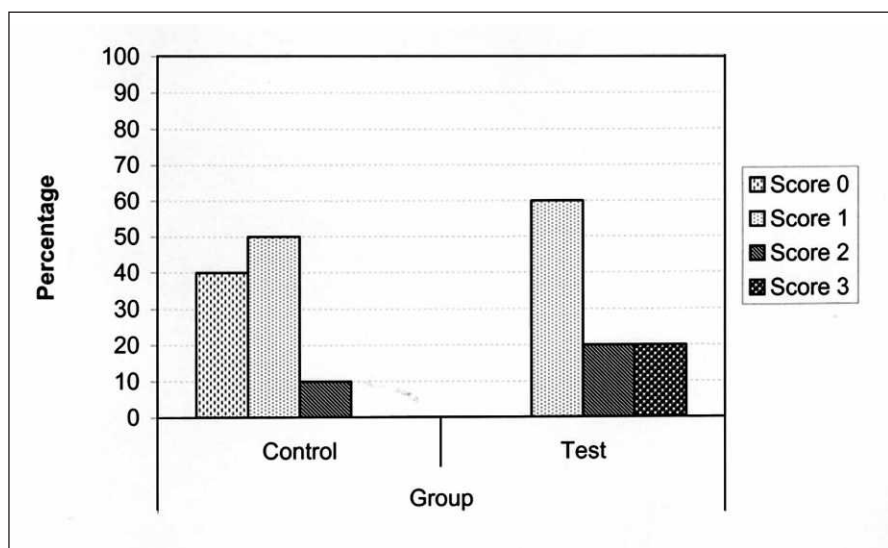


Figure 4. Leakage degree frequency distribution by group in enamel.

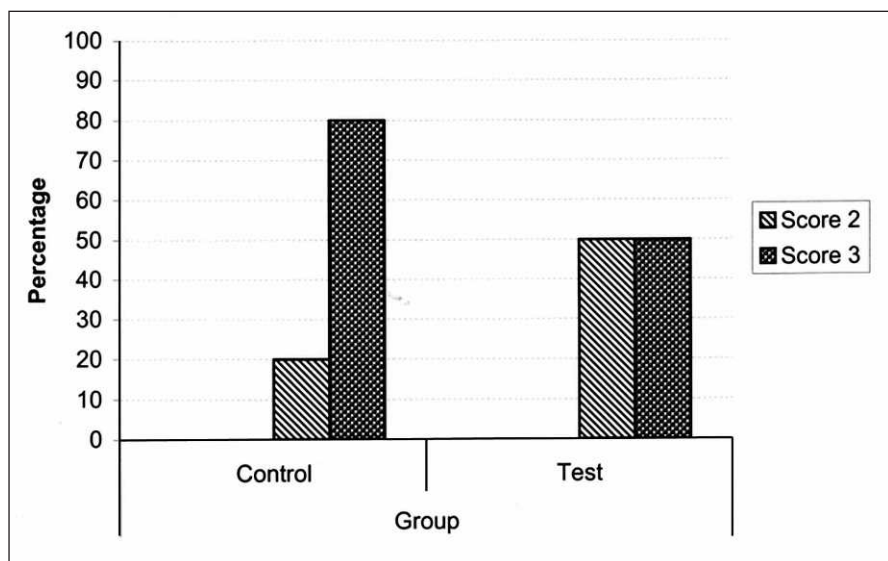


Figure 5. Leakage degree frequency distribution by group in dentin.

occlusal loading had a greater microleakage rate when compared to samples not submitted. Thus, loads with incidence in the occlusal surface of samples may promote a cusp deflection, producing stress next to the margins, thus causing an opening in the tooth/filling interface facilitating, dye leakage and, increasing microleakage. When adhesion behavior of the set tooth/filling is analyzed, it can clearly be noticed that, depending on the strength, intensity and duration, this bonding might break off, facilitating the occurrence of still more microleakage.

### CONCLUSIONS

It is concluded that fillings with cervical margins in cement/dentin had a greater microleakage rate than

those in enamel. When samples were submitted to occlusal loading, there was an increase in the marginal microleakage rate both in the enamel as cement/dentin

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### References

- Abdalla AI & Davidson CL (1993) Comparison of the marginal integrity of *in vivo* and *in vitro* Class II composite restorations *Journal of Dentistry* **21**(3) 158-162.
- Barnes DM, Blank LW, Thompson VP, Holston AM & Gingell JC (1991) A 5- and 8-year clinical evaluation of a posterior composite resin *Quintessence International* **22**(2) 143-151.
- Bouschlicher MR, Vargas MA & Boyer DB (1997) Effect of composite type, light intensity, configuration factor and laser polymerization on polymerization contraction forces *American Journal of Dentistry* **10**(2) 88-96.
- Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34**(6) 849-853.
- Carvalho RM et al (1996) A review of polymerization contraction: The influence stress development versus stress relief *Operative Dentistry* **21**(1) 17-24.
- Croll TP (1992) Glass ionomers and esthetic dentistry: What the new proprieties mean to dentistry *Journal of the American Dental Association* **123**(5) 51-54.
- Davidson CL & Abdalla AI (1994) Effect of occlusal load cycling on the marginal integrity of adhesive Class V restorations *American Journal of Dentistry* **7**(2) 111-114.
- Davidson CL & de Gee AJ (1984) Relaxation of polymerization contraction stresses by flow in dental composites *Journal of Dental Research* **63**(2) 146-148.
- Duncalf WV & Wilson NH (2001) Marginal adaptation of amalgam and resin composite restoration in Class II conservative preparations *Quintessence International* **32**(5) 391-395.
- Feilzer AJ et al (1955) Influence of light intensity on polymerization shrinkage and integrity restoration-cavity interface *European Journal of Oral Science* **103**(5) 322-326.
- Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66**(11) 1636-1639.
- Francci C, Perdigão J, Cardoso PE, Meira JB & Nunes MF (1999) The effect of composite resin, adhesive system and low-viscosity liner on microleakage *Journal of Dental Research* **78** (IADR Abstract #2263) p 388.

- Goracci G, Mori G & Casa De Martinis L (1996) Curing light intensity and marginal leakage of resin composite restorations *Quintessence International* **27**(5) 355-362.
- Gwinnett AJ (1973) Human prismless enamel and its influence on sealant penetration *Archives of Oral Biology* **18**(3) 441-444.
- Hansen EK & Asmussen E (1993) Correlation between depth of cure and surface hardness of a light-activated resin *Journal of Dental Research* **101**(1) 62-64.
- Koran P & Kurschner R (1998) Effect of sequential versus continuous irradiation of a light-cured resin composite on shrinkage, viscosity, adhesion, and degree of polymerizations *American Journal of Dentistry* **11**(1) 17-22.
- Leinfelder KF (1995) Posterior composite resins: The materials and their clinical performance *Journal of the American Dental Association* **126**(5) 663-676.
- Lundin SA & Noren JG (1991) Marginal leakage in occlusally loaded, etched, Class II composite resin restorations *Acta Odontologica Scandinavica* **49**(4) 247-254.
- Lutz F, Krejci I, Luescher B & Oldenburg TR (1986) Improved proximal margin adaptation of Class II composite resin restorations by use of light-reflecting wedges *Quintessence International* **17**(10) 659-664.
- Mandras RS, Retief DH & Russel CM (1991) The effects of thermal and occlusal stresses on the microleakage of the Scotchbond 2 dentinal bonding system *Dental Materials* **7**(1) 63-67.
- Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials Research* **16**(3) 265-273.
- Peutzfeldt A (1994) Correlation between recordings obtained with a light-intensity tester and degree of conversion of a light-curing resin *Scandinavian Journal of Dental Research* **102**(1) 73-75.
- Raadal M (1979) Microleakage around preventive composite fillings in loaded teeth *Scandinavian Journal of Dental Research* **87**(5) 390-394.
- Rueggeberg FA, Caughman WF & Curtis JW (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19**(1) 26-32.
- Rueggeberg FA & Jordan DM (1993) Effect of light-tip distance on polymerization of resin composite *International Journal of Prosthodontics* **6**(4) 364-370.
- Sakaguchi RL, Douglas WH & Peters MC (1992) Curing light performance and polymerization of composite restorative materials *Journal of Dentistry* **20**(3) 183-188.
- Takamizu M, Moore BK, Setcos JC & Phillips RW (1988) Efficacy of visible-light generators with changes in voltage *Operative Dentistry* **13**(4) 173-180.
- Taylor MJ & Lynch E (1992) Microleakage *Journal of Dentistry* **20**(1) 3-10.
- Tung FF, Estafan D & Scherer W (2000) Microleakage of a condensable resin composite An *in vitro* investigation *Quintessence International* **31**(6) 430-434.
- Wendt SL, McInnes PM & Dickinson GL (1992) The effect of thermocycling in microleakage analysis *Dental Materials* **8**(3) 181-184.
- Yearn JA (1985) Factors affecting cure of visible light activated composites *International Dental Journal* **35**(3) 218-225.



# Microtensile Dentin Bond Strength of Self-Etching Resins: Effect of a Hydrophobic Layer

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## Clinical Relevance

In resin composite restorations where maximum dentin adhesion is desirable, bonding of single component self-etching adhesives would likely be improved through the addition of a layer of a more hydrophobic adhesive.

## SUMMARY

In this study, the microtensile bond strength of resin composites to dentin was determined when hydrophilic self-etching resins were used with and without an additional layer of a more hydrophobic adhesive. Included were three single-step self-etching adhesives, Adper Prompt L-Pop (3M ESPE), iBond GI (Heraeus Kulzer, Inc) and Xeno III (Caulk/Dentsply), and as a negative control,

UniFil Bond (GC America), a self-etching primer with a separate adhesive. Each product was evaluated using a hybrid resin composite from its respective manufacturer, and each was used as directed and then used with an added layer of a more hydrophobic resin from its respective manufacturer. Testing was performed after 72 hours using a “non-trimming” microtensile test at a crosshead speed of 0.6 mm/minute. When the products were used according to manufacturers’ directions, iBond had a significantly higher bond strength to dentin than the other three products ( $p<0.001$ ), which were not significantly different from each other. For the three self-etching adhesive systems, the addition of a layer of a more hydrophobic resin produced significantly higher bond strengths to dentin ( $p<0.001$ ), while no significant effect was found for the self-etching primer ( $p=0.40$ ). A significant interaction was found between the variables product and adhesive treatment. The TEM evaluation of Prompt L-Pop and iBond demonstrated reduced nanoleakage with the additional resin layer.

## INTRODUCTION

The adhesion of resin to dentin became feasible with the advent of hydrophilic resins capable of infiltrating into and polymerizing within a moist decalcified

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dentinal surface to form a hybrid layer (Nakabayashi, Ashizawa & Nakamura, 1992). Dentin-adhesive resins were originally formulated with separate etchants, primers and adhesives but have evolved into products in which the adhesive and primer, or etchant and primer, or, in some cases, all three have been combined (Van Meerbeek & others, 2003). Self-etching resins with a separate adhesive are usually termed “self-etching primers” or “self-etching adhesives” when etchant, primer and adhesive are combined. Although driven by convenience, this combination of components appears to reduce dentin bond strengths relative to earlier formulations (Bouillaguet & others, 2001; Inoue & others, 2001; Molla, Park & Haller, 2002).

Throughout this evolution, resin monomers have also become more hydrophilic, which likely hastens hydrolytic degradation of resins in the hybrid layer (Tay & others, 2002b; Tay & others, 2003). Such degradation is also evident in studies that show greater nanoleakage (Tay & others, 2002a; Tay, Pashley & Yoshiyama, 2002c; Tay & Pashley, 2003b) and more rapid degradation in dentin bond strength over time (Armstrong & others, 2003) than is evident in previous systems in which a more hydrophobic layer is applied. Such evidence has led to the proposal that current self-etching adhesives are too hydrophilic and would perform better if covered by a layer of more hydrophobic resin (Tay & Pashley, 2003a).

This study determined whether the addition of a layer of more hydrophobic resin, comparable to the adhesive component of earlier systems, would increase the dentin bond strength of three self-etching adhesive systems and a self-etching primer system, which was added as a negative control. Microtensile bond strength testing is considered the most credible method of evaluating dentin adhesion, because the small size of the specimens provides optimal stress distribution at the resin/dentin interface and allows testing of different regions of the dentin, (Pashley & others, 1999; Shono & others, 1999) and was chosen for this study.

## METHODS AND MATERIALS

Extracted teeth were collected in accordance with human subjects' regulations at the Medical College of Georgia. Twenty unerupted third molars stored in water saturated with thymol at 4°C were used within three months of extraction. The occlusal enamel of each tooth was removed, then each tooth was hemisected facio-lingually using a slow-speed water-cooled saw equipped with a diamond-impregnated disk (Isomet, Buehler Ltd, Lake Bluff, IL, USA). A 320-grit silicon carbide paper disk was used under running water at 60 rpm on a polishing machine (Ecomet 3, Buehler, Ltd), followed by hand finishing for 30 seconds on wet 600-grit silicon carbide paper, to create a realistic smear layer on the surface of the occlusal mid-coronal dentin.

Four commercial self-etching resin systems were selected for the study, Adper Prompt L-Pop (3M ESPE, St Paul, MN, USA), iBond GI (Heraeus Kulzer, Inc, Armonk, NY, USA), Xeno III (Caulk/Dentsply, Milford, DE, USA) and UniFil Bond (GC America, Inc, Alsip, IL, USA). The first three are self-etching adhesives, while the last, added as a negative control, is a self-etching primer to which a layer of adhesive is applied. Half of each hemisected tooth was randomly assigned, alternating mesial and distal, to each of the four adhesive systems. For these samples, each adhesive was applied according to manufacturer's instructions, which are listed in Table 1. The other half of each tooth was assigned to the same adhesive, which was again applied as directed, except that an additional layer of a more hydrophobic unfilled resin from the same manufacturer was applied and light-cured before the addition of the appropriate resin composite restorative material: ScotchBond Multi-Purpose Plus Adhesive (3M ESPE), Gluma Solid Bond S (Heraeus Kulzer), Xeno III Part B (Caulk/Dentsply) or UniFil Bonding Agent (GC America). For Xeno III, this entailed an application of the more hydrophobic of the two liquids, which is usually mixed, and for UniFil, an additional application of adhesive. Sample size was five half-teeth for each adhesive treatment.

Following application of the adhesives by the eight methods listed, 6-mm thick cores of one of the same manufacturer's hybrid resin composites were built incrementally on the occlusal dentin surface of each hemisection. The resin composites were Z 250 (3M ESPE), Venus (Heraeus Kulzer), TPH Spektrum (Caulk/Dentsply) and Gradia Direct (GC America). Increment thickness was limited to 2 mm, and curing was accomplished from all directions using a fast halogen light source (VIP, BISCO, Inc, Schaumburg, IL, USA), for a total of five minutes' curing per specimen. Using the unit's built-in radiometer, light output was verified to be 600 mW/cm<sup>2</sup> throughout the study.

After storage in distilled water at 37°C for 24 hours, the restored teeth were sectioned occluso-gingivally into serial slabs approximately 0.9-mm thick using the same slow-speed water-cooled diamond saw. Each slab was then sectioned by the same method into resin composite and dentin beams approximately 0.9 x 0.9 mm in cross section, according to the “non-trimming” version of the microtensile test (Shono & others, 1999). Each restored half-tooth yielded 5-12 beams for bond strength evaluation. Occasional beams with premature failure during sectioning were excluded from compilation of the mean tensile bond strength but were recorded in the results of the study. After 48 hours of storage in distilled water at 37°C, the dimensions of each beam were measured with a digital caliper (Absolute Digimatic, Model CD 6" CS, Mitutoyo Corp, Kanagawa, Japan), accurate to  $\pm 5 \mu\text{m}$ . The beams were then

Table 1: <i>Manufacturer, Instructions/Technique, Composition and pH for Each Adhesive</i>			
Adhesive	Technique	Composition	pH
Adper Prompt L-Pop (PLP) Lot #149354	Mix using unit-dosed blister pack Apply with agitation, 15 seconds* Air dry, light cure	Methacrylated Phosphoric Acid Esters, Water BisPhenol A Diglycidyl Ether Dimethacrylate, 2-Hydroxyethyl Methacrylate	0.8
ScotchBond Multi-Purpose Plus Adhesive Lot #3AH/3NG	(Experimental 2 <sup>nd</sup> coating)	BisPhenol A Diglycidyl Ether Dimethacrylate, 2-Hydroxyethyl Methacrylate	
Filtek Z 250 Lot #20030509 (3M ESPE, St Paul, MN, USA)	(Resin composite restorative)		
iBond GI (IB) Lot #010042	Apply three coats with agitation, 30 seconds Air dry, light cure	4-Meta, Urethane Dimethacrylate, Glutar-aldehyde, Acetone, Water	1.6
Gluma Solid Bond S Lot #030041	(Experimental 2 <sup>nd</sup> coating)	Bis-GMA, TEGDMA, 2-Hydroxyethyl Methacrylate, carboxylic acid	
Venus Lot #180026 (Heraeus Kulzer, Armonk, NY, USA)	(Resin composite restorative)		
Xeno III (X) Lot #0303001499	Mix, apply thick coating, 30 seconds Air dry, light cure	Hydroxyethyl Methacrylate, water, ethanol, 2,6-Di-tert-butyl-p hydroxy toluene (Liquid A)	1.0
Xeno III Liquid B	(Experimental 2 <sup>nd</sup> coating)	Pyro-EMA-SK, PEM-F, Urethane Dimethacrylate, EPD, p-diethyl amine ethyl benzoate	
TPH Spektrum Lot #0301281 (Caulk/Dentsply, Milford, DE, USA)	(Resin composite restorative)		
UniFil Bond (UB) Lot #0204121	Apply primer, 20 seconds, air dry Apply bonding agent, light cure	Hydroxyethyl Methacrylate, Ethanol, 4-Methacryloxyethyltrimellitate (Primer)	2.0
UniFil Bonding Agent	(Experimental 2 <sup>nd</sup> coating)	Urethane Dimethacrylate, 2-Hydroxyethyl Methacrylate (Bonding Agent)	
Gradia Direct Lot #0305151 (GC America, Inc, Alsip, IL, USA)	(Resin composite restorative)		
*Subsequent to study, manufacturer increased recommendation to two coats.			

affixed to a Ciucchi device (Kuraray Co, Ltd, Osaka, Japan), using Zapit cyanoacrylate glue (Dental Ventures of America, Corona, CA, USA) and tested to failure under tension in a universal testing machine (Vitrodyne V1000; Chatillon, Largo, FL, USA) at a crosshead speed of 0.6 mm/minute. The type of failure was observed at 2.5x magnification and categorized as adhesive, cohesive or mixed.

In addition to microtensile testing, several teeth were bonded with Adper Prompt L-Pop and iBond, both alone and with additional layers of the same resins listed above. After light curing, the adhesive layer of these specimens was covered with a layer of Epic-TMPT (Parkell, Inc, Farmingdale, NY, USA), a resin com-

posite that can be cut with a diamond knife. These teeth were divided into beams that were immersed in 50 wt% ammoniacal silver nitrate for 24 hours to measure nanoleakage along the bonded interfaces. After rinsing, the specimens were exposed to photodeveloper and light to reduce silver nitrate to metallic silver. The beams were then dehydrated through ascending alcohols, embedded in epoxy resin, then cut into 70-90 nm-thick sections according to the protocol of Tay and others (2001). These were examined without staining in a Phillips TEM (Model CM20, Eindhoven, The Netherlands) operated at 120 kV.

Bond strength was calculated and data obtained for the eight subgroups were statistically analyzed using a



two-factor nested repeated measures ANOVA for the factors adhesive treatment and product at a confidence level of 5%. A Bonferroni adjustment was used in which the overall alpha level was divided by the number of pairwise comparisons so that, for all post-hoc tests, *p* was less than 0.0032 for differences considered to be significant.

RESULTS

When all the systems were used according to manufacturers’ directions, iBond had a significantly higher bond strength to dentin than the other three (*p*<0.001),

which were not significantly different from each other. For the three self-etching adhesives, the addition of one more layer of a more hydrophobic resin produced significantly higher bond strengths to dentin (*p*<0.001). For the self-etching primer, which was the negative control, no significant effect on bond strength was found (*p*=0.40). A significant interaction was found between the variables of product and adhesive treatment. Few cohesive and no mixed failures were observed. Complete microtensile bond strength results are presented in Table 2.

Table 2: Mean Microtensile Bond Strength to Dentin of Four Adhesive Systems, MPa (SD), Number of Cohesive Failures in Dentin, Beams Broken During Specimen Preparation				
Adhesive	n (from 5 teeth)	µTBS (SD)	Cohesive**	Broken
IB	51	41.0 (17.7)a	0	0
IB+	47	53.5 (19.0)*	3	0
PLP	33	16.4 (9.9)b	0	2
PLP+	43	56.4 (21.8)*	1	0
X	38	10.3 (8.3)b	2	3
X+	37	25.8 (8.0)*	2	2
UB	42	15.8 (6.8)b	0	0
UB+	40	18.0 (7.6)	2	0

\*Significantly higher than same product without added layer.  
\*\*All other failures adhesive.  
(PLP = Adper Prompt L-Pop; IB = iBond; X = Xeno III; UB = UniFil Bond)  
(+ = with experimental second layer)  
Groups assigned different letters are significantly different.

In the dentin-adhesive interfaces evaluated by TEM, Adper Prompt L-Pop demonstrated hybrid layers about 3-µm thick, while the adhesive layer separating the hybrid layer from the overlying resin composite was only 2-µm thick (Figure 1A). Large reticular silver deposits were seen in the hybrid layer, while the silver uptake in the adhesive layer was limited to tiny (10-20 nm) diameter spot-like deposits distributed throughout the adhesive. When the additional layer of Scotchbond Multi-Purpose adhesive was added, the total adhesive layer thickness

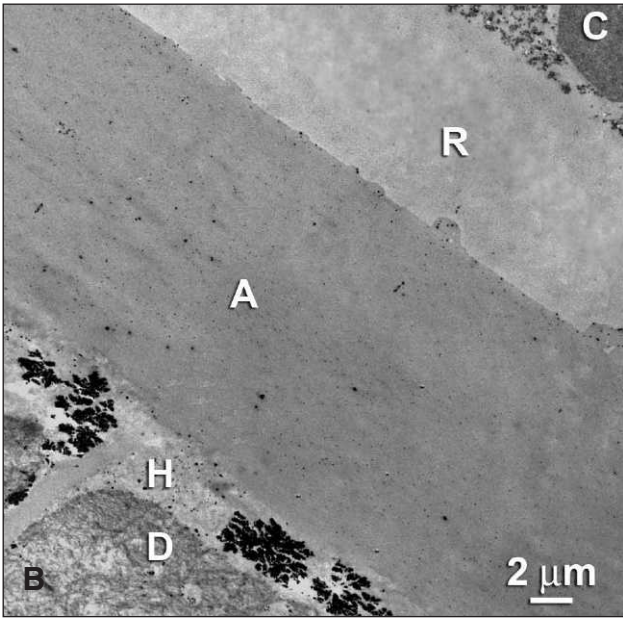
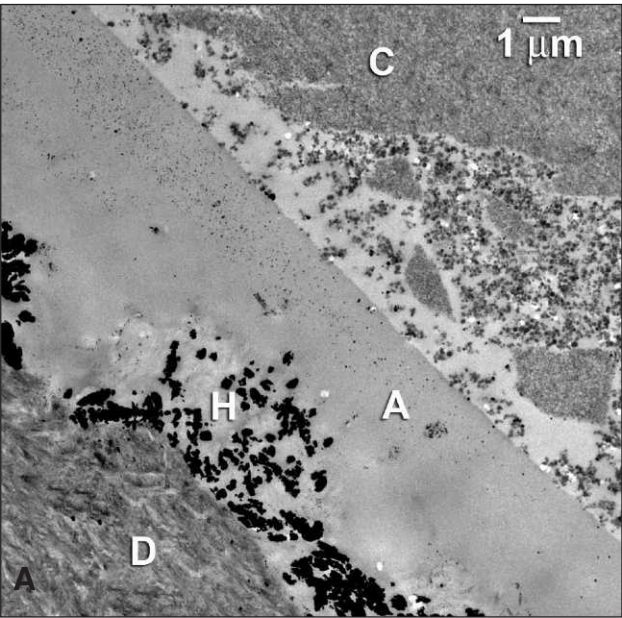


Figure 1: Unstained, transmission electron micrographs of human coronal dentin bonded with Prompt L-Pop. Figure 1A. Prompt L-Pop alone produced a 2-3 µm-thick hybrid layer (H) covered by a 2-3 µm-thick adhesive layer (A) that, in turn, was covered with composite (C). A moderate amount of silver was taken up into the hybrid layer in a heavy reticular pattern. Much less silver was taken up by the adhesive (A) where it was distributed in tiny spot-like deposits (10,000x). Figure 1B. After bonding with Prompt L-Pop, the bonded surface was sealed with an additional layer of Scotchbond Multi-Purpose (R). This resulted in less silver uptake by both the hybrid layer (H) and the adhesive layer (5000x).

increased but the degree of nanoleakage decreased, both in the hybrid layer and in the adhesive layer (Figure 1B). The Prompt L-Pop adhesive was more electron dense than the Scotchbond Multi-Purpose adhesive, making the junction between them very distinct (Figure 1B).

In specimens bonded with iBond only, the hybrid layer was only about 1- $\mu\text{m}$  thick (Figure 2A). Periodically, along the bonded interface, significant (ca 3-4  $\mu\text{m}$  diameter) accumulations of silver associated with the underlying dentinal tubules were seen extending into the adhesive from the hybrid layer. Few spot-like silver deposits were seen in the overlying adhesive layer (Figure 2A). In contrast, when the Gluma Solid Bond S additional layer was applied, the amount of silver uptake into the hybrid layer was reduced. Much less interfacial silver uptake was seen between the hybrid layer and the overlying adhesive layer, or between the adhesive layer and the overlying thick layer of Gluma Solid Bond S (Figure 2B).

### DISCUSSION

The authors elected to pair only adhesives and resin composites from the same manufacturer for each group of specimens, because this would be the most likely way resins would be purchased by clinicians. This also eliminated the risk of any incompatibility between any adhesive and a single resin composite, but dictated that the study was an evaluation of the adhesive systems, rather than of the adhesives themselves.

There are several possible explanations for the increased microtensile dentin bond strength observed for three of the products in this study when an additional layer of a more hydrophobic resin was applied. The additional layer of hydrophobic resin seems to limit diffusion of water through the hybrid layer to the interface between the adhesive and resin composite (Figures 1B and 2B), which could otherwise have occurred rapidly (Tay & others, 2002b; Tay & Pashley, 2003a). That could, in turn, inhibit polymerization (Jacobsen & Söderholm, 1995) and thereby weaken the adhesive/resin composite interface.

