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### Be Careful What You Wish For...

his issue of *Operative Dentistry* begins our fifth year as your editorial team. We started our tenure as a labor of love but, although we still feel the same excitement, affection and dedication, the love is sometimes buried under the labor. We started out in 2000 with a number of goals for the journal including a new look, larger issues with more articles, corporate sponsorship to provide money for color and expansion, a more useful and informative web site and more clinical research articles and clinical case report/technique papers. However, our primary objective was to cut publication time and ensure that all accepted manuscripts would appear in print within one year of submission. This, to me, was essential for both our contributors and readership if we were to disseminate important information in a timely manner. Thanks to our superb Editorial Board and diligent staff, we have accomplished all of these goals (at least to some degree). We are particularly proud of the improved publication time that we accomplished in our first year with the journal. We got what we wished for...and everything that goes with it.

As word spread that accepted manuscripts were published quickly in Operative Dentistry, we began to receive more submissions with a wider variety of topics from more and more internationally recognized researchers. The journal's higher ranking in various journal citation reports relative to impact factor and cited half-life was a testament to the quality of the publication and has generated even more interest by prospective authors. The end result has been a steadily increasing (read explosion) of submissions to our journal. We have gone from 95 submitted papers in 2000 to more than 275 in 2003. This has been extremely exciting and gratifying but has obviously created a tremendous increase in workload for our Editorial Board and journal staff. Because of this, we are forced to institute some changes in Editorial policy to shift some of the work to our contributors. Let me explain why we are making these changes, what they are and

what it means to authors submitting papers to *Operative Dentistry*.

We have a large Editorial Board (more than 130 members) for reviewing submitted papers, all of whom are volunteers receiving no remuneration for their time and expertise. However, our policy is to send each manuscript to three reviewers, and the tremendous increase in submissions has put a strain on all of our Board members. While we are constantly seeking additions to our cadre of reviewers, finding individuals with the appropriate background, expertise and willingness to participate is challenging. I cannot begin to express my appreciation for the thousands of hours of effort contributed by our reviewers, but I certainly recognize that I am constantly asking for more than they should have to give.

In contrast to the size of our Editorial Board, we have a small journal staff. The Editor, Associate Editor (3), Managing Editor and Assistant Managing Editor (2) positions are all unpaid, while the Editorial Associate and Editorial Assistant/Subscription Manager receive salaries. We also compensate additional individuals who provide various services during the preparation of each issue for publication. When our submission rate was lower, I personally provided many services to authors by proofing all manuscripts, correcting grammar, syntax and punctuation for international authors (whose native language was not English), converting various program and file formats to those used in our page-set routine, finding ways to open and read nonstandard files and disks and performing many touchups and re-dos of graphs and illustrations in Excel and Photoshop so that the quality met what our readership expects. The marked increase in the number of manuscripts received has made it impossible for me to continue to provide these services on a routine basis...unless I want to give up my full-time faculty position and private practice. In addition, the increase in the number of pages to proof and set, as well as the voluminous correspondence with authors and review-

ers, has taxed both our Editorial Associate and Subscription Manager to their limits.

Therefore, it has become necessary to ask our contributors to share the burden. The new Editorial policy is reflected in our Instructions to Contributors, which is posted on our web site and includes the following stipulations:

- All manuscripts must comply with the current Instructions to Contributors in relation to general formatting, reference formatting and submission form of tables, graphs and illustrations.
  - Manuscripts must be in Microsoft Word format
  - Tables must be in Microsoft Word format
  - Graphs must be in Microsoft Excel format with associated data
  - Illustrations must be submitted as TIFF or JPEG files of appropriate resolution (Black & White and Grayscale = 300 dpi minimum. Color = 600 dpi minimum) or as clear, high quality photographs or hard copy drawings
- The electronic submission of manuscripts is encouraged and generally saves considerable time and money for both the contributor and the journal. Much of the journal's correspondence, including transmission of page-proofs will be handled by e-mail, and all authors must provide an e-mail address as well as telephone and fax numbers. International authors should try to provide a complete phone and fax number, including access codes necessary to connect from the United

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- If manuscripts are submitted by mail, the appropriate number of copies must be present and any computer disks must be either 3 1/2" "floppy," Zip disks (100 or 200 MB) or standard CD-ROM.
- It is strongly suggested that international authors have their manuscripts reviewed and corrected by a native English speaker prior to submission. If the manuscript is not consistent with accepted English format, it will be returned.
- If any of the criteria outlined in our Instructions to Contributors are not met, the manuscript will be returned to the corresponding author, without review, for correction and resubmission

The bottom line is that we do not want to limit submissions or pages, nor discourage authors from submitting their work to *Operative Dentistry*...we merely need their help in making it possible to maintain our policy of printing all accepted manuscripts within one year of submission. Our goal is to continue to provide our contributors with an excellent forum for their papers and our readers with an outstanding source of quality information, and to accomplish this within the limitations imposed by our resources and abilities. At least that is what we are wishing for...and suggestions are always welcome.

Michael A Cochran Editor

### Quantitative Measurement of Marginal Disintegration of Ceramic Inlays

M Hayashi • Y Tsubakimoto F Takeshige • S Ebisu

### Clinical Relevance

The findings of this study lend support to the subjective view that marginal disintegration of ceramic inlays tends to be accelerated by occlusal force.

### **SUMMARY**

The objectives of this study include establishing a method for quantitative measurement of marginal change in ceramic inlays and clarifying their marginal disintegration *in vivo*. An accurate CCD optical laser scanner system was used for morphological measurement of the marginal change of ceramic inlays. The accuracy of the CCD measurement was assessed by comparing it with microscopic measurement. Replicas of 15

premolars restored with Class II ceramic inlays at the time of placement and eight years after restoration were used for morphological measurement by means of the CCD laser scanner system. Occlusal surfaces of the restored teeth were scanned and cross-sections of marginal areas were computed with software. Marginal change was defined as the area enclosed by two profiles obtained by superimposing two cross-sections of the same location at two different times and expressing the maximum depth and mean area of the area enclosed. The accuracy of this method of measurement was  $4.3 \pm 3.2 \,\mu m$  in distance and 2.0± 0.6% in area. Quantitative marginal changes for the eight-year period were 10 x 10 µm in depth and 50 x 10<sup>3</sup> µm<sup>2</sup> in area at the functional cusp area and 7 x 10 µm in depth and 28 x 103 µm2 in area at the non-functional cusp area. Marginal disintegration at the functional cusp area was significantly greater than at the non-functional cusp area (Wilcoxon signed-ranks test, p<0.05). This study constitutes a quantitative measurement of in vivo deterioration in marginal adaptation of ceramic inlays and indicates that occlusal force may accelerate marginal disintegration.

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### INTRODUCTION

The long-term clinical performance of ceramic inlays has recently been reported (van Dijken, Hoglund-Aberg & Olofsson, 1998; Frankenberger, Petschelt & Kramer, 2000; Hayashi & others, 2000; Pallesen & van Dijken, 2000; Reiss & Walter, 2000). In most cases, favorable results were obtained for long periods after restoration, so that this aesthetic restoration has been considered clinically acceptable. However, a longitudinal *in vivo* 

evaluation identified deterioration in marginal adaptation in more than 70% of the restorations (Hayashi & others, 2000).

To avoid marginal deterioration of the ceramic inlays, the mechanism of such disintegration should first be clarified quantitatively and qualitatively. Morphological changes in the marginal area were investigated qualitatively with a scanning electron microscope in previous studies (Hayashi & others, 1998, 2000), wherein the disintegration of marginal ceramics, wear of resin composite cement and chipping of marginal enamel were detected. A tiny microcrack in the ceramics could also induce a critical fracture. However, since only a few studies have investigated quantitative change of marginal deterioration of ceramic inlays (Roulet & others, 1997), the mechanism of deterioration is still uncertain.

The purpose of this ongoing series of studies, an element of which is reported in this paper, was to clarify the mechanism of *in vivo* marginal disintegration of ceramic inlays. The initial investigation was to establish a method for the quantitative measurement of marginal change in ceramic inlays.

### **METHODS AND MATERIALS**

### **Evaluated Teeth**

Fifteen permanent premolars restored with Class II fired ceramic inlays were employed for the quantitative measurement of marginal disintegration. Table 1 summarizes distribution of the teeth that were measured. At the Department of Conservative Dentistry of Osaka University Dental Hospital between October 1990 and March 1991, the inlays were placed in 12 patients using a fieldspathic porcelain system (G-Cera Cosmotech II, GC Co, Tokyo, Japan) and

a dual-cured resin composite cement (G-Cera Cosmotech II Composite, GC). The details of the clinical procedures have been described previously (Hayashi & others, 1998, 2000).

### **Preparation of Replicas**

For the quantitative analysis, replicas of the restored teeth were made at placement and eight years after restoration. A precise impression of the restored pre-

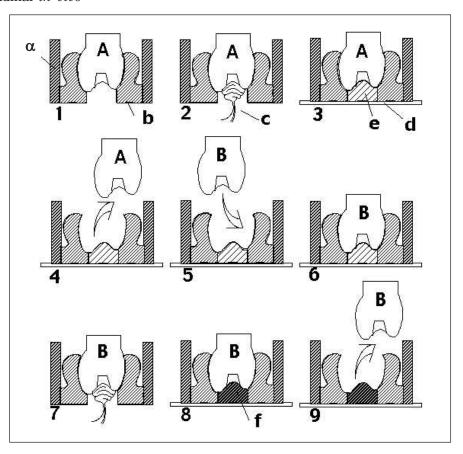


Figure 1. The apparatus for precise positioning.

A: replica at the time of placement; B: replica at eight years after restoration; a: metal frame, b: guide pattern, c: impression material, d: glass plate, e: impression of the occlusal surface of the initial replica, f: impression of the occlusal surface of the eight-year replica. 1: a replica taken at the time of placement was secured into a metal frame with a guide pattern; 2: an impression of the occlusal surface of the initial replica was made with a polyvinylsiloxane impression material; 3: the bottom of the apparatus was sealed with a glass plate; 4: the initial replica was removed from the apparatus and the surface of the impression was used for quantitative measurement; 5 and 6: a replica made eight years after restoration was inserted into the metal frame with the guide pattern and the initial impression of the occlusal surface; 7 and 8: an impression of the occlusal surface of the eight-year replica was made with a polyvinylsiloxane impression material; 9: the eight-year replica was removed from the apparatus and the occlusal surface of the impression was used for quantitative measurement.

Table 1: Distribution of Teeth Measured								
	First Premolar	Second Premolar	Total					
Upper premolar	5	2	7					
Lower premolar	2	6	8					
Total	7	8	15					

molar was taken with a polyvinylsiloxane impression material (Exafine, GC) that was then cast with epoxy resin (Araldite GY-1252 JP, CIBA-GEIGY GmbH, Wehr, Germany) to make an accurate replica.

### **Positioning Technique**

Since the accurate positioning of the replicas prepared at different times was essential for a precise analysis, a special apparatus was developed (Figure 1). First, a replica, made at the time the ceramic inlay was placed, was secured into a metal frame with a guide pattern made of MMA resin (Pattern Resin, GC) (Figure 1-1). Then, an impression material (Exafine, GC) was inserted onto the occlusal surface of the replica (Figure 1-2), after which the bottom of the apparatus was sealed with a glass plate (Figure 1-3). The replica was removed from the frame after curing the impression material and the impression pattern with a print of the occlusal surface was adopted as an initial pattern for quantitative measurement (Figure 1-4). Next, a replica made eight years after the initial placement was inserted into the metal frame guided by the resin pattern previously used and the initial occlusal pattern (Figures 1-5 and 1-6). Impression material was injected onto the occlusal surface of the eight-year replica after removing the initial pattern and sealing the bottom with the glass plate (Figures 1-7 and 1-8). When the eight-year replica was removed after curing the impression material, it was placed in the exact same position in the metal frame as the initial replica (Figure 1-9). Both impressions of the occlusal surface of the restored tooth could be used for superimposition for quantitative measurement.

### 3-D Morphological Measurement

Figure 2 shows a schematic diagram of the measuring device. It consists of a CCD laser displacement meter (LE-4000, Keyence Co, Osaka, Japan), an x-y stage (Mark-201/MSG-552, Sigma Koki Co, Tokyo, Japan) with an electric stage controller (Mark-201/MSG-552, Sigma Koki Co) and a personal computer (Mebius PC-PJ2, Sharp Co, Tokyo, Japan). The metal frame with the impression pattern prepared during the positioning procedure was fixed onto the x-y stage. The movement of the stage was controlled electrically by a 3-D measuring program (EMS98-3D, Sigma Koki Co) installed in the personal computer. The horizontal movement range of the stage was 50 mm in either direction. The morphological measurement of the marginal area was conducted at an interval of every 100 µm across the measurable whole margin of the restoration over a width of 5 mm at a measuring speed of 2000 µm/second.

A CCD laser scanner was employed for morphological measurement of the occlusal surfaces of the restorations. A sensor with a CCD laser was fixed onto the x-y stage and the surface of the replica was scanned with a 670 nm laser beam with a diameter of 30 µm. According

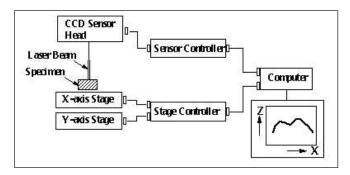


Figure 2. Schematic diagram of the hardware assembly.

to the manufacturer's instructions, the range of height measured was 5 mm and the measuring resolution of the device was  $0.1~\mu m$ .

The points generated by a single scan constituted a line profile. Data were accumulated in a controller and processed in the computer using the 3-D measuring program. Finally, a 3-D occlusal surface was constructed from a series of line profiles.

### **Superimposition of Profiles**

Two profiles of the same location obtained at the time of placement and eight years after the restoration were superimposed with graphic computer software (Claris Draw 1.0 version 2, Apple Computer Inc, Cupertino, CA, USA) using the outline of a restored tooth as a reference (Figure 3-upper). Quantitative marginal change was defined as the area enclosed by the two profiles and

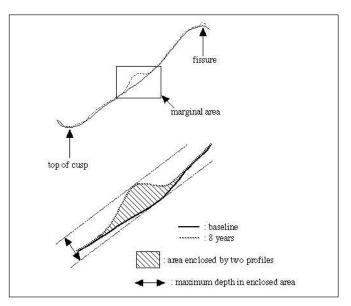


Figure 3. Superimposition of two profiles of marginal area. The area enclosed by two profiles, which were obtained at baseline and eight years after placement, was defined as the quantitative marginal change of a ceramic inlay. Two profiles were superimposed according to the outline of restored tooth as a reference (upper). The quantitative marginal change was represented by the maximum depth (µm) and the enclosed area (µm²)(lower).

was expressed as the maximum depth in µm and the area enclosed in µm² (Figure 3-lower). The maximum depth and area enclosed were calculated with picture analysis software (NIH image 1.61, NIMH, Bethesda, MD, USA).

### **Accuracy of Measurement**

The accuracy of the CCD measurement was analyzed in terms of distance and area. To assess the reproducibility of this method of distance measurement, a tooth model with a reference point  $80~\mu m$  in diameter and marked on the center of the occlusal surface was used. Five replicas with the reference point were duplicated from the original tooth using the same procedures that were used in the clinical situation. The original and duplicated specimens were positioned in the metal frame and the two occlusal surfaces were scanned with the CCD device. The profiles, including the reference points, were then constructed and superimposed, and the distances of the two profiles, including the reference point, were calculated every  $50~\mu m$ . The average dis-

crepancy was taken to represent the reproducibility of this method of measurement in terms of distance.

To determine the accuracy in area, the CCD measurement was compared with microscopic measurements. First, an epoxy replica was duplicated from a master tooth with reference points on the occlusal surface. Then, a gap, approximately 500  $\mu$ m in width and 1000  $\mu$ m in depth was prepared on the center of the occlusal surface of the master tooth. After preparing the gap, six epoxy replicas with gap and reference points were duplicated from the master tooth.

Second, an area of a cross-section of the gap was measured with the same procedures as measuring clinical models by means of the CCD laser scanner. The replicas before and after preparation of the gap were first positioned in the metal frame. Then, the two occlusal surfaces were scanned by CCD optical laser system, and the two profiles, including the gap and reference points, were superimposed with the guidance of the reference point and the outline of the teeth. The area enclosed by the two profiles was calculated by

Table 2: Comparison of Two Different Methods of Measurement Sample **Gap Area Measured Gap Area Measured Measuring Difference** x 100 (%) **Between Two Methods** Number by CCD Scanner (a) by Microscope (b) la-bl in Each Sample b  $(\mu m^2)$  $(\mu m^2)$ 1 617102 624519 1.19 2 633720 623893 1.58 3 616412 627583 1.78 628006 4 646626 2.96 5 643133 627916 2.42 6 613629 626628 2.07 628437 ± 14608  $2.00 \pm 0.63$ Mean ± SD 626424 ± 1797 \*: There was no significant difference in the area measured by two different methods (Mann-Whitney test, p>0.999)

	Disinteg	ration in	Maximum Depth	Disinteg	ration in		
	Mean	±	SE	Mean	±	SE	Number of
			(x10 µm)			(x10³ µm²)	<b>Measuring Points</b>
Premolars	8	±	0.5	40	±	3	285
Functional cusp area	10	±	0.8 ]*	50	±	5 ] ,	157
Non-functional cusp area	7	±	0.4	28	±	p=0.018	128
Upper premolars	8	±	0.6	42	±	4 7	137
Functional cusp area	9	±	0.9	47	±	6	81
Non-functional cusp area	7	±	0.5	35	±	3 <sub>#</sub> p=0.018	56
Lower premolars	8	±	0.8	38	±	4	148
Functional cusp area	11	±	1.3 7*	53	±	8 7.	76
Non-functional cusp area	6	±	0.6	22	±	$3 \int_{0}^{\infty} p < 0.0001$	72

software. The measurement was made five times for each replica and the mean value of the area calculated was taken to represent the area of the gap.