The additional layer may also have slowed the extraction of unpolymerized monomers or oligomers from the hybrid layer. Zones of poorly polymerized hydrophilic phases that permit water movement have been demonstrated within the hybrid layers and self-etching adhesives (Tay & others, 2002c). Although the 72-hour storage time in this study is relatively short, the small specimen size over the final 48 hours of storage could have accelerated aging of the bonds and allowed maximum water sorption. In both of the above scenarios, the additional adhesive layer would cause the self-etching adhesives to emulate earlier multiple-bottle systems. The additional layer of resin increased the thickness of the adhesive layer (Figures 1B and 2B), which is known to reduce polymerization stresses (Choi, Condon & Ferracane, 2000) and may improve stress distribution during testing. Both may have contributed to the higher bond strengths that were observed.

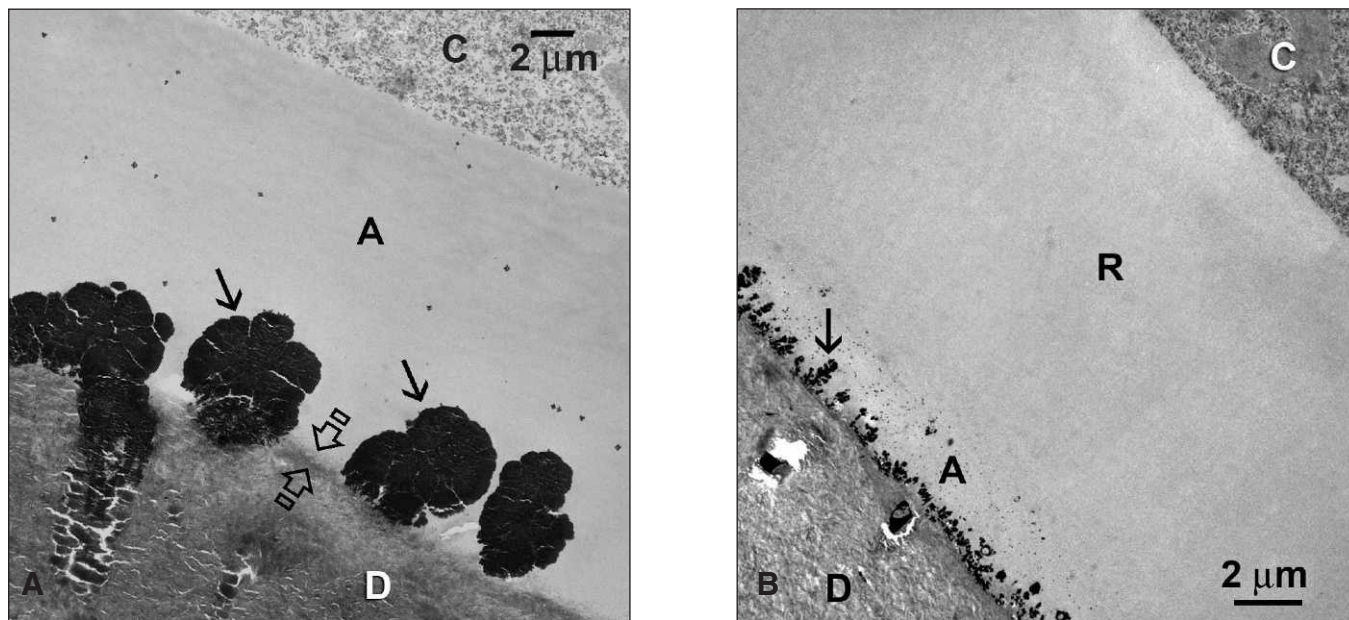


Figure 2: Unstained transmission electron micrographs of iBond treated specimens. Figure 2A. Three coats of iBond alone produced a 1  $\mu\text{m}$ -thick hybrid layer (H) covered by a 17-18  $\mu\text{m}$ -thick adhesive layer (A). Large amounts of silver traced-water accumulation at the interface between the adhesive and the hybrid layer (arrows). These spherical accumulations of silver (4-5  $\mu\text{m}$  in diameter) extended into the adhesive layer (A), which showed little silver uptake (5000X). Figure 2B. When the iBond adhesive layer (A) was covered by Gluma Solid Bond S, there was much less spherical silver uptake into the hybrid layer (arrows). No silver uptake was seen in the Gluma Solid Bond S (R).



Finally, the low pH of the self-etching adhesives may persist after polymerization and be sufficient to inhibit polymerization of the resin composite (Suh & others, 2003). This effect has been demonstrated when polymerization is delayed for 2.5 or more minutes and has been attributed to acidic monomers neutralizing tertiary amine initiators within the restorative material (Tay & others, 2001). The extent to which this effect would manifest in the estimated one-minute delay produced by sculpting the initial layer of resin composite in this study is uncertain and likely is product-specific. In this case, the addition of another layer of adhesive would improve adhesion, because the layer is polymerized immediately.

The relatively high degree of nanoleakage observed for iBond did not appear to influence the 72-hour bond strengths evaluated in this study, but future studies should address whether the incidence of these water-rich phases influences the long-term stability of the adhesive bond. The single-component design of this product may promote a hybrid layer thinner than that of a product such as Prompt L-Pop, which is mixed immediately prior to application.

Although its bond strength may be limited by its relatively high pH, an additional resin layer did not likely benefit UniFil Bond, the negative control in this study, because it is a self-etching primer and such a layer is already in place when the product is used as directed. The results of this study appear to support this approach to self-etching adhesive systems. All self-etching resins, no matter how they are labeled, are probably most effective as primers.

## CONCLUSIONS

The addition of a more hydrophobic resin layer significantly improved the bond strengths of single component self-etching adhesive resin systems.

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## References

- Armstrong SR, Vargas MA, Fang Q & Laffoon JE (2003) Microtensile bond strength of a total-etch 3-step, total-etch 2-step, self-etch 2-step, and a self-etch 1-step dentin bonding system through 15-month water storage *Journal of Adhesive Dentistry* **5**(1) 47-56.
- Bouillaguet S, Gysi P, Wataha JC, Ciucchi B, Cattani M, Godin C & Meyer JM (2001) Bond strength of composite to dentin using conventional, one-step, and self-etching adhesive systems *Journal of Dentistry* **29**(1) 55-61.
- Choi KK, Condon JR & Ferracane JL (2000) The effects of adhesive thickness on polymerization contraction stress of composite *Journal of Dental Research* **79**(3) 812-817.
- Inoue S, Vargas MA, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, Sano H & Van Meerbeek B (2001) Microtensile bond strength of eleven contemporary adhesives to dentin *Journal of Adhesive Dentistry* **3**(3) 237-245.
- Jacobsen T & Söderholm KJ (1995) Some effects of water on dentin bonding *Dental Materials* **11**(2) 132-136.
- Molla K, Park HJ & Haller B (2002) Bond strength of adhesive/composite combinations to dentin involving total- and self-etch adhesives *Journal of Adhesive Dentistry* **4**(3) 171-180.
- Nakabayashi N, Ashizawa M & Nakamura M (1992) Identification of a resin-dentin hybrid layer in vital human dentin created *in vivo*: Durable bonding to vital dentin *Quintessence International* **23**(2) 135-141.
- Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA & Tay F (1999) The microtensile bond test: A review *Journal of Adhesive Dentistry* **1**(4) 299-309.
- Shono Y, Ogawa T, Terashita M, Carvalho RM, Pashley EL & Pashley DH (1999) Regional measurement of resin-dentin bonding as an array *Journal of Dental Research* **78**(2) 699-705.
- Suh BI, Feng L, Pashley DH & Tay FR (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part III. Effect of acidic resin monomers *Journal of Adhesive Dentistry* **5**(4) 267-282.
- Tay FR, Hashimoto M, Pashley DH, Peters MC, Lai SC, Yiu CK & Cheong C (2003) Aging affects two modes of nanoleakage expression in bonded dentin *Journal of Dental Research* **82**(7) 537-541.
- Tay FR, King NM, Chan KM & Pashley DH (2002a) How can nanoleakage occur in self-etching adhesive systems that demineralize and infiltrate simultaneously? *Journal of Adhesive Dentistry* **4**(4) 255-269.
- Tay FR, King NM, Suh BI & Pashley DH (2001) Effect of delayed activation of light-cured resin composites on bonding of all-in-one adhesives *Journal of Adhesive Dentistry* **3**(3) 207-225.
- Tay FR & Pashley DH (2003a) Have dentin adhesives become too hydrophilic? *Journal of the Canadian Dental Association* **69**(11) 726-731.
- Tay FR & Pashley DH (2003b) Water treeing—a potential mechanism for degradation of dentin adhesives *American Journal of Dentistry* **16**(1) 6-12.
- Tay FR, Pashley DH, Suh BI, Carvalho RM & Itthagarun A (2002b) Single-step adhesives are permeable membranes *Journal of Dentistry* **30**(7-8) 371-382.
- Tay FR, Pashley DH & Yoshiyama M (2002c) Two modes of nanoleakage expression in single-step adhesives *Journal of Dental Research* **81**(7) 472-476.
- Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P & Vanherle G (2003) Buonocore memorial lecture. Adhesion to enamel and dentin: Current status and future challenges *Operative Dentistry* **28**(3) 215-235.



# Assessing the Tooth-Restoration Interface Wear Resistance of Two Cementation Techniques: Effect of a Surface Sealant

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R Cilli • RFL Mondelli

## Clinical Relevance

The “resin coating” technique did not provide an increase in tooth-restoration interface width or wear after the toothbrushing abrasion test when compared to the conventional cementation technique. The application of a restoration surface sealant improved interface wear resistance for both cementation techniques.

## SUMMARY

**This study compared (1) the tooth-restoration interface width of conventional and “resin coating” cementation techniques, (2) the toothbrushing wear resistance of the two interfaces and (3) this study evaluated the influence of a restoration surface sealing on toothbrush wear**

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resistance on both cementation technique interfaces. Mid-coronal buccal surfaces of 40 bovine teeth were ground to obtain a flat enamel surface. For each specimen, a 3 mm x 4 mm x 3 mm dimension rectangular cavity was prepared. The teeth were divided into four groups. Two groups (RC) received a “resin coating” (ED Primer + Tetric Flow) prior to cementation. The remaining two groups (NC) served as non-coated groups. All teeth were restored with composite inlays (Z250) fabricated by the indirect method and were cemented with dual cure resin cement (Panavia F). After finishing the margins, one group from each of the cementation techniques (RC+S and NC+S) had the tooth-restoration interface protected with a restoration surface sealant (Biscover). The specimens were subjected to 100,000 brushing abrasion cycles. The tooth-restoration width was obtained using a Hommel Tester T 1000—basic profilometer and Turbo Datawin NT 1.34 Software ( $\mu\text{m}$ ). The interface wear (vertical loss/ $\mu\text{m}$  and area/ $\mu\text{m}^2$ ) was calculated with Image Tool 3.0 Software. Data were analyzed with Student *t*-test, one-way analysis of variance and Tukey test ( $\alpha=0.05$ ). Mean interface width for the NC group was 67  $\mu\text{m}$  and 72  $\mu\text{m}$  for

the RC group. The student *t*-test showed no significant differences between groups ( $p=0.53$ ). ANOVA showed significant differences ( $p<0.01$ ) in vertical loss among groups (NC: 49.30  $\mu\text{m}$ ; NC+S: 7.90  $\mu\text{m}$ ; RC: 27.15  $\mu\text{m}$ ; RC+S: 4.74  $\mu\text{m}$ ). Also, ANOVA showed significant differences ( $p<0.01$ ) in worn areas among groups (NC: 2,008  $\mu\text{m}^2$ ; NC+S: 128  $\mu\text{m}^2$ ; RC: 1,580  $\mu\text{m}^2$  and RC+S: 88  $\mu\text{m}^2$ ). No differences were found in tooth-restoration interface width and worn area between conventional and "resin coating" techniques. "Resin coating" interface presented reduced vertical loss. Restoration surface sealing provided reduced wear in tooth-restoration interface for both techniques.

### INTRODUCTION

Applications of resin luting agents have increased considerably in recent years (el-Mowafy, 2001) because of their superior mechanical properties and increased retentive capability (White & Yu, 1993; el-Mowafy & Milenkovic, 1994). Nevertheless, there are still some limitations concerning the clinical behavior of these materials and researchers are eager to overcome them. They can be associated with initial post-cementation sensitivity (Sjogren & others, 1992), polymerization shrinkage (Peumans & others, 2000), wear and its linear correlation with interface width (Guzman, Moore & Andres, 1997) and resin composite matrix dissolution (Roulet & Walti, 1984).

Recently, a "resin coating" cementation technique was developed in which a hybrid layer and tight sealing film are produced with a dentin adhesive agent and a low viscosity microfilled resin. This technique is eminent for the potential to minimize pulp irritation and post-operative sensitivity and enhance resin cement bond strength (Nikaido & others, 2003). Although there are claims that fitting problems do not occur as impressions are taken after coating the cavity, it is still unclear whether this technique is prone to an increase in tooth-restoration interface.

With regard to the wear of composite restoratives, even after performing the appropriate finishing and polishing techniques, the surface exhibits micro irregularities that inherently lead to material wear, deterioration and marginal leakage resulting mainly from the abrasive processes the restoration is subjected to in the oral environment (Takeuchi & others, 2003). Clinically, restoration wear may result from centric and functional contacts, attrition of food bolus, interproximal contact areas and toothbrushing through the mechanical action of the toothbrush and dentifrice (Kanter, Koski & Martin, 1982; Goldstein & Lerner, 1991; Buchalla, Attin & Hellwig, 2000).

During service, restorative material wear can result in loss of contour, increase in surface roughness, staining (Hachiya & others, 1984) and plaque retention, which can lead to an increased risk of both caries and periodontal inflammation (Bollen, Lambrechts & Quirynen, 1997). Considering that in any indirect restoration the weak link is at the tooth-restoration interface (Peumans & others, 2000), these are undesirable characteristics, since they can affect the longevity and esthetics of indirect restorations.

In an attempt to overcome composite wear problems, the use of a thin layer of low viscosity resin over polymerized composite surfaces has been investigated (Takeuchi & others, 2003; des Santos & others, 2003). The so-called surface penetrating sealant or re-bonding agent should be able, by capillary action, to fill the structural microdefects and microfissures that are formed on composite surfaces. This approach is assumed to provide a more uniform and regular surface, thereby enhancing wear resistance and the ability to penetrate deeply into the interfacial microgaps, providing improved marginal sealing (Takeuchi & others, 2003).

Some studies have supported the idea that the use of rebonding agents improves the mechanical properties of polymerized resin composite restorations (Dickinson & others, 1990; des Santos & others, 2003). However, there is still a lack of studies that report whether the surface integrity of cementation interface may be enhanced by the use of a low viscosity surface sealant. In this context, this study evaluated the width and wear resistance of the tooth-restoration interface obtained by the "conventional" cementation technique and the "resin coating" technique before and after the mechanical toothbrushing abrasion test. Also, the influence of a restoration surface sealing on abrasion wear resistance in both cementation interfaces was assessed.

### METHODS AND MATERIALS

#### Experimental Design

The factors under study were the different cementation techniques and the influence of a restoration surface penetrating sealant, both at two levels. The association between the two factors resulted in four groups. The experimental sample was comprised of 40 specimens ( $n=10$ ) made in random sequence. The quantitative response variable was the width and wear of the cementation interface (vertical loss and area).

#### Materials

The materials used in this study are outlined in Table 1.

#### Specimen Preparation

Forty extracted bovine incisors, stored in a solution of 1% chloramine T at 4°C, were chosen. They were cut in

Table 1: *Material Used*

Materials/Manufacturer	Composition	Batch #
Resin cement Panavia F/Kuraray Medical Inc	Paste A—Quartz glass, micro-filler, MDP, methacrylate, photoinitiator/Paste B—barium glass, NaF, methacrylates, chemical initiator	00120A
Dentin Bonding System ED Primer/Kuraray Medical Inc	Primer A—MDP, HEMA, 5-NMSA, chemical initiator Primer B—5-NMSA, water	00030A
Low viscosity resin Tetric Flow/Ivoclar/Vivadent	Bis-GMA, Urethane dimethacrylate, triethylene glycol dimethacrylate, barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, silicone dioxide	F56810
Resin Composite Z250/3M ESPE	Bis-GMA, Urethane dimethacrylate, Bis-EMA, silica, zircon	2MR
Restoration Surface Sealant Biscover/BISCO	Ethoxylated Bisphenol A, diacrylate, Urethane acrylate ester, polyethyleneglycol diacrylate	30000848

the mid-coronal buccal region into rectangular specimens (15 mm x 5 mm) using a low speed diamond saw under water cooling (South Bay Technology Inc, model 650, San Clemente, CA, USA) and embedded in slow setting epoxy resin (15 mm x 5 mm x 4 mm). A flat enamel surface was produced by wet polishing with 600-grit silicon carbide abrasive papers. Care was taken so that the ground surface consisted entirely of enamel. A rectangular cavity was prepared on the flattened enamel surface of the tooth using a superfine bur (SF 145, Shofu Co Inc, Kyoto, Japan) mounted on a high-speed handpiece under water cooling. The cavity dimensions were approximately 3 mm x 4 mm wide and 3 mm deep. The prepared teeth were randomly divided into the following four groups (n=10):

(NC): Conventional cementation technique

(NC+S): Conventional cementation technique with restoration surface sealing

(RC): Resin coating technique

(RC+S): Resin coating technique with restoration surface sealing

Next, for the specimens in the resin-coating group, the cavity surface was prepared using a self-etching primer bonding system (ED Primer, Kuraray Medical Inc, Okayama, Japan) according to the manufacturer's instructions. The cavities were then coated with a low viscosity microfilled resin (Tetric Flow, Ivoclar/Vivadent, Schaan, Liechtenstein) and polymerized for 20 seconds (Optilux Demetron, VLC 403, Demetron Research Co, Danbury, CT, USA; performing 500mW/cm<sup>2</sup>, verified by a radiometer—Model 100, Demetron).

Cavity impressions were taken with a condensation silicone material (Optosil-Xantopren, Heraeus-Kulzer, Hanau, Germany) and die stone casts were fabricated with type IV stone (Durone IV, Dentsply, Petrópolis, RJ, Brazil). The next day, resin composite inlays (Z250, 3M-ESPE, St Paul, MN, USA) were made indirectly on those die stone replicas. Trial insertion prior to cemen-

tation was performed to ensure a good fit for each inlay. In the cementation procedure, the enamel and dentin cementing surfaces of non-coated teeth were primed with ED primer for 60 seconds and dried. The cavities in the resin-coated teeth were etched with 37% phosphoric acid gel

(3M-ESPE) for 10 seconds, rinsed and dried to remove debris. Primer was again applied to the surface. The fitting surfaces of the inlays were also etched with 37% phosphoric acid gel for 10 seconds and silanized (Angelus, Londrina, Brazil) for 60 seconds according to manufacturer's instructions. Equal amounts of the two dual cured resin cement pastes (Panavia F, Kuraray Medical Inc) were mixed and placed in the cavities, and the inlays were seated and polymerized for 60 seconds in three different locations under a 1 Kg load. The teeth were stored in water at 37°C for 24 hours, then the margins were finished with 600, 1000 and 1200-grit silicon carbide abrasive papers and water, and 1 µm, 0.3 µm and 0.05 µm finishing aluminum oxide pastes (Arotec S/A Ind e Com, Brazil) after which the first analysis of surface profile was made.

Then, Groups NC+S and RC+S had the tooth-restoration interface area etched with 37% phosphoric acid gel for 20 seconds, rinsed with water spray for 40 seconds and dried with an air stream for 10 seconds. A uniform layer of the restoration surface sealant (Biscover, BISCO Inc, Schaumburg, IL, USA) was applied over the etched area with a small, fine brush; gently air-thinned for five seconds and light cured for 15 seconds (VLC 403, Optilux Demetron), after which another analysis of surface profile was made. The specimens were then individually stored in deionized water at 37°C for 24 hours.

### Baseline Surface Profile Measurement

Each specimen was gently dried with absorbent paper and surface profile measurements were taken using the Hommel Tester T 1000—basic profilometer and Turbo Datawin—NT 1.34 Software (Hommelwerke GmbH, Schwenningen, Germany). The tracing parameters were established as Lt (Assessment length): 4.8 mm and Lc (Cut-off): 0.800 mm. For each cementation interface, three perpendicular tracings were performed at three different locations before and after the restoration surface sealant application.



## Brushing Abrasion Test

Subsequently, the specimens were subjected to brushing abrasion in an automatic toothbrushing machine that imparted the reciprocating motion of 10 soft nylon bristle toothbrush heads (Oral-B 40, Osasco, SP). The machine was equipped with a stainless steel base, with 10 independent devices for positioning the specimens (15 mm x 5 mm x 4 mm). Brushing was performed in a thermostatically controlled environment to simulate the temperature in the mouth ( $37 \pm 2^\circ\text{C}$ ). One hundred thousand strokes were performed with abrasive slurry at a speed of 4.5 strokes per second at a load of 300 grams (Mondelli & others, 2003). The slurry consisted of a dentifrice (Colgate Total, SB Campo, SP) and distilled deionized water at a ratio of 2:1 by weight (ISO, 1996), which was independently and simultaneously injected beside each brush head at a frequency of 0.4 ml for each two-minute interval. After brushing abrasion, the specimens were rinsed thoroughly with distilled water, ultrasonically cleaned for 10 minutes and stored in deionized water at  $37^\circ\text{C}$ .

## Final Surface Profile Measurement

Final profile measurements were evaluated as previously described for the baseline condition. The tooth-restoration interface width was determined by the

average of the three measurements obtained for each specimen in Turbo Datawin—NT 1.34 Software (Hommelwerke GmbH, Germany) by positioning vertical bars at the interface borders. The program supplied the distance between bars. Using these profiles as reference, the interface wear was determined by the average of the worn area and by the average of depth (vertical loss) obtained among the three random tracings on the dimensional analysis Image Tool 3.0 Software (UTHSCSA, San Antonio, TX, USA).

## Scanning Electron Microscope Examination

Random samples of each tested group were selected for microscopic examination. The surface of each selected specimen was replicated with additional silicone (Express, 3M ESPE) and epoxy-die (RD-6921, Redelease, SP, Brazil). The replicas were gold sputter-coated (MED 010, Balzers, Liechtenstein) and the surfaces analyzed by scanning electron microscope (LEO 435 VP, England) at 1000x magnification.

## Statistical Analysis

Student's *t*-test was used to compare the cementation interface width between the two techniques, Groups NC and RC ( $\alpha=0.05$ ). The Student's paired *t*-test ( $\alpha=0.05$ ) was used for each group to compare significant wear alterations (vertical loss and worn area) after the

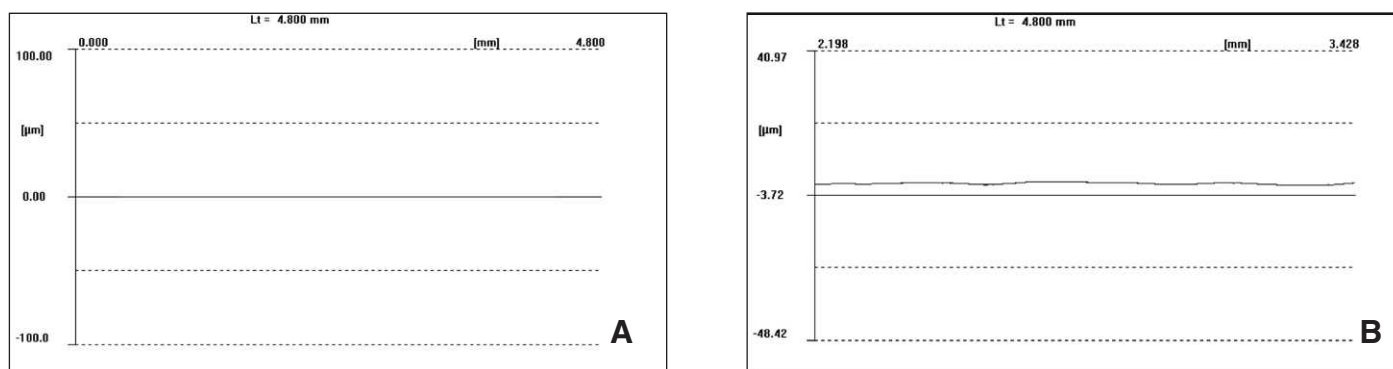


Figure 1. Representative baseline linear profiles on tooth-restoration interfaces. Vertical axis represents the interface wear depth. Horizontal axis represents the tracing across interface. (Figure 1A: before restoration sealing; Figure 1B: after restoration sealing).

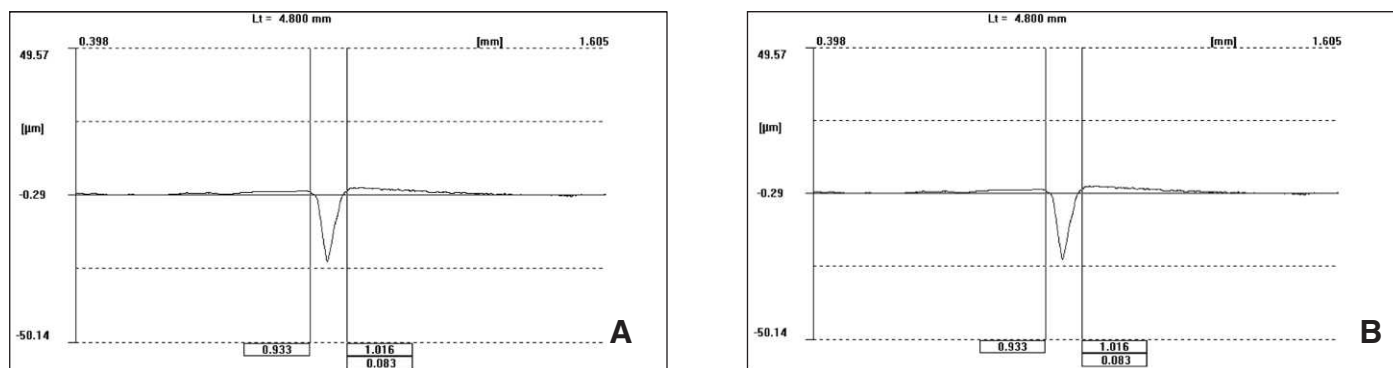


Figure 2. Conventional cementation technique: representative tooth-restoration interface profile after toothbrushing abrasion test. Vertical axis represents the interface wear depth. Horizontal axis represents the tracing across interface. (Figure 2A: unsealed; Figure 2B: sealed).