Third, the size of the gap in the replica models previously measured by the CCD system was measured microscopically. A cross-sectional specimen of a replica model that included both the gap and the reference points was prepared by means of a slow-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA). The profile of the gap in the cross-sectional specimen was then captured by means of a reflection microscope (SMZ-U, Nikon, Tokyo, Japan), and the cross-sectional area of the gap was calculated using the software.

Finally, values of the cross-sectional areas of the gap obtained from the two different methods were compared in each specimen. The difference between the measurements was presented as a percentage of the value obtained from the microscopic measurement. The mean difference in percentage was taken to represent the measuring accuracy in area.

### Statistical Analysis

The marginal changes were compared in terms of the type of tooth using the Mann-Whitney test and location of margin using the Wilcoxon signed-ranks test at a 95% level of confidence. The results of the accuracy test in the two methods of measurement were also compared with the Mann-Whitney test at a 95% level of confidence.

### **RESULTS**

### **Accuracy of Measurement**

Measurement of the discrepancy of the two profiles, including the reference point, was made in 10 positions for each specimen. The average discrepancy was calculated as  $4.3 \pm 3.2 \, \mu m$ , which was defined as the reproducibility of the measuring method in distance.

Table 2 summarizes the results of the measurement and differences between the two methods. The mean area of the gap was measured as  $628437 \pm 14608 \ \mu m^2$  using CCD scanning and as  $626424 \pm 1794 \ \mu m^2$  using microscopic measurement. There was no significant difference in the area measured by the two methods (Mann-Whitney test, p > 0.999). The mean difference in the areas between the two methods of measurement was  $2.0 \pm 0.6\%$ . This measuring error was defined as the accuracy of the CCD measurement in area.

### **Quantitative Marginal Change**

Table 3 presents quantitative marginal changes in the ceramic inlays over an eight-year period. Quantitative marginal change is expressed as the maximum depth in the area enclosed by the two profiles and the average area enclosed.

When the marginal change is expressed as a distance, the relevant figures are presented as Ax10 µm, with the inclusion of a measurement error of 4.3 µm. When expressing marginal change in area, the relevant figures are presented as Ax10³µm², with the inclusion of a measurement error of 2.0%.

The quantitative marginal changes of Class II ceramic inlays in premolars eight years after placement were  $10x10~\mu m$  in depth and  $50x10^3~\mu m^2$  in area for the functional cusp and  $7x10~\mu m$  in depth and  $28x10^3~\mu m^2$  in area for the non-functional cusp area. There were significant differences in marginal disintegration in both depth and area between the two areas (Wilcoxon signed-ranks test, p<0.05). Furthermore, disintegration of the upper premolars was significantly greater than the lower premolars in area (Mann-Whitney test, p<0.05).

### DISCUSSION

Surface changes in posterior restorations have previously been measured quantitatively with the aid of 3-D digitization and a computer graphic system (Williams & others, 1983; Lambrechts & others, 1994; DeLong, Pintado & Douglas, 1985; Braem & others, 1986; Hewlett, Orro & Clark, 1992; Dastane & others, 1996; Mehl & others, 1997; Roulet & others, 1997). The accuracy of measurement used for these investigations is extremely important and has been extensively reported and discussed.

In previous studies, the surface topography of the restorations was obtained by means of optical contouring of the surface using a laser, and the surface change was calculated by superimposing two contour maps generated at different times (Williams & others, 1983). The accuracy of this method was reported to be 2% to 5% in volume. A method capturing and displaying anatomic surface contours was developed by using a combination of computer graphics and servohydraulics with an accuracy of 12% in volume (Delong & others, 1985). Another quantitative measurement of wear of posterior composite restorations used a newly developed optical 3-D device in combination with reference-free automated 3-D superimposition software (Mehl & others, 1997). The in vivo accuracy of this system was reported as 10 µm in distance.

Accuracy of the CCD measurement in this study was 4.3 µm in distance and 2.0% in area. This accuracy can be considered comparable to other previous studies. The data obtained in the current study should be worth evaluating if accuracy of the measurement is given proper consideration.

Various patterns of marginal disintegration observed with a scanning electron microscope in the authors' previous study (Hayashi & others, 2000) demonstrated that area, depth and width of disintegration varied

widely depending on the restoration. Moreover, disintegration even showed different patterns in different areas of a single tooth. In some areas, for example, disintegration was advanced accompanied by obvious marginal fracture, and in other areas in the same restoration, only slight wear of resin composite cement was detected along the margin. Therefore, the quantitative marginal change measured did not show normal distributions. Even after a logarithmic transformation had been performed, distributions were far from normal. Hence, non-parametric analyses were performed to compare marginal change in terms of type of tooth and location of margin.

The statistical analysis of these findings suggests that occlusal force is clinically significant to accelerate marginal disintegration, since disintegration at the functional cusp side was significantly more severe than that at the non-functional cusp side. This means that for clinical procedures of ceramic inlay restorations, marginal design should be decided carefully and accurately to avoid excessive loading on the margin.

Further investigations are needed to assess the longitudinal disintegration of ceramic inlays by evaluating replicas made at various periods with the quantitative measuring system developed for the study presented here.

### **CONCLUSIONS**

This study constituted a quantitative measurement of *in vivo* marginal disintegration of ceramic inlays by means of a CCD optical laser scanner system. Occlusal force accelerated marginal disintegration.

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## Comparative Depths of Cure Among Various Curing Light Types and Methods

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### Clinical Relevance

Depth of cure of LED and QTH curing lights is light unit and exposure mode dependent.

### **SUMMARY**

This study evaluated the depth of cure associated with commercial LEDs (light-emitting diodes) (Elipar FreeLight [FL], 3M-ESPE; GC e-Light [EL], GC), high intensity (Elipar TriLight [TL], 3M-ESPE) and very high intensity (Astralis 10 [AS], Ivoclar Vivadent) Quartz Tungsten Halogen (QTH) curing lights. Depth of cure of the various lights/curing modes were compared to a conventional QTH light (Max [Mx], Dentsply-Caulk). Ten exposure regimens were investigated: FL1 - 400 mW/cm² [40 seconds]; FL2 - 0-400 mW/cm² [12 seconds] → 400 mW/cm² [28 seconds]; EL1 - 750 mW/cm² [10 pulses x 2 seconds], EL2 - 350 mW/cm² [40 seconds]; EL3 - 600 mW/cm² [20 seconds];

 $EL4 - 0 - 600 \text{ mW/cm}^2 [20 \text{ seconds}] \rightarrow 600 \text{ mW/cm}^2$ [20 seconds]; TL1 - 800 mW/cm<sup>2</sup> [40 seconds];  $TL2 - 100-800 \text{ mW/cm}^2 [15 \text{ seconds}] \rightarrow 800 \text{ mW/cm}^2$ [25 seconds]; AS1 - 1200 mW/cm<sup>2</sup> [10 seconds]; MX - 400 mW/cm<sup>2</sup> [40 seconds]. Depth of cure was determined by penetration, scraping and microhardness techniques. The results were analyzed using one-way ANOVA/Scheffe's post-hoc test and Pearson's correlation at significance level 0.05 and 0.01, respectively. All light curing regimens met the ISO depth of cure requirement of 1.5 mm with the exception of EL1-EL3 with the microhardness technique. Curing with most modes of EL resulted in significantly lower depths of cure than the control [MX]. No significant difference in depth of cure was observed among the control and the two modes of FL. Curing with TL1 resulted in significantly greater depth of cure compared to MX with all testing techniques. No significant difference in depth of cure was observed between the control and AS1 for all testing techniques except for the penetration technique. The depth of composite cure is light unit and exposure mode dependent. Scraping and penetration techniques were found to correlate well but tend to overestimate depth of cure compared to microhardness.

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### INTRODUCTION

LED (light-emitting diodes) light curing units (LCUs) were developed to overcome several inherent disadvantages of Quartz Tungsten Halogen (QTH) LCUs. Fujibayashi and others (1998) have found that an LED source with the same irradiance as a QTH source produced a significantly greater depth of cure than a QTH source. In addition, Mills, Jandt and Ashworth (1999) reported that LED LCUs with lower irradiance than halogen LCUs can produce greater depths of cure. Other cited advantages of LED LCUs are operational lifetimes of more than 10,000 hours, little or no degradation of light output over time and no need for filters (Mills & others, 1999). The small, compact size and low power consumption also make LED curing lights portable. Most importantly, the narrower spectral output of blue LED falls within the absorption profile of the photoinitiator, camphorquinone (CQ) (450-500 nm with peak absorption at 470 nm) (Lee & others, 1993).

The use of high intensity light sources was introduced to decrease exposure time and increase depth of cure (Tanoue, Matsumura & Atsuta, 1999). However, curing composites with a high intensity light may demonstrate significant disadvantages due to increased shrinkage stress (Unterbrink & Muessner, 1995). High intensity lights provide higher values of degree of conversion and superior mechanical and physical properties but produced higher contraction strain rates during polymerization of composites (Uno & Asmussen, 1991). The use of very high light intensities for short durations (turbo cure) has also been developed. These curing regimens were established primarily to reduce clinical time and have been shown not to increase polymerization stresses if the total light energy density (intensity x time) is maintained (Yap, Wong & Siow, 2003).

Depth of cure can be defined as the extent of quality resin polymerization deep from the surface of composite restoratives. The extent of resin cure is affected by filler size, light source intensity, duration of exposure and resin shade (Rueggeberg & others, 1993). Studies show that darker shades exhibit lower depth of cure when compared to lighter shades (Newman, Murray & Yates, 1983; Swartz, Phillips & Rhodes, 1983). However, Ferracane and others (1986) demonstrated that depth of cure of light-activated resin composites of the darkest shade were equivalent to that of the lightest shade, and hence, suggested that depth of cure may be less dependent upon shade than translucency. Intensity of the light source and attenuating power of the material are two important factors that influence the depth of cure (Rueggeberg & others, 1993). Light attenuation in the material is controlled by both absorption and scattering of filler particles (Ruyter & Øysæd, 1982). Hence, light transmission of resin composite and the use of a specific light source system are two important factors in achieving greater cure depth (McCabe & Carrick, 1989). The presence of inadequate polymerization throughout the restoration bulk can lead to undesirable effects such as gap formation, marginal leakage, recurrent caries, adverse pulpal effects and ultimate failure of the restoration (Ferracane, 1993).

Depth of cure for light-activated dental composites may be assessed directly or indirectly. Indirect methods of assessment include scraping (Cook, 1980), visual (Murray, Yates & Newman, 1981) and surface hardness (Asmussen, 1982). Microhardness testing with increasing depth in a composite has been used in many studies, because surface hardness has been shown to be an indicator of the degree of polymerization (Asmussen, 1982). Direct methods that assess the degree of conversion, such as infrared spectroscopy and laser Raman spectroscopy, have not been accepted for routine use, as they are complex, expensive and time-consuming (Rueggeberg & Craig, 1988). DeWald and Ferracane (1987) compared four commonly used methods for evaluating depth of cure in light-activated composites. They found that visual and scraping methods correlated well, but severely overestimated depth of cure as compared with microhardness testing or degree of conversion. Degree of conversion appeared to be the most sensitive test for cure. A good correlation between the results of Knoop Hardness and infrared spectroscopy was also reported. Microhardness testing appears to be the most popular method for investigating factors that influence cure because of the relative simplicity of the method.

This study determined the depth of cure of LED, high intensity and very high intensity QTH lights using different curing modes. Curing depths were determined by three different test methods. The depth of cure with these lights were compared with conventional QTH light.

### **METHODS AND MATERIALS**

A mini-filled resin composite (Shade A2, Z100; 3M-ESPE, St Paul, MN, USA; Lot #20010517) was used in this study. The five LCUs selected for this study are detailed in Table 1. The 10 light exposure regimens are also featured in Table 1. Intensity of all light units was measured with a hand-held dental curing radiometer (Cure Rite, EFOS INC, Ontario, Canada) to ensure consistency in output. The depth of cure was determined by three methods: scraping, penetration and microhardness.

Scraping Method: This test methodology was performed according to methods of ISO 4049 (2000) (International Organization for Standardization for polymer based filling materials). Uncured composite was placed in black Teflon molds with square cavities 6.7-mm deep and 4-mm wide and confined between two opposing acetate strips (Hawe-Neos Dental, Bioggio,

Switzerland). A white delrin base was used beneath the molds. 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 exposure modes. Immediately after light exposure, the acetate strips were removed and the specimens released from their molds. Uncured material was removed using a plastic spatula. The height of the remaining cured material was measured with a digimatic caliper (Model CD-6CS; Mitutovo Corporation, Kanagawa, Japan) with precision of 0.01 mm. Depth of cure was also reported as the total remaining height after uncured material was removed (ISO 4049, 1988) [S1] divided by 50% of the remaining length (ISO 4049, 2000) [S2]. Five specimens were prepared for each exposure mode.

Penetration Method: The methodology used in this evaluation was based upon that used by Harrington and Wilson (1993). A microtester (Model 5848, Instron Corporation, Canton, MA, USA) was used as a penetrometer. Specimen preparations were identical to the scraping method. Immediately after light curing, the specimens in their molds were inverted with the unexposed surface facing the penetration needle (Figure 1). A force of 12.5 N was exerted after light exposure through a 0.5 mm-diameter needle at a rate of 1-mm/minute in the middle of the unexposed, bottom surface.

Depth of cure of the specimens was computed using the formula:



Figure 1. Pictorial illustration of depth of cure by penetration technique.

LCU	Curing Modes	Curing Profiles	
Elipar FreeLight (LED)	Standard (FL1)	400 mW/cm² (40 seconds)	
3M-ESPE, Seefeld, Germany	Exponential (FL2)	0-400 mW/cm² → (12 seconds)	400 mW/cm <sup>2</sup> (28 seconds)
GC e-Light (LED)	Pulse Curing (EL1)	750 mW/cm² (10 pulses x 2 seconds)	
GC Europe, Leuven, Belgium	Standard (EL2)	350 mW/cm <sup>2</sup> (40 seconds)	
	Turbo (EL3)	600 mW/cm <sup>2</sup> (20 seconds)	
	Soft-start curing A (EL4)	0-600 mW/cm² → (20 seconds)	600 mW/cm <sup>2</sup> (20 seconds)
Max (Halogen)	Standard (MX)	400 mW/cm <sup>2</sup> (40 seconds)	
Dentsply-Caulk, Milford, DE, USA			
Elipar TriLight (Halogen)	Standard (TL1)	800 mW/cm² (40 seconds)	
3M-ESPE, Seefeld, Germany	Exponential (TL2)	100-800 mW/cm² → (15 seconds)	800 mW/cm <sup>2</sup> (25 seconds)
Astralis 10 (Halogen)	High Power (AS1)	1200 mW/cm² (10 seconds)	
Ivoclar-Vivadent, Schaan, Liechtenstein			

Depth of cure = depth of mold (6.7 mm)—depth of penetration (1)

Microhardness Method: The specimen fabrication for this method is similar to that described in the scraping protocol. Immediately after light exposure, all acetate strips were removed and the specimens were positioned in their molds centrally beneath the indenter of a digital microhardness tester (FM7, Future-Tech Corp, Tokyo, Japan) to assess the microhardness (Knoop Hardness Number [KHN]) of the top surface. A 500g load was 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 using the formula:

$$KHN = 14.2 \times (F/d^2)$$
 (2)

where F is the test load in kgf and d is the longer diagonal length of an indentation in millimeters. KHN values of side surfaces were measured at 1-mm intervals from the top surface, using the same testing parameters (Figure 2). 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. Depth of cure was set to at least 80% of the top surface hardness.

Statistical analysis was determined using two-way analyses of variance (ANOVA). Data were also subjected to one-way ANOVA/Scheffe's post-hoc test at a significance level of 0.05. Data from the three testing methods were also analyzed using Pearson's Correlation at significance level 0.01.

### **RESULTS**

Table 2 summarizes and compares the depth of cure of different light curing regimens as evaluated by the different testing methods. The depth of cure evaluated by the scraping and penetration techniques was higher than the microhardness technique. Table 3 shows the mean KHN obtained at increasing depths using the microhardness method.

Table 4 shows the significant differences in mean depth of cure between the various LCUs modes and the control light source (MX). Two-way ANOVA revealed significant interaction between exposure modes and testing methods. Therefore, the effect of exposure modes on depth of cure was test method-dependent. For the S1 and S2 techniques, all four exposure modes (EL1-EL4) of GC e-Light had significantly lower depth of cure than the control (MX), while both exposure modes (TL1, TL2) of Elipar TriLight had greater depth of cure than MX. For the penetration technique, depth of cure of MX was significantly greater than all four exposure modes of GC e-Light (EL1-EL4) and AS1 but lower than TL1. For the microhardness technique, depth of cure of MX was significantly greater than EL1 and EL3, and lower than TL1.

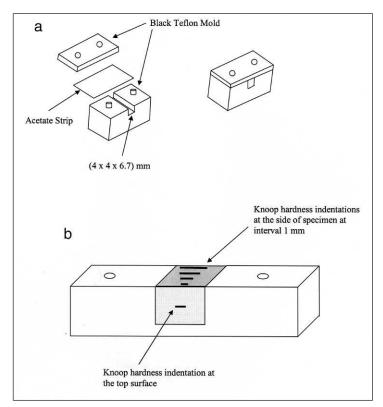


Figure 2. Schematic illustration of (a) the preparation of specimens for Knoop Hardness indentations and (b) increasing Knoop Hardness indentations with depth in a cross-sectional plane of a composite mold.