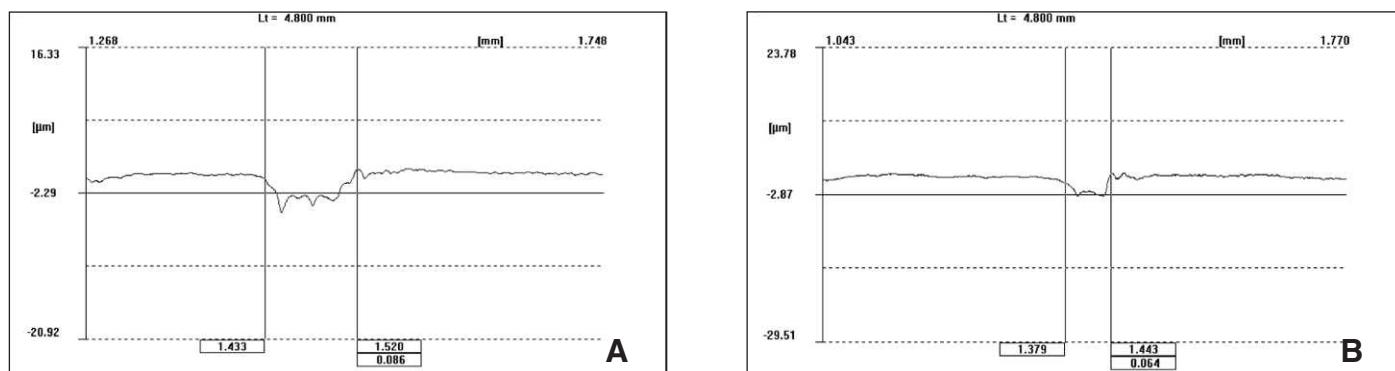


Figure 3. Resin coating technique: representative tooth-restoration interface profile after toothbrushing abrasion test. Vertical axis represents the interface wear depth. Horizontal axis represents the tracing across interface. (Figure 3A: unsealed; Figure 3B: sealed).

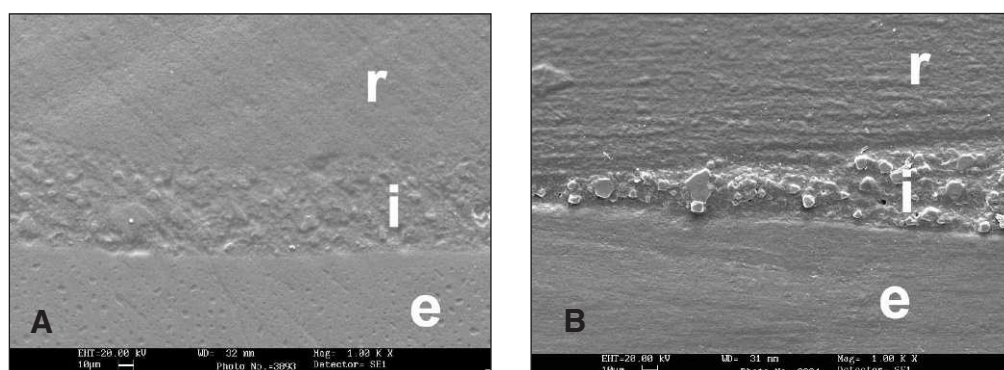


Figure 4. Conventional cementation technique—unsealed specimens/tooth-restoration interface. Enamel surface (e); Tooth-restoration interface (i); Resin composite (r). (Figure 4A: before toothbrushing; Figure 4B: after toothbrushing) (SEM 1000x).

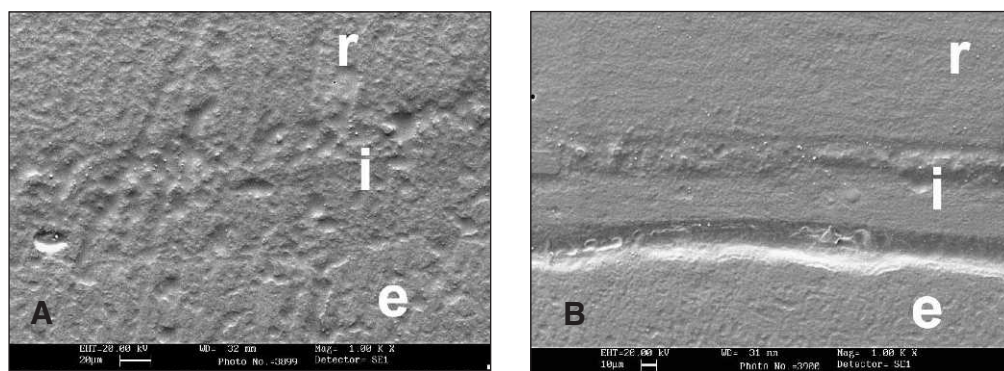


Figure 5. Resin coating cementation technique—unsealed specimens/tooth-restoration interface. Enamel surface (e); Tooth-restoration interface (i); Resin composite (r). (Figure 5A: before toothbrushing; Figure 5B: after toothbrushing) (SEM 1000x).

brushing abrasion test. The Analysis of Variance and Tukey test ( $\alpha=0.05$ ) for vertical loss and worn areas were used to compare differences among studied factors.

## RESULTS

At the baseline tracings, all groups demonstrated the same linear profile pattern with or without restoration surface sealant. Initial wear measurements were considered to be zero (Figure 1). Thus, the Student's paired

*t*-test ( $p<0.01$ ) showed significant wear alterations (vertical loss and area) for all groups (Figures 2 and 3). When the interface width of both cementation techniques was compared (Groups NC and RC), no statistical difference was observed ( $p=0.53$ ). The mean interface width for the conventional technique was  $67 \mu\text{m} (\pm 18)$  and  $72 \mu\text{m} (\pm 24)$  for the resin coating technique (Table 2).

Table 3 shows the mean vertical loss for each group and indicates significant differences among them ( $F=32.20$  and  $p<0.01$ ). Vertical loss for the conventional cementation technique was statistically more pronounced than for the resin coating technique. Both groups that had the cementation interface sealed did not present differences between them, but were statistically more wear resistant than the unsealed groups.

Table 4 shows the worn area mean for each group and also indicates differences among them ( $F=27.90$  and  $p<0.01$ ). No differences were found between the unsealed conventional and resin coating techniques, and no differences were found between the sealed conventional and resin coating techniques, whereas, statistical differences were found between the sealed and unsealed groups.

Scanning electron micrographs of the conventional and resin coating interfaces before and after the

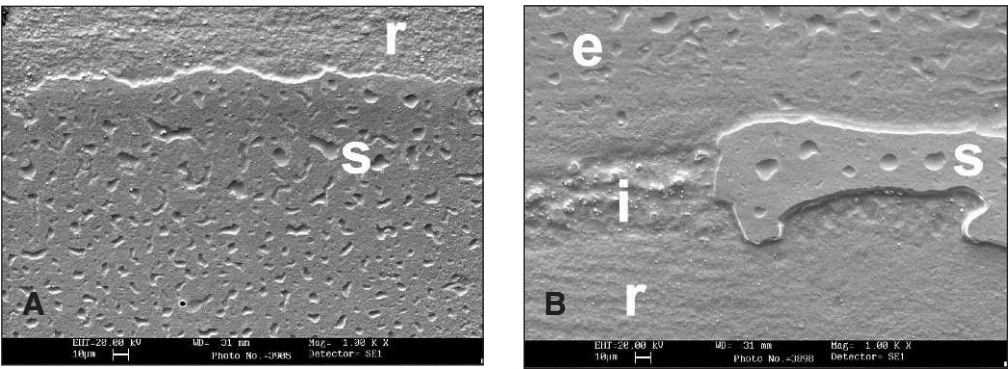


Figure 6. Sealed specimens after toothbrushing abrasion test. Enamel surface (e); Tooth-restoration interface (i); Resin composite (r); Restoration surface sealant (s). (Figure 6A: restoration sealant remained and protected the interface; Figure 6B: restoration sealant partially removed from the interface) (SEM 1000x).

Table 2: Mean of Tooth-restoration Interface Width (μm) and Statistical Analysis		
Groups	Tooth-restoration Interface Width Mean (SD)	Student's t-Test
NC	67 (18)	p=0.53
RC	72 (24)	
Student's t-test, n=10, SD: Standard Deviation, α=0.05.		

Table 3: Mean of Vertical Loss ( $\mu\text{m}$ ) and Statistical Analysis			
Groups	Mean of Vertical Loss (SD)	Tukey	
NC	49.30 (22)	a	
NC+S	7.90 (2.9)		c
RC	27.15 (5.3)	b	
RC+S	4.74 (1.9)		c
Analysis of variance and Tukey, $n=10$ , SD: Standard Deviation, $\alpha=0.05$ . Same letters indicate no statistical differences.			

Table 4: Mean of Worn Area ( $\mu\text{m}^2$ ) and Statistical Analysis			
Groups	Worn Area (SD)	Tukey	
NC	2,008 (790)	d	
NC+S	128 (55)		e
RC	1,580 (640)	d	
RC+S	88 (22)		e
Analysis of variance and Tukey, $n=10$ , SD: Standard Deviation, $\alpha=0.05$ . Same letters indicate no statistical differences.			

brushing abrasion test are shown in Figures 4 and 5. After the abrasion test, some filler particles protruded from the worn surface of the cement layer. For the resin coating groups, small interfacial gaps were detected between enamel and low viscosity resin composite and between low viscosity composite and indirect restoration. With regard to the sealed groups, Figure 6 shows that the restoration surface sealant in some regions was removed and in others remained and protected the interface.

DISCUSSION

The present findings revealed that toothbrushing provided significant wear of the tooth-restoration interface for all groups studied ( $p<0.01$ ). These results are in agreement with those reported by Guzman and others (1997), who noticed changes in the cementation interface after the brushing abrasion test.

The resin coating technique permits coverage and protection of the prepared dentin immediately after cavity preparation in order to provide high resin cement bond strength (Jayasooriya & others, 2003b), minimizing pulp irritation and post-operative sensitivity (Nikaido & others, 2003). In addition, it has been reported that one-step self-etch adhesives (such as ED primer) behave like permeable membranes after polymerization (Tay & others, 2002), allowing water to diffuse from dentin across to the adhesive. In the presence of a slow setting composite, this diffusion process tends to intensify, providing an adhesive-composite incompatibility leading to premature decoupling of dual-cured cements (Tay & others, 2003). Thus, a resin coating technique was recommended for Panavia F (Jayasooriya & others, 2003a). Although fitting problems have been claimed not to occur as the impressions are taken after coating the cavity, it was still unclear whether there was an increase in tooth-restoration interface width. The results of this study showed no statistical differences in interface width between the two techniques. Presumably, the increase in the low viscosity composite layer thickness was partially compensated by a reduction in the thickness of the resin cement layer (Carvalho & others, 2004).

Although both techniques presented different wear patterns (Figures 2 and 3) after brushing strokes, no statistical differences were found between the worn areas. In addition, the unsealed coated groups presented lower vertical loss than the unsealed conventional cementation groups. Since both adhesive and resin cement seem to be less wear resistant than low viscosity resin composite, the latter possibly protected them from the wear process. As the adhesive began to wear away, the toothbrush/slurry could no longer contact its surface.

A relation between vertical loss and the worn area for tested techniques did not occur, since they presented



different wear patterns. Both techniques presented similar interfacial widths. In the conventional cementation procedure, this width, associated with higher vertical loss, resulted in a lower worn area. This is probably because this vertical loss value resulted from a single "reduction" or "depression" shown in the profile (Figure 2). On the other hand, for the resin coating cementation procedure, although vertical loss was smaller, the profile showed several "reductions" (Figure 3) that probably led to an increase in the mean worn area.

Even though the use of restoration surface sealing was not effective for reducing cementation interface wear in the study by Shinkai and others (1994), the findings of the current study showed increased wear resistance for the sealed groups. Some factors could account for the disagreement between the two studies, such as differences in the composition of the materials used and the characteristics and type of wear test (400,000 cycles of a three-body wear test with a load of 17 lbs by means of a dial micrometer strain gauge). In this regard, Shinkai and others (1994) stated that, during early periods of the wear test, the sealant increased wear resistance. However, after 400,000 cycles, the sealant had worn away. Also, after a period of time, the wear process of cementation interfaces had decreased, because the food bolus (used as third-body) could no longer contact and abrade the interface. For this reason, their sealed and unsealed interfaces probably had the amount of wear leveled up by the end of the test. According to the authors (Shinkai & others, 1994), an intermittent application of the restoration surface sealant would be more effective in reducing the wear of a luting interface.

On the other hand, the results of the current study agree with other studies (Dickinson & others, 1990; des Santos & others, 2003), which show that the application of a resin without filler was able to increase composite wear resistance. This may be explained by the presence of a thicker organic cover on the restoration sealed surfaces, which may undergo wear, delaying the exposure of underlying restorative material. According to the material manufacturer, reapplication at follow-up is indicated for 6 to 12 months. Considering that 100,000 brush strokes correspond to 3.5 to 4 years of clinical service (Wang & others, 2004), the partial restoration sealant debonding, shown in Figure 6, is expected to occur. Also, the clinical difficulty of sealing sub-gingival margins and inaccessible areas must be considered. Fortunately, it is as difficult to apply the restoration surface sealant in these areas as it is for the toothbrush and food bolus to contact them directly. Further clinical studies are necessary to demonstrate the longevity of these restoration sealants and to provide guidelines for proper handling. In this study, the authors offered a feasible approach for improving wear resistance of the cementation interface. This technique is relatively simple and requires little additional time by the clinician.

## CONCLUSIONS

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

1. After the toothbrushing abrasion test, all groups demonstrated significant wear alterations (vertical loss and area).
2. No statistical differences were found in the tooth-restoration interface width and worn area between conventional and resin coating cementation techniques.
3. The resin coating technique presented reduced vertical loss compared to the conventional cementation technique.
4. Surface sealing of the tooth-restoration interface provided reduced wear for both techniques.

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## References

- Bollen CM, Lambrechts P & Quirynen M (1997) Comparison of surface roughness of oral hard materials to the threshold surface for bacterial plaque retention: A review of the literature *Dental Materials* **13**(4) 258-269.
- Buchalla W, Attin T & Hellwig E (2000) Brushing abrasion of luting cements under neutral and acidic conditions *Operative Dentistry* **25**(6) 482-487.
- Carvalho RM, Pegoraro TA, Tay FR, Pegoraro LF, Silva NR & Pashley DH (2004) Adhesive permeability affects coupling of resin cements that utilizes self-etching primers to dentine *Journal of Dentistry* **32**(1) 55-65.
- des Santos PH, Consani S, Correr Sobrinho L & Coelh Sinhoreti MA (2003) Effect of surface penetrating sealant on roughness of posterior composite resins *American Journal of Dentistry* **16**(3) 197-201.
- Dickinson GL, Leinfelder KF, Mazer RB & Russell CM (1990) Effect of surface penetrating sealant on wear rate of posterior composite resins *Journal of the American Dental Association* **121**(2) 251-255.
- el-Mowafy OM (2001) The use of resin cements in restorative dentistry to overcome retention problems *Journal of the Canadian Dental Association* **67**(2) 97-102.
- el-Mowafy OM & Milenkovic M (1994) Retention of paraposts cemented with dentin-bonded resin cements *Operative Dentistry* **19**(5) 176-182.
- Goldstein GR & Lerner T (1991) The effect of toothbrushing on hybrid composite resin *Journal of Prosthetic Dentistry* **66**(4) 498-500.

- Guzman AF, Moore BK & Andres CJ (1997) Wear resistance of four luting agents as a function of marginal gap distance, cement type, and restorative material *International Journal of Prosthodontics* **10**(5) 415-425.
- Hachiya Y, Iwaku M, Hosoda H & Fusayama T (1984) Relation of finish to discoloration of composite resins *Journal of Prosthetic Dentistry* **52**(6) 811-814.
- Jayasooriya PR, Pereira PN, Nikaido T, Burrow MF & Tagami J (2003a) The effect of a "resin coating" on the interfacial adaptation of composite inlays *Operative Dentistry* **28**(1) 28-35.
- Jayasooriya PR, Pereira PN, Nikaido T & Tagami J (2003b) Efficacy of a resin coating on bond strengths of resin cement to dentin *Journal of Esthetic and Restorative Dentistry* **15**(2) 105-113.
- Kanter J, Koski RE & Martin D (1982) The relationship of weight loss to surface roughness of composite resins from simulated toothbrushing *Journal of Prosthetic Dentistry* **47**(5) 505-513.
- Mondelli RFL, Prakki A, Cilli R, Navarro MFL & Mondelli J (2003) Surface roughness and electron microscopic observations of resin luting agents *Journal of Applied Oral Science* **11** 327-331.
- Nikaido T, Cho E, Nakajima M, Tashiro H, Toba S, Burrow MF & Tagami J (2003) Tensile bond strengths of resin cements to bovine dentin using resin coating *American Journal of Dentistry* **16**(Spec No) 41A-46A.
- Peumans M, Van Meerbeek B, Lambrechts P & Vanherle G (2000) Porcelain veneers: A review of the literature *Journal of Dentistry* **28**(3) 163-177.
- Roulet JF & Walti C (1984) Influence of oral fluid on composite resin and glass-ionomer cement *Journal of Prosthetic Dentistry* **52**(2) 182-189.
- Shinkai K, Suzuki S, Leinfelder KF & Katoh Y (1994) Effect of surface penetrating sealant on wear resistance of luting agents *Quintessence International* **25**(11) 767-771.
- Sjogren G, Bergman M, Molin M & Bessing C (1992) A clinical examination of ceramic (Cerec) inlays *Acta Odontologica Scandinavica* **50**(3) 171-178.
- Takeuchi CY, Orbegoso Flores VH, Palma Dibb RG, Panzeri H, Lara EH & Dinelli W (2003) Assessing the surface roughness of a posterior resin composite: Effect of surface sealing *Operative Dentistry* **28**(3) 281-286.
- Tay FR, Pashley DH, Suh BI, Carvalho RM & Itthagarum A (2002) Single-step adhesives are permeable membranes *Journal of Dentistry* **30**(7-8) 371-382.
- Tay FR, Pashley DH, Yiu CK, Sanares AM & Wei SH (2003) Factors contributing to incompatibility between simplified-step adhesives and self-cured or dual-cured composites. Part I. Single-step self etching adhesive *Journal of Adhesive Dentistry* **5**(1) 27-40.
- Wang L, Garcia FC, Amarante de Araujo P, Franco EB & Mondelli RF (2004) Wear resistance of packable resin composites after simulated toothbrushing test *Journal of Esthetic and Restorative Dentistry* **16**(3) 303-314.
- White SN & Yu Z (1993) Physical properties of fixed prosthodontic, resin composite luting agents *International Journal of Prosthodontics* **6**(4) 384-389.
- Working draft for wear test by tooth brushing. Part I. ISO, 1996. International Organization for Standard Technical Specification 14569-1 Switzerland.

# Immediate Dentin Sealing of Onlay Preparations: Thickness of Pre-cured Dentin Bonding Agent and Effect of Surface Cleaning

MM Stavridakis • I Krejci • P Magne

## Clinical Relevance

The film thickness of the Dentin Bonding Agent (DBA) used for the “immediate dentin sealing” of onlay preparations prior to the final impression for indirect restorations presents a vast range of values, depending on both the type of DBA and the topography of the tooth preparation. Curing the DBA in the absence of oxygen (air blocking) is mandatory to maintain a minimum DBA thickness. The filled DBA presented a more uniform thickness compared to the unfilled one. Air abrasion and polishing used for cleaning the pre-cured DBA prior to final cementation reduces the thickness of the DBA in a non-uniform manner.

## SUMMARY

**This study evaluated the thickness of Dentin Bonding Agent (DBA) used for “immediate dentin sealing” of onlay preparations prior to final impression making for indirect restorations. In addition, the amount of DBA that is removed when the adhesive surface is cleaned with polishing or air abrasion prior to final cementation was evaluated. For this purpose, a standardized onlay preparation was prepared in 12 extracted**

**molars, and either OptiBond FL (Kerr) or Syntac Classic (Vivadent) was applied to half of the teeth and cured in the absence of oxygen (air blocking). Each tooth was bisected in a bucco-lingual direction into two sections, and the thickness of the DBA was measured under SEM on gold sputtered epoxy resin replicas at 11 positions. The DBA layer of each half tooth was treated with either air abrasion or polishing. The thickness of the DBAs was then re-measured on the replicas at the same positions. The results were statistically analyzed with non-parametric statistics (Mann-Whitney U test and Kruskal-Wallis test) at a confidence level of 95% ( $p=0.05$ ).**

**The film thickness of the DBA was not uniform across the adhesive interface ( $121.13 \pm 107.64 \mu\text{m}$ ), and a great range of values was recorded (0 to  $500 \mu\text{m}$ ). Statistically significant differences ( $p<0.05$ ) were noted, which were both material (OptiBond FL or Syntac Classic) and position (1 to 11) dependent. Syntac Classic presented a higher thickness of DBA ( $142.34 \pm 125.10 \mu\text{m}$ ) than OptiBond FL ( $87.99 \pm 73.76 \mu\text{m}$ ). The higher film thickness of both DBAs was at the deepest part of**

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the isthmus (the most concave part of the preparation), while the lowest was at the line angles of the dentinal crest (the most convex part of the preparation). OptiBond FL presented a more uniform thickness around the dentinal crest of preparation; Syntac Classic pooled at the lower parts of the preparation.

The amount of DBA that was removed with air abrasion or polishing was not uniform ( $11.94 \pm 16.46 \mu\text{m}$ ), and a great range of values was recorded (0 to  $145 \mu\text{m}$ ). No statistically significant differences ( $p < 0.05$ ) were found either between different DBAs (OptiBond FL or Syntac Classic) or between different treatments (air abrasion or polishing). As far as the effect of different treatments at different positions, polishing removed more DBA from the top of the dentinal crest, but the difference was not statistically significant. Air abrasion removed less DBA from the corners of the dentinal crest (Positions 4 and 6) than the outer buccal part of the preparation (Positions 1 and 2). Neither air abrasion nor polishing removed the entire layer thickness of the DBA in the majority of the cases.

## INTRODUCTION

The demand for tooth-colored restorations has grown considerably during the past decade. Due to residual polymerization stresses, direct resin composite restorations are often contraindicated in large cavities. In these situations, indirect porcelain or polymer restorations are commonly the choice of the dental clinician, as they are more conservative than full coverage crowns. Many aspects of the adhesive luting procedures of such restorations have been thoroughly investigated in order to find the ideal method that would ensure excellent clinical results (Hansen & Asmussen, 1987; Millstein & Nathanson, 1992; Dietschi, Magne & Holz, 1995; Bertschinger & others, 1996; Bachmann & others, 1997; Paul, 1997a,b; Paul & Scharer, 1997a,b; Peter & others, 1997; Dietschi & Herzfeld, 1998; Magne & Douglas, 1999).

Successful adhesion to enamel has been achieved with relative ease. On the contrary, the development of predictable bonding to dentin has been more problematic. Only in the past decade have dentin bonding agents produced results that approach those of enamel bonding and have achieved a predictable level of clinical success with direct resin composite restorations. As earlier bonding agents achieved lower bond strengths when applied to dentin compared to enamel, the presence of dentin in a significant percentage of the luting interface was one of the main issues that made dentists skeptical about placement of indirect bonded restorations in the posterior region. The dentin-adhesive resin interface of indirect restorations has also been thor-

oughly investigated, and different luting procedures have been proposed. In the case of a substantial accessible area of dentin being exposed by tooth preparation (inlay, onlay or veneer), the local application of a dentin bonding agent (DBA) is recommended. At least two methods have been presented for use of the DBA in order to promote dentin adhesion of indirect restorations.

In the *classical approach*, dentin exposures are initially ignored; the DBA is applied only at the last treatment stage when proceeding to lute the restoration. In this case, the DBA must be initially left uncured to allow for complete seating of the restoration. It has been postulated that the pressure of the luting composite during the seating of the restoration may create a collapse of demineralized dentin (collagen fibers) and subsequently affect the adhesive interface cohesiveness (Dietschi & Herzfeld, 1998; Dietschi & others, 1995; Magne & Douglas, 1999). Thinning the adhesive layer has been proposed in order to allow its curing before insertion of the restoration. However, because methacrylate resins show an inhibition layer up to  $\sim 40 \mu\text{m}$  when they are light-cured (Rueggeberg & Margeson, 1990), excessive thinning can prevent the curing of light-activated DBAs.

More recently, a *new approach* was proposed to optimize the DBA application (Paul, 1997a; Paul & Scharer, 1997a; Bertschinger & others, 1996; Magne & Douglas, 1999). Because the DBA appears to have a superior potential for adhesion when it is precured (Frankenberger & others, 1999) and applied to freshly prepared dentin, its application is recommended immediately after completion of the tooth preparation, before the final impression. A substantial clinical advantage of the so-called "immediate dentin sealing" (Magne & Douglas, 1999; Magne & Belser, 2002a) is that this precautionary measure seals and protects the pulpodentinal organ and, by the same token, prevents sensitivity and bacterial leakage during the provisional phase. Further adhesion of the luting agent to the preexisting adhesive layer must be promoted by surface cleaning just prior to luting (Magne & Douglas, 1999; Magne & Belser, 2002b) in order to remove remnants of provisional cements that may cause a significant decrease in the bond strength of the luting agent (Paul & Scharer, 1997b; Millstein & Nathanson, 1992). This is especially important when the luting agent contains eugenol, which inhibits resin polymerization (Hansen & Asmussen, 1987; Millstein & Nathanson, 1992).

Initially, pumice slurry was used as a means of removing remnants of provisional cements (Gerbo & others, 1992), even though its efficacy has been questioned (Bachmann & others, 1997). Prophylaxis pastes have also been proposed for cleaning the dental surfaces prior to cementation (Aboush, Tareen & Elderton,

1991). Another fast and effective final cleansing method is the use of an intraoral sandblaster. There are no data, however, providing information about the effect of surface treatments on the thickness of the pre-cured adhesive layer when “immediate dentin sealing” has been applied. Therefore, this study evaluated the effect of surface cleaning on the remaining thickness of the precured DBA. A standardized onlay preparation was chosen to compare filled and unfilled DBAs and evaluate various sites of the cavity. The null hypothesis was that there was no statistically significant difference between the thickness of the different DBAs at various sites of the adhesive interface before and after surface cleaning of the pre-cured DBA.

## METHODS AND MATERIALS

Twelve extracted caries-free human lower molars with completed root formation were stored in 0.1% thymol solution for the time between extraction and use in this *in vitro* test. After scaling and pumicing of the root surface, a standardized onlay cavity without a marginal bevel in enamel was prepared. Figure 1 illustrates a bucco-lingual section of the cavity design used in this study. The lingual one-third of each tooth was left unprepared. A deep, straight isthmus was prepared in a mesio-distal direction, which extended all the way to the proximal surfaces of the teeth. To that purpose, 80 µm diamond burs (Universal Prep Set, Intensiv SA, Lugano, Switzerland) were used under continuous water cooling in a direction where their long axis was perpendicular to the long axis of the teeth and parallel to their lingual surface. The isthmus had the same depth across the tooth, to a depth where the proximal enamel was left intact 1 mm from the cemento-enamel junction. The enamel was then prepared at the buccal surface of the teeth to the same horizontal level as proximally. The next step was to remove the occlusal enamel and dentin from the buccal cusps, so as to leave a crest of dentin below the buccal cusps, which was approximately 2 mm in height and width. The height of this dentinal crest was the same across the tooth, with rounded bucco-occlusal and linguo-occlusal line angles. Finally, enamel and dentin were removed at the mesio-buccal and disto-buccal line angles, so as to have a continuous 1 mm rounded shoulder margin at the same horizontal level, which extended from the mesial to the distal side of the tooth. The entire cavity was finished using 25 µm finishing diamond burs (Intensiv SA). Cavity preparations were checked for marginal imperfections, such as fractures or chipping, under a stereo microscope (Wild M5, Wild AG, Heerbrugg, Switzerland)

at 12x magnification. If present, the imperfections were corrected.

The teeth were then randomly divided into two groups according to the DBA that would be applied to them and prepared for simulation of the intratubular fluid flow. To that purpose, the apices were sealed with two coats of nail varnish. Then the teeth were mounted on custom-made specimen holders, with their roots in the center, using an auto-polymerizing resin (Paladur, Kulzer & Co, Wehrheim, Germany). A custom-made device assured that the isthmus of the preparation was placed parallel to the base of the specimen holder. A cylindrical hole was drilled into the pulpal chamber approximately in the middle third of the root. A metal tube with a diameter of 1.4 mm was then adhesively luted using a DBA (Syntac Classic, Vivadent, Schaan, Liechtenstein). The pulpal tissue was not removed. This tube was connected by a flexible silicone hose to an infusion bottle placed 34 cm vertically above the test tooth. The infusion bottle was filled with horse serum diluted in a 1:3 ratio with 0.9% PBS (Maita & others, 1991; Pashley & others, 1981) to simulate the dentinal fluid under normal hydrostatic pressure of about 25 mm Hg (Tao & Pashley, 1989; Mitchem, Terkla &

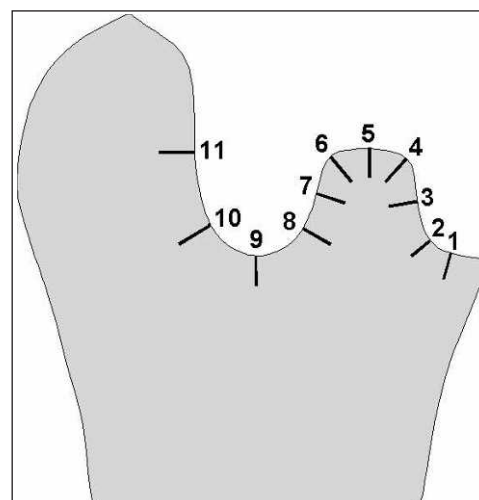


Figure 1. Bucco-lingual section of the cavity design used in this study. Eleven orientation lines were marked to certain areas of dentin perpendicular to the adhesive layer.

Table 1: Manufacturers, Lot Numbers and Expiration Dates of the Materials Used

Material	Manufacturer	Lot #	Exp Date
Ultra-Etch	Ultradent Products Inc	35PQ	2003-04
OptiBond FL primer	Kerr USA	008C00	2002-06
OptiBond FL adhesive	Kerr USA	006122A	2001-11
Syntac primer	Vivadent	C16315	2002-10
Syntac adhesive	Vivadent	C15085	2002-08
Heliobond	Vivadent	C06372	2005-02

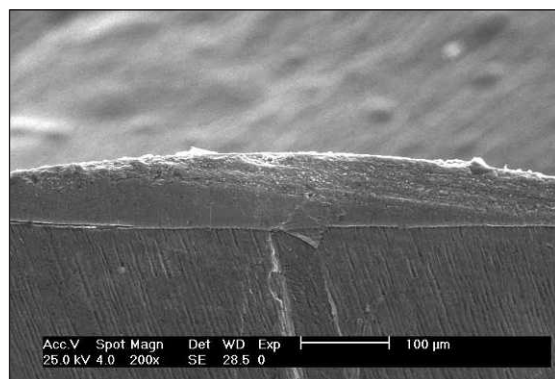


Figure 2. Film thickness of the DBA was measured in a direction parallel to the orientation line that was marked in dentin adjacent to the adhesive interface (200x magnification).

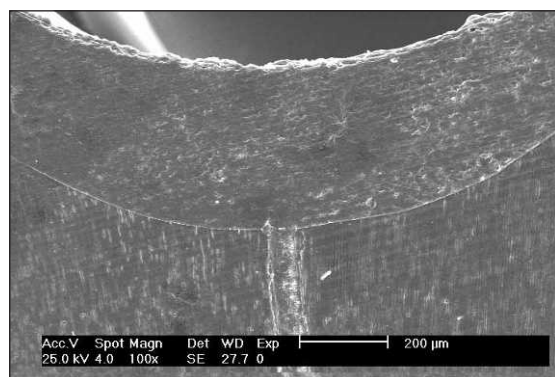


Figure 3. Film thickness of the DBA at Position 9 prior to application of air abrasion (100x magnification).

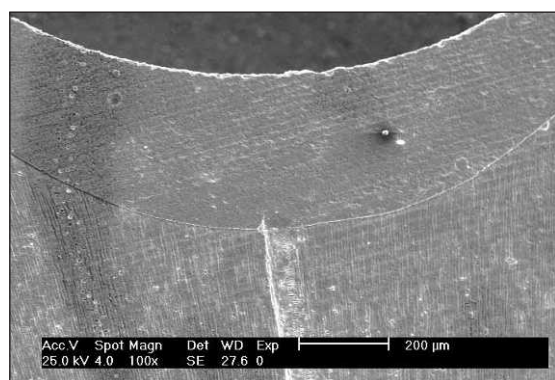


Figure 4. Film thickness of the DBA at Position 9 after application of air abrasion (100x magnification).

Gronas, 1988). Twenty-four hours before starting application of the DBA, the pulp chambers were evacuated with a vacuum pump and subsequently filled with the diluted, bubble-free horse serum by using a three-way valve. From this time forward, the intrapulpal pressure was maintained at 25 mm Hg throughout application of the DBA. The manufacturers, lot numbers and expira-

tion dates of the materials used in this study are listed in Table 1.

OptiBond FL (Kerr, Orange, CA, USA) was applied to the first group of six teeth. Both enamel and dentin were etched with 35% phosphoric acid gel (Ultra-Etch, Ultradent, South Jordan, UT, USA) for 30 and 15 seconds, respectively. The dental surfaces were washed with water for 30 seconds in order to remove the etchant and gently air dried for approximately five seconds, making sure that the dentin was not desiccated. OptiBond FL primer was applied over the enamel and dentin with a light scrubbing motion for 30 seconds using a Kerr Applicator Tip. The dental surfaces were gently air dried for approximately five seconds, being careful once again to not desiccate the dental surfaces. OptiBond FL adhesive was uniformly applied over the enamel and dentin with a Kerr Applicator Tip, attempting to create a uniform thin layer of adhesive resin. The adhesive resin was left to penetrate into the demineralized hard dental tissues for 30 seconds, before being polymerized for 30 seconds, using a tip with an exit window diameter of 11 mm (Demetron 500; Demetron/Kerr, Danbury, CT, USA; irradiance according to the Demetron Curing Radiometer:  $\sim 800 \text{ mW/cm}^2$ ). A water soluble glycerin gel (K-Y Lubricating Jelly, Johnson & Johnson Consumer France SAS, Sezanne, France) was applied over the teeth, and the DBA was polymerized for another 30 seconds and subsequently washed and air dried.

Syntac Classic was applied to the second group of six teeth. The enamel was etched with 35% phosphoric acid gel (Ultra-Etch) for 60 seconds, washed with water for 30 seconds in order to remove the etchant and air dried for approximately five seconds. Syntac primer was applied over the enamel and dentin with a light scrubbing motion for 15 seconds, and the dental surfaces were thoroughly air dried for approximately five seconds. Syntac adhesive was applied over the enamel and dentin for 10 seconds and dried. Heliobond was applied uniformly with a Kerr Applicator Tip, trying to create a uniformly thin layer of adhesive resin. Heliobond was also left for 30 seconds to penetrate into the demineralized hard dental tissues before it was polymerized for 30 seconds using the same curing unit. A water soluble glycerine gel (K-Y Lubricating Jelly) was applied over the teeth, and the DBA was polymerized for another 30 seconds and subsequently washed and air dried.

Each tooth was bisected in a bucco-lingual direction perpendicular to the long axis of the isthmus and parallel to its long axis with the aid of a slowly rotating diamond disc (Isomet Low Speed Saw 11-1180, AB Bühler Ltd, Chicago, IL, USA) under water cooling. The sectioned surface of each half was polished using rotating sandpaper discs of descending abrasivity to the level of 4000 grit. The surface was relieved using 35% phosphoric acid for one second, washed and dried. Eleven



Position	1	2	3	4	5	6	7	8	9	10	11
	64	96	69	13	150	35	43	25	25	48	104
	91	106	54	17	153	34	96	222	251	154	54
	74	125	48	15	115	25	24	61	99	96	27
	90	115	44	21	114	20	52	71	56	25	30
	102	118	29	18	179	29	93	188	281	86	115
	206	257	176	17	207	59	79	205	321	240	40
	54	78	53	11	145	30	37	21	17	62	118
	112	119	52	70	157	48	87	219	277	206	130
	92	143	39	9	90	35	35	44	102	49	37
	97	108	48	0	124	36	32	58	82	27	49
	59	101	40	15	125	51	27	87	300	59	117
	199	251	125	23	209	85	40	101	363	260	65
Mean	103.33	134.75	64.75	19.08	147.33	40.58	53.75	108.50	181.17	109.33	73.83
St Dev	49.61	57.95	42.63	17.12	36.92	17.88	27.11	77.60	128.14	84.15	39.63

Position	1	2	3	4	5	6	7	8	9	10	11
	162	182	37	0	245	0	187	417	482	87	31
	311	434	299	9	19	0	92	237	273	62	25
	254	284	147	19	129	0	67	142	178	126	58
	111	124	30	0	0	108	149	316	436	267	40
	331	269	37	25	0	0	103	248	298	121	67
	206	190	107	0	96	0	109	344	413	187	52
	119	196	90	0	245	55	13	248	500	316	138
	332	442	399	0	0	0	13	149	273	153	36
	191	238	246	144	0	125	46	18	64	179	89
	120	119	27	130	115	25	49	209	444	200	28
	228	257	106	28	128	21	35	179	266	214	57
	137	182	193	153	0	84	14	56	226	396	280
Mean	208.50	243.08	143.17	42.33	81.42	34.83	73.08	213.58	321.08	192.33	75.08
St Dev	83.20	104.73	119.53	61.34	93.58	46.59	56.24	114.97	134.32	96.39	71.85

orientation line marks were marked to certain areas of dentin perpendicular to the adhesive layer, as illustrated in Figure 1. These lines were made with a fine surgical blade (#11 Bard-Parker Stainless Steel Surgical Blades, Becton Dickinson AcuteCare, Franklin Lakes, NJ, USA) under 20x magnification. Impressions were made of the sectioned surface of each half using a polyvinylsiloxane impression material (President light body, Coltène AG, Altstätten, Switzerland). Subsequently, epoxy replicas were prepared for the computer-assisted measurement of the thickness of the adhesive layer in a scanning electron microscope (Philips XL20, Philips, Eindhoven, Netherlands) at 200x magnification. The thickness of the adhesive layer was measured in a direction parallel to the orientation lines

that were marked in dentin adjacent to the adhesive interface and which were easily identified (Figure 2).

The adhesive layer of each half of the tooth was treated with one of the two most common methods used for the removal of temporary cement prior to the final cementation of indirect restorations. One half was air abraded with 50 µm aluminum-oxide powder (Dento-Prep Microblaster, Ronvig Dental Mfg, Daugaard, Denmark) that was propelled at 4.5 bars pressure with three Z-shaped sweeping motions over the preparation for approximately five seconds. The other half was cleaned with a prophylaxis paste (Depurder, Dr Wild & Co AG, Basel, Switzerland) in a rotary nylon brush (Nylon brush, Hawe-Neos, Bioggio, Switzerland) at 1,000 rpm for approximately five seconds. Impressions and epoxy

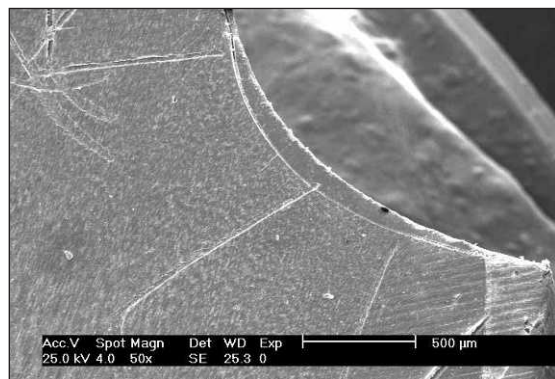


Figure 5. Representative specimen with "thin" film thickness of DBA at Positions 1, 2 and 3 (50x magnification).

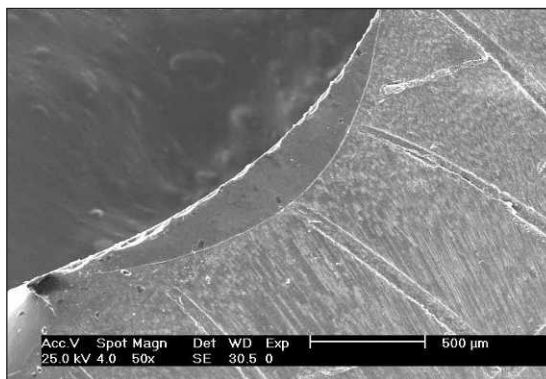


Figure 6. Representative specimen with "thick" film thickness of DBA at Positions 1, 2 and 3 (50x magnification).

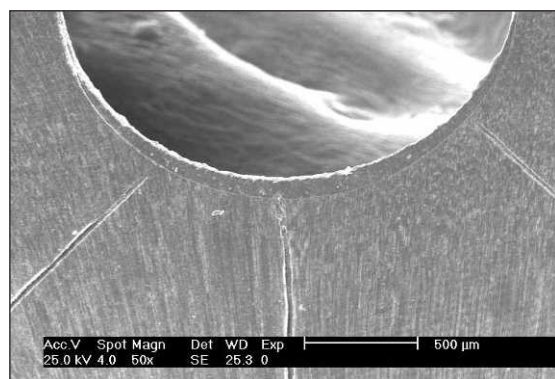


Figure 7. Representative specimen with "thin" film thickness of DBA at Positions 8, 9 and 10 (50x magnification).

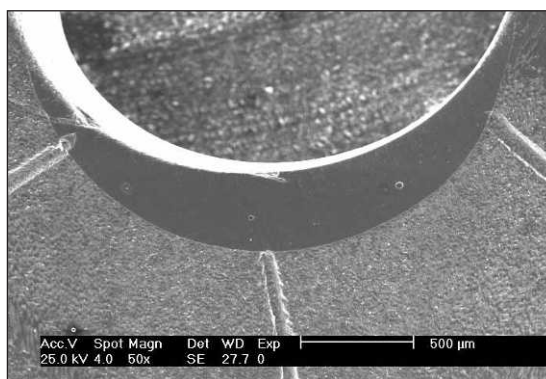


Figure 8. Representative specimen with "thick" film thickness of DBA at Positions 8, 9 and 10 (50x magnification).

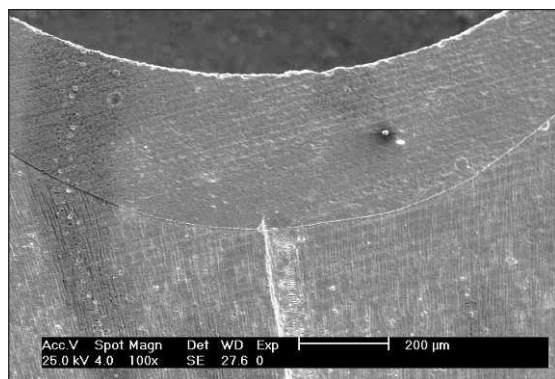


Figure 9. Representative specimen with "thin" film thickness of DBA at Position 6 (200x magnification). The thickness of the DBA increases at the top-right side toward Position 5.

replicas were made from the sectioned surface of each half of the tooth as previously described. The thickness of the adhesive layer was once again evaluated in SEM at 200x magnification in a direction parallel to the same orientation lines. Using these orientation lines assured that the measurements before and after treatment of the adhesive interface with air abrasion or polishing were comparable. The amount of the adhesive

layer that was removed by air abrasion or polishing was calculated by subtracting the thickness of the adhesive layer before (Figure 3) and after (Figure 4) each treatment.

As the data for the adhesive layer that was collected did not come from a normal distribution, non-parametric statistics were used for the statistical evaluation. The Mann-Whitney U and Kruskal-Wallis tests were used for comparison of the medians of the sample groups at a confidence level of 95% ( $p=0.05$ ).

## RESULTS

The film thickness of the DBA of OptiBond FL and Syntac Classic at different positions of the adhesive layer prior to application of air abrasion or polishing is presented in Tables 2 and 3. The film thickness of the DBA was not uniform across the adhesive interface ( $121.13 \pm 107.64 \mu\text{m}$ ), and a significant range of values was recorded (0 to 500  $\mu\text{m}$ ) (Figures 5 to 9). Statistically significant differences ( $p<0.05$ ) were noted, which were both material (OptiBond FL or Syntac Classic) and position (1 to 11) dependent. Syntac Classic presented statistically significant higher thickness of DBA ( $142.34 \pm 125.10 \mu\text{m}$ ) than OptiBond FL ( $87.99 \pm 73.76 \mu\text{m}$ ).

The statistically significant differences ( $p<0.05$ ) of the DBA thickness between the different positions of OptiBond FL are presented in Table 4. The smallest film thickness of DBA was found in Position 4 (bucco-occlusal line angle of the dentinal crest), which was statistically different from all other areas. The film thickness of DBA was higher in Position 9 (the deepest part of the isthmus), which was not statistically different from most other areas of the preparation (Positions 5, 2, 1, 8, 10, 11 and 7).

The statistically significant differences ( $p<0.05$ ) of the DBA thickness between the different positions of

Table 4: *OptiBond FL: subgroups of statistical significance prior to the application of air abrasion or polishing (Kruskal-Wallis test,  $p < 0.05$ ,  $n = 12$ ). Positions that were different are marked with the—symbol in the table below. Positions with x were not different.*

Positions	4	6	7	3	11	10	8	1	2	5	9
4	-	-	-	-	-	-	-	-	-	-	-
6	-		x	x	x	x	-	-	-	-	-
7	-	x		x	x	x	x	x	-	-	x
3	-	x	x		x	x	x	-	-	-	-
11	-	x	x	x		x	x	x	x	-	x
10	-	x	x	x	x		x	x	x	x	x
8	-	-	x	x	x	x		x	x	x	x
1	-	-	x	-	x	x	x		x	-	x
2	-	-	-	-	x	x	x	x		x	x
5	-	-	-	-	-	x	x	-	x		x
9	-	-	x	-	x	x	x	x	x	x	

Table 5: *Syntac Classic: Subgroups of statistical significance prior to the application of air abrasion or polishing (Kruskal-Wallis test,  $p < 0.05$ ,  $n = 12$ ). Positions that were different are marked with the—symbol in the table below. Positions with x were not different.*

Positions	6	4	11	5	7	3	10	1	2	8	9
6		x	x	x	x	x	-	-	-	-	-
4	x		x	x	x	x	-	-	-	-	-
11	x	x		x	x	x	-	-	-	-	-
5	x	x	x		x	x	x	-	-	-	-
7	x	x	x	x		x	-	-	-	-	-
3	x	x	x	x	x		x	x	x	x	-
10	-	-	-	x	-	x		x	x	x	x
1	-	-	-	-	-	x	x		x	x	x
2	-	-	-	-	-	x	x	x		x	x
8	-	-	-	-	-	x	x	x	x		x
9	-	-	-	-	-	-	x	x	x	x	

Syntac Classic are presented in Table 5. Areas located on the dentinal crest (Positions 4, 6 and 5) and inclined planes at the upper half of the preparation (Positions 11, 7 and 3) presented thinner films of DBA with no statistically significant differences among them. Concave parts at the bottom half of the preparation (Positions 9, 8, 2, 1 and 10) presented thicker films of DBA, also with no statistically significant differences among them.

The film thickness of the DBA that was removed after application of air abrasion or polishing at different positions of the adhesive layer is presented in Table 6. The amount of DBA that was removed was not uniform across the adhesive interface ( $11.94 \pm 16.46 \mu\text{m}$ ), and a great range of values was recorded ( $0\text{--}145 \mu\text{m}$  for polishing and  $0\text{--}63 \mu\text{m}$  for air abrasion). In OptiBond FL specimens, air abrasion ( $8.47 \pm 8.63 \mu\text{m}$ ) removed less DBA than polishing ( $16.45 \pm 24.34 \mu\text{m}$ ), but the difference, due to the large standard deviations, was not statistically significant. In Syntac Classic specimens, air abrasion ( $11.41 \pm 14.11 \mu\text{m}$ ) removed similar thickness-

es of DBA to polishing ( $11.42 \pm 14.05 \mu\text{m}$ ). The amount of DBA removed by polishing was not dependent on the position. Polishing removed more DBA from the top of the dentinal crest, but the difference was not statistically significant. On the contrary, when air abrasion was used, the amount of DBA removed depended on the position. As Table 7 illustrates, air abrasion removed less DBA from the corners of the dentinal crest (Positions 4 and 6) and more DBA from the outer buccal part of the preparation (Positions 1 and 2).

## DISCUSSION

The film thickness of DBAs presented a vast range of values around different positions of the adhesive layer. This was to be expected, as other researchers have reported similar results for direct resin composite preparations (Watson, 1989) and complete coverage preparations (Peter & others, 1997; Pashley & others, 1992). Even though a non-uniform distribution of DBA around the adhesive interface was expected, OptiBond FL and Syntac Classic presented some dissimilarity in



Table 6: Means and standard deviations of the film thickness (in microns) of OptiBond FL and Syntac Classic that was removed after the application of air abrasion or polishing at different positions of the adhesive layer (n=6).

GROUP		1	2	3	4	5	6	7	8	9	10	11
OBFL/AA	Mean	15.83	20.67	16.33	4.33	9.50	1.50	2.00	3.33	5.33	5.00	9.33
	SD	11.53	10.33	13.07	0.95	5.96	2.41	2.29	2.94	3.67	3.68	3.95
SYN/AA	Mean	19.00	15.50	10.83	0.00	5.50	1.17	14.67	20.17	16.00	13.00	9.67
	SD	20.75	9.62	5.68	1.51	6.80	3.18	22.82	19.55	13.47	8.08	7.60
OBFL/PO	Mean	13.83	16.50	7.17	3.00	35.83	13.33	5.00	12.83	26.83	20.33	26.33
	SD	12.70	13.48	10.52	3.85	52.48	10.75	4.27	16.51	27.36	21.94	28.82
SYN/PO	Mean	10.67	15.50	11.33	15.67	14.33	14.50	4.67	11.50	12.00	9.67	5.83
	SD	13.70	14.19	16.46	20.46	16.56	17.05	4.83	11.59	14.03	8.73	5.44

OBFL: OptiBond FL; SYN: Syntac Classic; AA: air abrasion; PO: polishing.

Table 7: Air abrasion: subgroups of statistical significance of the film thickness (in microns) of DBA that was removed (n=12). Positions that were different are marked with the—symbol in the table below. Positions with an X were not different.

Positions	6	4	7	8	5	9	10	11	3	1	2
6		x	x	x	x	-	-	-	-	-	-
4	x		x	x	x	x	x	-	-	-	-
7	x	x		x	x	x	x	x	-	x	-
8	x	x	x		x	x	x	x	x	x	x
5	x	x	x	x		x	x	x	x	x	x
9	-	x	x	x	x		x	x	x	x	x
10	-	x	x	x	x	x		x	x	x	x
11	-	-	x	x	x	x	x		x	x	x
3	-	-	-	x	x	x	x	x		x	x
1	-	-	x	x	x	x	x	x	x		x
2	-	-	-	x	x	x	x	x	x	x	

their behavior. Both DBAs presented their maximum thickness at Position 9 (the deepest part of the isthmus) and a substantial thickness of DBA in concave parts of the preparation (Positions 2, 8 and 10). During the application of the DBA, the teeth were positioned with their vertical axis perpendicular to the horizontal plane, thus resembling the clinical conditions of mandibular molars of a patient whose occlusal plane of the lower arch is parallel to the floor. Therefore, the influence of different inclinations of teeth, as well as the possible effects of gravitational forces while working on maxillary teeth to the thickness of DBA at different positions of the adhesive layer, was not evaluated. Nevertheless, the increased thickness due to pooling of the DBA at the inner line angles of the preparation is in accordance with the dental literature (Peter & others, 1997). The minimum thickness of both DBAs was observed at convex areas of the preparation, such as Positions 4 and 6 (bucco-occlusal and linguo-occlusal line angles of the dentinal ridge). OptiBond FL produced a thicker layer at Position 5, which was located at the top of the dentinal crest ( $147.33 \pm 36.92 \mu\text{m}$ ) than Syntac Classic ( $81.42 \pm 93.58 \mu\text{m}$ ), even though in most positions Syntac Classic surpassed OptiBond FL.

A closer observation of the thickness of DBA in all specimens at the top of the dentinal crest (Positions 4, 5 and 6) revealed that Syntac Classic often failed to produce a measurable layer at these positions, even though a substantial thickness of DBA was observed in other positions of the same specimens. The bonding resin of Syntac Classic (Heliobond) did not rest on the position where it was placed, and it pooled to lower parts of the preparation, as lower positions (10, 1, 8, 2 and 9) exhibited increased thickness than higher positions (6, 4, 7, 11, 5 and 3). The pooling of the bonding resin may be attributed to the difference in viscosity between the primers (very low viscosity because of the high solvent and relatively low resin content) and the unfilled Heliobond (low-to-medium viscosity because of its bis-GMA composition) (Paul, 1997b).

A closer observation of the thickness of the DBA in all specimens at the top of the dentinal crest (Positions 4, 5 and 6) revealed that OptiBond FL produced a distinct layer at these positions in practically all specimens. Nevertheless, the observed thickness at Position 4 (bucco-occlusal line angle of the dentinal crest) and Position 6 (linguo-occlusal line angle of the dentinal crest) was most often below  $20 \mu\text{m}$ , and  $40 \mu\text{m}$ , respec-

tively. If no glycerine gel (K-Y Lubricating Jelly) was applied over the teeth, a possible problem could have been observed at these areas, as the inhibition layer of polymerization due to oxygen inhibition of the radicals that normally induce the polymerization reaction has been reported to reach 40  $\mu\text{m}$  (Rueggeberg & Margeson, 1990). As the thickness of the DBA is smaller than that of the oxygen inhibition layer, a significant portion of the top layers of the DBA would be left unpolymerized. The unpolymerized DBA would be subsequently easily removed during cleaning of the adhesive interface by air abrasion or polishing, along with a portion of the thin polymerized lower layers of the DBA. This could result in areas of exposed dentin. Therefore, unless future research indicates otherwise, it seems that using air block at the first stage is mandatory to avoid more dentin exposure during cleaning of the adhesive interface at a later stage.