Table 5 shows the correlations among the different methods. A significantly strong (correlation coefficient, r=0.93) and positive relationship between penetration and scraping (S1 and S2) tests was observed. No significant correlation was observed between microhardness and penetration methods and between microhardness and scraping (S1 and S2) methods.

### DISCUSSION

Shade A2 was selected to minimize the effects of colorants on light polymerization (Bayne, Heymann & Swift, 1994). As a minimum intensity of 400 mW/cm² has been suggested for routine polymerization (Rueggeberg, Caughman & Curtis, 1994; Tate, Porter & Dosch, 1999), this light intensity (Max polymerization unit), together with the manufacturer's recommended exposure duration of 40 seconds was used as control in this study.

The depth of cure decreased with increasing depth, as observed in the results that used the microhardness method (Table 3). Material nearer to the light source underwent more complete polymerization and was thus harder. In six out of 10 exposure modes, higher hardness values were observed at 1 mm below the surface as compared to the top. This result may be due to oxygen inhibition, a finding corroborated by Unterbrink and Muessner (1995). The depth of cure of

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EL, which consisted of 64 LEDs, was significantly lower than that using a conventional QTH light (MX). However, depth of cure of TL, the high intensity light, was found to be significantly higher than MX for all test methods used in this study. FL, consisting of 19 LEDs, and AS1 had depths of cure comparable MX. A possible reason for this finding may be related to differences in total light energy delivered to the specimen (intensity x time). Energy density of standard the mode of EL, FL, MX, TL and AS1, computed according to the manufacturer profiles, was found to be 14,000, 16,000, 16,000, 32,000 and 12,000 mJ/cm<sup>2</sup>, respectively. The inferior depth of cure observed in EL may be due to lower light ener-

Modes (	Using the Differe	nt Testing Techniq	ues (n=5)	•						
LCU	Curing		Curin Depth (mm)							
	Modes	Scraping 1 (ISO 1988)	Scraping 2 (ISO 2000)	Penetration	Micro- indentation					
Elipar	FL1	6.15 (0.02)	3.07 (0.01)	6.01 (0.07)	2.00 (0.00)					
FreeLight	FL2	5.84 (0.11) 2.92 (0.06) 5.96 (0.07) 2.0								
GC e-Light	EL1	4.85 (0.20)	2.43 (0.10)	4.89 (0.08)	1.00 (0.00)					
	EL2		2.66 (0.05)	4.95 (0.20)	1.20 (0.45)					
	EL3	4.67 (0.12)	2.34 (0.06)	4.75 (0.11)	1.00 (0.00)					
	EL4	4.97 (0.04)	2.49 (0.02)	5.06 (0.08)	2.00 (0.00)					
Max	MX	5.86 (0.19)	2.93 (0.10)	6.04 (0.11)	1.80 (0.45)					
Elipar	TL1	6.41 (0.15) 3.21 (0.08) 6.48 (0.1		6.48 (0.11)	3.00 (0.00)					
TriLight	TL2	6.26 (0.10)	3.13 (0.05)	6.31 (0.06)	2.40 (0.55)					
Astralis 10	AS1	5.94 (0.10)	2.97 (0.05)	5.62 (0.24)	2.00 (0.00)					

Table 2: Mean Depths of Cure (SD) Observed for the Different LCUs and Their Respective Exposure

LCU	Curing	Тор		Distance	from Top Su	ırface (mm)	
	Modes	KHN	1	2	3	4	5
Elipar FreeLight	FL1	77.5 (0.6)	77.4 (1.6)	67.6 (1.9)	51.3 (4.0)	28.0 (4.8)	9.7 (0.7)
	FL2	71.4 (1.1)	77.9 (2.3)	69.5 (2.2)	54.5 (1.6)	30.8 (2.4)	9.4 (0.0)
GC e-Light	EL1	74.2 (1.9)	67.5 (2.8)	49.8 (4.4)	27.0 (4.0)	9.7 (0.9)	-
	EL2	75.1 (1.3)	72.5 (2.2)	51.6 (9.3)	37.7 (6.4)	14.5 (5.6)	-
	EL3	73.4 (1.0)	74.9 (1.3)	54.0 (2.7)	30.0 (5.0)	9.8 (0.6)	-
	EL4	76.8 (0.2)	77.7 (4.8)	64.3 (1.7)	40.1 (1.2)	10.0 (0.5)	-
Max	MX	70.2 (2.5)	74.3 (6.1)	62.6 (7.9)	42.9 (5.4)	-	-
Elipar TriLight	TL1	76.8 (1.6)	81.2 (2.1)	75.0 (1.3)	64.1 (1.8)	40.5 (4.8)	16.5 (6.2)
	TL2	74.8 (0.6)	77.4 (1.0)	69.3 (3.0)	57.4 (4.8)	36.8 (5.4)	11.8 (2.8)
Astralis 10	AS1	75.4 (0.5)	73.7 (0.7)	63.5 (2.1)	44.9 (5.0)	22.0 (5.8)	-

gy density, wider emission spectrum and lower thermal emission produced when compared to the QTH lights. It was speculated that heat produced by the curing lights may be useful for the polymerization process. The very high intensity LCU (AS1), which has an energy density lower than EL, had a depth of cure comparable to the control. This may be attributed to the high thermal energy produced by the curing unit. The thermal emission of the different LCUs has been examined in an earlier study. Results suggest that heat produced by the different LCUs may play a role in the polymerization process of composites (Yap & Soh, 2003).

n=5 specimens per test condition

Depth of cure using the different light curing regimens met the ISO depth of cure requirement of 1.5 mm, except for most modes of GC e-Light evaluated using the microhardness technique. Differences in depths of cure were observed among the three different methods evaluated in this study. Despite the slight variation in results observed for penetration and scraping methods, good correlation between the two techniques was observed. The ISO scraping technique used to determine depth of cure was easy to perform and required minimal instrumentation. However, this test provides no indication of quality of cure at any point, including the lower layers adjacent to the soft resin that was

removed (Yearn, 1985). While the ISO defines depth of cure as 50% of the length of composite specimens after removal of the uncured material, some studies (Swartz & others, 1983; DeWald & Ferracane, 1987; Baharav & others, 1988: Hansen & Asmussen. 1993) have defined depth of cure as the total remaining length after uncured material is removed. Results obtained in this study demonstrate that S1 severely overestimated depth of cure, while S2 was found to be more reasonable in determining this parameter, with values closer to the microhardness technique. Depth of cure for most exposure modes or LCUs, except some modes of EL, was found to be 2 mm, while a value of 3 mm was achieved by TL1 using microhardness and supported by S2 method.

The penetration method proved to be simple, reproducible and better than

the simple scrape test adopted for the standard specifications. Measurements were made near the center of the composite mold and well away from mold walls. Both the penetration and scraping methods measure the height of cured specimen, the main difference is that the penetration technique applies a constant force, allowing for consistency of results. Both methods were found to correlate well, and this excellent relationship accounted for the almost similar results obtained. However, both techniques overestimated depth of cure when compared to hardness testing. This corroborated with the results of DeWald and Ferracane (1987), Yearn (1985) and McCabe and Carrick (1989).

The microhardness technique indicates that cure within the body of the composite falls from that at the surface. This method does not quantitatively predict the actual levels of conversion achieved. Good correlation was, however, observed between the degree of conversion and hardness testing at the top surfaces (Hansen & Asmussen, 1993). A laboratory technique using microhardness has been shown to provide a convenient means of assessing cure throughout the depth of a composite sample that relates to the clinical situation. Using this method, it is possible to evaluate the influence of chemical formulation factors, the nature of the light source and the control exercised by clinicians in determining the quality of cure achieved and hence long-term performance of the restoration (Yearn, 1985).

Studies (Fujibayashi & others, 1998; Mills & others, 1999; Mills & others, 2002; Mills, Uhl & Jandt, 2002) have shown that an LED LCU, with a lower or similar irradiance than the QTH LCU, is capable of achieving a greater depth of cure. These observations were attrib-

Table 4: Statistical Analysis of Depth of Cure of Various LCUs Modes Compared to a Conventional QTH LCU for the Different Techniques

Techniques

Significance

Scraping 1 (ISO 1988)

EL1, EL2, EL3, EL4 < MX < TL1, TL2

Scraping 2 (ISO 2000)

EL1, EL2, EL3, EL4 < MX < TL1, TL2

Penetration

EL1, EL2, EL3, EL4, AS1 < MX < TL1

Microhardness

EL1, EL3 < MX < TL1

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

Table 5: Correlations Among the Different Techniques Used for the Determination of Depth of Cure									
Techniques	Penetration	Microhardness	Scraping 1 (ISO 1988)	Scraping 2 (ISO 2000)					
Penetration	-	NC	0.931 (S)	0.931 (S)					
Microhardness	NC	-	NC	NC					
Scraping 1 (ISO 1988)	0.931 (S)	NC	-	1.00 (S)					
Scraping 2 (ISO 2000)	0.931 (S)	NC	1.00 (S)	-					
S indicates statistical signi	ificance while NC denotes	s no correlation.	1						

uted to the LED LCUs emission spectra, which coincide with the absorption spectrum of the camphorquinone (CQ) photoinitiator present in the composite. Although LED LCUs achieve greater or similar depths of composite cure, not all LED LCUs have the same performance, as the results of this study show. Depth of cure with most exposure modes of EL was found to be lower than the QTH control, while all modes of FL were found to be comparable to the control. Hence, more studies on blue LED technology are warranted.

### **CONCLUSIONS**

Within the conditions imposed in this *in-vitro* study and by using the specific exposure duration values recommended by the manufacturers, the following conclusions can be made:

- Depth of cure associated with LED and QTH curing lights was light unit and exposure mode dependent.
- 2. Both penetration and scraping techniques correlated well but overestimated depth of composite cure.
- 3. Depth of cure of all exposure modes of GC e-Light was generally significantly lower than the conventional QTH light.
- 4. Depth of cure of Astralis 10 was comparable to conventional QTH light when evaluated by S1, S2 and microhardness techniques.
- 5. Depth of cure of Elipar FreeLight was comparable to conventional QTH light.
- 6.Depth of cure of Elipar TriLight was higher than the conventional QTH light.

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# Simultaneous Release of Fluoride and Aluminum from Dental Materials in Various Immersion Media

MF Hayacibara • GMB Ambrosano • JA Cury

### Clinical Relevance

The anticariogenic potential of dental materials that release fluoride and aluminum can change depending on the media used to evaluate them.

### **SUMMARY**

Since fluoride (F<sup>-</sup>) and aluminum (Al<sup>3+</sup>) present anticariogenic properties and F release from ionomeric materials depends on the media used in the evaluation, this study tested the hypothesis whether release of Al3+ also depends on the testing media. The simultaneous release of Fand Al3+ was assessed over 15 days in three media: (i) distilled and deionized water (H<sub>2</sub>O), (ii) artificial saliva (AS) and (iii) solutions simulating a cariogenic challenge (pH-cycling in demineralizing and remineralizing solutions, De-Re-). Six cylindrical samples of each tested material (Ketac-Fil, Vitremer, Fuji Ortho LC and F 2000) were prepared and suspended individually in 1.0 mL of each solution. All solutions were changed daily. F<sup>-</sup> and Al<sup>3+</sup> were determined by ion-selective electrode and atomic absorption, respectively.

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ANOVA showed statistical significance for the interaction of material, time, media (p<0.05), either for F<sup>-</sup> or Al<sup>3+</sup> release. The resin-modified glass ionomer Vitremer released the highest amount of F<sup>-</sup> and Al<sup>3+</sup> in De-Re- solutions compared to the other materials (p<0.05); differences among the materials in H<sub>2</sub>O and AS were statistically consistent. The data suggest that the media used to evaluate the simultaneous release of F<sup>-</sup> and Al<sup>3+</sup> should be taken into account when the anticariogenic potential of different dental materials is assessed.

### INTRODUCTION

Glass ionomer cement (GIC) has been recommended for the prevention of secondary caries, which so far, is the most frequent reason for replacement of restorations (Kidd, Toffenetti & Mjör, 1992; Mjör, 1997). The anticariogenic properties of glass ionomer cements have been attributed to their ability to release fluoride (F<sup>-</sup>) (Wilson & McLean, 1988; Forsten, 1998), although they also release other substances (Forss, 1993; Czarnecka, Limanowska-Shaw & Nicholson, 2002).

The amount of F<sup>-</sup> released by GIC has been considered an indicator of the anticariogenic potential of these materials. The F<sup>-</sup> release evaluations have been made *in vitro* using different storage media (El Mallakh & Sarkar, 1990; Lavis & others, 1997; Savarino & others, 2000; Dhondt, De Maeyer & Verbeeck, 2001;

Material	Manufacturer	Batch #	Nomenclature
			(McLean, Nicholson & Wilson, 1994
Ketac-Fil	ESPE—Seefeld, Germany	# FW0043052	Glass-ionomer cement
Vitremer	3M—Dental Products Division, St Paul, MN, USA	# 19960904	Resin-modified glass ionomer
Fuji Ortho LC	GC Corporation - Tokyo, Japan	# 260971	Resin-modified glass ionomer
F 2000	3M—Dental Products Division, St Paul, MN, USA	# 19970819	Polyacid-modified resin

Czarnecka & others, 2002) and most of them are subject to criticism, because they do not simulate saliva composition or they do not mimic the process of caries development. Furthermore, since F<sup>-</sup> release depends on the media used, comparison of the anticariogenic potential of different GIC can be misunderstood, depending on the media used in the evaluation (Carvalho & Cury, 1999).

Among the other substances released by GIC, aluminum ( $Al^{3+}$ ) should be mentioned for several reasons. It is the major constituent of these materials (Wilson & McLean, 1988) and it is leached over an extended period of time (Andersson & Dahl, 1994; Nakajima, Komatsu & Okabe, 1997; Czarnecka & others, 2002). In addition, it may have synergistic anticariogenic properties, because the inhibition of *Streptococcus mutans* ATPase is highest when F<sup>-</sup> and  $Al^{3+}$  are combined (Sturr & Marquis, 1990). Furthermore, recent data have shown that dental materials that simultaneously release F<sup>-</sup> and  $Al^{3+}$  are more efficient in reducing *S mutans* acidogenicty than those that basically release F<sup>-</sup> (Hayacibara & others, 2003).

However, while  $F^-$  release has been extensively studied, the data concerning  $Al^{3+}$  are still scarce. In addition there is no information on whether the release of  $Al^{3+}$  is dependent on the media used, as shown for  $F^-$ . Considering that  $F^-$  and  $Al^{3+}$  present anticariogenic properties, evaluating their simultaneous release should be done under more appropriate conditions.

Therefore, this study tested the hypothesis that the release of Al<sup>3+</sup> from glass-ionomer cements, polyacid-modified resins and resin-modified glass ionomer depends on the media used in the evaluation.

### **METHODS AND MATERIALS**

### **Experimental Design**

Eighteen disc specimens (10.5 mm in diameter and 2.5 mm in thickness) of each material (Table 1) were prepared and randomly distributed for immersion among three different storage media: (i) deionized distilled water ( $\rm H_2O$ ), (ii) artificial saliva (AS) and (iii) solutions simulating a cariogenic challenge (pH-cycling in demineralizing and remineralizing solutions, De-Re). The storage solution was changed daily during an experi-

mental period of 15 days (Carvalho & Cury, 1999) and  $F^-$  and  $Al^{3+}$  released were analyzed in the remaining solutions. Six specimens were used for each immersion media that was tested (Carvalho & Cury 1999).

### **Preparation of Specimens**

A total of 72 specimens (18 of each material, six disks for each medium) were prepared at room temperature (23±1.0°C and 50±5% relative humidity), according to the ISSO #7489 specification. The materials were mixed according to the manufacturers' recommendations, placed in plastic molds and pressed between polyester matrix and glass plates. A piece of nylon was incorporated into the cements during setting to suspend the samples in the test medium. The materials were light cured for 40 seconds on both upper and lower surfaces, and the conventional glass-ionomer cement, Ketac-Fil, was allowed to set under pressure for 10 minutes. After hardening, the specimens were stored in an oven (37°C, 100% relative humidity) for 24 hours before starting the experimental phase (Carvalho & Cury 1999).

### **Experimental Phase**

All specimens were suspended individually in 1.0 mL of each studied solution in polyethylene tubes cleaned by soaking in 10% HNO<sub>3</sub> for 24 hours and rinsed with distilled and deionized water. The storage media used were: distilled and deionized water; artificial saliva comprised of Ca 1.5 mM, PO<sub>4</sub> 0.9mM, KCL 150 mM and Tris [tris-(hydroxymethyl)aminomethane] buffer 20 mM, pH 7.0, containing NaN3 0.02%; and pH cycling system—Demineralizing (Ca 2.0 mM, PO<sub>4</sub> 2.0 mM and acetate buffer 75 mM, pH 4.3 containing NaN<sub>3</sub> 0.02%) and remineralizing (the same as artificial saliva) solutions. The tubes were placed in a shaking bath at 37°C. The storage media were changed daily as follows: distilled and deionized water and artificial salivachanged every 24 hours; pH-cycling system—the specimens were immersed for six hours in demineralization solution (pH 4.3) and 18 hours in remineralization solution (pH 7.0) as described by Carvalho and Cury (1999). After the prescribed immersion time, the disks were removed from the solutions in which F- and Al3+ concentrations were determined.

### Fluoride and Aluminum Analysis

An initial pilot study was undertaken to verify which method would provide more accuracy in the F- determination considering the high calcium and Al<sup>3+</sup> concentrations present in the solutions released from some materials. Among the use of (i) TISAB II (acetate buffer 1.0 M, pH 5.0 containing NaCl 1.0 M and 1,2 cyclohexanediaminetetracetic 0.4%), (ii) TISAB III (total ionic strength adjustment buffer, Orion, no 940911, Boston, MA, USA) and (iii) Microdifusion Technique (Taves, 1968), TISAB III was chosen because the result

found did not differ statistically from that observed by microdiffusion. Furthermore, TISAB II was not able to avoid F complex with other ions released from some material.