OptiBond FL exhibited less pooling than Syntac Classic. This could be attributed to the fact that its adhesive resin is viscous due to the incorporation of filler particles in its composition. The maximum thickness of DBA recorded in this study was superior to that reported in the dental literature. This may be attributed to the fact that, in this study, no air thinning of the DBA was performed prior to its polymerization, which is the recommended clinical procedure when using "immediate dentin sealing" (Magne & Douglas, 1999; Magne, 2005). Air thinning would spread the adhesive beyond the preparations' margins, subsequently altering the margin definition and complicating finishing procedures. The decision not to air-thin the DBA was also due to the fact that one of the purposes of this investigation was to examine the effect of air abrasion and polishing, and air-thinning would increase the chance of having very little or no DBA at the bucco- and linguo-occlusal line angles of the dentinal crest. This factor could have complicated the evaluation of the effect of air abrasion and polishing. Additionally, in a subsequent part of this investigation, the effect of whether or not to use a water soluble glycerin gel in order to polymerize the oxygen-inhibited layer would be investigated. In any case, the thickness of the DBA in the borders of the preparation (not reported as placement of the orientation line marks were nearly impossible to be placed perpendicular to the adhesive layer, and they also often damaged the adhesive layer) was well within the range reported in the literature, as the DBA tended to pool in the interior of the preparation. The significant standard deviations in all positions around the adhesive layer illustrate the point that, even though the experienced operator who performed the research intended to achieve an even distribution of the DBA in all specimens, this was not feasible, even though he worked *in vitro* under ideal conditions. Specimens where Syntac Classic was used as DBA presented a

thicker layer than OptiBond FL. This could result from the fact that the adhesive resin of Syntac Classic (Heliomolar) is practically transparent, thus making the visual evaluation of the quantity of adhesive resin placed onto the preparation difficult. One could only speculate what kind of variation of thickness of DBA would be encountered in clinical cases, as a similar *in vivo* study would be most difficult to complete.

There is no similar study which compares the results of the film thickness of the DBA that was removed after the application of air abrasion or polishing in the dental literature. No difference was found between these two treatments when the authors examined the results of both DBAs. Nevertheless, a closer observation of the results of Tables 6 and 7 revealed that there was a difference in the behavior of these two treatments at different positions. The results of Table 7 reveal that air abrasion removed more DBA from the outer buccal part of the preparation (Positions 2 and 1) than the corners of the dentinal crest (Positions 6 and 4). This may be explained by the fact that the air abrasion particles, in their descending course after colliding with the buccal wall of the dentinal crest, may then be deflected to collide with the lower parts of the preparation at the buccal side, thus maximizing the amount of DBA being removed in these areas. On the other hand, the results of Table 6 show that polishing removed more DBA from the top of the preparation (Position 5 at the mid-height of the dentinal crest and Position 11 at the mid-lingual wall of the preparation), the very lowest parts of the preparation (Position 9 at the depth of the isthmus and Position 2 at the axio-buccal line angle of the dentinal crest) and less DBA from the vertical walls of the dentinal crest (Positions 3 and 7). Once again, few differences were found to be statistically significant due to the large standard deviations. The amount of DBA removed by polishing seemed to be related to the accessibility of the bristles of the rotating polishing brush to specific areas of the preparation.

Polishing removed more DBA than air abrasion in OptiBond FL, even though the difference was not statistically significant due to the ample standard deviations, whereas similar amounts of DBA were removed by the two treatments in Syntac Classic. This may be explained by the fact that OptiBond FL adhesive is a filled adhesive, in contrast to Heliobond, which is a non-filled adhesive resin. The fillers make OptiBond FL more resistant to air abrasion, as they increase its mechanical properties (compression, tensile strength and elastic modulus). On the other hand, resistance to wear from polishing is a more complex phenomenon that depends on several intrinsic and extrinsic factors. Several possible wear mechanisms have been proposed for dental composites. In one of them, it has been suggested that filler particles transmit considerable stress to the matrix, possibly resulting in microcracking and

subsequent loss of material. There are strong indications in the dental literature that adding inorganic filler particles to a resin, even in small amounts, greatly enhances the long-term wear resistance of such materials (Ulvestad, 1977; Raadal, 1978). Nevertheless, as the polishing procedure lasted only five seconds, the presence of filler particles in OptiBond FL adhesive resin could have contributed to the significant loss of material in the short term via the aforementioned mechanism.

In the vast majority of cases, both air abrasion and polishing did not remove the entire thickness of the DBA. There were only two exceptions: one specimen in the Syntac Classic—air abrasion group, where 19  $\mu\text{m}$  were removed from Position 5, and one specimen in the OptiBond FL—polishing group, where 11  $\mu\text{m}$ , 145  $\mu\text{m}$  and 30  $\mu\text{m}$  were removed from Positions 4, 5 and 6, respectively. This illustrates the fact that both treatments may be used in the manner described in the Methods and Materials section without fear of removing the entire thickness of the DBA in large areas of the preparation. Even though the measurements of this study were made at only 11 sites of one cross section of the preparation, the visual aspect of all teeth after air abrasion and polishing did not give the impression that the DBA was totally removed from large areas.

### CONCLUSIONS

Under the limitations of the experimental set-up, several conclusions may be drawn from this study. The film thickness of the DBA was not uniform across the adhesive interface, and a great range of values was recorded. Syntac Classic presented higher thickness of DBA than OptiBond FL. The higher film thickness of both DBAs was at the deepest part of the isthmus, while the lowest was at the line angles of the dentinal crest. OptiBond FL presented a more uniform thickness around the dentinal crest of preparation, while Syntac Classic pooled at the lower parts of the preparation. The amount of DBA that was removed with air abrasion and polishing was not uniform across the adhesive interface, and a great range of values was also recorded. There was no statistically significant difference in the amount of DBA removed between the two treatments (air abrasion and polishing) for both DBAs (OptiBond FL and Syntac Classic). Even though few differences were found that were related to the effect of air abrasion and polishing to different positions, polishing showed a tendency to remove more DBA from the top of the dentinal crest, while air abrasion tended to remove less DBA from the corners of the dentinal ridge of the preparation. Both treatments did not remove the entire thickness of the DBA in the majority of the cases, and overall, the precured adhesive was maintained despite cleaning with air abrasion or polishing. Nevertheless, emphasis should be given to the

fact that, in this study, a glycerine gel was used in order to polymerize the oxygen-inhibited layer; a step that, until proven otherwise, seems to be mandatory in order to avoid more exposure of dentin at later stages, during cleaning of the adhesive interface with either air abrasion or polishing. Taking into consideration all of the above, OptiBond FL treated with air abrasion seems to be more appropriate than Syntac for “immediate dentin sealing,” as it produced a more uniform thickness of DBA, which was also visibly detectable, a fact that made the evaluation of the DBA during placement easier, as well as after surface cleaning prior to final cementation.

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### References

- Aboush YE, Tareen A & Elderton RJ (1991) Resin-to-enamel bonds: Effect of cleaning the enamel surface with prophylaxis pastes containing fluoride or oil *British Dental Journal* **171**(7) 207-209.
- Bachmann M, Paul SJ, Luthy H & Scharer P (1997) Effect of cleaning dentine with soap and pumice on shear bond strength of dentine-bonding agents *Journal of Oral Rehabilitation* **24**(6) 433-438.
- Bertschinger C, Paul SJ, Luthy H & Scharer P (1996) Dual application of dentin bonding agents: Effect on bond strength *American Journal of Dentistry* **9**(3) 115-119.
- Dietschi D & Herzfeld D (1998) *In vitro* evaluation of marginal and internal adaptation of Class II resin composite restorations after thermal and occlusal stressing *European Journal of Oral Sciences* **106**(6) 1033-1042.
- Dietschi D, Magne P & Holz J (1995) Bonded to tooth ceramic restorations: *In vitro* evaluation of the efficiency and failure mode of two modern adhesives *Schweizerische Monatsschrift für Zahnmedizin* **105**(3) 299-305.
- Frankenberger R, Sindel J, Kramer N & Petschelt A (1999) Dentin bond strength and marginal adaptation: Direct composite resins vs ceramic inlays *Operative Dentistry* **24**(3) 147-155.
- Gerbo LR, Lacefield WR, Wells BR & Russell CM (1992) The effect of enamel preparation on the tensile bond strength of orthodontic composite resin *The Angle Orthodontist* **62**(4) 275-281; discussion 282.



- Hansen EK & Asmussen E (1987) Influence of temporary filling materials on the effect of dentin-bonding agents *Scandinavian Journal of Dental Research* **95(6)** 516-520.
- Magne P (2005) Immediate dentin sealing: A fundamental procedure for indirect bonded restorations *Journal of Esthetic and Restorative Dentistry* **17(3)** 144-155.
- Magne P & Belser U (2002a) Tooth preparation, impression and provisionalization. In: Bonded Porcelain Restorations in the Anterior Dentition—A Biomimetic Approach 1<sup>st</sup> Berlin Quintessence Publishing Co, Inc 270-271.
- Magne P & Belser U (2002b) Try-in and adhesive luting procedures. In: Bonded Porcelain Restorations in the Anterior Dentition—A Biomimetic Approach 1<sup>st</sup> Berlin Quintessence Publishing Co, Inc 358-363.
- Magne P & Douglas WH (1999) Porcelain veneers: Dentin bonding optimization and biomimetic recovery of the crown *The International Journal of Prosthodontic* **12(2)** 111-121.
- Maita E, Simpson MD, Tao L & Pashley DH (1991) Fluid and protein flux across the pulpodentine complex of the dog *in vivo* *Archives of Oral Biology* **36(2)** 103-110.
- Millstein PL & Nathanson D (1992) Effects of temporary cementation on permanent cement retention to composite resin cores *Journal of Prosthetic Dentistry* **67(6)** 856-859.
- Mitchem JC, Terkla LG & Gronas DG (1988) Bonding of resin dentin adhesives under simulated physiological conditions *Dental Materials* **4(6)** 351-353.
- Pashley DH, Nelson R, Williams EC & Kepler EE (1981) Use of dentine-fluid protein concentrations to measure pulp capillary reflection coefficients in dogs *Archives of Oral Biology* **26(9)** 703-706.
- Pashley EL, Comer RW, Simpson MD, Horner JA, Pashley DH & Caughman WF (1992) Dentin permeability: Sealing the dentin in crown preparations *Operative Dentistry* **17(1)** 13-20.
- Paul SJ (1997a) Effect of a dual application of dentin-bonding agents on shear bond strength of various adhesive luting systems on dentin. Adhesive luting procedures Berlin Quintessence Publishing Co, Inc 89-98.
- Paul SJ (1997b) Thickness of various dentin-bonding agents. Adhesive luting procedures Berlin Quintessence Publishing Co, Inc 67-74.
- Paul SJ & Scharer P (1997a) The dual bonding technique: A modified method to improve adhesive luting procedures *The International Journal of Periodontics & Restorative Dentistry* **17(6)** 536-545.
- Paul SJ & Scharer P (1997b) Effect of provisional cements on the bond strength of various adhesive bonding systems on dentine *Journal of Oral Rehabilitation* **24(1)** 8-14.
- Peter A, Paul SJ, Luthy H & Scharer P (1997) Film thickness of various dentine bonding agents *Journal of Oral Rehabilitation* **24(8)** 568-573.
- Raadal M (1978) Abrasive wear of filled and unfilled resins *in vitro* *Scandinavian Journal of Dental Research* **86(5)** 399-403.
- Rueggeberg FA & Margeson DH (1990) The effect of oxygen inhibition on an unfilled/filled composite system *Journal of Dental Research* **69(10)** 1652-1658.
- Tao L & Pashley DH (1989) Dentin perfusion effects on the shear bond strengths of bonding agents to dentin *Dental Materials* **5(3)** 181-184.
- Ulvestad H (1977) Hardness testing of some fissure-sealing materials *Scandinavian Journal of Dental Research* **85(7)** 557-560.
- Watson TF (1989) A confocal optical microscope study of the morphology of the tooth/restoration interface using Scotchbond 2 dentin adhesive *Journal of Dental Research* **68(6)** 1124-1131.

# Curing Efficacy of a New Generation High-power LED Lamp

AUJ Yap • MS Soh

## Clinical Relevance

New generation high-power LED lamps may cure composites as effectively as conventional LED/halogen in half the time.

## SUMMARY

This study investigated the curing efficacy of a new generation high-power LED lamp (Elipar Freelight 2 [N] 3M-ESPE). The effectiveness of composite cure with this new lamp was compared to conventional LED/halogen (Elipar Freelight [F], 3M-ESPE; Max [M], Dentsply-Caulk) and high-power halogen (Elipar Trilight [T], 3M-ESPE; Astralis 10 [A], Ivoclar Vivadent) lamps. Standard continuous (NS, FS, TS; MS), turbo (AT) and exponential (NE, FE, TE) curing modes of the various lights were examined. Curing efficacy of the various lights and modes were determined by measuring the top and bottom surface hardness of 2-mm thick composite specimens (Z100, 3M-ESPE) using a digital microhardness tester ( $n=5$ ; load=500 g; dwell time=15 seconds) one hour after light polymerization. The hardness ratio was computed by dividing HK (Knoop Hardness) of the bottom surface by HK

of the top surface. The data was analyzed using one-way ANOVA/Scheffe's test and Independent Samples *t*-test at significance level 0.05. Results of the statistical analysis were as follows: HK top—E, FE, NE > NS and NE > AT, TS, FS; HK bottom—TE, NE > NS; Hardness ratio—NS > FE and FS, TS > NE. No significant difference in HK bottom and hardness ratio was observed between the two modes of Freelight 2 and Max. Freelight 2 cured composites as effectively as conventional LED/halogen and high-power halogen lamps, even with a 50% reduction in cure time. The exponential modes of Freelight 2, Freelight and Trilight appear to be more effective than their respective standard modes.

## INTRODUCTION

Light-activated resin composites have revolutionized the practice of clinical dentistry by maximizing working time and reducing setting time. Camphoroquinone (CQ), which has an absorption spectrum between 450 and 500 nm (Cook, 1982; Lee & others, 1993), is the most commonly used photoinitiator in light-activated composites. Traditionally, halogen-based curing lamps have been used to activate the photoinitiator system, which will then start the resin polymerization reaction. Halogen bulbs generate light by the electrical heating of a small tungsten thread to extremely high temperatures. As such, mostly heat radiation, which is in the infrared range of the electromagnetic spectrum, is generated (Althoff & Hartung, 2000). Only a few percent of

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the light output is in the visible part, including the blue range desired for polymerization. The latter is usually obtained by the use of filters. One shortcoming of halogen curing lamps is that their optical power and spectral content degrade over time, often due to the aging of bulbs and filters. The aforementioned is accelerated by high operating temperatures and the large quantity of heat produced during curing cycles (Jandt & others, 2000). The drop in optical power, which is compounded by the failure of clinicians to adequately maintain and use curing lights according to manufacturer's directions, adversely affects the polymerization process, resulting in restoration failures and adverse pulpal responses to unpolymerized monomers (Asmussen, 1982a; Caughman & others, 1991). A number of clinical studies have shown that many halogen lamps used by clinicians do not reach the minimum power output specified by manufacturers (Martin, 1998; Miyazaki & others, 1998).

LED (Light-emitting Diode) curing lamps were introduced to overcome the limitations of halogen-based lights. They are solid-state semiconductor devices that convert electrical energy directly to light. LED bulbs have lifetimes of more than 10,000 hours and undergo little degradation of light output during that time. This represents a distinct advantage when compared to halogen bulbs, which have effective lifetimes of less than 100 hours (Rueggeberg & others, 1996). In addition, LED lamps do not require cooling fans and filters to produce blue light. Their relatively low power consumption also makes them suitable for portable use. The light generated by gallium nitride LEDs results in high efficacy, as most of the energy radiated falls within the absorption spectrum of the CQ photoinitiator (Mills, Jandt & Ashworth, 1999). Most commercially available LED curing lamps have pre-set exponential (soft-start) curing regimens incorporated. These curing regimens, involving pre-polymerization at low light intensity, followed by final cure at high intensity, were designed to minimize shrinkage during composite polymerization. Studies have shown that exponential curing results in smaller marginal gap, increased marginal integrity and improved material properties (Uno & Asmussen, 1991; Mehl,

Hickel & Kunzelmann, 1997). The modulus, strength and depth of cure of composites polymerized with LED lights were found to be equivalent to those cured with conventional halogen lamps (Mills & others, 1999; Stahl & others, 2000; Jandt & others, 2000). Several studies (Kurachi & others, 2001; Dunn & Bush, 2002; Leonard & others, 2002; Soh, Yap & Siow, 2003) have, however, reported that LED lamps may not be as effective as halogen lights when used for curing composites.

LED technology has advanced significantly since the above studies were carried out. To overcome the inadequacies of the first generation LED-based curing lamps and to reduce curing time, high-power LED lamps have been developed. Similar irradiances to conventional halogen lights can now be achieved with these new lamps (Mills & others, 2002). In some devices, the high irradiance can be achieved with a single LED instead of multiple LED arrays. This study investigated the curing efficacy of a new generation high-power single LED lamp (Elipar Freelight 2 [N], 3M-ESPE). The effectiveness of composite cure associated with this new lamp was compared to conventional LED/halogen (Elipar Freelight [F], 3M-ESPE; Max [M], Dentsply-Caulk) and high-power halogen (Elipar Trilight [T], 3M-ESPE; Astralis 10 [A], Ivoclar Vivadent) lamps. For lamps that offer exponential curing regimens (T, F and N), the curing efficacy between standard (S) continuous and exponential (E) modes were also compared.

## METHODS AND MATERIALS

The five curing lamps and their curing regimens are detailed in Table 1. Max, the conventional continuous

Table 1: Details of the Curing Lights and the Various Curing Modes Evaluated

LCU	Curing Modes	Curing Profiles
Elipar Freelight 2 (LED)	Standard (NS)	1000 mW/cm <sup>2</sup> (20 seconds)
3M-ESPE Seefeld, Germany	Exponential (NE)	0-1000 mW/cm <sup>2</sup> (5 seconds) → 1000 mW/cm <sup>2</sup> (15 seconds)
Elipar Freelight (LED)	Standard (FS)	400 mW/cm <sup>2</sup> (40 seconds)
3M-ESPE Seefeld, Germany	Exponential (FE)	0-400 mW/cm <sup>2</sup> (12 seconds) → 400 mW/cm <sup>2</sup> (28 seconds)
Max (Halogen) Dentsply-Caulk Milford, DE, USA	Standard (MS)	400 mW/cm <sup>2</sup> (40 seconds)
Astralis 10 (Halogen) Ivoclar-Vivadent Schaan, Liechtenstein	High Power (AT)	1200 mW/cm <sup>2</sup> (10 seconds)
Elipar TriLight (Halogen)	Standard (TS)	800 mW/cm <sup>2</sup> (40 seconds)
3M-ESPE Seefeld, Germany	Exponential (TE)	100-800 mW/cm <sup>2</sup> (15 seconds) → 800 mW/cm <sup>2</sup> (25 seconds)

Curing profiles are based on manufacturers' data.



cure halogen lamp, was used as the comparison light source. A minifill composite (Z100, 3M-ESPE, St Paul, MN, USA) of A2 shade was selected for the study. The composite material was placed into the square recesses (4 mm long, 4 mm wide and 2 mm deep) of black delrin molds and confined between two opposing acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide (1 mm thick) was then placed on the molds and excess material was extruded by applying pressure. The composite was then irradiated from the top through the glass slide and acetate strip with the different curing lamps and modes. The acetate strips were removed and the specimens were held in air at room temperature ( $25 \pm 2^\circ\text{C}$ ) for one hour prior to microhardness testing.

The Knoop's Hardness (HK) of the top and bottom surfaces was assessed with a digital microhardness tester (FM7, Future-Tech Corp, Tokyo, Japan). The specimens, in their molds, were positioned centrally beneath the indenter of the hardness tester. A 500g load was then applied through the indenter, with a dwell time of 15 seconds. HK corresponding to each indentation was computed by measuring the dimensions of the indentations and using the formula  $\text{HK (kgf/mm}^2\text{)} = 14.23 \times (F/d^2)$ , where F is the test load in kilograms and d is the diagonal length of an indentation in millimeters. Five specimens were made for each curing light-mode combination. The mean HK and hardness ratio of the five specimens were then calculated and tabulated using the following formula: Hardness ratio = HK of bottom surface/HK of top surface. Differences in HK top, bottom and hardness ratio between MS/FS/FE and the other curing lights/ modes were analyzed using one-way ANOVA and Scheffe's post-hoc test. For lamps pre-set with two curing regimens, Independent Sample's *t*-test was used to determine significant differences in HK data between standard and exponential modes. All statistical analyses were conducted at significance level 0.05.

## RESULTS

The mean HK for the various curing lights and modes is shown in Table 2, while mean hardness ratios are

Table 2: Mean (SD) HK for the Various Curing Lights and Modes

Curing Lamp	Mode	HK Top (kgf/mm <sup>2</sup> )	HK Bottom (kgf/mm <sup>2</sup> )
Elipar Freelight 2	Standard (NS)	75.00 (1.41)	73.44 (1.93)
	Exponential (NE)	82.94 (2.25)	78.30 (1.34)
Elipar Freelight	Standard (FS)	75.42 (0.81)	74.58 (0.53)
	Exponential (FE)	81.10 (0.65)	73.68 (0.76)
Max	Standard (MS)	79.60 (2.02)	75.90 (4.28)
Astralis 10	Turbo (AT)	77.32 (1.00)	75.54 (1.09)
Elipar Trilight	Standard (TS)	78.20 (0.59)	77.86 (0.57)
	Exponential (TE)	81.90 (0.72)	78.38 (0.36)

Table 3: Comparison of Hardness Data Between Max/Standard and Exponential Modes of Freelight 2 and the Other Curing Lights/Modes

Curing lights/modes	Variables	Differences
Freelight 2/ Standard (NS)	HK top	TE, FE & NE > NS
	HK bottom	TE & NE > NS
	Hardness ratio	NS > FE
Freelight 2/ Exponential (NE)	HK top	NE > AT, TS, FS & NS
	HK bottom	NE > NS
	Hardness ratio	FS, TS > NE
Max /Standard (MS)	HK top	MS > FS & NS
	HK bottom	No significant difference
	Hardness ratio	TS > MS > FE

> denotes significant differences in HK values or hardness ratio. Results of one-way ANOVA/Scheffe's post-hoc tests ( $p < 0.05$ ).

Table 4: Comparison of Hardness Data Between Standard and Exponential Modes

Curing Lights/Modes	Variables	Differences
Freelight 2	HK top	Exponential > Standard
	HK bottom	Exponential > Standard
	Hardness ratio	Standard > Exponential
Freelight	HK top	Exponential > Standard
	HK bottom	No significant difference
	Hardness ratio	Standard > Exponential
Trilight	HK top	Exponential > Standard
	HK bottom	No significant difference
	Hardness ratio	Standard > Exponential

> denotes significant differences in HK values or hardness ratio. Results of Independent Samples *t*-test ( $p < 0.05$ ).

reflected in Figure 1. Results of statistical analysis are shown in Tables 3 and 4.

For both HK top and HK bottom, the lowest values were observed for NS. Mean HK top ranged from 75.00 to 82.94 for NS and NE, respectively. Mean HK bottom ranged from 73.44 to 78.38 for NS and TE, respectively. Hardness ratios ranged from 0.91 to 1.00 for FE and TS. At the top surfaces, HK values observed with TE, FE and NE were significantly greater than with NS. At the bottom surfaces, curing with TE and NE resulted in significantly higher values than NS. The top surface of composites cured with NE was significantly harder than those cured with AT, TS, FS and NS. The HK bottom of NE was significantly greater than NS.

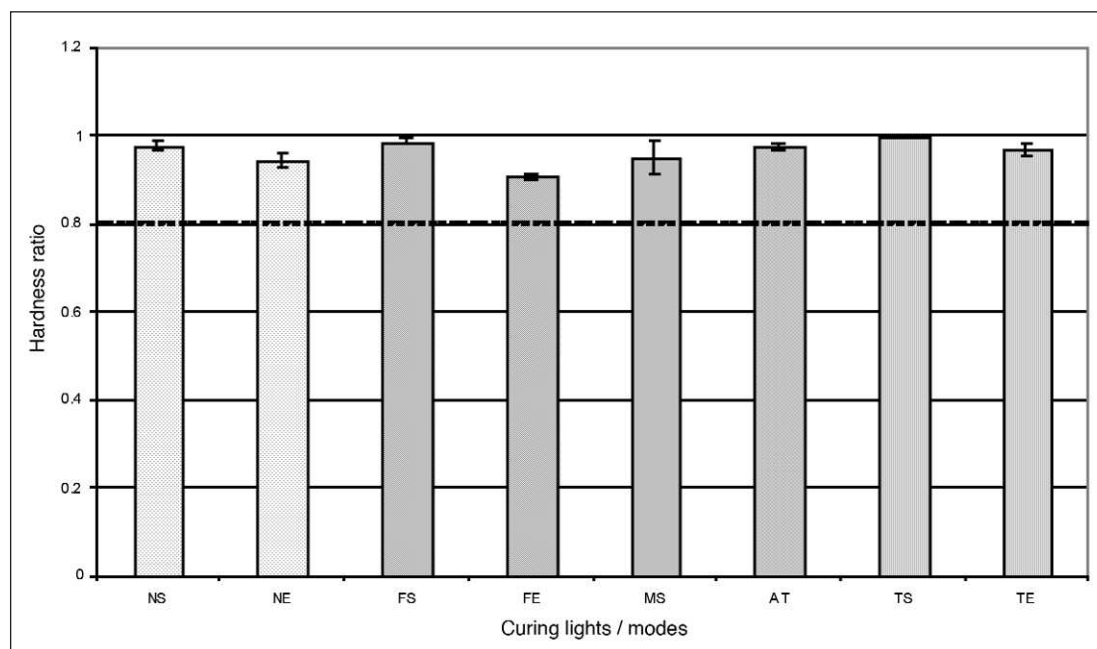


Figure 1. Mean hardness ratio of the various curing lights and modes.

Significant differences in hardness ratios for NS and NE are shown in Table 3.

Although no significant difference in HK bottom was observed between MS and the other curing lights/modes, significant differences in HK top and hardness ratios were observed. The HK top of MS was significantly greater than that of FS and NS. The hardness ratio of MS was significantly lower than TS but greater than FE. For Freelight 2, Freelight and Trilight, curing with the exponential mode resulted in significantly harder top surfaces and lower hardness ratio. Although no significant difference in HK bottom was observed between standard and exponential modes for Trilight and Freelight, a significant difference was observed for Freelight 2. For the latter, curing with the exponential mode resulted in significantly harder bottom surfaces than curing with the standard mode.

## DISCUSSION

Curing efficacy can be measured by direct and indirect methods. Direct methods that assess the degree of conversion, such as infrared spectroscopy and laser Raman spectroscopy, are complex, expensive and time-consuming (Rueggeberg & Craig, 1988). These techniques are also more qualitative than quantitative in nature. Indirect methods have included visual, scrape and hardness testing. The hardness testing method of determining curing efficacy is widely used (Dunn & Bush, 2002; Mills & others, 2002; Soh & others, 2003) and was selected for this study due to its relative simplicity and efficiency. In addition, the physical properties of composites are clinically important. DeWald and Ferracane

(1987) compared four commonly used methods for evaluating composite cure. They found that visual and scraping methods correlated well but severely overestimated depth of cure as compared to hardness testing or degree of conversion. The correlation between Knoop Hardness and degree of conversion was found to be good (Asmussen, 1982a,b; DeWald & Ferracane, 1987). To minimize the effects of colorants and cure-tip distance on polymerization (Bayne, Heymann & Swift, 1994; Denehy

& others, 1993), A2 shade composite and a 1-mm glass spacer was used. Two millimeter thick composite specimens were evaluated, as they gave maximum and uniform polymerization (Yap, 2000). Hardness readings were taken at one hour intervals, as composite reaction and shrinkage has been shown to continue after removal of the light source and is greatest at one hour (Pilo & Cardash, 1992; Yap & others, 2000). Max was selected as the comparison light source for all curing lights/modes, as it had an intensity of approximately 400 mW/cm<sup>2</sup>, which has been suggested for routine curing (Rueggeberg, Caughman & Curtis, 1994; Tate, Porter & Dosch, 1999).

Several studies have found that the top surface hardness of composites is less dependent on light intensity than bottom surface hardness (Denehy & others, 1993; Fowler, Swartz & Moore, 1994). Despite this, significant differences in HK top were still observed between Max and the standard modes of both LED lights. As the hardness of a material is related to properties such as strength, proportional limit, ductility and wear resistances (Anusavice, 1996), curing with the standard modes of both LED lights may compromise the clinical longevity of restorations. When the standard modes of these LED lights are used, longer exposure duration times (cure time) are recommended (Leonard & others, 2002). No significant difference in HK top values was observed between Max and the exponential modes of both Freelight and Freelight 2. In view of this and the lack of statistical difference in HK bottom between Max and the exponential modes of both LED lights, the clinical use of the exponential modes of Freelight and

Freelight 2 is advocated.

In contrast to Freelight, which has an array of 19 LEDs, Freelight 2 uses a single high-intensity LED for light generation. This high-intensity LED uses a substantially larger semi-conductor, which increases both the illuminated area and light intensity, enabling a 50% reduction in cure time. Despite the reduction in cure time, the HK top of Freelight 2 was significantly higher than that of most curing lights/modes with the exception of MS, TE and FE. This may be attributed, in part, to the emission spectra of Freelight 2 and its high light energy density (up to 20 J/cm<sup>2</sup>). When the light intensities of LED lamps were measured with a commercial radiometer (Cure Rite, EFOS Inc, Ontario, Canada), readings of 260 ± 3 and 1055 ± 7 mW/cm<sup>2</sup> were observed for Freelight and Freelight 2, respectively. The lower irradiance of Freelight could be accounted for by the narrower emission spectra when compared to that of halogen lamps. In the case of Freelight 2, irradiance remained very high in spite of the narrow emission spectra, attesting to the power of this new LED. Heat development occurs in the single LED due to high irradiance. Although the thermal emission of LED lamps is significantly lower than their halogen counterparts (Yap & Soh, 2003), the dissipation of heat is still crucial, as it may affect LED durability. Heat is dissipated by a heat sink of highly thermally conductive aluminum integrated into the housing. Other prototype high-power LED lamps with multiple arrays have been evaluated (Mills & others, 2002). The performance of these new lights was also comparable to that of halogen lamps and offers the advantage of a 50% reduction in cure time.

The HK top of the exponential mode of Freelight 2 was significantly greater than its standard mode. Similar trends were also observed between the exponential and standard modes of Freelight and Trilight. Although significant differences in HK bottom were observed between the exponential and standard modes of Freelight 2, HK values for bottom surfaces were not statistically different between the two curing regimens for Freelight and Trilight. Observations from these three lamps suggest that the curing efficacy of the exponential modes was generally superior to that of standard continuous mode. For 2-mm thick composite specimens, light intensity and exposure time are the two most important factors influencing composite cure (Rueggeberg & others, 1993). The total light energy density (intensity x time) of standard modes is theoretically greater than the exponential modes based on manufacturers' curing profiles (Table 1). Greater HK values are therefore expected with standard modes. Contradictory findings were observed in a recent paper by Soh and others (2003). While the use of the exponential mode of Trilight resulted in significantly harder top and bottom surfaces compared to the standard

mode, HK values for the exponential mode of Freelight was similar or lower than its standard mode. The difference between this and the current study may be attributed to composite post-cure (Pilo & Cardash, 1992; Tarumi & others, 1999) arising from the time difference prior to hardness testing. Hardness measurements were made one hour after removal of the light source in the current study as compared to immediately for Soh and others (2003). Exponential curing was developed to slow down the polymerization reaction (Davidson & Feilzer, 1997), and this may also be transferred to the post-irradiation polymerization of composites. Although Mehl and others (1997) found no significant difference in composite hardness between standard and exponential modes, they reported increased flexural modulus and strength with exponential curing. Reasons for the improved physical properties associated with exponential curing cannot be ascertained by the current study, and the associated polymerization kinetics warrants further investigations.

The hardness ratio should be 1 if polymerization is completely effective, as the hardness of the bottom surface should be the same as the top surface. However, as light passes through the bulk of a composite, light intensity is reduced due to light scattering by the resin matrix and filler particles (Ruyter & Øysæd, 1982). Pilo and Cardash (1992) suggested that the hardness gradient should not exceed 10% to 20% (hardness ratio of 0.8 or greater) for light-activated composites to be adequately polymerized. As all curing light-mode combinations fulfilled this criterion (Figure 1), statistically significant differences in hardness ratio may not be imperative clinically. The significantly lower hardness ratios observed with the exponential modes of Freelight and Freelight 2 (Table 3) are contributed to by the higher HK top achieved with the exponential modes of these LED lamps.

## CONCLUSIONS

Under the conditions of this *in vitro* study:

1. The curing efficacy of the high-power single LED lamp was generally comparable to that of conventional LED/halogen and high-power halogen lamps even with a 50% reduction in cure time.
2. The exponential modes of LED and halogen lamps were more effective than their standard modes.

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## References

- Althoff O & Hartung M (2000) Advances in light curing  
*American Journal of Dentistry* **13** 77D-81D.



- Anusavice KL (1996) Mechanical properties of dental materials in *Phillip's Science of Dental Materials* 10<sup>th</sup> edition Philadelphia WB Saunders Co p 69.
- Asmussen E (1982a) Restorative resins: Hardness and strength vs quantity of remaining double bonds *Scandinavian Journal of Dental Research* **90(6)** 484-489.
- Asmussen E (1982b) Factors affecting the quantity of remaining double bonds in restorative resin polymers *Scandinavian Journal of Dental Research* **90(6)** 490-496.
- Bayne SC, Heymann HO & Swift EJ Jr (1994) Update on dental composite restorations *Journal of the American Dental Association* **125(6)** 687-701.
- Caughman WF, Caughman GB, Shiflett RA, Rueggeberg & Schuster GS (1991) Correlation of cytotoxicity, filler loading and curing time of dental composites *Biomaterials* **12(8)** 737-740.
- Cook WD (1982) Spectral distribution of dental photopolymerization sources *Journal of Dental Research* **61(12)** 1436-1438.
- Davidson CL & Feilzer AJ (1997) Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives *Journal of Dentistry* **25(6)** 435-440.
- Denehy GE, Pires JAF, Cuitko E & Swift EJ Jr (1993) Effects of curing tip distance on light intensity and composite resin microhardness *Quintessence International* **24(7)** 517-521.
- DeWald JP & Ferracane JL (1987) A comparison of four modes of evaluating depth of cure of light-activated composites *Journal of Dental Research* **66(3)** 727-730.
- Dunn WJ & Bush A (2002) A comparison of polymerization by light-emitting diode and halogen-based light-curing units *Journal of the American Dental Association* **133(3)** 335-341.
- Fowler CS, Swartz ML & Moore BK (1994) Efficacy testing of visible-light-curing units *Operative Dentistry* **19(2)** 47-52.
- Jandt KD, Mills RW, Blackwell GB & Ashworth SH (2000) Depth of cure and compressive strength of dental composites cured with blue light emitting diodes (LEDs) *Dental Materials* **16(1)** 1379-1385.
- Kurachi C, Tuboy AM, Magalhaes DV & Baganto VS (2001) Hardness evaluation of a dental composite polymerized with experimental LED-based devices *Dental Materials* **17(4)** 309-315.
- Lee SY, Chiu CH, Boghosian A & Greener EH (1993) Radiometric and spectroradiometric comparison of power outputs of five visible light-curing units *Journal of Dentistry* **21(6)** 373-377.
- Leonard DL, Charlton DG, Roberts HW & Cohen ME (2002) Polymerization efficiency of LED curing lights *Esthetic Dentistry* **14(5)** 286-295.
- Martin FE (1998) A survey of the efficiency of visible light curing units *Journal of Dentistry* **26(3)** 239-243.
- Mehl A, Hickel R & Kunzelmann KH (1997) Physical properties and gap formation of light-cured composite with and without soft-start polymerization *Journal of Dentistry* **25(3-4)** 321-330.
- Mills RW, Jandt KD & Ashworth SH (1999) Dental composite depth of cure with halogen and blue light emitting diode technology *British Dental Journal* **186(8)** 388-391.
- Mills RW, Uhl A, Blackwell GB & Jandt KD (2002) High power light emitting diode (LED) arrays versus halogen light polymerization of oral biomaterials: Barcol hardness, compressive strength and radiometric properties *Biomaterials* **23(14)** 2955-2963.
- Miyazaki M, Hattori T, Ichiishi Y, Kondo M, Onsoe H & Moore BK (1998) Evaluation of curing units used in private dental offices *Operative Dentistry* **23(2)** 50-54.
- Pilo R & Cardash HS (1992) Post-irradiation polymerization of different anterior and posterior visible light-activated resin composites *Dental Materials* **8(5)** 299-304.
- Rueggeberg FA, Caughman WF, Curtis JW & Davis HC (1993) Factors affecting cure at depths within light-activated resin composites *American Journal of Dentistry* **6(2)** 91-95.
- Rueggeberg FA, Caughman WF & Curtis JW (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19(1)** 26-32.
- Rueggeberg FA & Craig RG (1988) Correlation of parameters used to estimate monomer conversion in a light-cured composite *Journal of Dental Research* **67(6)** 932-937.
- Rueggeberg FA, Twigg SW, Caughman WF & Khajotia S (1996) Lifetime intensity profiles of 11 light-curing units *Journal of Dental Research* **75(SI)** Abstract #2897 p 380.
- Ruyter IE & Øysæd H (1982) Conversion in different depths of ultraviolet and visible light activated composite materials *Acta Odontologica Scandinavica* **40(3)** 179-192.
- Soh MS, Yap AU & Siow KS (2003) Effectiveness of composite cure associated with different curing modes of LED lights *Operative Dentistry* **28(4)** 371-377.
- Stahl F, Ashworth SH, Jandt KD & Mills RW (2000) Light-emitting diode (LED) polymerization of dental composites: Flexural properties and polymerization potential *Biomaterials* **21(13)** 1379-1385.
- Tarumi H, Imazato S, Ehara A, Kato S, Ebi N & Ebisu S (1999) Post-irradiation polymerization of composites containing BisGMA and TEGDMA *Dental Materials* **15(4)** 238-242.
- Tate WH, Porter KH & Dosch RO (1999) Successful photocuring: Don't restore without it *Operative Dentistry* **24(2)** 109-114.
- Uno S & Asmussen E (1991) Marginal adaptation of a restorative resin polymerized at reduced rate *Scandinavian Journal of Dental Research* **99(5)** 440-444.
- Yap AU (2000) Effectiveness of polymerization in composite restorative claiming bulk placement: Impact of cavity depth and exposure time *Operative Dentistry* **25(2)** 113-120.
- Yap AU & Soh MS (2003) Thermal emission by different light-curing units *Operative Dentistry* **28(3)** 260-266.
- Yap AU, Wang HB, Siow KS & Gan LM (2000) Polymerization shrinkage of visible-light-cured composites *Operative Dentistry* **25(2)** 98-103.

# Curing of Pit & Fissure Sealants Using Light Emitting Diode Curing Units

JA Platt • H Clark • BK Moore

## Clinical Relevance

Adequate polymerization of opaque light-activated sealants should not be assumed and is dependent upon the material and light-curing unit.

## SUMMARY

Light Emitting Diode (LED) curing units are attractive to clinicians, because most are cordless and should create less heat within tooth structure. However, questions about polymerization efficacy have surrounded this technology. This research evaluated the adequacy of the depth of cure of pit & fissure sealants provided by LED curing units. Optilux (OP) and Elipar Highlight (HL) high intensity halogen and Astralis 5 (A5) conventional halogen lights were used for comparison. The Light Emitting Diode (LED) curing units were Allegro (AL), LE Demetron I (DM), FreeLight (FL), UltraLume

2(UL), UltraLume 5 (UL5) and VersaLux (VX). Sealants used in the study were UltraSeal XT plus Clear (Uclr), Opaque (Uopq) and Teethmate F-1 Natural (Kclr) and Opaque (Kopq). Specimens were fabricated in a brass mold (2 mm thick x 6 mm diameter) and placed between two glass slides (n=5). Each specimen was cured from the top surface only. One hour after curing, four Knoop Hardness readings were made for each top and bottom surface at least 1 mm from the edge. The bottom to top (B/T) KHN ratio was calculated. Groups were fabricated with 20 and 40-second exposure times. In addition, a group using a 1 mm-thick mold was fabricated using an exposure time of 20 seconds. Differences between lights for each material at each testing condition were determined using one-way ANOVA and Student-Newman-Keuls Post-hoc test ( $\alpha=0.05$ ). There was no statistical difference between light curing units for Uclr cured in a 1-mm thickness for 20 seconds or cured in a 2 mm-thickness for 40 seconds. All other materials and conditions showed differences between light curing units. Both opaque materials showed significant variations in B/T KHN ratios dependent upon the light-curing unit.

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## INTRODUCTION

The use of pit & fissure sealants is recognized as an important component of preventive dentistry (Weintraub, 1989; Wendt, Koch & Birkhed, 2001). These sealants are also utilized as part of preventive resin restorations (Simonsen, 1978) and, as such, are an important material for the dental practitioner. The presence of an intact sealant or sealed restoration may decrease the need for, or delay the onset of, a more invasive restoration (Mertz-Fairhurst & others, 1998). This effect is dependent upon the presence of the sealant. Therefore, issues that impact the retention of resin-based sealants are important when selecting a sealant for use.

Partial loss of the sealant may be associated with decreased effectiveness (Weintraub, 1989). Because visualizing a partial loss with clear resins can be difficult, opaque materials have been developed in an effort to enhance detection (Rock & others, 1989). These materials have opacifiers, such as titanium oxide, added to the resin-matrix. Also, clinicians seem to favor light-activated materials that provide a rapid initiation of polymerization. This rapid initiation may aid ease of placement, particularly in the pediatric population. It has long been recognized that the shade of a resin has a significant impact on the polymerization of a resin composite (Kanca, 1986; Shortall, Wilson & Harrington, 1995). By their nature, opacifiers would be expected to cause significant amounts of light

Table 1: *Light-activating Units Used*

Curing Units	Manufacturer	Type	Power Density (mW/cm <sup>2</sup> )
Highlight (HL)	3M ESPE, St Paul, MN, USA	QTH	764
Optilux (OP)	SDS Kerr, Danbury, CT, USA	QTH	894
Astralix 5 (A5)	Ivoclar-Vivadent, Liechtenstein	QTH	504
LE Demetron I (DM)	SDS Kerr, Danbury, CT, USA	LED	740
Allegro (AL)	Den-Mat, Santa Maria, CA, USA	LED	1033
UltraLume 5 (UL5)	Ultradent Products, South Jordan, UT, USA	LED	793
UltraLume 2 (UL)	Ultradent Products, South Jordan, UT, USA	LED	589
FreeLight (FL)	3M ESPE, St Paul, MN, USA	LED	273
Versalux (VX)	Centrix, Shelton, CT, USA	LED	142

Table 2: *Sealant Materials Tested*

Sealant	Manufacturer	Lot #
UltraSeal XT Clear (Uclr)	Ultradent	4W44
UltraSeal XT Opaque (Uopq)	Ultradent	4VYC
Teethmate F-1 Natural (Kclr)	Kuraray	067BA
Teethmate F-1 Opaque (Kopq)	Kuraray	187BA

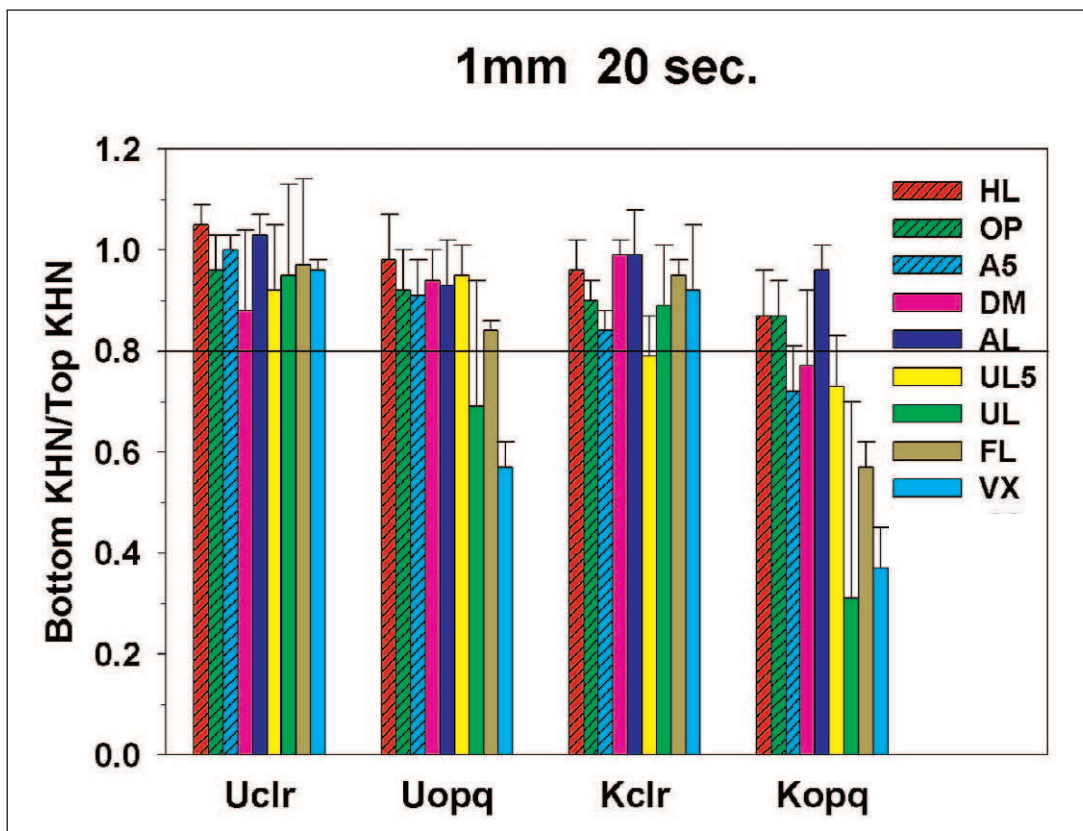


Figure 1: 1-mm thick—20 second cure: bottom/top KHN. (n=5)

reflection, scattering and absorption. They should decrease the amount of light energy that penetrates through the bulk of the resin. Therefore, the opacity of



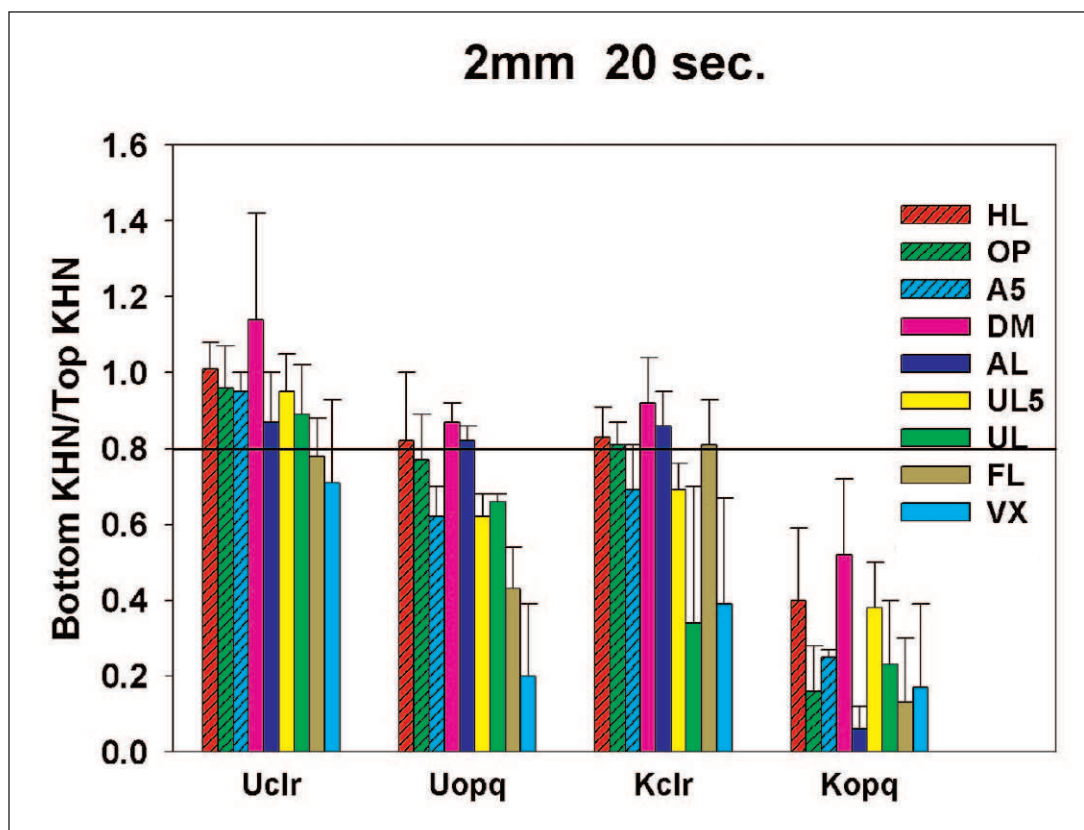


Figure 2: 2-mm thick—20 second cure: bottom/top KHN. (n=5)

some currently marketed pit & fissure sealants should be expected to have a similar impact on the polymerization process.

The clinician has the choice of several light sources for the activation of light-activated sealants, including quartz-tungsten-halogen (QTH), Light-Emitting Diode (LED), plasma arc and laser units. There have been reports on the impact of LED light-activation units on the polymerization of restorative resin composites (Besnault & others, 2003; Price & others, 2003; Uhl, Sigusch & Jandt, 2004). Higher degrees of conversion in a resin system provide increased mechanical properties that, in turn, should provide increased longevity of the restoration. Ferracane (1985) demonstrated that Knoop Hardness Numbers (KHN) predict the relative degree of conversion for a specific resin under variable conditions. Because the impact of the light source on the sealant's degree of conversion is less well known, this study investigated this relationship using bottom/top KHN ratios (B/T KHN). The hypothesis tested was that B/T KHN for light-activated pit & fissure sealants is not affected by the light-curing unit used.

#### METHODS AND MATERIALS

Nine different light curing units (Table 1) were used. Irradiance for the activation lights was measured using a USB2000 Spectrometer with a FOIS-1 integrating

sphere (Ocean Optics Inc, Dunedin, FL, USA). Calibration of the spectrophotometer in absolute spectral irradiance units was conducted with an LS-1-CAL-INT NIST traceable light source using OOIrrad-C software, also from Ocean Optics. The power spectrum was integrated from 380-520 nm to obtain intensity values in mW/cm<sup>2</sup>. Four different sealant materials (Table 2) were investigated using three curing conditions for each combination of light/material: 1-mm thickness—20-second cure, 2-mm thickness—20-second cure and 2-mm thickness—40-second cure.

Each specimen was fabricated in a brass mold of appropriate thickness, with a 6 mm internal diameter. The mold was placed between two glass slides and on top of a white background. Each specimen was cured for the appropriate amount of time from the top surface only. The specimens were stored in the dark under 100% relative humidity and 37°C for one hour. Four Knoop Hardness readings were made at least 1 mm from the edge of each top and bottom surface after an indenter dwell time of 15 seconds and a load of either 10 or 25 grams (M-400 Hardness Tester, LECO Corp, St Joseph, MI, USA). A B/T KHN ratio was calculated for each specimen. Top surface and B/T KHN means were determined for each group (n=5). Statistical analysis was performed using one-way ANOVA and Student-Newman-Keuls Post-hoc tests ( $\alpha=0.05$ ) for each material in each of the three testing conditions. The independent variable was the light-curing unit. In addition, a linear regression followed by an ANOVA for the regression was performed for each material in each testing condition ( $\alpha=0.05$ ).