TISAB III was added to the solutions in the proportion 1:10. The remineralizing and demineralizing solutions were joined and analyzed. The F<sup>-</sup> electrode was previously calibrated with a series of five standard solutions (0.065, 0.125, 0.250, 0.500 and 1.000 mg F<sup>-</sup>/ mL) in triplicate and the samples analyzed in duplicate.

Aluminum in the solutions (De-Re solutions were joined) was determined in duplicate by atomic absorption using nitrous oxide, acetylene flame and a hollow cathode lamp at 309.3 nm. The spectrophotometer (VARIAN—AA-50, Mulgrave, Australia) was calibrated with five standard solutions ranging from 0.5 to 25.0 µg Al³+/mL and all samples were analyzed straight, with no pre-treatment procedure. The sensitivity limit of this analysis was 0.1 µg Al³+/mL.

The results obtained from each sample were divided by the area of the specimen  $(250 \mu m^2)$  and expressed in  $\mu g/cm^2$ . The cumulative release during the 15 days was considered for statistical analysis of the difference between the materials and medium.

### **Statistical Analysis**

The results were analyzed by two-way ANOVA followed by Tukey test (p<0.05). Pearson's correlation analysis was undertaken between total fluoride and aluminum release in each media.

### **RESULTS**

The pattern of F<sup>-</sup> and Al<sup>3+</sup> release in deionized water ( $\rm H_2O$ ), artificial saliva (AS) and the pH-cycling solutions (De-Re) over time are illustrated in Figures 1, 2 and 3. Vitremer released the highest amount of F<sup>-</sup> and F2000 the lowest throughout the period

analyzed considering the media De-Re solutions. However, these results changed when the media  $H_2O$  or AS were considered. Similar to F-, Vitremer released the highest amount of  $Al^{3+}$  throughout the time evaluated. The concentration of  $Al^{3+}$  in  $H_2O$  and AS found in the solutions after the initial few days of immersion was, for most of the materials, below the quantification limit of the analysis method used.

Considering the statistical significance for the interaction material x media, Table 2 shows the cumulative release of F<sup>-</sup> according to the materials and storage

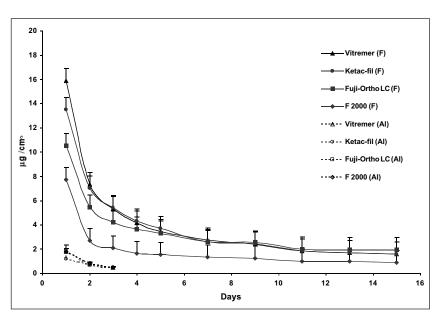


Figure 1. Fluoride (F-) and aluminum (A(I) released (mg/cm) from the materials in deionized water over time. (Avg; SD; n=6).

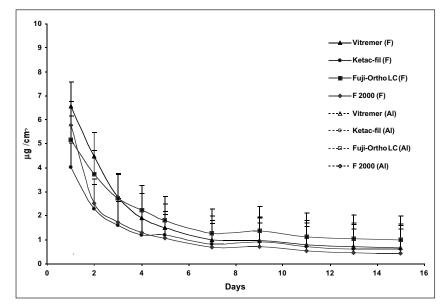


Figure 2. Fluoride (F) and aluminum (Al³+) released (mg/cm²) from the materials in artificial saliva over time. (Avg; SD; n=6).

media. With regard to the media and each material, it may be seen that the materials released different amounts of  $F^-$  in the three media and, for most of them, higher values were found in De-Re than in the other solutions (p<0.05). Considering the materials and each medium, statistically significant differences (p<0.05) among all materials were observed in De-Re solutions

 $(p{<}0.05)$  but not in  $\rm H_2O$  or AS. The resinmodified glass ionomer Vitremer released the highest amount of  $\rm F^-$  in De-Re solutions, but in  $\rm H_2O$  or AS, it did not differ statistically from Fuji-Ortho  $(p{>}0.05)$ . The polyacid-modified resin F2000 released the lowest amount of  $\rm F^-$ , both in De-Re and  $\rm H_2O$ , but in AS, it did not differ statistically from the glass-ionomer cement Ketac-Fil  $(p{>}0.05)$ .

Table 3 shows the cumulative release of  $Al^{3+}$  according to the materials and the storage media. Considering the media and each material, they released different amounts of  $Al^{3+}$  in the three media and for all materials, a higher concentration was found in De-Re than in the other solutions (p<0.05). Considering the materials and each medium, a statistically significant difference among all materials was found in De-Re solutions (p<0.05) but not in  $H_2O$  or AS. The resin-modified glass ionomer Vitremer released the highest amount of  $Al^{3+}$  in De-Re solutions and the polyacid-modified resin

F2000 released the lowest. It was possible to differentiate all the materials with regard to Al<sup>3+</sup> release in De-Re solutions but not in the other media.

A very high correlation was found between  $F^-$  and  $Al^{3+}$  release in De-Re solutions ( $r^2$ =0.96, p<0.001), although no correlation was found in  $H_2O$  and AS.

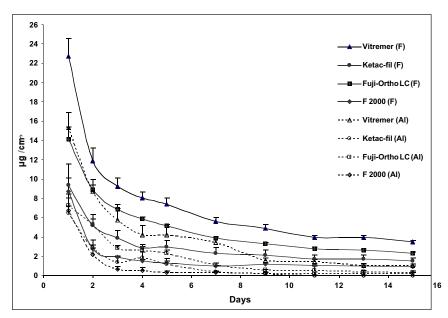


Figure 3. Fluoride ( $F^-$ ) and aluminum ( $Al^{3+}$ ) released ( $mg/cm^2$ ) from the materials in pH-cycling solutions over time. (Avg; SD; n=6).

		Materials		
Media				
	Vitremer	Ketac-Fil	Fuji Ortho	F 2000
De-Re	81.4 ± 4.8	33.8 ± 6.6	56.0 ± 2.0	21.3 ± 2.9
AS	21.3 ± 2.0	14.0 ± 1.3	21.5 ± 2.1	15.2 ± 1.2
H <sub>2</sub> O	46.5 ± 6.8	44.3 ± 16.0	38.3 ± 4.7	21.2 ± 2.4

		Materials		
Media				
	Vitremer	Ketac-Fil	Fuji Ortho	F 2000
De-Re	46.92 ± 4.90	14.96 ± 2.88	23.25 ± 2.86	11.04 ± 2.31
AS	$0.34 \pm 0.02$	0.07 ± 0.07	0.10 ± 0.04	0.23 ± 0.10
H <sub>2</sub> O	3.17 ± 0.17	2.48 ± 0.27	3.05 ± 0.42	3.15 ± 0.25

Means connected by brackets are not statistically different (p<0.05); comparisons among media for each material (vertical direction) and materials for each medium (horizontal direction).

### **DISCUSSION**

The anticariogenic potential of dental materials can be estimated *in vitro* by the release of substances known to have an effect on dental caries. F<sup>-</sup> is the most recognized anticariogenic substance contained in some dental materials (Forss & Seppä, 1990; Benelli & others, 1993; Modesto & others, 1997) and the evaluation of its release in media that do not simulate the caries process has been criticized (Carvalho & Cury, 1999). Al<sup>3+</sup> is another substance released from some dental materials and it presents anticariogenic properties, enhancing F<sup>-</sup> effect (Sturr & Marquis, 1990; Hayacibara & others, 2003). However, no study has been made that evaluated the simultaneous release of F<sup>-</sup> and Al<sup>3+</sup> in media simulating caries development.

Kinetic findings have shown that the patterns of F-release from all materials in all media were similar (Figures 1, 2 and 3) and are in accordance with previous reports (Forsten, 1991; Forss, 1993; de Araujo & others, 1996; Carvalho & Cury, 1999). The F- release mechanism is not completely understood and is out of the scope of this study and the hypothesis being tested. According to Dhondt and others (2001), the process involves not only the loss of loosely bound F- in the cement matrix, but also the release of F-, which becomes strongly bound during the setting reaction and induces long-term release.

With regard to  $Al^{3+}$ , the release in De-Re solutions showed the same pattern observed for F<sup>-</sup>; a high release during the first days followed by a sharp decline and a continued release of small amounts over time (Figures 1, 2 and 3). The release of  $Al^{3+}$  in  $H_2O$  was below the quantification limit of the analysis after the third day; in AS, a detectable amount of  $Al^{3+}$  was found only after the first 24 hours of immersion. Nevertheless, the pattern of  $Al^{3+}$  release over time is in agreement with Fukazawa, Matsuya and Yamane (1987), Nakajima and others (1997) and Savarino and others (2000).

The findings of this study suggest that solutions simulating the caries process (pH-cycling with demineralizing and remineralizing solutions) are more appropriate for evaluating the anticariogenic potential of dental materials with regard to releasing ions. This suggestion is initially supported by the fact that the release of Al³+ from the polyacid–modified resin F 2000 in H<sub>2</sub>O and AS is very low and it was only possible to quantify it on the first days. However, in De-Re solutions, it was possible to quantify the release of Al³+ during the 15 days of evaluation and, furthermore, a high correlation (0.96) was found between the concentrations of F⁻ and Al³+ released.

In addition to these qualitative considerations which suggest that De-Re solutions should be the elected media for evaluating the release of anticariogenic substances, the quantitative data shown in Tables 2 and 3

provide additional support. Table 2 shows that when the media are compared, each material has its own pattern of F<sup>-</sup> release. In general, the highest release is found in De-Re solutions and the lowest in AS. These data confirm the authors' previous study that uses the same media to evaluate F<sup>-</sup> release from different dental materials (Carvalho & Cury, 1999). The highest release found in De-Re solutions can be explained by the fact that the demineralizing solution presents pH 4.3 and a high release of F- in acidic solutions has been shown by other authors (Savarino & others, 2000; Modesto & others, 1997; Forsten, 1991). The lowest release in AS can be explained by an effect of common ions reducing the solubility of salts present in the materials (El Mallakh & Sarkar, 1990; Carvalho & Cury, 1999). The fact that most materials released higher amounts of F- in De-Re solutions than in other media should not be considered as a reason to prefer them, because the minimum F concentration capable of reducing caries is not well known (Forsten, 1998). However, De-Re solutions should be chosen for evaluating F- release from dental materials because they mimic the pH-cycling that occurs during caries development. In addition, the constant presence of F in dental plaque when the pH drops soon after sugar exposure or during further pH increase, is important for reducing enamel-dentin demineralization and enhancing remineralization, respec-

Furthermore, it is relevant to emphasize that in De-Re solutions it was possible to differentiate all the materials statistically in terms of F- release (Vitremer>Fuji Ortho>Ketac-Fil>F 2000). When H<sub>2</sub>O or AS were used, respectively, it was not possible to differentiate the three materials (Vitremer=Ketac-Fil=Fuji Ortho>F 2000) and 2 (Fuji Ortho=Vitremer> Ketac-Fil= F 2000). Another aspect that should be mentioned regarding the data in Table 2 is that the two resin-modified glass ionomers evaluated showed the same relative F- release pattern with regard to the medium. For both, the order of F- release was De-Re> $H_2O$  >AS (p>0.05). However, the order found, either for conventional glass-ionomer cement or polyacidmodified resin, was De-Re=H<sub>2</sub>O>AS. The explanation for these differences is out of the scope of this study but suggests that the F<sup>-</sup> release pattern for resin-modified glass ionomer may be a consequence of a material more homogeneous in composition than the other materials.

Al<sup>3+</sup> is the major constituent of glass ionomer cements. During mixing and setting Al<sup>3+</sup> is released from the glass into the polyalkeonic acid solution. Part of this Al<sup>3+</sup> may not combine with the polyalkeonic acid but may be released from the cement (Andersson & Dahl, 1994). All the materials (Table 3) showed the same order of Al<sup>3+</sup> release in the medium (De-Re>H<sub>2</sub>O>AS). The highest release in De-Re is in accordance with Forss (1993), who observed an increase in Al<sup>3+</sup> release in

lactic acid when compared to distilled water. These findings suggest that although a high correlation between  $F^-$  and  $Al^{3+}$  release was observed (0.96), the mechanisms of  $Al^{3+}$  release may be different from those for  $F^-$  release when different materials are considered. If  $F^-$  release is not yet completely understood (Dhondt & others, 2001), the knowledge about  $Al^{3+}$  release is even scarcer. Considering that  $Al^{3+}$  can enhance the anticariogenic effect of  $F^-$ , the simultaneous release of these two ions should be adequately determined. This is relevant because the authors have shown that the effect of dental materials on the reduction of sugar fermentation by S mutans is more dependent on the combining effect of  $F^-$  and  $Al^{3+}$  released than on  $F^-$  alone (Hayacibara & others, 2003).

The findings showed (Table 3) that in using De-Re solutions, it was statistically possible to differentiate all the materials with regard to Al³+ release (Vitremer>Fuji Ortho>Ketac-Fil>F 2000). In AS, the only differentiation was that Vitremer released a higher amount of Al³+ than Keta-fil, but among them, the differences were not statistically significant. When  $\rm H_2O$  was used as storage media, no difference among the materials was found. The same arguments were used to suggest that using De-Re solutions for evaluating F¹ release from dental materials are valid for Al³+ release.

### **CONCLUSIONS**

The authors' findings suggest that electing media for evaluating the anticariogenic potential of dental materials is valid not only when F<sup>-</sup> release is being assessed, but also for Al<sup>3+</sup> release.

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# Biocompatibility of a Flowable Composite Bonded with a Self-etching Adhesive Compared with a Glass Ionomer Cement and a High Copper Amalgam

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### Clinical Relevance

A flowable composite bonded with a self-etching adhesive showed an acceptable biological compatibility with monkey pulps.

### **SUMMARY**

This study evaluated the pulpal response and *invivo* microleakage of a flowable composite bonded with a self-etching adhesive and compared the results with a glass ionomer cement and amalgam. Cervical cavities were prepared in monkey

teeth. The teeth were randomly divided into three groups. A self-etching primer system (Imperva FluoroBond, Shofu) was applied to the teeth in one of the experimental groups, and the cavities were filled with a flowable composite (SI-BF-2001-LF, Shofu). In the other groups, a glass ionomer cement (Fuji II, GC) or amalgam (Dispersalloy, Johnson & Johnson) filled the cavity. The teeth were then extracted after 3, 30 and 90 days, fixed in 10% buffered formalin solution and prepared according to routine histological techniques. Five micrometer sections were stained with hematoxylin and eosin or Brown and Brenn gram stain for bacterial observation. No serious inflammatory reaction of the pulp, such as necrosis or abscess formation, was observed in any of the experimental groups. Slight inflammatory cell infiltration was the main initial reaction, while deposition of reparative dentin was the major long-term reaction in all groups. No bacterial penetration along the cavity walls was detected in the flowable composite or glass ionomer cement except for one case at 30 days in the glass ionomer cement. The flowable composite bonded with self-etching adhesive showed an acceptable biological com-

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patibility to monkey pulp. The *in vivo* sealing ability of the flowable composite in combination with the self-etching adhesive was considered comparable to glass ionomer cement. Amalgam restorations without adhesive liners showed slight bacterial penetration along the cavity wall.

### INTRODUCTION

Resin composites have been available to the dental profession for nearly four decades. With the development of dentin bonding systems, high bond strengths to tooth structure have been achieved with composite restorations (Nakabayashi, Kojima & Masuhara, 1982; Watanabe, Nakabayashi & Pashley, 1994). However, marginal adaptation still remains an unavoidable problem for composite restorations, especially at the gingival wall of cervical or Class II restorations (Ferdianakis, 1998; Chuang & others, 2001; Kubo & others, 2001; Jayasooriya & others, 2003). In an attempt to improve marginal sealing, many techniques and lining materials have been designed. To reduce stress generated by polymerization shrinkage, applying and curing of resin composites in layers is often recommended. It has also been reported that using a thick adhesive layer or a low-viscosity resin may, due to its elastic properties, serve as a flexible intermediate layer and compensate for the polymerization stress created in resin composites (Hosoda & others, 1991; Jayasooriya & others, 2003).

"Flowable composites" were introduced in 1996 as liners for "packable composites." These materials are a modification of restorative resin composites and, thus, tend to contain a lower filler content (weight: 60%-70%; volume: 46%-65%) than their hybrid analogs (weight: 70%-80%; volume: 60-75%) (Bayne & others, 1998). In previous studies, the use of flowable composites as liners appeared to reduce microleakage and internal voids in Class I and II restorations (Ferdianakis, 1998; Leevailoj & others, 2001; Jayasooriya & others, 2003).

However, little information is available on the biocompatability of flowable resins on pulp tissue. The cytotoxic responses to resin composites and their components still need to be discussed (Wataha & others, 2003). Because of less filler loading of flowable composites, they contains more resin matrix and resin components which might affect pulpal tissue (Wataha & others, 2003). In addition, flowable composites are reported to exhibit a significantly high initial degree of polymerization shrinkage (Tolidis & Setcos, 1999).

Since the development of glass ionomer cement, many investigators have reported the biological effects of glass ionomer cements and low irritant effects on pulp tissue (Tobias & others, 1978; Hosoda & others, 1991;

Shimada, 1992). Dental amalgam has also been used successfully as a restorative material for more than a century. Although its appearance is unaesthetic, dental amalgam is a remarkably durable and long-lasting restorative material (Wahl, 2001; Murray & others, 2002).

This study evaluated the pulp response and microleakage of a flowable composite histologically and compared the results with glass ionomer cement and an amalgam.