#### RESULTS

The B/T KHN ratios are given in Tables 3, 4, 5 and Figures 1, 2 and 3. Uclr showed no significant difference between lights when curing 1 mm for 20 seconds or 2 mm for 40 seconds. There were differences when curing

Table 3: 1-mm Thick—20 Second Cure: Bottom/Top KHN. Vertical Means with the Same Letters Are Not Significantly Different (n=5)

Curing Units	Uclr (sd)	Uopq (sd)	Kclr (sd)	Kopq (sd)
HL	1.05 (0.04)a	0.98 (0.09)a	0.96 (0.06)a	0.87 (0.09)a
OP	0.96 (0.07)a	0.92 (0.08)a	0.90 (0.04)ab	0.87 (0.07)a
A5	1.00 (0.03)a	0.91 (0.07)a	0.84 (0.04)ab	0.72 (0.09)ab
DM	0.88 (0.16)a	0.94 (0.06)a	0.99 (0.03)a	0.77 (0.15)ab
AL	1.03 (0.04)a	0.93 (0.09)a	0.99 (0.09)a	0.96 (0.05)a
UL5	0.92 (0.13)a	0.95 (0.06)a	0.79 (0.08)b	0.73 (0.10)ab
UL	0.95 (0.18)a	0.69 (0.25)b	0.89 (0.12)ab	0.31 (0.39) d
FL	0.97 (0.17)a	0.84 (0.02)a	0.95 (0.03)a	0.57 (0.05) b,c
VX	0.96 (0.02)a	0.57 (0.05)b	0.92 (0.13)ab	0.37 (0.08) cd

Table 4: 2-mm Thick—20 Second Cure: Bottom/Top KHN. Vertical Means with the Same Letters Are Not Significantly Different (n=5)

Curing Units	Uclr (sd)	Uopq (sd)	Kclr (sd)	Kopq (sd)
HL	1.01 (0.07)ab	0.82 (0.18)ab	0.83 (0.08)a	0.40 (0.19)ab
OP	0.96 (0.11)abc	0.77 (0.12)ab	0.81 (0.06)a	0.16 (0.12) bc
A5	0.95 (0.05)abc	0.62 (0.08) b	0.69 (0.12)a	0.25 (0.02) bc
DM	1.14 (0.28)a	0.87 (0.05)a	0.92 (0.12)a	0.52 (0.20)a
AL	0.87 (0.13)abc	0.82 (0.04)ab	0.86 (0.09)a	0.06 (0.06) c
UL5	0.95 (0.10)abc	0.62 (0.06) b	0.69 (0.07)a	0.38 (0.12)ab
UL	0.89 (0.13)abc	0.66 (0.02) b	0.34 (0.36) b	0.23 (0.17) bc
FL	0.78 (0.10) bc	0.43 (0.11) c	0.81 (0.12)a	0.13 (0.17) bc
VX	0.71 (0.22) c	0.20 (0.19) d	0.39 (0.28) b	0.17 (0.22) bc

Table 5: 2-mm Thick—40 Second Cure: Bottom/Top KHN. Vertical Means with the Same Letters Are Not Significantly Different (n=5)

Curing Units	Uclr (sd)	Uopq (sd)	Kclr (sd)	Kopq (sd)
HL	1.04 (0.06)a	0.88 (0.08)a	0.81 (0.05) c	0.59 (0.06)ab
OP	0.97 (0.06)a	0.87 (0.12)a	0.88 (0.08) bc	0.59 (0.06)ab
A5	0.98 (0.06)a	0.81 (0.09)a	0.77 (0.05) c	0.41 (0.10)ab
DM	0.93 (0.04)a	0.93 (0.07)a	0.96 (0.06)ab	0.65 (0.11)a
AL	0.88 (0.27)a	0.95 (0.05)a	1.00 (0.05)a	0.45 (0.35)ab
UL5	0.87 (0.12)a	0.78 (0.12)a	0.77 (0.06) c	0.64 (0.07)a
UL	1.01 (0.03)a	0.79 (0.10)a	0.84 (0.11) c	0.50 (0.16)ab
FL	0.83 (0.14)a	0.57 (0.06) b	0.80 (0.09) c	0.31 (0.04) b
VX	0.84 (0.07)a	0.24 (0.10) c	0.81 (0.05) c	0.31 (0.07) b

Table 6: 1-mm Thick—20 Second Cure: Top KHN. Vertical Means with the Same Letters Are Not Significantly Different (n=5)

Curing Units	Uclr (sd)	Uopq (sd)	Kclr (sd)	Kopq (sd)
HL	29.9 (2.0)ab	27.6 (1.9) bc	17.9 (0.4)a	18.3 (0.6)a
OP	30.9 (0.9)a	29.4 (1.0)ab	17.4 (0.7)a	17.9 (0.7)a
A5	31.7 (1.0)a	29.4 (1.8)ab	18.8 (0.1)a	18.3 (0.6)a
DM	29.3 (3.2)ab	26.8 (1.2) bc	14.1 (0.6) b	14.0 (1.0) b
AL	28.8 (2.6)ab	26.8 (1.8) bc	14.5 (1.1) b	14.3 (1.2) b
UL5	31.6 (1.3)a	30.6 (1.7)a	17.9 (1.0)a	17.5 (0.5)a
UL	28.3 (2.3)ab	27.5 (1.9) bc	14.3 (2.1) b	14.9 (0.9) b
FL	26.2 (2.7) b	26.2 (1.3) c	13.1 (0.7) b	13.6 (0.9) b
VX	21.4 (2.1) c	23.9 (1.5) d	9.9 (2.5) c	10.4 (1.0) c

2 mm for 20 seconds, although the differences appear to be related to the brand of curing unit and were not related to the use of QTH versus LED. Uopq, Kclr and Kopq showed significant differences for all curing conditions that were dependent upon the brand of light used.

The top KHN values are given in Tables 6, 7 and 8 and Figures 3, 4 and 5. There were differences for all materials dependent upon the light-curing unit used to activate the polymerization.

The results of the linear regression are provided in Table 9. The predicted required power density for each material to produce a B/T KHN of 0.80 is included.

## DISCUSSION

Currently, ANSI/ADA Specification 39 for Pit & Fissure Sealants (1992) requires a 0.75-mm depth of cure determined by wiping the bottom surface after polymerizing. ISO Specification 6874 (1988) requires a 1.5-mm depth of cure. Although many areas of sealants would fall within these thickness requirements, a pilot study indicated that areas greater than 2 mm in thickness were commonly present in molar sealants. Covey, Johnson and Hopper (2004) supported this finding. Though most fissures have signifi-

cant portions that result in thin areas of sealant, inadequate cure of the thicker areas may be associated with partial sealant loss and failure of the preventive therapy or restoration. Therefore, 2-mm thick specimens were used in this study.

A B/T KHN ratio of 0.80 has been used to identify an acceptable level of conversion in resin-matrix systems (Pilo & Cardash, 1992; Shortall & Harrington, 1996; Yap & Seneviratne, 2001). Only Uclr met this requirement with all of the lights when curing 1 mm for 20 seconds and 2 mm for 40 seconds. No material consistently reached this level when curing 2 mm for 20 seconds. It should be noted that the times tested were not necessarily those recommended by the manufacturers. However, the results support activation, with QTH or LED units being most predictable when accomplished, for 40 seconds.

All but one of the test conditions demonstrated the presence of LED and QTH lights in the same statistical groups. In the 2-mm thickness-40 seconds Kclr group, the LED AL provided the highest B/T KHN. This provides evidence that current LED light curing units are capable of initiating polymerization of these materials.

Table 7: 2-mm Thick—20 Second Cure: Top KHN. Vertical Means with the Same Letters Are Not Significantly Different (n=5)

Curing Units	Uclr (sd)	Uopq (sd)	Kclr (sd)	Kopq (sd)
HL	30.2 (1.1)a	28.7 (1.8)abc	18.2 (0.3)a	16.8 (0.5) b
OP	31.0 (2.1)a	28.5 (2.7)abc	17.8 (1.1)a	17.6 (1.0)ab
A5	31.3 (0.9)a	30.4 (1.5)a	19.0 (1.6)a	18.5 (0.4)a
DM	25.0 (5.2) bc	26.2 (2.3) bc	14.1 (0.8) b	13.6 (0.9) c
AL	28.9 (2.5)a	25.6 (2.5) c	14.8 (0.7) b	13.8 (0.7) c
UL5	30.6 (0.9)a	30.0 (1.1)ab	19.0 (0.4)a	17.6 (0.4)ab
UL	27.6 (1.4)ab	28.0 (2.9)abc	13.9 (0.5) b	14.1 (0.7) c
FL	27.0 (0.5)ab	26.7 (0.6)abc	12.2 (0.9) c	14.3 (1.9) c
VX	22.2 (1.9) c	24.8 (1.5) c	9.1 (0.7) d	11.4 (0.8) d

Table 8: 2-mm Thick—40 Second Cure: Top KHN. Vertical Means with the Same Letters Are Not Significantly Different (n=5)

Curing Units	Uclr (sd)	Uopq (sd)	Kclr (sd)	Kopq (sd)
HL	30.0 (2.4)ab	28.8 (1.0)ab	18.3 (0.4)ab	18.4 (0.6)a
OP	30.5 (1.4)ab	29.4 (2.0)ab	17.7 (2.2) b	18.3 (0.8)a
A5	33.0 (1.1)a	31.6 (0.9)a	19.8 (0.3)a	18.6 (0.3)a
DM	28.8 (1.1) b	27.0 (2.5) bc	15.8 (1.1) c	14.0 (1.0) c
AL	28.1 (2.1) b	29.0 (0.8)ab	14.7 (0.6) c	14.1 (0.4) c
UL5	32.1 (1.4)a	29.5 (2.0)ab	19.2 (0.7)ab	18.4 (0.6)a
UL	30.1 (1.6)ab	28.2 (2.4) bc	14.6 (1.0) c	15.8 (0.9) b
FL	28.0 (2.5) b	27.7 (0.7) bc	14.1 (1.3) c	14.0 (0.6) c
VX	25.4 (1.4) c	25.4 (1.5) c	11.5 (0.7) d	13.7 (1.4) c

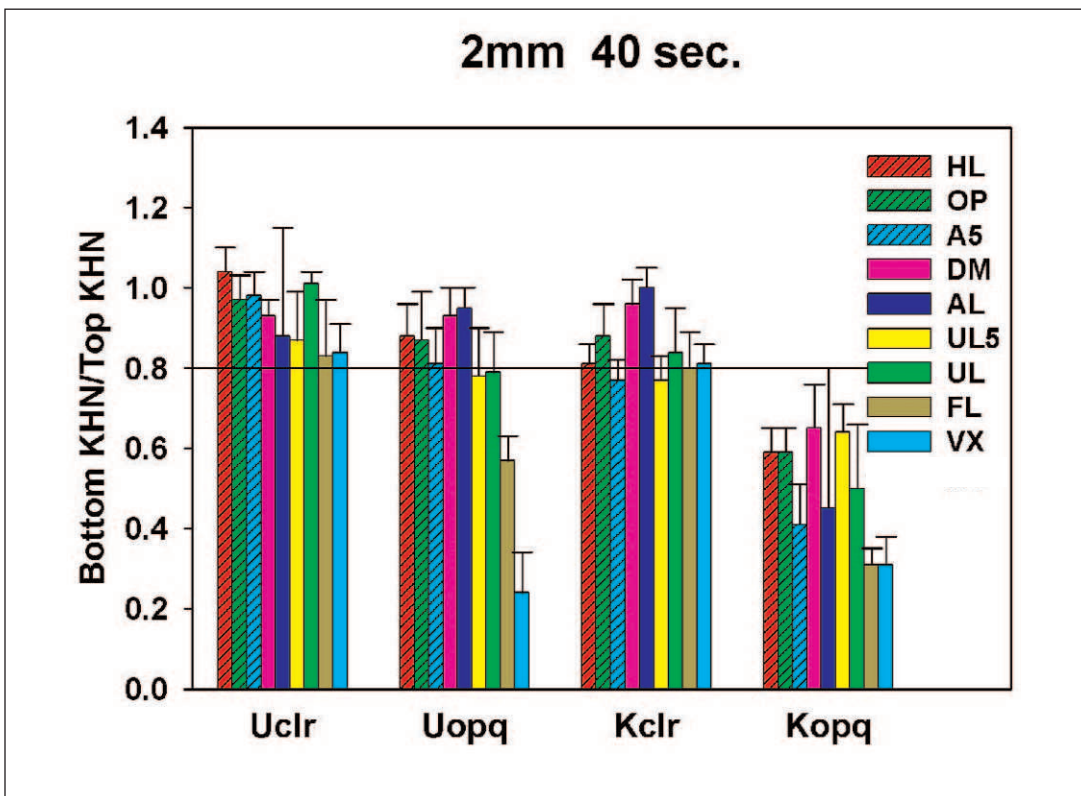


Figure 3: 2-mm thick—40 second cure: bottom/top KHN. (n=5)



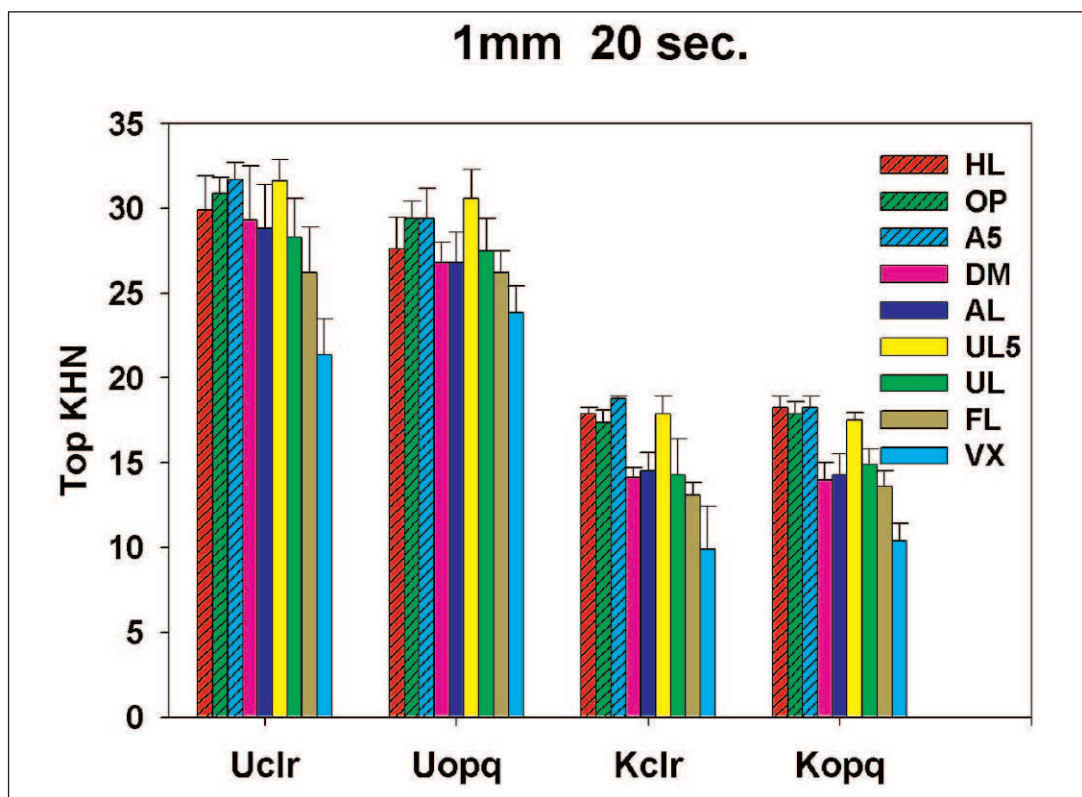


Figure 4: 1-mm thick—20 second cure: top KHN. (n=5)

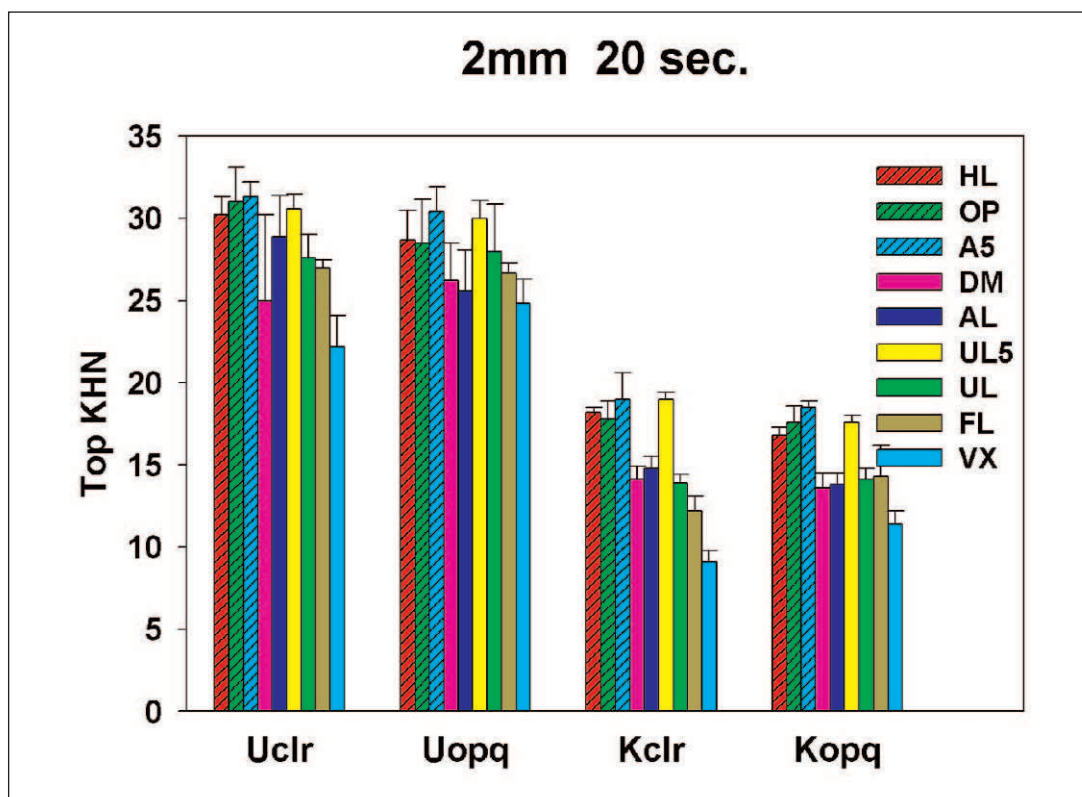


Figure 5: 2-mm thick—20 second cure: top KHN. (n=5)

The power density produced by a light-curing unit is one factor that influences the monomer conversion in methacrylate resin systems. Five of the six opaque material groups in this study exhibited at least modest correlation between the hardness ratios and power density. The fact that the correlation for the clear materials was quite low would indicate that other factors may be more important in influencing the polymerization of these materials.

Another important factor in the polymerization process is the spectrum of light output as it relates to the absorption of the photoinitiator/s of the resin. A camphoroquinone-amine system is used for photoinitiation in the Kclr and Kopq materials. The Uclr and Uopq materials also contain a second proprietary initiator. This may account for some of the apparent differences noted between materials, particularly with the UltraLume 5 light, which contains a second diode with an intensity peak at approximately 410 nm likely activating the proprietary initiator. However, this study did not pursue a comparison between materials.

It seems apparent that the opaque materials were more difficult to polymerize in

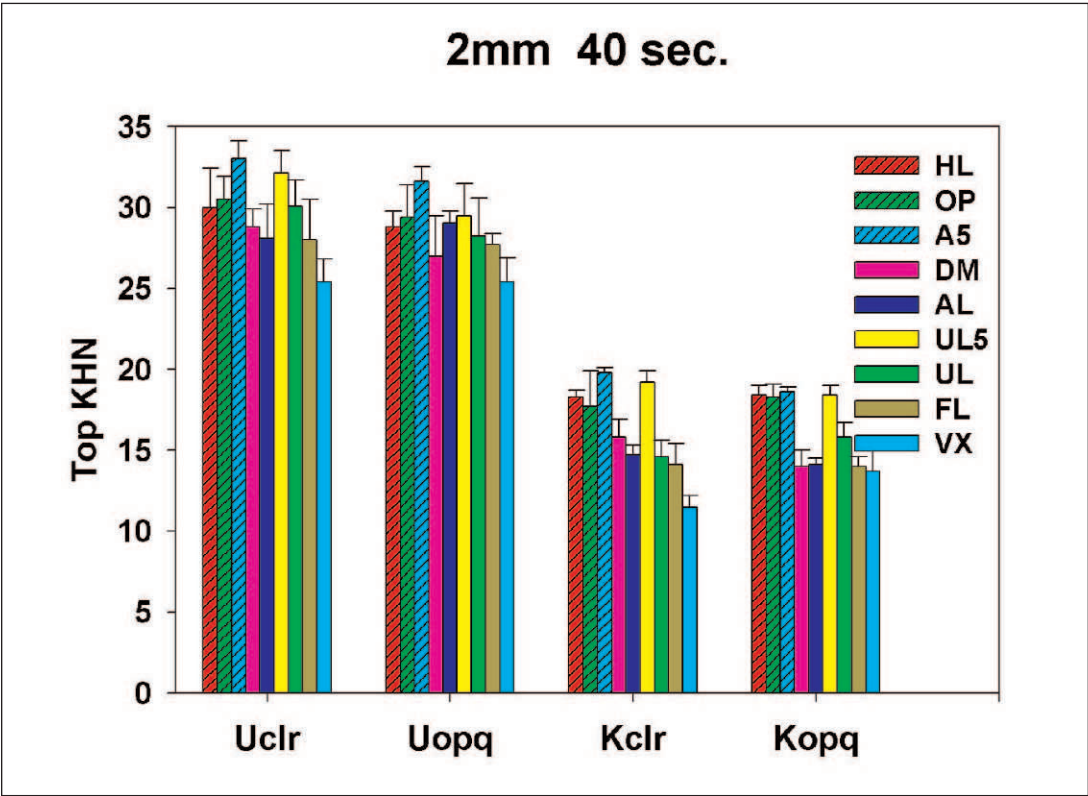


Figure 6: 2-mm thick—40 second cure: top KHN. (n=5)

Table 9: Linear Regression Analysis of Testing Condition Versus Power Density ( $r^2$ reported), Analysis of Variance of the Regression (p-value reported) and Power Density Predicted by the Regression to Obtain a B/T KHN of 0.80 (reported as PD)			
	1 mm – 20 seconds	2 mm – 20 seconds	2 mm – 40 seconds
Uclr	$r^2=0.018$ $p=0.733$ PD=513.034	$r^2=0.387$ $p=0.074$ PD=468.409	$r^2=0.333$ $p=0.104$ PD=537.465
Uopq	$r^2=0.527$ $p=0.027^*$ PD=547.378	$r^2=0.787$ $p=0.001^*$ PD=820.649	$r^2=0.777$ $p=0.002^*$ PD=685.006
Kclr	$r^2=0.010$ $p=0.874$ PD=587.687	$r^2=0.298$ $p=0.128$ PD=710.138	$r^2=0.137$ $p=0.327$ PD=457.754
Kopq	$r^2=0.605$ $p=0.014^*$ PD=751.116	$r^2=0.024$ $p=0.693$ PD=799.574	$r^2=0.546$ $p=0.023^*$ PD=1129.919

general. This could have significant clinical implications. The 2-mm 20-second group provides some indication of what might occur in any area of compromised light access. Cusp tips increasing the distance between the curing wand and the sealant surface and patients with limited opening would challenge sealant polymerization. It should be expected that most light curing units would not perform adequately with opaque sealants in these situations. The results of this study support the use of a clear light-activated sealant, a chemically-activated sealant or prolonged curing times for opaque light-activated sealants with a high output

light-curing unit to overcome this concern.

CONCLUSIONS

The ability to initiate polymerization of resin-matrix pit & fissure sealants was dependent upon the light-curing unit used. Differences were not related to the energy source being QTH or LED. Under the conditions of this study, clear light-activated resin-matrix sealants are more predictable than opaque materials when light exposure may be compromised.

Acknowledgement

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References

ANSI/ADA Specification No 39 (1992) Pit and fissure sealants American Dental Association Chicago, IL.

Besnault C, Pradelle-Plasse N, Picard B & Colon P (2003) Effect of a LED versus halogen light cure polymerization on the curing characteristics of three composite resins *American Journal of Dentistry* **16**(5) 323-328.

Covey DA, Johnson WW & Hopper LR (2004) Penetration of various pit and fissure sealants into occlusal grooves *Journal of Dental Research* **83**(Special Issue A) Abstract 3471.

ISO 6874 (1988) Dental resin-based pit and fissure sealants International Standards Organization Geneva, Switzerland.

Ferracane JL (1985) Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins *Dental Materials* **1**(1) 11-14.

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

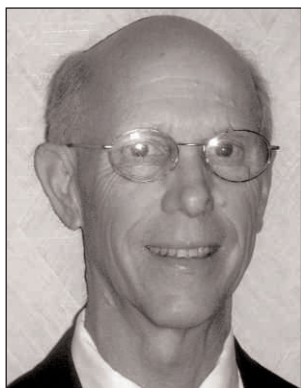
- Mertz-Fairhurst EJ, Curtis JW Jr, Ergle JW, Rueggeberg FA & Adair SM (1998) Ultraconservative and cariostatic sealed restorations: Results at year 10 *Journal of the American Dental Association* **129**(1) 55-66.
- Pilo R & Cardash HS (1992) Post-irradiation polymerization of different anterior and posterior visible light-activated resin composites *Dental Materials* **8**(5) 299-304.
- Price RB, Ehrnford L, Andreou P & Felix CA (2003) Comparison of quartz-tungsten-halogen, light-emitting diode, and plasma arc curing lights *Journal of Adhesive Dentistry* **5**(3) 193-207.
- Rock WP, Potts AJ, Marchment MD, Clayton-Smith AJ & Galuszka MA (1989) The visibility of clear and opaque fissure sealants *British Dental Journal* **167**(11) 395-396.
- Shortall AC, Wilson HJ & Harrington E (1995) Depth of cure of radiation-activated composite restoratives—influence of shade and opacity *Journal of Oral Rehabilitation* **22**(5) 337-342.
- Shortall AC & Harrington E (1996) Effect of light intensity on polymerisation of three composite resins *European Journal of Prosthodontics and Restorative Dentistry* **4**(2) 71-76.
- Simonsen RJ (1978) Preventive resin restorations *Quintessence International* **9**(1) 69-76.
- Uhl A, Sigusch BW & Jandt KD (2004) Second generation LEDs for the polymerization of oral biomaterials *Dental Materials* **20**(1) 80-87.
- Weintraub JA (1989) The effectiveness of pit and fissure sealants *Journal of Public Health Dentistry* **49**(5)(Special Issue) 317-330.
- Wendt LK, Koch G & Birkhed D (2001) On the retention and effectiveness of fissure sealant in permanent molars after 15-20 years: A cohort study *Community Dentistry and Oral Epidemiology* **29**(4) 302-307.
- Yap AU & Seneviratne C (2001) Influence of light energy density on effectiveness of composite cure *Operative Dentistry* **26**(5) 460-466.



## Awards

# American Academy of Gold Foil Operators Clinician of the Year Award

## Dr Barry O Evans



Barry O Evans

**T**his year's recipient of the Outstanding Clinician Award is Dr Barry Evans. Barry is a past president of our academy, serving on the Executive Council starting in 1990 and culminating with his presidency in 1996. As a presenter to our academy, who could forget Barry's intriguing program featuring Dr Hollenbeck's direct gold restorations that were placed in the late Katherine

Hepburn. How Barry obtained those pictures is an interesting story of its own. Barry has been an active operator at our annual meetings, never missing an opportunity to transport his instruments to distant clinics. I am convinced it is his love of the "art" of direct gold that has motivated him to operate so willingly.

Barry and his wife, Yvonne, have two children. His daughter, Brianna, and her husband have recently elevated Barry and Yvonne to grandparent status. Their son, Bryce, is a recent graduate of Oregon Health Sciences University School of Dentistry. He has a dental practice in Seaside, Oregon and has recently married.

I first came to know Barry in the fall of 1977. Since that chance meeting, our paths have crossed many times and, occasionally, in unusual ways. In fact, I actually owe a great deal of gratitude to him for a few of my own accomplishments. You see, in 1977, I was a first-year dental student. Barry was helping me pay for my school and living expenses. Now, before you heap on the praise, let me explain. At that time I was married to Barry's hygienist, Martha Bibb. Unfortunately, the employment opportunity was short lived, but the little I knew about dentistry told me that Barry was anything but ordinary.