### **METHODS AND MATERIALS**

The animals, their handling and the facilities were approved by the Committee on Ethical Guidelines for Animal Care of Tokyo Medical and Dental University. Three monkeys (Macaca fuscata), weighing 10 to 15 kg and ranging in age from seven to eight years, were anesthetized by intramuscular injection of 2 mg/kg ketamine (Ketaral, Sankyo Co, Tokyo Japan) and intravenous injection of 2 mg/kg pentobarbital sodium (Nembutal Sodium Solution, Abbott Laboratories, Abbott Park, IL, USA). Cervical cavities were prepared in the teeth using diamond stones (ISO #SB2, GC, Tokyo, Japan) at high-speed under water spray coolant. Thirty cavities were prepared in one animal; for the mandibular first molar, two cavities were prepared in each tooth. The cavosurface margin of the cavities was always in enamel, and one operator prepared all cavities, approximately 2-mm deep.

A total of 90 cavities were equally divided into three groups of the different restorative materials as follows:

Group 1: A flowable composite was bonded with a self-etching adhesive system. A self-etching primer was applied to the cavity for 20 seconds, followed by application of an adhesive resin that was light-cured for 10 seconds (Imperva FluoroBond, Shofu, Kyoto, Japan, Lot #1001). The cavity was then filled with a flowable composite (SI-BF-2001-LF, Shofu, Lot #07010304) and photo-irradiated for 40 seconds.

Group 2: Glass ionomer cement (Fuji II, GC, Tokyo, Japan, Lot #101211) was hand-mixed for 30 seconds and placed into the cavity using a composite injection syringe. After the initial setting phase, a thin coat of varnish was placed on the surface to prevent dehydration and cracking of the restoration.

Group 3: High-copper amalgam alloy and mercury (Dispersalloy, Johnson & Johnson, East Windsor, NJ, USA, Lot #30GH) were mixed in an amalgamator for 15 seconds and condensed into the cavity.

The experimental periods were 3, 30 and 90 days. Three experimental groups and three experimental periods were randomly assigned to each animal. After the experimental periods, the monkeys were sacrificed by an intravenous injection of 250 mg/kg of thiopental

sodium (Ravonal, Tanabe Pharmaceutical Co, Osaka, Japan), and the teeth were removed from their jaws. Following fixation in 10% neutral buffered formalin solution for seven days, the teeth were demineralized with Plank-Rychlo's decalcifying solution at 4°C for four days, neutralized with 5% sodium sulfate for six hours and washed with running water for six hours. After removing the restorations, the teeth were dehydrated and finally embedded in paraffin.

Histological serial sections 5-µm thick were prepared, obtaining 50 to 60 sections per cavity. They were stained with hematoxylin and eosin staining for routine histological evaluation and Brown and Brenn gram staining to assess the bacteria alternately. For all sections, three histological features, inflammatory cell

infiltration, reparative dentin formation and bacterial staining, were evaluated and the features were then graded under a light microscope as none (0), slight (1), moderate (2) and severe (3) (Shimada, 1992; Kitasako & others, 2000). These criteria are shown as follows:

### **Inflammatory Cell Infiltration**

None (0): characterized by the absence of inflammatory cells;

Slight (1): characterized by the scattering of a small number of inflammatory cells;

Moderate (2): characterized by a distinct increase in inflammatory cells;

Severe (3): characterized by abscess formation in the pulp or pulpal necrosis.

### **Reparative Dentin Formation**

None (0): no additional or abnormal increased thickness in circumpulpal dentin below the cut dentinal tubules of

cavity preparation;

Slight (1): a small, thin rim of reparative dentin below the cut dentinal tubules of the cavity floor (the thickness of the reparative dentin was less than twice the thickness of predentin);

Moderate (2): a moderate bulk of new reparative dentin below the cut dentinal tubules of the cavity floor (the thickness of the

reparative dentin was more than twice the thickness of predentin but less than half the pulpal thickness);

Severe (3): a large bulk of new reparative dentin below the cut dentinal tubules of the cavity floor (the thickness of the reparative dentin was more than half that of the pulpal thickness).

### **Bacterial Staining**

None (0): the absence of stained bacterial profiles in any of the sections;

Slight (1): positive bacterial staining profiles along the coronal walls or axial floor of the cavity;

Moderate (2): positive stained bacterial profiles within the cut dentinal tubules of the axial floor;

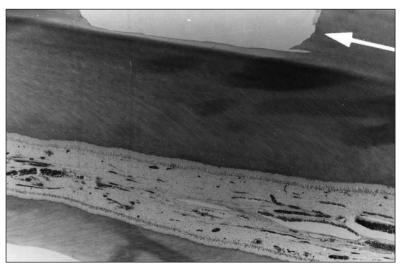


Figure 1. Flowable composite—30 days. Stained with H & E. The remaining dentin thickness is approximately 0.45 mm. No inflammatory cell infiltration is seen. The flowable composite is remaining in the cavity (arrow) (magnification 40x).

	RDT (mm)	Inf	Inflammation			Reparative Dentin				Bacteria Presence					
	Mean (SD)	0 1	2	3	Αv	0	1	2	3	Av	0 1		2	3	Αv
Flowable Composite															
3 days	0.59 (0.30)	10 0	0 (	0	0.0	10	0	0	0	0.0	10	0	0	0	0.0
30 days	0.60 (0.31)	9 (	) 1	0	0.2	8	2	0	0	0.2	10	0	0	0	0.0
90 days	0.53 (0.23)	10 (	0	0	0.0	8	2	0	0	0.2	10	0	0	0	0.0
Glass Ionomer Cement															
3 days	0.46 (0.30)	9 (	) 1	0	0.2	10	0	0	0	0.0	10	О	0	0	0.0
30 days	0.42 (0.18)	10 0	0 (	0	0.0	7	3	0	0	0.3	9	1	0	0	0.1
90 days	0.57 (0.15)	9 1	1 0	0	0.1	6	2	2	0	0.6	10	)	0	0	0.0
High Copper Amalgam															
3 days	0.36 (0.26)	7 (	) 2	1	0.7	10	0	0	0	0.0	5	4	1	0	0.6
30 days	0.52 (0.26)	10 0	0 (	0	0.0	6	2	2	0	0.6	9	1	0	0	0.1
90 days	0.44 (0.32)	8 (	) 2	0	0.2	3	4	2	1	1.1	7	3	0	0	0.3

Severe (3): positive stained bacterial profiles within dental pulp.

Results of the inflammatory cell infiltration, reparative dentin deposition and bacterial staining were statistically analyzed using the non-parametric multiple comparison (Steel Dwass test) with a statistical significance of p<0.05. The remaining dentin thickness between the cavity floor and pulp was also recorded, and the variance of the thickness was analyzed using one-way ANOVA (p<0.05).

### **RESULTS**

Table 1 shows a summary of the findings on the histological sections and the thickness of remaining dentin. No statistically significant differences of remaining dentin thickness were found among the groups (one way ANOVA, *p*=0.44).

### **Inflammatory Cell Infiltration**

No inflammatory cell infiltration was detected in the flowable composite, except one moderate reaction at 30 days (Figure 1). The glass ionomer cement only showed one moderate reaction at three days and one slight reaction at 90 days (Figure 2). In amalgam, one severe reaction and two moderate reactions were detected at three days, and two cases of moderate reaction at 90 days were also seen (Figure 3). However, no significant differences in inflammatory cell infiltration were found among the restorative materials or periods (Steel Dwass test, p<0.05).

### **Reparative Dentin Formation**

At three days in all three groups, there was no indication of any reparative dentin below the cut tubules of the remaining dentin.

Only slight reparative dentin formation was seen in the flowable composite group and two slight formations of 10 specimens at 30 days and 90 days, respectively. The glass ionomer also showed slight reparative dentin formation at 30 days; whereas, two moderate and two slight formations of reparative dentin in 10 specimens at 90 days were observed. In the case of high copper amalgam, two moderate formations and two slight formations of reparative dentin were seen at 30 days, and one severe formation, two moderate formations and four slight formations were observed at 90 days.

Non-parametric multiple comparison analysis of reparative dentin formation showed that a significant difference existed between the flowable composite and the amalgam groups only at 90 days (Steel Dwass test, p < 0.05).

### **Bacterial Penetration**

Bacterial penetration along the cavity walls could not be detected in any of the flowable composite or glass ionomer cement specimens, except for one case at 30 days in the glass ionomer cement. The leakage ratings of the flowable composite and glass ionomer cement showed no significant difference. Amalgam showed five cases of bacterial penetration at three days, one case at 30 days and three cases at 90 days. Bacterial penetration of amalgam at three days showed a significantly higher level compared with flowable composite and glass ionomer cement (Steel Dwass test, p < 0.05).



Figure 2. Glass ionomer cement—90 days. Stained with H & E. the remaining dentin thickness is approximately 0.50 mm. Reparative dentin formation can be seen (score 2) (magnification 40x).

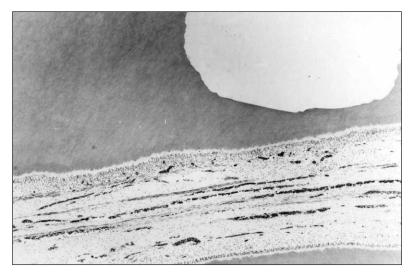


Figure 3. Hi-copper amalgam—3 days. Stained with H & E. Remaining dentin thickness is approximately 0.35 mm. No inflammatory cell infiltration is seen (magnification 40x).

### DISCUSSION

This in vivo study examined the pulpal irritation and bacterial penetration of a flowable composite bonded with a self-etching adhesive system. In the ISO standard (ISO 7405.6.3, 1997) for in vivo biological testing of materials, zinc oxide eugenol (ZOE) cement is suggested as the non-irritating control restorative agent for placement in non-exposed cervical cavities of non-human primates. Hosoda and others (1991) used ZOE cement as a control material and have indicated that the irritational effect of this cement on monkey teeth was moderate and without bacterial invasion. Silicate cement was also used in *in vivo* pulp tests as a positive control material, however, this material caused a severe pulp response and led to necrosis (Cox & others, 1987). However, because of their poor physical properties in terms of solubility, setting shrinkage and low tensile strength, these materials are seldom used clinically for permanent restorations (Cox & others, 1987; Shimada, 1992; Murray & others, 2002). Therefore, it may be necessary to use control materials other than ZOE or silicate cement for pulp tissue research.

In this study, an attempt was made to use glass ionomer cement and an amalgam as control materials. No significant difference in inflammatory cell infiltration or reparative dentin formation was seen among the materials placed in cavities. In the case of bacterial penetration, glass ionomer cement showed good sealing properties; whereas, slight bacterial penetration occurred under the amalgam restorations, which was also found in previous studies (Boston & Graver, 1994; Wahl, 2001). The higher frequency of bacterial penetration in amalgam restorations can be attributed to the inability to provide a complete seal along cavity walls and to lack of adhesion (Cox & others, 1987; Hosoda & others, 1991; Kitasako & others, 2000; Murray & others, 2002). However, the sealing ability of flowable composite bonded with a self-etching adhesive appeared comparable to glass ionomer cement, in that it had sealing properties that prevented bacterial penetration. The pulpal response of restored teeth is associated with the microleakage of bacteria compared with the absence of bacteria (Cox & others, 1987; Hosoda & others, 1991; Kitasako & others, 2000; Murray & others, 2002; Mjör & Ferrari, 2002). The results of this study also showed that a low level of inflammatory activity was detected in the absence of bacteria. Bacterial infiltration through marginal leakage rather than the toxicity of restorative materials, is the greatest threat to pulp (Cox & others, 1987; Hosoda & others, 1991; Kitasako & others, 2000; Murray & others, 2002; Mjör & Ferrari, 2002). In this study, no liner or adhesive was used for amalgam or glass ionomer restorations. Particularly in the case of amalgam, one moderate and three slight bacterial penetration cases were seen at three days, which seemed to be related to the one severe and two moderate inflammation cases at the same time. Even though no statistical significance for inflammatory cell infiltration was detected among groups, it is highly probable that applying an adhesive liner would prevent bacteria microleakage and reduce the inflammatory level (Cox & others, 1987; Murray & others, 2002; Mjör & Ferrari, 2002).

The amount of reparative dentin formation may reflect the results of inflammatory reaction of pulp tissue at three days. Scores of inflammatory cell infiltration of flowable composite and glass ionomer cement were none or slight at three days (flowable composite, score 0.0; glass ionomer cement, score 0.2), and the scores of reparative dentin formation were also found at a low level (flowable composite, score 0.2; glass ionomer cement, score 0.6); whereas, high copper amalgam showed slightly higher reactions at three days (score 0.7) and reparative dentin formation in 90 days (score 1.1). However, it is highly likely that the irritation effect of amalgam is not injurious to pulp tissue, because the tissue produced reparative dentin without any significant inflammatory reactions at 30 or 90 days, with a score of only 0.2 at 90 days.

Enamel bonding of self-etching adhesive systems is still a point of discussion. Several studies have shown that using self-etching adhesive with a resin composite did not provide as good a bond to the enamel surface as a phosphoric acid etching system (Abate & others, 1997; Hara & others, 1999; Finger & Fritz, 1996). However, several studies reported good bonding performance of the self-etching adhesive system by Kuraray Co, Clearfil SE Bond on enamel surfaces (Shimada & others, 1999). While bacteria are perhaps the most useful biological tracers for detecting microleakage in vivo, marginal sealing or enamel bonding of the self-etching adhesive system appeared to be sufficient in this study. Acidic monomers in primer, such as 4-acryloxyethyl trimet (4-AET) and 2-hydroxyethyl methacrylate (HEMA), may promote the penetration of bonding resin into dentin and enamel and/or may increase the wetability of bonding agent on tooth surface, resulting in an effective seal of flowable composite (Nakabayashi & others, 1982). Due to its elastic properties, the use of the flowable composite may also be considered as compensating for the polymerization stress that leads to the reduction of gap-formation and bacterial penetration along the cavity wall (Ferdianakis, 1998).

In a study by Bayne and others (1998), the mechanical properties of several commercial flowable composites were about 60% to 90% of conventional composites. Results of the current research are limited to the biocompatability and *in vivo* microleakage. Further study on the wear resistance and bonding durability of a flowable composite under occlusal stress is needed.

### **CONCLUSIONS**

A flowable composite in combination with a self-etching adhesive showed an acceptable biological compatibility with monkey pulps without bacterial penetration along the cavity walls. The *in vivo* sealing ability of this system was considered equal to glass ionomer cement.

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## Fracture Resistance of Compomer and Composite Restoratives

AUJ Yap • SM Chung • WS Chow KT Tsai • CT Lim

### **Clinical Relevance**

Since componers are less resistant to crack propagation than composites, their use in load bearing areas of posterior teeth should be approached with caution.

### **SUMMARY**

This study evaluated and compared the fracture toughness of compomers and composites. Three compomer (Compoglass F [CG], Vivadent; F2000 [FT], 3M-ESPE; Dyract Posterior [DP], Dentsply) and three composite (Tetric Ceram [TC], Vivadent; Z250 [ZT], 3M-ESPE; Esthet X [EX], Dentsply) restoratives were selected for the study. Single-edged notched specimens (25 x 2 x 2 mm) were fabricated according to manufacturers' instructions and conditioned in distilled water at  $37^{\circ}$ C for one week prior to testing. Seven

specimens were made for each material. The specimens were loaded to failure using an Instron microtester with a crosshead speed of 0.5 mm/minute. Data were subjected ANOVA/Scheffe's test and Independent Samples T-test at significance level 0.05. The mean fracture toughness (K<sub>IC</sub>) ranged from 0.97 to 1.23 MPam<sup>1/2</sup> for componers and 1.75 to 1.92 MPam<sup>1/2</sup> for composites. The fracture toughness of compomers was significantly lower than their composite counterparts. No significant difference in K<sub>IC</sub> values was observed among the different composites. When the compomers were compared, FT had significantly higher fracture toughness than DP and CG. In view of their poorer resistance to crack propagation, compomers are not recommended for use in stressbearing areas.

### INTRODUCTION

Compomers, also referred to as polyacid-modified resin composites, were developed to combine the advantages of glass ionomer cements (chemical bonding to tooth structure, fluoride release and good biocompatibility) with the ease of handling and aesthetic properties of composites. They are a subgroup of composites that contain either or both of the essential components of glass ionomers but at levels insufficient to promote the acid-base cure reaction in the dark (McLean, Nicholson & Wilson, 1994). Like composites, the matrix in com-

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pomers is formed mainly during the light-activated, radical polymerization reaction of monomers. These monomers are essentially modified methacrylates and new bifunctional monomers that contain two carboxylic groups and two double bond functions. The bifunctional monomer reacts simultaneously with methacrylate by radical polymerization and via an acid-base reaction with cations liberated from the glass particles by the action of water (Meyer, Cattani-Lorente & Dupuis, 1998).

Compomers are indicated for restoring Class III and V cavities in anterior teeth. Some products have, however, been re-formulated for use in stress-bearing Class I, II and IV cavities. While many clinical studies have been conducted on Class III and V compomer restorations (Folwaczny & others, 2001; Di Lenarda, Cadenaro & De Stefano Dorigo, 2000; van Dijken, 1999), few have reported their use in posterior stress-bearing areas of permanent teeth (Luo & others, 2000). Although results from the latter study indicate that compomers are promising materials for restoring Class I and II cavities, caution should still be exercised in view of the short-term nature of the study and data reflecting the lower strength of some compomers as compared to composites (Yap & others, 2000; Meyer & others, 1998).

Further *in-vitro* mechanical characterization of componers is warranted to substantiate their routine use in restoring posterior teeth.