About 10 years later, Barry helped me more than he will ever know. It was about then that Dr Louis Schoel retired from practice. A lovely patient of Louis transferred to Barry's office at the advice of a friend. After her first visit with him, she was so overwhelmed by his thorough treatment recommendations that she decided to

seek another opinion. She called the local component of the Multnomah Dental Society and was given the names of three dentists willing to accept new patients. Lucky for me, I was the first name on the list. Twelve years later I would marry this lovely woman, and to this day, I have Barry to thank for this chance meeting. Barry is passionate about dentistry. He is a member of several operating study clubs spanning all disciplines of dentistry. He mentors three cast gold inlay clubs. He started a gold study club for "new" graduates, feeling that starting the graduates out with a good foundation would help them to experience what he likes to call "Excellence." As a former member of one of his cast gold clubs, I have fond memories of his dedication as a mentor. I will always remember his tedious use of an explorer. After what seemed like an eternity, he would look up and proclaim, "It's a good start." You can imagine my surprise, since I thought I had completed the operation. I am still puzzled by the meaning of "there are no straight lines in nature," when he describes how a preparation should flow from one area to the next. From time to time, I try to find those straight lines and think of him.

All of us are involved in continuing education. I calculated that the average dental graduate experiences about 5700 hours of instruction. About half of those hours are clinical. In 35 years of private practice, I estimate that Barry has participated in well over 8000 hours of continuing education. This is a real testament to his dedication to the profession and to those people who trust themselves to his care.

In my opinion, everyone in this academy is deserving of this award, but we only honor one member each year. Barry Evans has been chosen to receive the Outstanding Clinician Award this year not just because he places a fine direct gold restoration, but more importantly, because of his dedication to his profession and his love of dentistry has influenced the lives of so many people. He represents all that is good about our profession. He is willing to learn, is excited to share and, when necessary, is open to change. Dr Barry Evans continues in his own way to make our profession better for all of us. I am proud to have been asked to introduce this year's recipient of the Outstanding Clinician Award, Dr Barry Evans.

Scott B Barrett

# American Academy of Gold Foil Operators Distinguished Member Award

**Dr Michael A Cochran**



*Michael A Cochran*

**I**t is my honor and pleasure to present our Distinguished Member Award to Dr Michael Cochran. Hopefully, these remarks will provide evidence of his merit as a recipient of this award.

We first catch sight of Michael in the Upper Peninsula of Michigan in the 5<sup>th</sup> grade. His family is one of the few year-round residents in the area, and the size of his class is just 30 students, so he obviously did not have

much competition. Transportation to school was by bus, and the walk to the bus stop from his home on Lake Michigan was about 3/4 of a mile...reportedly uphill both ways. Now, Michigan winters are long, cold and snowy, but schools rarely close due to weather. One morning in near blizzard conditions, Michael is on his way to the bus stop and sees a fuzzy outline standing in his usual spot, which he hopes might be a new classmate. As he gets closer, however, he sees that the outline isn't fuzzy because of the falling snow but because it is a large bear. This discovery prompted a hasty retreat home...an example of the good judgment that Mike has shown throughout his life. He is not sure whether or not the school bus picked up the bear.

After graduating high school, Michael earned his BS degree from Northern Michigan University, majoring in Biology, with minors in Chemistry and Art, the latter avocation serving him well in his dental career. He then attended the University of Michigan School of Dentistry as a member of the class of 1969. He was the cartoonist for the dental school newsletter, and one might wonder as to the impact this may have had on the faculty and administration, since Mike is not known for his lack of opinions.

Upon graduation from dental school, Mike joined the US Navy Dental Corps and served at Great Lakes, Illinois; San Juan and Roosevelt Roads, Puerto Rico; Norfolk, Virginia and on the USS Forrestal aircraft carrier. Between assignments in Puerto Rico and Virginia, the Navy sent Mike to the Graduate Operative Dentistry program at Indiana University School of Dentistry for two years, where he earned his Master's degree. Among the

hurdles of his graduate studies was an event known as the oral and written examination. After handling numerous questions on Dental Materials, Statistics and Preventive Dentistry, Mike suddenly drew a blank on his first question on Operative Dentistry, which was to describe the different preparation designs for Class III direct gold restorations. Can anyone imagine Mike speechless? Aware of his artistic ability, I made the suggestion that he go to the board and draw the preparations. This shattered his mental block and the exam continued. After his tours at Norfolk and on the Forrestal, Michael left the Navy and joined the faculty at Indiana University.

At this point Michael began a career that has led him to this recognition. Academically, he progressed from director of the Operative Dentistry Clinic to chair of the Department of Operative Dentistry to his current position as director of the Graduate Operative Dentistry Program. During this time, he has taught the techniques and materials of Operative Dentistry to approximately 2,700 dental students, as well as more than 130 Graduate Operative students. He has been recognized by his students with numerous awards as outstanding lecturer and teacher. His illustrations and writing have appeared in seven editions of four different textbooks, and Lloyd Baum and I have especially appreciated his art activity in our Operative Dentistry texts. He has presented more than 180 continuing education programs around the world, many involving direct gold. His association with the Navy Dental Corps has continued over the years, with numerous presentations at Bethesda and San Diego and a long-term commitment as consultant on Operative Dentistry to the Naval Post-Graduate Dental School. His service was recognized in 1992 when he received the "Civism" award from the National Naval Dental Center.

Michael has been an author or co-author on more than 35 abstracts and 70 articles, many as a result of his research activity with his graduate students. He is a strong supporter of organized dentistry and holds membership in 11 professional organizations including Omicron Kappa Upsilon, the American Dental Association, the International Association for Dental Research, the Academy of Operative Dentistry and this group, the American Academy of Gold Foil Operators, where he served as President in 1991 and received the Clinician of the Year Award in 1992. His most recent membership is with the American Association of Dental Editors.

Since 1999, Mike has been editor of *Operative Dentistry* and, of all the activities that support tonight's award, the editorship of our journal is one of the most prominent. During his tenure, the journal has increased in size, content and recognition. This does not, however, diminish the activity of prior editors.

Obviously, his professional career has been extensive and time consuming, but his family is of even greater importance to Mike. His lovely wife, Christianne, who is also a full-time faculty member at our School of Dentistry, has been a tremendous influence and support. His two

daughters, Holly and Shannon, his son Sean (a 6 ft 6 in basketball player at age 14) and his five grandchildren by his daughter Holly (Brandon, Josh, Jake, Kylie and Brady) are all the joy and foundation of his life.

As a result of all this supporting data and my long-standing knowledge of and friendship with Mike Cochran, it is my pleasure to present to him the Distinguished Member Award of the American Academy of Gold Foil Operators.

Melvin R Lund



## Departments

### Abstracts



The editor wishes to thank the second-year Comprehensive Dentistry residents at the Naval Postgraduate Dental School, Bethesda, Maryland, for their assistance in preparing these abstracts.

**The effect of the distance between post and residual gutta-percha on the clinical outcome of endodontic treatment. Moshonov J, Slutzky-Goldberg I, Gottlieb A & Peretz B *Journal of Endodontics* (2005) 31(3) 177-179.**

(Department of Endodontics, Hadassah School of Dental Medicine, Jerusalem, Israel)

After endodontic therapy, teeth are often restored with a post and core. In many cases after post cementation, there is a gap between the apical end of the post and the remaining gutta percha. This study evaluated *in vivo* the outcome of endodontic therapy in teeth with varying amounts of space between cemented post and gutta percha.

A total of 94 patients, who had previously undergone endodontic therapy followed by post and core restoration, were selected. The group consisted of 26 males and 68 females, 23 to 88 years of age.

The endodontically treated teeth fit the following criteria:

1. periapical tissue of normal appearance before treatment
2. irreversible pulpitis before treatment
3. pulp exposure during caries removal
4. elective root canal treatment before prosthetic treatment

All teeth had been cleaned and shaped under rubber dam isolation and obturated within 1 mm of the radiographic apex, using laterally condensed gutta percha with AH-26 sealer. Cases with complicating factors (separated files, over or underextension of root canal filling, root fracture, residual root canal fill < 3 mm) were excluded.

The selected cases were divided into three groups, based on measurements from post-treatment radiographs:

Group I: no gap between gutta-percha and post.

Group II: a gap of >0 to 2 mm between gutta-percha and post.

Group III: a gap >2 mm between gutta-percha and post.

Follow-up radiographs, taken between one and five years post-treatment, were evaluated according to the following criteria:

Normal: no periapical radiolucency, intact PDL.

Disease: periapical radiolucency or widening of the PDL space.

Radiographs were "masked" coronally with cardboard to reduce bias. Clinical outcomes related only to the roots in which posts were placed. The results were as follows:

Group I: 16.7% disease, 83.3% normal.

Group II: 46.4% disease, 53.6% normal.

Group III: 70.6% disease, 29.4% normal.

This study illustrates the need to exhibit care when cementing posts in endodontically treated teeth, seating the post properly to eliminate space between the post and residual gutta percha.

**Five-year follow-up with Procera all-ceramic crowns. Fradeani M, D'Amelio M, Redemangi M & Corrado M (2005) *Quintessence International* 36(2) 105-113.**

(University of Milan; private practices in Milan, Mestre, Lomazzo and Monselice, Italy)

This study evaluated the clinical performance of Procera AllCeram crowns placed over a five-year period at three different private dental practices.

Two-hundred and five Procera AllCeram crowns placed in 106 patients were evaluated over a period ranging from a minimum of six months to a maximum of 60 months, with a mean of 23.52 months. The clinical procedures were performed by three dentists in their private practices. The crowns were fabricated by three dental technicians following manufacturers' instructions. One hundred and fifty-one crowns were cemented with Panavia 21 TC (Kuraray), 40 with Fuji Plus (GC) and 14 with RelyX Luting (3M). Patients were reexamined by the authors one month after cementation and at three or six-month intervals for the following period. A restoration was considered a failure when it impaired esthetic quality or function, thus necessitating remake of the crown. Patients with severe parafunction, periodontitis, serious gingival inflammation, or poor oral hygiene or caries were excluded from the study.

The survival rate was determined with the use of the Kaplan-Meier method, which gave an overall survival rate of 96.7% (100% for the anterior crowns and 95.15% for the posterior crowns). Of the 50 anterior crowns, there were no failures. Of the 155 posterior crowns, there were four failures. All four failures were molars. Two involved fracture of the veneer and alumina coping. One involved fracture of the veneering porcelain only, and one involved de-lamination of the veneering porcelain. The results of this study match results reported in other similar studies on Procera Allceram crowns. Within the limits of this study, it was concluded that the Procera AllCeram system seems to have a good prognosis for the posterior teeth and an excellent prognosis for the anterior teeth.

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**Microhardness of composites in simulated root canals cured with light transmitting posts and glass-fiber reinforced composite posts. Yoldas O & Alaçam T *Journal of Endodontics* (2005) 31(2) 104-106.**

(Department of Conservative Dentistry and Endodontics, Çukurova University, Adana, Turkey)

This study compared the microhardness of resin composite cured in simulated root canals using light-transmitting plastic posts (LTPP), glass-fiber-reinforced composite posts (GFRCP) and conventional light curing methods (control group).

Thirty black plastic cylinders, measuring 15 mm in length and 4 mm in internal diameter, were divided into three groups of 10 specimens each. Tetric Ceram (Ivoclar Vivadent) composite was firmly packed into the simulated canals. The LTPP (No 4, Luminex, Dentatus) and GFRCP (No 1, Postec, Ivoclar Vivodent) with the same diameters (1.5 mm) were inserted into the simulated canals using a parallelometer. All samples were then light cured (Hilux Dental Curing Light, Model No 200, Benlioglu Dental, Inc) with a constant-type exposure at 460 mW/cm<sup>2</sup> for 90 seconds. After 24 hours, the plastic cylinders were removed from the samples and a microhardness test was performed using a Micromet Microhardness Tester (MMT-3 Digital Microhardness Tester, Buehler Ltd) with a load of 100 g for 10 seconds. Three test indentations of each sample were made at randomly selected areas of the polymerized resin composite samples at depths of 2, 4, 6, 8, 10, 12 and 14 mm from the light exposed surface. All microhardness measurements were recorded as a Knoop Hardness Number (KHN), and the results were evaluated statistically using a one-way analysis of variance and the Tukey post hoc test between groups. Paired *t*-tests and repeated measure analysis were used to compare KHN within groups.

There was a significant increase in microhardness of the resin composite for both LTPP and GFRCP compared with the control group ( $p < 0.01$ ). The microhardness test could not be performed on the control group due to the lack of polymerization below 4 mm. There were no significant differences in microhardness between LTPP and GFRCP until 10 mm ( $p > 0.01$ ). At 10 mm, the microhardness of resin composite was significantly higher with LTPP than GFRCP ( $p < 0.01$ ). After 10 mm, the microhardness of GFRCP could not be performed because of the lack of polymerization. With increasing distance from the curing tip, the measurement of resin composite microhardness was decreased in all groups.

**Conclusions**

The depth of cure of resin composite in a simulated root canal is significantly increased with LTPP and GFRCP. After 10 mm, the polymerization of resin composite could not be achieved by GFRCP.

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**The effect of adhesive and flowable composite on post-operative sensitivity: 2-week results. Perdigão J, Anauate-Netto C & others *Quintessence International* (2004) 35(10) 777-784.**

(University of Minnesota, Minneapolis, MN, USA; University of Mogi das Cruzes, SP, Brazil)

This study compared the effect of adhesive and flowable composites on post-operative sensitivity over a two-week interval. The authors state that post-operative sensitivity in posterior teeth restored with resin composites has been a problem experienced by clinicians for more than 15 years. Several factors, such as polymerization shrinkage, bulk filling technique, incomplete coating to the dentin surface with dentin adhesives and traumatic occlusion have been the culprit of the post-operative sensitivity.

Providers inserted 100 posterior composite restorations in Class II cavity preparations in patients' molars and premolars, either to replace an existing faulty restoration or to treat primary carious lesions. Patients' ages ranged from 20 to 54 years. All preparations were of conventional design, and cavosurface angles were entirely in enamel without any intentional bevel. All operative procedures were performed under local anesthesia with rubber dam isolation. The enamel and dentin walls of the preparation were treated either with a self-etching dentin/enamel primer (SE Primer, Kuraray America) for 20 seconds or were etched with 34% phosphoric acid (Caulk Etchant, Dentsply Caulk) for 15 seconds. While the self-etching primer was not rinsed, the phosphoric acid was washed for 10 seconds

and the dentin was left visibly moist or was remoistened to an acceptable level prior to application of Prime & Bond NT (Dentsply Caulk). The flowable composite Filtek Flow (3M ESPE) was inserted as the first increment in the cervical area of each box for half of the restorations receiving each adhesive. The composite restorative SureFil (Dentsply Caulk) was then inserted in one increment (when the flowable material was used as the cervical increment) or two increments (when no flowable material was used). Each increment was polymerized for 40 seconds with a light source as recommended by manufacturer. Finishing and polishing were performed on each restoration.

The authors evaluated the restorations clinically for the following characteristics: a) marginal discoloration; b) post-operative sensitivity to air and cold and c) post-operative sensitivity to masticatory forces. The results were that all restorations had no discoloration at the post-operative period; change from baseline to two weeks in sensitivity for cold or air stimulus was not significant; there were no cases of post-operative sensitivity due to masticatory forces.

The authors found that there were no differences in post-operative sensitivity between a self-etch adhesive and a total-etch adhesive at two weeks. A flowable composite did not decrease post-operative sensitivity.

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**The efficiency of three different films and radiovisiography in detecting approximal carious lesions. Erten H, Akarslan ZZ & Topuz O *Quintessence International* (2005) 36(1) 65-70.**

(University of Minnesota, Minneapolis, MN, USA; University of Mogi das Cruzes, SP, Brazil)

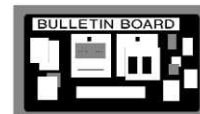
The investigators designed a project to determine if one type of x-ray film (D, E or F-speed category) or digital sensor provided a significant advantage over the others in detecting approximal caries. Forty molars and premolars were imaged using the same settings on the x-ray machine; 70kVp, 8mA and 16-inch focal spot-film distance. They varied the time of exposure to comply with manufacturers' recommendations. The images were randomized and viewed under subdued lighting without the use of magnification or post-processing manipulation. Three clinicians graded the lesions using a five-point rating system. The teeth were then sectioned mesiodistally for microscopic evaluation to determine the true extent of caries.

There was no significant difference among the four diagnostic modalities in the ability to detect interproximal caries lesions. The dental professional has an obligation to produce radiographs of the highest diagnostic quality using the least amount of radiation.

Insight film and RVG produced diagnostic images comparable to Ultraspeed and Ektaspeed Plus film utilizing reduced radiation exposure times.

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## Announcements



### 35<sup>th</sup> ANNUAL MEETING of the ACADEMY OF OPERATIVE DENTISTRY 22-24 February 2006, Fairmont Hotel, Chicago, IL

This is a personal invitation to join your friends, colleagues and fellow members at the Academy of Operative Dentistry's 35<sup>th</sup> Annual Meeting in beautiful downtown Chicago. Once again, we have an outstanding essay program, informative table clinics and entertaining social events that will please everyone.

**SCIENTIFIC SESSION:** Thursday's program begins with Dr Gregory Kinzer and Dr Vincent Kokich, Jr, with a two-hour presentation on "Interdisciplinary Management of Anterior Esthetic Dilemmas." Dr Jacques Nor will follow with this year's Buonocore Memorial Lecture, "Tooth Tissue Engineering and Molecular Biology in Restorative Dentistry." Thursday's luncheon will honor Dr William Douglas with the Hollenback Memorial Prize. Thursday afternoon features Dr Gerard Chiche presenting "Long-Distance Communication: Setting High Esthetic Standards for the Dental Team," followed by Dr Van Haywood with an informative presentation on bleaching titled "White Teeth or Little White Lies." The afternoon concludes with Dr Terry Donovan and Dr Richard Simonsen presenting highlights of the exemplary clinical dentistry of Dr Richard V Tucker with "A Pictorial Retrospective of Clinical Dentistry."

Dr Jeffrey Okeson leads off Friday morning with "Occlusion, Temporomandibular Disorders and Restorative Dentistry." Dr James Dunn follows with an essay on "Digital Dental Photography," and the essay programs conclude with Dr W Bruce Howerton, Jr speaking on "Three-Dimensional Imaging in Dentistry." The Friday luncheon will be held in the Mid-America Club and will feature presentation of the Academy's Award of Excellence to Dr Joel M Wagoner. The 2005 annual session will conclude with Friday afternoon's table clinics. This year, more than 30 clinicians will offer concise, focused presentations that provide a wealth of "pearls" for us to take back to our offices.

**COMPANION PROGRAM:** The Companion Activities Program is particularly exciting this year. On Thursday, Arnie Bernstein, author of *Hollywood on*



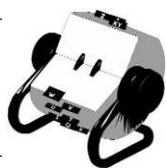
*Lake Michigan*, will provide a group tour of the Windy City to see why Chicago has become a favorite filming location for many current movies including "The Breakup," "Batman Begins," "Il Mare" and "Stranger Than Fiction." The Illinois Film Office estimates that filming of these movies in and around Chicago resulted in \$68 million in revenues in 2005. The tour will be followed by a luncheon at Osteria Via Stato, a fantastic concept Italian restaurant by Rich Melman.

This year, Friday is being left open as a day for shopping, sightseeing and visiting with friends and family.

**RECEPTION:** Finally, our Gala Reception on Thursday evening will once again provide a wonderful, once-a-year, platform for socializing with all our friends and colleagues from across the country and around the world.

Please do not miss this fantastic opportunity for education, information exchange and fun. See you in Chicago in February. For more meeting information, contact Dr Gregory Smith, PO Box 14996, Gainesville, FL 32604-2996, USA; Fax: (352) 371-4882; e-mail: gesaod@ufl.edu.

## Instructions to Contributors



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**NOTE:** As of May 1, 2005, all papers must be submitted electronically.

## Faculty Positions



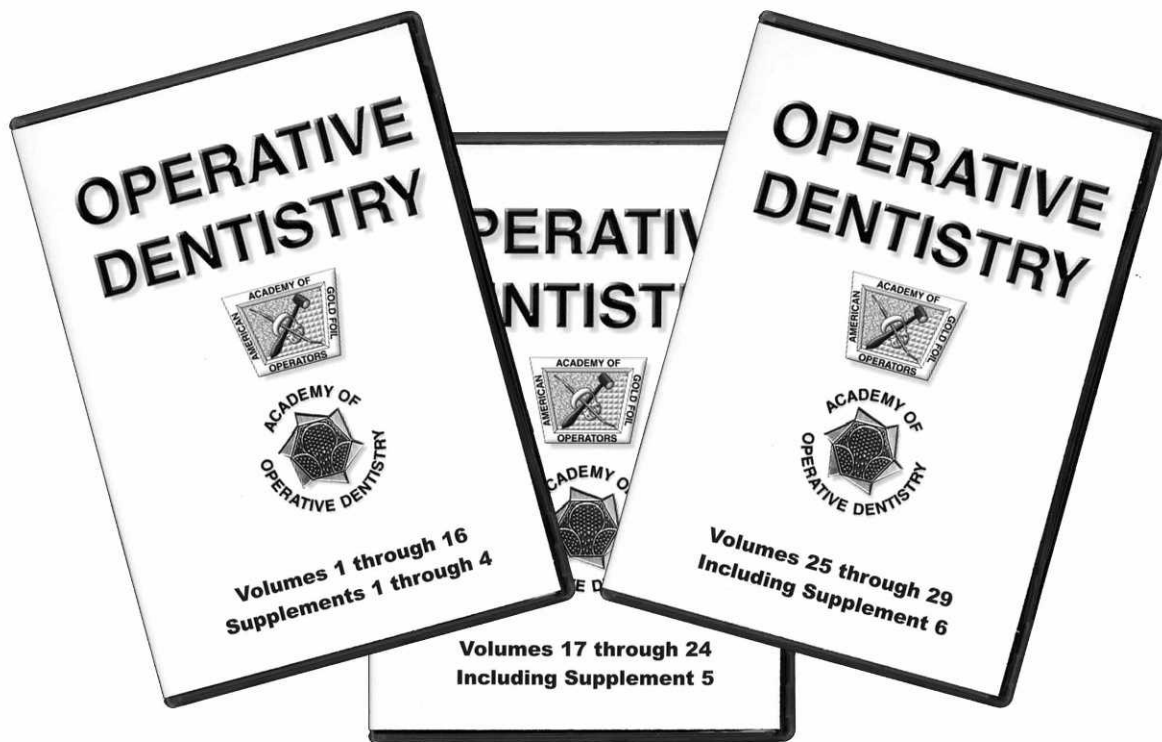
For a complete list of faculty positions featured in *Operative Dentistry*, go to the Operative Dentistry Home Page: <http://www.jopdent.org/>, click on Announcements, then Academic Positions.

### University of Iowa College of Dentistry

The University of Iowa's College of Dentistry is searching for a tenure track Chair of the Department of Prosthodontics. For a list of duties, go to: [www.dentistry.uiowa.edu/public/administrtaion/hr\\_search.html](http://www.dentistry.uiowa.edu/public/administrtaion/hr_search.html). The position is available immediately; screening begins immediately. Candidate must have a DDS/DMD from an ADA-accredited dental school or foreign equivalent, a record of effective teaching/evidence of scholarly activity and demonstrated experience working effectively in a diverse environment. Desirable qualifications include advanced education in prosthodontics and/or restorative dentistry, relevant administrative experience and clinical practice. Rank/salary are commensurate with qualifications/experience. Submit DV with three names of reference to: Dr William T Johnson, c/o Mary Ann Sevcik, S414 Dental Science South, College of Dentistry, University of Iowa, Iowa City, IA 52242-1011, USA. AA/EEO employer; women/minorities are encouraged to apply.

### CORRECTION:

The Acknowledgment for "Recommendations for Clinical Practice—Reasons for Replacement of Restorations," *Operative Dentistry* July/August 2005 **30-4** 409-416, should have included the name of Dr Ivar Mjør as the principal contributor. While the Recommendations for Clinical Practice are the work of a committee and, therefore, do not carry the name of an author, it is appropriate to acknowledge the efforts of individuals who contribute the majority of the information, and we apologize for the oversight.



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# OPERATIVE DENTISTRY JOURNAL

Official Journal of the Academy of Operative Dentistry,  
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There have been many questions sent to us with regard to the significant changes that have taken place, seemingly all at once, with Volume 31. We would like to assure you that all the changes have been thought about, discussed, changed and resubmitted countless times...all with the best interests of our subscribers in mind. We would also like to take this opportunity to clarify the new policies, explain the new procedures and expound upon why we think these changes will ultimately benefit you, our reader.

On page 781 (facing), you will see a synopsis of our recent mailing: Subscription Rates for Volume 31 (2006). You will also find various superscripted numbers (<sup>1</sup>); these same numbers will appear below, with any pertinent explanations.

We hope that this information will answer the majority of questions you may have. If you have additional questions or concerns, please do not hesitate to e-mail us at any time at [editor@jopdent.org](mailto:editor@jopdent.org). Thank you all for your continued support of *Operative Dentistry*.

Editorial Staff

1. We welcome the RV Tucker Academy to our journal family. Dues do not change if you belong to multiple academies; the second (or third) subscription goes to the journal for its continued improvement.

2. Our rates have jumped significantly. In reviewing our financial books, we have found that we were fiscally sound, but not enough to take advantage of any of the technological advances that have been requested of us. We wanted to create a pricing structure that was fair to all types of subscribers at a price that would not have to be increased any time soon. Online journals are an expensive proposition, but one for which we have received many requests. We are pleased to have partnered with Allen Press to be able to provide our readers with electronic options. The price increase also takes into account the upcoming technologies that will be made available to you as it becomes feasible to provide them to you, such as video and audio clips of procedures and processes, online subscription payments and more. We want to point out that we still are one of the highest quality, lowest cost, non-advertising journals on the market, and we hope to keep it that way for many years to come.

3. Online Subscriptions. We are excited to have this option available to all subscribers this year. Please take note of the following instructions.

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be considered, fraud and subject the institution to cancellation and forfeiture of all access rights.

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5. We recognize that occasionally issues go astray. In the past, the journal has absorbed the high cost of replacements. Now, due to full online access to all subscribers, there will be a USD \$5.00 processing fee that needs to accompany all claims.

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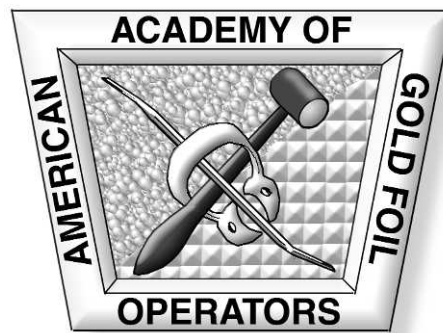
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