Margin and bulk fractures are common reasons for replacing composite restorations (Burke & others, 1999; York & Arthur, 1993). Both defects are suggested to be related to inadequate fracture resistance or fracture toughness of the materials (Ferracane, Antonio & Matsumoto, 1987). Fracture toughness (K<sub>IC</sub>) has also been used to predict in vivo wear of resin composites (Truong & Tyas, 1988). It is a measure of the stress intensity at the tip of a crack or flaw from which a crack propagates through a material in an unstable manner (Fujishima & Ferracane, 1996). The subscript I refers to the case when the crack is propagated in mode I or tensile opening. Although many researchers have examined the fracture toughness of composites resins (Knobloch & others, 2002; Manhart & others, 2000; Fujishima & Ferracane, 1996; Johnson, Dhuru & Brantley, 1993; Truong & Tyas, 1988; Ferracane & others, 1987), little is known about the fracture toughness of componers. This study determined and compared the fracture toughness of three compomer restoratives and their composite counterparts.

Table 1: Technical Profiles and Manufacturers of the Materials Evaluated Material Filler Type Filler Content Vol % Filler Size (µm) Manufacturer **Resin Matrix** (Lot #) **Cure Time** Compoglass F Ba-Al-55 UDMA, Vivadent, 1.0 (mean) (D51370) fluorosilicate. PEGDMA. Schaan. Ytterbium **DCDMA** Liechtenstein 40 seconds trifluoride F2000 CDMA. Al-fluorosilicate. 67 3-10 3M-ESPE. (20010122)Silica **GDMA** St Paul, MN, USA 40 seconds **Dyract Posterior** Strontium-UDMA. Dentsply, 47 0.8 (mean) (0107001376)fluorosilicate **TCB** Konstanz, Germany 40 seconds Tetric Ceram Barium glass, 60 0.7 (mean) Bis-GMA. Vivadent. (D54267) Ba-Al-fluorosilicate. UDMA. Schaan, Liechtenstein Mixed oxide, **TEGDMA** 40 seconds Silica. Ytterbium trifluoride Z250 Zirconia silica 60 0.6 (mean) BisGMA, 3M-ESPE, St Paul, MN, USA (20010402)UDMA, 20 seconds BisEMA Esthet X Ba-Al-fluorosilicate, 60 0.7 (mean) Bis-GMA, Dentsply, (0108102)Silicon dioxide Bis-EMA, Konstanz, Germany **TEGDMA** 20 seconds

SBisEMA = Ethoxylated bisphenol-A-glycidyl methacrylate

BisGMA = Bisphenol-A-dimethacrylate

CDMA = Dimethacrylate functional oligomer derived from citric acid

DCDMA = Cycloaliphatic dicarbonic acid dimethacrylate

GDMA = Glyceryl methacrylate

PEGDMA = Polyethylene glycoldimethacrylate

TEDGMA = Triethylene glycol dimethacrylate

TCB = Reaction product butane tetracarboxylic acid and HEMA

UMDA = Urethane dimethacrylate

2.5

2

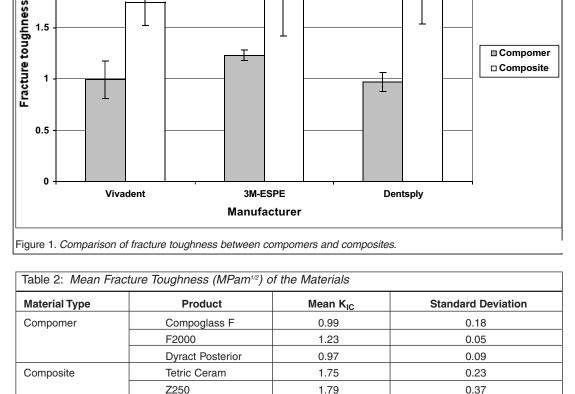
1.5

□ Compomer

□ Composite

### **METHODS AND MATERIALS**

Three compomer (Compoglass F [CG]; F2000 [FT]; Dyract Posterior [DP]) and three composite (Tetric Ceram [TC]: Z250 [ZT]; Esthet X [EX]restoratives from the same manufacturers were selected for the study. Table 1 shows technical profiles of the restoratives and their manufacturers. Fracture toughness (K<sub>IC</sub>) was determined using seven single-edge notched (SEN)-bend specimens of each material. The specimens (25mm long x 2-mm thick x 2-mm wide) were fabricated using customized Teflon molds. The restorative materials were placed in the molds that were positioned on top of a glass slide using a plastic instrument. A second glass slide was then placed on top of



1.92

Esthet X

each mold and gentle pressure was applied to extrude excess material. The top and bottom surfaces of the specimens were then light polymerized in three overlapping irradiations with the Max polymerization unit (Dentsply, Milford, DE, USA) according to manufacturers' cure times. The exit window of the curing tip was 13 mm in diameter and mean intensity of the light source was 402 ± 4 mW/cm<sup>2</sup>. The latter was determined with a commercial light radiometer (CureRite, EFOS) Inc, Ontario, Canada).

Immediately after removing the specimens from their molds, they were visually inspected for defects, sized with sandpaper and measured (width and height) with a digital vernier caliper (Mitutoyo Corp, Kanagawa, Japan) to an accuracy of 0.01 mm. A notch (0.3-mm wide and approximately 0.5-mm deep) was machined into each specimen with a diamond saw (15C; Buehler, Lake Bluff, IL, USA) using water coolant. A fresh surgical blade was then placed into the notch in the specimen to create a distinct flaw from which the crack propagated. Notch length was observed with a stereomicro-

Table 3: Results of Statistical Analysis						
Variables Differences						
All materials	EX, ZT, TC > FT, CG, DP					
Compomers	FT > CG, DP					
Composites	NS					

0.38

NS denotes no statistically significant difference, while > denotes significantly greater fracture toughness (Results of one-way ANOVA/Scheffe's test [p<0.05]).

scope (Olympus, Tokyo, Japan) at 40x magnification and measured against a calibrated scale bar. This was done for individual specimens as notch or crack length was a critical variable in the calculation of  $K_{\rm IC}$ . The specimens were then conditioned in distilled water at 37°C for one week and tested in three-point bending (span = 20 mm) with an Instron Microtester (5848; Instron Corp, Canton, OH, USA) at a crosshead speed of 0.5 mm/minute. Testing was conducted at 37°C in distilled water and the peak load required to fracture the specimens was established. The fracture toughness, K<sub>IC</sub> (MPam<sup>1/2</sup>), was calculated from the ASTM

(American Society for Testing and Materials) Designation E 399-90 (1997) equation:

$$K_{IC} = [(PS)/(BW^{1.5})] \times f(a/W)$$

where P=load at fracture, S=span or distance between supports, B=specimen thickness, W=specimen width, a=crack length and f=a function of (a/W) whose value is obtained from the ASTM standard.

Fracture toughness data were subjected to statistical analysis using one-way ANOVA and Scheffe's post-hoc test at significance level 0.05. Compomer-composite comparisons were made for products from the same manufacturer using Independent Samples *T*-test.

### **RESULTS**

Table 2 and Figure 1 show the mean fracture toughness of the different materials. Results of statistical analysis are shown in Table 3. Mean fracture toughness ( $K_{\rm IC}$ ) ranged from 0.97 to 1.23 MPam<sup>1/2</sup> for componers and 1.75 to 1.92 MPam<sup>1/2</sup> for composites.  $K_{\rm IC}$  values observed for TC, ZT and EX were significantly greater than for CG, FT and DP. Independent Samples *T*-test showed that fracture toughness of the composite restoratives was significantly higher that their compomer counterparts (Figure 1). No significant differences in  $K_{\rm IC}$  values were observed among the different composites. When the compomers were compared, FT had significantly higher fracture toughness that DP and CG. No significant difference in  $K_{\rm IC}$  values was observed between the latter two compomers.

### DISCUSSION

Dental resin composites, like ceramics, are brittle (they fracture after virtually no plastic deformation) and fail by catastrophic crack propagation (Xu & others, 1999). Previous studies have reported widely different values of fracture toughness for the same dental composites (Kovarik & Fairhurst, 1993). As fracture toughness is a characteristic property of a material, its values should be independent of the mode of measurement. Fujishima and Ferracane (1996), however, found that fracture toughness of composites was method dependent. Four common methods for fracture toughness testing (SEN, compact tension, short rod with chevron notch and double torsion) were compared in their study. They found that values obtained from the short rod test were significantly higher that those obtained from the other three tests and results obtained from the double torsion test were lower than values obtained from the SEN and compact tension methods. The authors concluded that the double torsion method provides the most information about crack initiation and propagation and may be the most indicative of true fracture toughness of dental composites. This method was, however, not chosen for this study as it is very technique sensitive, difficult to conduct and has a very low (50%) success rate (Fujishima & Ferracane, 1996). The SEN method was selected due to its relative simplicity and acceptance by both ASTM and British Standards Institution. In addition, it remains the more widely used method for determining fracture toughness of dental composites. The specimens were not thermal cycled, as cyclic temperature changes do not alter the fracture toughness of composites (Mair & Vowles, 1989).

Mode I fracture toughness of dental composites ranges between 0.7 and 2.0, depending on filler type, fillermatrix ratio and testing method (Scherrer & others, 2000). Composite K<sub>IC</sub> values observed in this study were slightly higher than those of previous SEN tests (Fujishima & Ferracane, 1996; Johnson & others, 1993; Ferracane & others, 1987). The aforementioned can be attributed in part to differences in specimen preparation. In earlier studies, the notch was created by placing materials around a razor blade contained within the mold. This results in flaws that are resin rich and more susceptible to crack propagation due to a lack of fillers. By machining the notch into specimens, a more accurate representation of material behavior is obtained. The composite  $K_{IC}$  values observed were comparable to those obtained by Manhart and others (2000), who used a similar specimen preparation technique.

The K<sub>IC</sub> values of composites generally increase as filler volume fraction is increased (Fujishima & Ferracane, 1996; Johnson & others, 1993). The toughening mechanisms are assumed to be crack pining, crack deflection and matrix-filler interactions. Since filler volume of the composites were identical, no significant difference in fracture toughness was expected between TC, ZT and EX. The significantly higher fracture toughness of the composites and FT, as compared to CG and DP, can be attributed to differences in filler volumes. Although the filler volume of FT was 7% higher than the composites (Table 1), its fracture toughness was significantly lower. Draughn (1981) reported superior mechanical properties for a lower, rather than a higher filler volume fraction. This negative effect was assumed to relate to inherent flaws, especially air porosities included in the composite material. Although the latter may contribute to the significantly lower fracture toughness of FT, a more feasible explanation is the incorporation of glass ionomer components into compomers. An acid-base reaction takes place between the fluorosilicate glass and ionized carboxylic groups once water penetrates the polymer matrix (Meyer & others, 1998). This reaction is a surface phenomenon, and development of a carboxylate-rich surface layer (Eliades, Kakaboura & Palaghias, 1998) at the crack front may explain the lower fracture resistance of FT despite its higher filler loading. The lack of chemical bonding between the large fluorosilicate glass particles and resin matrix may also contribute to the significantly lower fracture toughness of FT compared with the composites. The fracture toughness of the componers tested corresponded to their previous *in-vitro* wear ranking. Latta and others (2001) reported that FT was significantly more wear resistant than CG and Dyract AP using the Leinfelder machine. According to the manufacturer, the formulation of DP is similar to Dyract AP.

Despite good preliminary clinical results (Luo & others, 2000), the routine use of componers for the restoration of posterior teeth should be approached with caution in view of their poor fracture resistance. In addition, componers are not a homogenous class of material and significant differences in mechanical properties exist between products (Abu-Bakr & others, 2000). Water sorption, which is necessary for activation of the carboxylic groups, may also result in decreased mechanical properties over time due to the plasticizing effect of water. A recent paper has, however, reported a significant increase in flexural strength and modulus of FT after aging in water (Yap & others, 2002). In view of their ability to release fluoride and inhibit caries (Yap, Khor & Foo, 1999; Forsten, 1998; Donly & Grandgenett, 1998), the re-formulation of current componers for use in posterior teeth should be further explored.

### **CONCLUSIONS**

Within the limitations of this *in-vitro* study:

- 1. Componers were significantly less resistant to crack propagation than composites.
- 2. The fracture toughness of F2000 was significantly greater than Dyract Posterior and Compoglass F.
- 3.No significant difference in fracture toughness was observed among Tetric Ceram, Z250 and Esthet X.
- 4. The use of componers in stress-bearing areas is not recommended.

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# Microleakage and Marginal Hybrid Layer Formation of Compomer Restorations

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#### Clinical Relevance

The different componer materials tested in this study revealed poor marginal adaptation, leading to possible microleakage over time due to an insufficient quality of marginal hybrid layer.

#### **SUMMARY**

The tested hypotheses of this study were that dentin enamel bonding agents (DBAs) proposed for compomers create a hybrid layer (HL) that seals the margins of Class V restorations and HL is free from voids or gaps on both enamel and dentin margins. For purposes of this study, Class V restorations (n=70) were made *in vitro* at the CEJ in extracted third molars. Different systems (bonding agent + compomer) were selected. After finishing with discs, each margin was polished

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with diamond polishing paste for one minute, treated with a 2.5% NaOCl gel for 10 seconds and washed with deionized water to remove polishing debris and non-infiltrated collagen. All restorations were immersed in dye solution for 24 hours, then inspected along the margins. SEM analysis was used to evaluate the morphology of the marginal HL and microleakage tests to evaluate their ability to seal the margins of restorations. Marginal leakage was observed along the dentin and enamel margin. A thin marginal HL (0.5-1.2 µm) was detected only along the dentin margin of several bonding systems but not along the enamel margin. Porosities and gaps were detected along margins when no HL was observed. The results demonstrated that the tested bonding agents for compomers produced a thin marginal hybrid layer, especially along the dentin margin. Microleakage had a relationship with the morphology (gap, porosities and thickness) of this hybrid smear layer. In conclusion, the DBAs tested specifically developed for compomers did not ensure an intimate interfacial adaptation, because microleakage was detected along the enamel and dentin interfaces and the marginal hybrid layer was only partially homogeneous.

#### INTRODUCTION

Many manufacturers have developed resin-modified composites—or compomers. These materials release fluoride for potential caries prevention and have mechanical and esthetic properties that are less than resin-based composites but better than glass-ionomer cements (Blunck & Roulet, 1999). New self-etching dentin-enamel bonding agents (DBAs) have been specifically developed for compomers; they may reduce clinical problems related to the use of etching procedures, such as the collapse of the collagen-fiber network and contribute to sealing dentin and enamel margins (Gwinnett, Tay & Wei, 1996; Pashley, 1996; Perdigão & others, 1996; 2000).

Recent studies have demonstrated that compomer restorations present several problems, such as marginal gaps along dentin and microleakage, when used without etching procedures, because of incomplete formation of resin tags (Chersoni & others, 1997; Ferrari & others, 1998; García-Godoy & Hosoya, 1998). However, other studies have demonstrated an improved marginal adaptation in enamel (Blunck & Roulet, 1999).

Some studies demonstrated that the marginal hybrid layer (HL), the area of HL close to the external margin of the restorations, may be affected by porosities and may be different from the HL obtained in the deeper area (internal HL) of the restoration (Prati & others, 1998; 2000). The quality of marginal-external HL may influence the microleakage (*in vivo* and *in vitro*) and durability of the restoration (*in vivo*).

Recent studies revealed that the hybrid layer may be similar to a permeable membrane (Tay & others, 2002a, 2002b) and may play a critical role in creating marginal microleakage.

De Munck and others (2003) demonstrated bond reduction in dentin-adhesive interface directly exposed to water in dentin-adhesive interface directly exposed to water.

This study evaluated the morphology of the margins of restorations made with different types of compomers and different self-etching DBAs.

### METHODS AND MATERIALS

#### **Cavity Preparation**

Human teeth were obtained from young patients (mean age 30

years old) and stored at 4C° in saline solution for up to one month. A non-retentive V-shaped cavity (3.5 mm diameter and 3.0 mm depth) was prepared just in correspondence to the cemento-enamel junction (CEJ) in an attempt to produce a conventional Class V restoration (N°=90). In this way, about one-third of the cavity was in enamel. Cavities were prepared only in the buccal surface of each tooth. A medium-grit diamond bur (Intensiv #2552, Intensiv, Lugano, Switzerland) and a fine diamond bur (Intensiv #2552) were used on a high-speed water-cooled handpiece (W&H, Austria). Three operators prepared all the cavities. Table 1 lists the dentin-enamel bonding agents (DBAs) and componers used for the restorations. All materials were used according to the manufacturers' instructions at room temperature (20°C-21°C) and at a relatively constant (50-62%) relative humidity. DBAs were applied using small brushes (no etching procedure was made for enamel and dentin) and lightcured with a previously tested unit (Visilux Command 2, 3M/ESPE Dental Products Division, St Paul, MN, USA) working at 400 mW/cm<sup>2</sup> for 20 seconds.

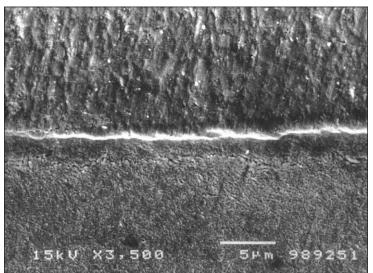


Figure 1. Scanning Electron Microscopic evaluation of the marginal hybrid layer formed along a dentin margin with a thickness of about 3 µm (SEM, 3500x).

DBA/Compomer	Company
Clicker /F2000	3M/ESPE Dental Products, St Paul, MN, USA
EBS/Hytac	3M/ESPE, Seefeld, Germany
Prime & Bond 2.1/Dyract AP	Dentsply, Konstanz, Germany
Prompt L-Pop/F 2000	3M/ESPE, Seefeld, Germany
Prompt L2-Pop/F2000	3M/ESPE, Seefeld, Germany
Syntac Single Component/Compoglass	Ivoclar, Schaan, Liechtenstein
Syntac Single Component F/Compoglass	Ivoclar, Schaan, Liechtenstein
Syntac Sprint/Compoglass F	Ivoclar, Schaan, Liechtenstein
Syntac Sprint/Compoglass Flow	Ivoclar, Schaan, Liechtenstein

Componers were applied using a stainless steel instrument in a single increment and light cured for 60 seconds. Two operators placed all the restorations.

#### **Polishing Procedures**

Each restoration was immediately polished along the margins with wet silicon carbide papers (#600, #800, #1000 and #1200) and with Enhance (Dentsply DeTrey GmbH, Konstanz, Germany). The specimens were then stored in water at room temperature for 20 minutes and gently washed with deionized water. All samples were cleaned along the margins with Ace 1.5% NaOCl gel (Procter & Gamble, Cincinnati, OH, USA) for 10 seconds, washed under deionized water and stored in deionized water for 24 hours at room temperature. This procedure selectively dissolves any collagen fibers that are not enveloped by resin and removes any polishing debris (Prati & others, 1998).

#### Microleakage Procedures

The teeth were washed in tap water for one minute, gently dried, covered with a layer of finger nail varnish to cover only the root surface and the apex and immersed in dye solution (Erythrosin B, John O Butler Co, Chicago, IL, USA) for 24 hours at room

temperature. Each tooth was then carefully washed in tap water and inspected under a stereomicroscope (Zeiss Stemi 2000-C, Carl Zeiss Jena GmbH, Zeiss Group, Jena, Germany, at 12x and 24x magnification) to examine the presence and distribution of dye solution deposited along the dentin and enamel margins. The visualization of dye along the margins was considered an index of the presence of marginal gaps and/or marginal fractures. One experienced operator assessed the microleakage. For each restoration, the percentage of margin that showed dye penetration with respect to the total length of margin was calculated. Dentin and enamel were evaluated separately.

# Scanning Electron Microscopic (SEM) Examination of the Tooth-restoration Interface

Additional restorations (four for each group; n=36) were prepared as previously described and used for SEM analysis. After polishing, each tooth was immersed in 2% glutaraldehyde (pH 7.4) for 24 hours, then slowly dehydrated at constant room temperature (21°C-22°C) through ascending alcohols to 100%. Finally, each sample

was gold coated and inspected under SEM (JEOL 5400, JEOL, Tokyo, Japan).

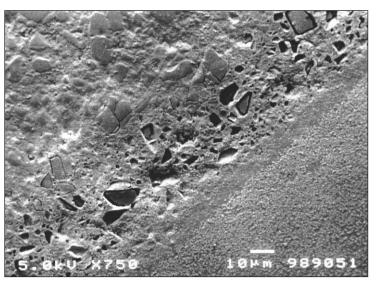


Figure 2. Dentin margin of a Class V restoration. No gaps are visible. The layer of bonding agent is evident. Several porosities and fractures around the fillers are visible near the margin (SEM, 750x).

Table 2: Mean and SD of Enamel Marginal Microleakage (EML) Measured Along the Enamel-Compomer Interface after NaOCI Post-polishing Treatment

Materials	EML
Prompt L2-Pop/F2000	6.1±2.0 μm <sup>a</sup>
Prompt L-Pop/F2000	10.4±4.1 μm <sup>a</sup>
Prime & Bond 2.1/Dyract AP	13.2±21.6 μm²
Syntac Single Component/Compoglass	22.5±14.6 μm²
Syntac Single Component/Compoglass F	22.2±11.4 μm <sup>a</sup>
Syntac Sprint/Compoglass Flow	72.6±30.7 μm <sup>ь</sup>
Clicker/F2000	74.2±28.8 μm <sup>ь</sup>
Syntac Sprint/Compoglass F	78.0±25.1 μm <sup>ь</sup>
EBS/Hytac	86.6±16.1 μm <sup>b</sup>
Different superscript letters are related to statistically different value	es (p>0.05).

Table 3: Mean and SD of Dentin Marginal Microleakage (DML) Measured Along the Dentin-Compomer Interface after NaOCI Post-polishing Treatment

Materials	EML
Prompt L2-Pop/F2000	12.4±9.1 μm²
Clicker/F2000	19.0±16.8 μm²
Prime & Bond 2.1/Dyract AP	26.3±28.9 μm <sup>a</sup>
Prompt L-Pop/F2000	64.5±20.5 μm <sup>ь</sup>
EBS/Hytac	68.3±24.7 μm <sup>b</sup>
Syntac Sprint/Compoglass Flow	67.3±31.7 μm <sup>δ</sup>
Syntac Sprint/Compoglass F	74.6±25.6 μm <sup>ь</sup>
Syntac Single Component/Compoglass	75.7±37.6 μm <sup>ь</sup>
Syntac Single Component/Compoglass F	95.5±10.3 μm <sup>ь</sup>
Different superscript letters are related to statistically different value	nes (p>0.05).

The cervical margin of the restorations was inspected under SEM from one side of the CEJ to the other. The thickness of the marginal hybrid layer was evaluated every  $100\text{-}150~\mu m$ . All the values were averaged to calculate the mean marginal hybrid layer thickness for the inspected sample. In several restorations, the marginal hybrid layer was completely absent and marginal gaps and/or many porosities were observed along the margin. These alterations were visible along the dentin margin and frequently close to the CEJ. In this case, no measurements were done, but the presence of gaps and the length of margin free from HL were calculated.

#### **Statistical Analysis**

The means and standard deviation of marginal microleakage were calculated by averaging the individual values of each sample. Enamel microleakage was evaluated separately from dentin microleakage. A one-way ANOVA was performed to determine whether there were any statistically significant differences in hybrid layer thickness. Post-hoc multiple comparisons were made with Tukey's test using SigmaStat (SPSS, Chicago, IL, USA). The means and standard deviations of the marginal hybrid layer thickness were calculated by averaging the individual values measured in four standardized areas of the margin for each sample. These areas were located along the dentin-componer interface and a one-way ANOVA was performed to determine whether there were any statistically significant differences in hybrid layer thickness. Post-hoc multiple comparisons were made with Tukey's test.

#### **RESULTS**

#### **Microleakage Evaluation**

Tables 2 and 3 report the mean microleakage for enamel and dentin margins of the materials tested. For enamel-compomer evaluation, Prompt L2-Pop, Prime & Bond 2.1 and Syntac Single Component showed statistically lower microleakage (p<0.05) compared to all the other materials, indicating a better sealing capacity. For dentin, Prompt L2-Pop, Clicker/F 2000 and Prime & Bond 2.1 showed statistically (p 0.05) lower microleakage, while the others showed microleakage values ranging from 67.3% to 95.5% of the total length of the dentin-componer interface. If stain was detected along the entire margin, the microleakage score would be 100%.

#### **SEM Evaluation of the Marginal Hybrid Layer**

Table 4 shows the thickness of dentin marginal HL for each material calculated. The morphology of dentin marginal HL was largely dependent on the DBA used. The amount of dentin marginal HL ranged none present to a thickness of 3.3 µm (Figure 1). Many porosites were observed within the marginal hybrid layer (Figure 2). Gaps were frequently observed close to the CEJ area, suggesting that this area represented a critical zone for obtaining a standardized morphology (Figure 3). Gap thickness was 2-5 µm (Figure 3).

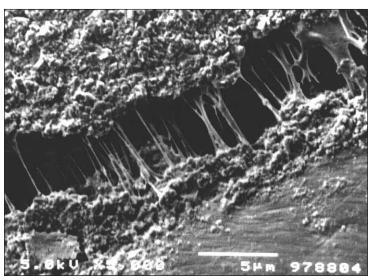


Figure 3. High magnification of a deep gap observed along a dentin margin. The presence of resin debris along the dentin wall suggested that the gap was formed between the hybrid layer and the compomer restoration (SEM, 5000x).

Table 4: Thickness of Marginal Hybrid Layer (Marginal HL) (mean ± SD)
Calculated Along the Dentin Margin After the Removal of Polishing
Debris with a Gently NaOCI Treatment and Percentage of Margin Free
from the Presence of Marginal HL Calculated with Respect to the Total
Length of Dentin Margin (from one side to the other side). The Margins
That Were Free of Marginal Hybrid Layer Showed Frequently Gaps
and/or Voids

Materials DBA/Compomer	Thickness of Dentin Marginal HL	Percentage of Margin Free from HL (± SD)*
Clicker/F 2000	1.0 ± 0.8 µm <sup>a</sup>	10.2 ± 5.7 μm°
Prompt L2-Pop/F2000	0.5 ± 0.3 μm <sup>b</sup>	30.5 ± 10.0 μm <sup>d</sup>
EBS/Hytac	0.3 ± 0.7 μm <sup>b</sup>	67.3 ± 12.7 μm°
Syntac Sprint/Compoglass Flow	0.3 ± 0.7 μm <sup>b</sup>	70.5 ± 12.0 μm°
Syntac Sprint/Compoglass F	0.6 ± 0.6 μm <sup>b</sup>	70.0 ± 10.5 μm°
Syntac Single Component/ Compoglass	0.3 ± 0.6 μm <sup>b</sup>	80.4 ± 5.9 μm <sup>e</sup>
Syntac Single Component/ Compoglass F	0.5 ± 0.3 μm <sup>ь</sup>	82.0 ± 9.5 μm°
Prompt L-Pop/F2000	0.0 ± 0.0 μm <sup>ь</sup>	97.1 ± 4.9 μm <sup>e</sup>

Different superscript letters are related to statistically different values (p>0.05).

\*In this area, it was not possible to evaluate (by SEM) the presence of hybrid layer.

The length of dentin-componer margins with marginal gaps was 5%-100% of the total length of the margin. The absence of dentin marginal HL was frequently associated with gaps or a combination of gaps and fractures along the margins. As Table 4 shows, Prompt L2-Pop, Clicker/F2000 and Prime & Bond 2.1 exhibited a low percentage of gaps, as confirmed by the reduced length of margins free of hybrid layer (Figures 4-5). Fractures and cracks were rarely seen along the dentin margin but were frequently noticed along the enamel margins (not calculated in the study). No marginal HL was detected along the enamel margin. Enamel prisms were partially identified. Filler particles were frequently observed (Figure 6). Marginal HL was frequently noticed as a groove or depression along the interface, suggesting that the NaOCl treatment removed a significant amount of non-resin infiltrated dentin matrix. As visible in the illustrations, most of the cervical samples were free of root cementum which was probably fully removed during polishing procedures.

#### **DISCUSSION**

This study demonstrated that dentin marginal HL produced by specific DBA-compomer restorations without etching/rinsing procedures is extremely thin (0-1 µm) and may present many porosities. Some DBA-componer systems exhibited large gaps and numerous marginal porosities that may be responsible for marginal microleakage. It is possible that the presence of smear layer (which remained in place because of a lack of acid-etching procedures) reduced the penetration of DBA resin into sound dentin, decreasing micro-mechanical retention and allowing for a high rate of compomer shrinkage. These conditions may increase the dimension of voids in the thickness of HL and the width of marginal gaps (Ferrari & others, 1998; Prati & others, 2000; Perdigão & others, 2000). The use of etching procedures with DBAs in future experiments may also increase retention and sealing along dentin margins, ensuring a superior clinical performance of these materials.

The laboratory post-polishing treatment with NaOCl before SEM analysis removed dentinal collagen that was not infiltrated by resin. Yoshiyama and others (1995, 1996) and Prati and others (1998) previously proposed a similar treatment. This laboratory procedure creates a "clean" dentin surface free from debris and smear layer produced during the finishing steps and may help to calculate the "true" thickness of marginal HL and the presence of non-infiltrated dentin areas. In previous studies, Prati and others (1998, 2000) calculated the thickness of the HL (called resin infiltrated dentin layer = RIDL) of many DBAs that require etching procedures. They observed that several

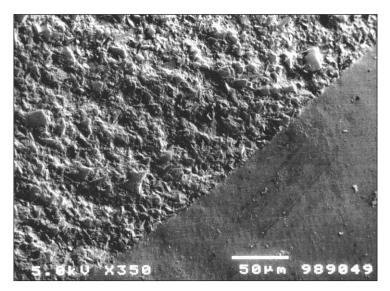


Figure 4. Dentin margin of a Class V restoration (SEM, 350x). No marginal gap or porosities are seen along the dentin margin.

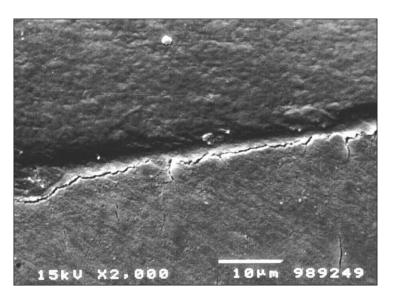


Figure 5. Marginal hybrid layer observed along the margin of a restoration. This is the typical morphology detectable along the margin. Fractures are probably artifacts of the high vacuum of the SEM. A large filler particle is positioned close the dentin margin. The thickness of marginal hybrid layer is 1-2 µm (SEM, 2000x).

DBAs produced HL thicknesses ranging from 5-6 µm in deep dentin (dentin about one-half millimeter from the pulp chamber) to 2-3 µm in superficial dentin close to the dentin-enamel junction. They concluded that HL thickness (calculated in longitudinal sections of dentin discs) had a strong relationship with the dentin location and, obviously, with the type of dentin treatment and bonding system used. Dentin close to peripheral cementum (the area of dentin involved in the preparation of the Class V cavity) has a relatively low number of dentinal tubules, and they are oriented parallel to the cavity wall (Pashley, 1996). The reduced thickness

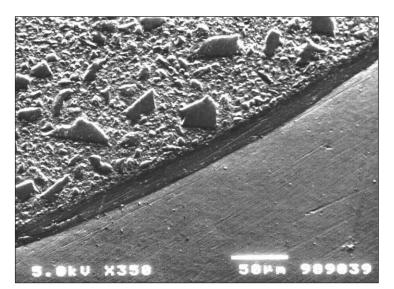


Figure 6. Scanning Electron Microscopic (SEM) image of the enamel margin of a Class V restoration (350x). Particles of compomer are well noticeable. A thick layer of bonding agent is visible at the enamel-restoration interface. This was rarely observed.

of the marginal HL observed in this study may be due to the reduced permeability of cervical dentin which blocks the infiltration of bonding resins toward deeper areas. The lower diameter of dentinal tubules in cervical root dentin and the reduced number of lateral branches of this area (Pashley, 1996) may contribute to the reduced penetration of resins into dentin, creating a thin HL along the margin of the restoration. The presence of voids may be due to what has been previously described as blister-like areas and the formation of resin globules (Tay & others, 1994; Tay, Gwinnett & Wei, 1996; Titley & others 1994). It is possible that several air bubbles and/or water and alcohol bubbles of bonding systems remained entrapped inside the smear layer along the cervical angle at the dentin-restoration interface (Tay & others, 1994, 1996; Titley & others 1994). The large dimensions of componer fillers may also alter the uniformity of interface along the hybrid layer, as demonstrated by our SEM images. After the polishing procedures, all the porosities may be filled with debris (dentin smear layer and composite particles) that covers the surface and masks gaps and voids (Prati & others, 1998). NaOCl laboratory treatment may remove this debris and open all the porosities created by application of the bonding system and compomer shrinkage. In practice, this marginal dentin area is the "open" door for micro- and nanoleakage (Van Meerbeek & others 1992; Titley & others, 1994; Sano & others, 1995; Tay & others, 1996; Stiesch-Scholz & Hannig, 2000) towards the pulp and must be "closed" with a well-sealed marginal hybrid layer. Few materials closed the porosities and reduced gaps as reported in previous investigations (Prati & others, 2000). In this study, several DBA-componers showed very high microleakage that may be corrected by adequate etching procedures. These DBA-compomers were bonded directly to the enamel smear layer that had previously been shown to produce low bond strengths (Titley & others, 1994). The significant amount of microleakage observed in many restorations in this study, both at the enamel and dentin margins, suggests that these materials have several limitations in their clinical use. DBAs that do not require a separate etching/rinsing procedure have overly porous enamelrestorative interfaces that cannot prevent dye penetration. Similar conditions were observed for dentinrestorative interfaces. The possibility of using compomers with DBAs and/or etching procedures to limit interference of the smear layer (still present on the dentin surface when these DBAs are applied) and to allow for formation of a thicker, higher quality hybrid layer along the dentin-restoration interface must be evaluated.

#### CONCLUSIONS

The results of this study demonstrated that the tested DBAs were able to produce only a thin marginal hybrid layer in compomer restorations. Gaps and porosities were frequently observed. In conclusion, the DBAs specifically developed for compomer restorations were not able to completely ensure an intimate interfacial adaptation, because microleakage was detected along the entire enamel and dentin interfaces.

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# Thermal and Mechanical Load Cycling on Microleakage and Shear Bond Strength to Dentin

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#### Clinical Relevance

Simulation of thermal and mechanical stresses presented in the oral environment does not interfere with microleakage and bond strength values.

#### **SUMMARY**

This study evaluated the influence of mechanical and thermal cycling on microleakage at the cervical margins of proximal slot restorations and shear bond strength on flat dentin surfaces.

Microleakage Evaluation: One hundred and twenty slot cavity restorations were performed on bovine incisors. The restorations were randomly divided into four groups (n=30): control (no thermal and mechanical load cycling), thermal cycling (2,000 cycles, 5°C-55°C), mechanical load

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cycling (50,000–80N) and thermal and load cycling (2,000 5°C-55°C/50,000–80N). The specimens were sealed with acid resistant varnish, leaving a 1-mm window around the cervical margin interface. To detect marginal leakage, a 2% methylene blue buffered solution was used for four hours. The specimens were sectioned longitudinally and qualitatively evaluated by stereomicroscopy (45x) following a ranked score for the dentin cervical margin. The data were analyzed by Kruskal-Wallis test ( $\alpha$ =0.05).

Shear Bond Strength Evaluation (SBS): Eighty bovine incisors were embedded and polished to obtain a flat standard surface on dentin. The surfaces were restored with Single Bond adhesive system and a resin composite subsequently inserted in a bipartite Teflon matrix. The specimens were randomly divided into the four groups (n=20) described above for microleakage. Shear bond strengths were measured in a universal testing machine with a crosshead speed of 0.5 mm/minute. The data were analyzed by one way ANOVA test ( $\alpha$ =0.05). No statistically significant influence of thermocycling, mechanical load cycling or the combination was observed for both microleakage and shear bond strength.

#### INTRODUCTION

A major goal of successful restorative treatment is the effective replacement of natural tooth structure. To effectively replace tooth structure, the restoration must be durable and functional. The durability of a restoration is largely based on maintenance of the tooth/restoration interface. To help maintain the integrity of the restoration, the tooth/restoration interface must resist dimensional changes to prevent developing leakage and possible further deterioration of the restoration (Leibrock & others, 1999). In addition to durability and function, the esthetic aspect of a restoration is also a growing concern. The extensive use of resin composites and adhesive systems in anterior and posterior teeth has been made possible by the continued advancement of their mechanical and physical properties.

Control of polymerization shrinkage is an important step in achieving and maintaining the integrity of the restoration interface. Polymerization shrinkage resulting in stress at the dentin/restoration interface allows for misadaptation of the material (Jorgensen & others, 1985) and, consequently, accelerates marginal leakage and possible deterioration of the restoration (Abdalla & Davidson, 1993; Alani & Toh, 1997; Dietschi & Herzfeld, 1998; Miyazaki & others, 1998). Even after controlling the effects of polymerization shrinkage, deterioration of the restoration could subsequently occur due to chemical, thermal and mechanical load stresses (Jorgensen & others, 1985; Abdalla & Davidson, 1993, 1996; da cunha Mello & others, 1997).

The gold standard for evaluating the clinical potential of a restorative material is the controlled clinical trial. The constant, rapid evolution of adhesive materials increases the number of long-term clinical trials needed. However, due to increasing costs and the immediate demand for information, there is a need for surrogate methods. The establishment of an *in vitro* methodology capable of reproducing some *in vivo* challenges is crucial for better understanding of restorative materials behavior. The use of mechanical and thermal cycling in laboratory studies of dental materials has been considered potential methods to simulate *in vivo* challenges (Abdalla & Davidson, 1996; da cunha Mello & others, 1997; Leibrock & others, 1999).

The use of thermal cycling is frequently seen in laboratory studies that evaluate microleakage (Darbyshire, Messer & Douglas, 1988; Yap, Stokes & Pearson, 1996; Alani & Toh, 1997; Yap, 1998; Hakimeh & others, 2000) and bond strength (Miyazaki & others, 1998; Leibrock & others, 1999). Several groups have questioned the effectiveness of this method in altering the restoration interface (Alani & Toh, 1997; Yap, 1998; Hakimeh & others, 2000). It is difficult to compare these studies,

because the temperatures, number of cycles and immersion times have not been standardized.

Several studies suggest that occlusal mechanical cycling could accelerate deterioration of the dentin/restoration interface (DeLong & Douglas, 1983; Abdalla & Davidson, 1993, 1996; da cunha Mello & others, 1997; Dietschi & Herzfeld, 1998). Some studies evaluating marginal leakage (Munksgaard & Irie, 1988; Abdalla & Davidson, 1996; Yap, Stokes, Pearson, 1996; Hakimeh & others, 2000) and bond strength (Williamson, Mitchell & Breeding, 1993; Leibrock & others, 1999) have included mechanical load cycling in their experimental protocol, although, as with thermal cycling, it is difficult to compare studies since they employed different load forces, number of cycles and different cycle frequencies.

This study evaluated the influence of mechanical and thermal cycling on microleakage at the cervical margins of proximal slot restorations and shear bond strength on flat dentin surfaces. The null hypotheses tested were that there was no relationship between thermal and mechanical cycling to microleakage and shear bond strength.

#### **METHODS AND MATERIALS**

#### **Microleakage Evaluation**

Specimen Preparation: One hundred and twenty fresh bovine incisors were cleaned of debris with curettes and pumice paste using a slow-speed handpiece and immersed in a 0.5% sodium azide saline solution. The incisal portion of the tooth was sectioned transversally 4 mm above the cemento-enamel junction (CEJ) at the mesial and distal surfaces, allowing the configuration of a flat standard occlusal surface.

Proximal slot cavities on mesial (MO) surfaces were prepared with a #245 high-speed carbide bur (KG Sorensen Ltd—Barueri, SP, Brazil) under water spray. The cavity dimensions were 3-mm wide, 5 mm-high (1 mm below the CEJ) and 1.5-mm deep.

The cavities were restored with Single Bond adhesive system (3M/ESPE Dental Products, St Paul, MN, USA) following manufacturer's instructions: acid etched for 15 seconds, rinsed for 15 seconds, blotted dry, two consecutive coats of the adhesive were applied, lightly air dried and light-cured for 10 seconds. The preparation was filled with a micro-hybrid resin composite Z-250 (3M/ESPE Dental Products) in two horizontal increments and light-cured for 40 seconds each, leaving a 1 mm overfill on the occlusal surface for mechanical load testing. During all restorative procedures the light intensity of the curing unit (Optilux/Demetron/Kerr Corp, Orange, CA, USA) was measured periodically by a radiometer (Demetron/Kerr Corp) and found to range from 520 to 560 mW/cm2. After the restorative procedure, the specimens were stored in distilled water at

 $37^{\circ}$ C for 24 hours. They were then finished and polished with  $Al_2O_3$  abrasive discs (Sof-Lex Pop-on 3M/ESPE Dental Products). The teeth were randomly divided into four groups (n=30):

G1= Control group (no thermal and mechanical load cycling)

G2= Thermal cycling (2,000 cycles; 5°C–55°C)

G3= Mechanical load cycling (50,000 cycles; 80N)

G4= Thermal cycling (2,000 cycles; 5°C–55°C) and mechanical cycling (50,000 cycles; 80N)

Thermal Cycling and Mechanical Load Cycling Procedure: Specimens from Groups 2 and 4 were subjected to 2,000 cycles in a thermocycling machine

(MCT2-AMM-2 INSTRUMENTAL, Sao Paulo, SP, Brazil) with two baths at  $5 \pm 2^{\circ}$ C and  $55 \pm 2^{\circ}$ C with a dwell time of 60 seconds and a transfer time of seven seconds between each bath.

Specimens from Groups 3 and 4 were submitted to mechanical load cycling. The specimens had part of their root embedded in epoxide resin (Buehler Ltd, Lake Bluff, IL, USA) in order to obtain a flat occlusal surface perpendicular to the long axis of the tooth. The cyclic mechanical loading device consisted of four stainless steel pistons to which a polyacetal cylinder tip was attached to the end. The polyacetal tips were placed in contact with the restoration (Figure 1). The loading device delivered an intermittent axial force of 80 N at 3 cycles/seconds totaling 50,000 cycles.

Immersion inDyeSolution andMicroleakage Evaluation: After thermal and mechanical load cycling, the apices and occlusal portion were filled with epoxy resin (Araldite Ciba—São Paulo, SP, Brazil) to avoid infiltration of the dye solution. The entire surface of each tooth was then coated with two layers of acid resistant varnish, except for a 1-mm width around the cervical margin. The teeth were immersed in a 2% methylene blue buffered solution for four hours and thoroughly rinsed under tap water for 10 minutes.

The teeth were hemi-sectioned longitudinally in a mesio-distal direction through the center of the restoration with a double-faced diamond disc (KG Sorensen Ind e Com Ltda) mounted on a straight low speed handpiece with water spray. For each restoration, two sections were obtained; the section with the highest

leakage score was considered for microleakage analysis since it represented the most severe dye penetration of the restoration. The sections were evaluated by three independent calibrated examiners on a stereomicroscope (MEIJI-EMZ-TR/Meiji Techno Ltd San Jose, CA, USA) at 45x magnification. The following criteria were used to score the extent of leakage at the cervical margin (Figure 2):

0 = No dye penetration;

1 =Dye penetration up to 1/3 of the cavity depth;

2 =Dye penetration up to 2/3 of the cavity depth;

3 = Dye penetration up to the entire cavity depth;

4 = Dye penetration into axial wall of cavity.

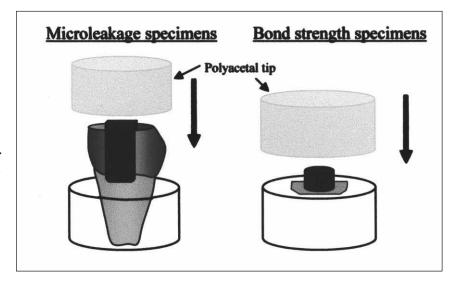


Figure 1. Illustration of the specimen shapes and incidence of the polyacetal tip for mechanical loading cycling. The load was applied only to resin composite. Black arrows show the axial direction of the force.

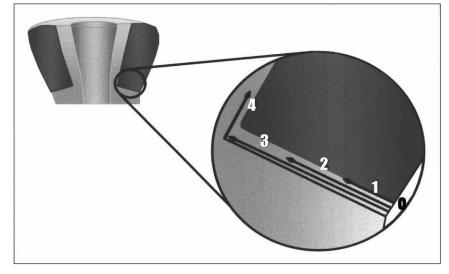


Figure 2. Schematization of the criteria employed to analyze the extent of leakage on cervical margin (dentin).

#### **Shear Bond Strength Test**

Specimen Preparation: Eighty extracted bovine incisors were collected, cleaned and stored in a 0.5% sodium azide saline solution. In each tooth, a piece from the coronal facial surface was sectioned with a double face diamond disk (KG Sorensen-Baruei, SP, Brazil) and mounted in a 3/4 inch diameter PVC ring parallel to the base of the ring. The rings were then filled with self-curing epoxide resin (Buehler Ltd) to set the teeth. The embedded teeth were ground on a watercooled mechanical grinder (Maxigrind-Solotest-São Paulo, SP, Brazil) using 320 grit Al<sub>2</sub>O<sub>3</sub> abrasive paper (Carborundum Abrasivos—Ribeirao Preto, SP, Brazil) to expose the dentin and polished with 400 and 600 grit Al<sub>2</sub>O<sub>3</sub> abrasive paper to obtain a 5-6 mm area of flat standardized dentin surface. They were then stored in distilled and deionized water at 37°C for 24 hours.

Before surface treatment, a 3-mm circular area was left uncovered as a bonding site by placing a piece of vinyl tape with a 3-mm diameter punched hole over the dentin. The Single Bond adhesive system was then applied following manufacturer's instructions.

A 3-mm diameter and 3-mm high bipartite Teflon ring mold was clamped to the dentin surfaces such that the mold was positioned over the treated dentin. The mold was bulk filled (Z-250) and light-cured (Optilux/Demetron) for 40 seconds, then light-cured for an additional 40 seconds after removing the mold. The light intensity was measured periodically by a radiometer. It ranged from 520 to 560 mW/cm². The specimens were immersed in distilled and deionized water at 37°C for 24 hours.

The teeth were then randomly divided into four groups (n=20):

G1= Control group (No Thermal and Mechanical Load Cycling)

G2= Thermal cycling (2,000 cycles)

G3= Mechanical load cycling (50,000 cycles)

G4= Thermal cycling (2,000 cycles) and mechanical cycling (50,000 cycles)

Thermal and Mechanical Load Cycling Procedure: Thermal and mechanical load cycling procedures were performed as previously mentioned. The specimens from Groups 3 and 4 were submitted to mechanical load cycling with the polyacetal tips placed in contact with the top of the resin composite cylinder. (Figure 1).

Bonding Test: Each specimen was mounted in a custom apparatus attached to a universal testing machine (EMIC Ltd São José dos Pinhais, PR, Brazil) with the dentin surface parallel to the machine's trajectory. A compressive load was applied using a steel knife-edge placed over the specimens so that the shear force was applied directly to the interface. The specimens were

loaded to fail at a crosshead speed of 0.5 mm/minute. The values were recorded and the shear bond strengths were calculated for each sample. Means and standard deviations were expressed in MPa. For fracture mode analysis, specimens were evaluated under a stereomicroscope (MEIJI-EMZ-TR/Meiji Techno Ltd) at 20x magnification and classified as adhesive, cohesive (in dentin and composite) or mixed failures (adhesive and cohesive).

#### Statistical Analysis

For microleakage evaluation, the scores of the three examiners were analyzed using the reproducibility Kappa test between examiners. To determine significant differences between groups, the median scores obtained by the three examiners were analyzed using the Kruskall-Wallis non-parametric analysis of variance test ( $\alpha$ =0.05). Shear bond strength data were analyzed using the parametric analysis of variance test (one-way ANOVA - $\alpha$ =0.05).

#### **RESULTS**

#### Microleakage Analysis

The Kappa Test showed 0.79 agreement between examiners 1 and 2, 0.95 between examiners 1 and 3 and 0.74 between examiners 2 and 3. The mean agreement was 0.83, indicating a good level of reliability for the scores evaluated. The Kruskal-Wallis non-parametric test indicated no statistically significant differences between the control group (G1) and the experimental groups—G2, G3, G4 (p=0.52). Thermal and mechanical load cycling did not have any effect on microleakage values when compared to the control group. Table 1 lists the mean rank of the dye penetration values and Table 2 describes the frequency of microleakage scores. The most common score was 1 (Figure 3); it demonstrated a low extent of dye penetration.

#### **Shear Bond Strength Analysis**

No statistically significant differences (p=0.49) were found for the bond strength values among the four groups evaluated. Table 1 describes the mean and standard deviations obtained for all groups. The use of thermal and mechanical load cycling did not affect the bond strength values of the experimental groups (G2, G3, G4) when compared to the control group (G1). For the fracture mode analysis, adhesive failure was most commonly observed. All groups presented with more than 95% adhesive failure and no differences were observed among the groups. Mixed fracture was observed for all other specimens (5%) and no cohesive failures were observed.

#### DISCUSSION

The unpredictability of the dentin substrate is a serious problem when bonding restorative materials (Pashley & others, 1993). For the microleakage evaluation, the

cervical margins of the restorations were positioned 1 mm below the CEJ due to critical adhesion in this area (Cagidiaco & others, 1997). The tubules in the cervical region are parallel to the cavity preparation, which impedes the formation of conventional tags. In addition, more intertubular dentin and less dentin tubules are exposed, increasing the area of hybrid layer formation resulting in higher bond strength values when compared to surfaces where the tubules are perpendicular to the bonded interface (Ogata & others, 2001).

Shear bond strength specimens were prepared on flat superficial dentin from the facial surface. The shear bond strength test has been criticized due to the occurrence of cohesive failures with current adhesive systems. This test reproduces a compressive stress situation found in restorations. In addition, it has been shown to be a reliable test and is currently used for bond strength evaluation in different substrates and conditions (Cardoso, Braga & Carrilho, 1998; Miyazaki & others, 1998; Leibrock & others, 1999).

In this study, bovine teeth were used in place of human teeth. Previous studies (Nakamichi, Iwaku & Fusayama, 1983; Reeves & others, 1995) have shown the similarity of bovine and human teeth in bonding tests. The major advantage of employing bovine teeth is the possibility of controlling for the age of the substrate (two years).

The application of thermal cycling and mechanical load cycling did not influence the values of microleakage and shear bond strength in this study. The use of thermocycling has been thoroughly investigated (Prati & others, 1994; Miyazaki & others, 1998; Hakimeh & others, 2000; Yoshida, Kamada & Atsuta, 2001). Variation can be observed depending on the dye stains, number of cycles, temperatures and materials

employed. The most common dye penetration stains employed are silver nitrate and methylene blue; both are considered acceptable markers for microleakage evaluation (Alani & Toh, 1997). The quantity of cycles and the temperatures used seem to be the major difference among the different studies (Prati & others, 1994; Yap, Stokes & Pearson, 1996; Cardoso & others, 1999; Hakimeh & others, 2000). In this study, 2,000 cycles were used as an average of recent articles. The use of intermediary baths and different temperatures has been described but the use of ISO

standardization ( $5^{\circ}$ - $55^{\circ}$ C) allows for a better comparison between studies.

Controversial results have been reported related to the influence of thermal cycling on microleakage. Darbyshire and others (1988); Prati and others (1994) and Chan and Glyn-Jones (1994) reported no effect of thermal cycling on microleakage. Recently, Hakimeh and others (2000) reported increased microleakage in Class V restorations after 2,880 cycles (4°-60°C). Several differences in the methodology of all these studies can be seen, such as the number of cycles, temperature and number of baths. In addition, the type of restorative material (amalgam, glass ionomer cement,

composite) used and the preparation (Class I, II or V) also varied. It is difficult to compare data from this study with that of other studies due to differences in methodology; however, the number cycles seems to be of major importance since different values may result, according to the number of cycles

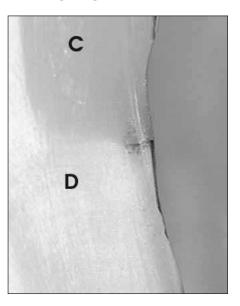


Figure 3. Photograph of microleakage with dye penetration score 1 between dentin (D) and composite (C).

Table 1				
	ı	Results		
Groups	Shear Bond Strengths Values n=20		Microle n=3	eakage Values 30
	Mean	SD	Median	Mean Ranks
G1 - Control	20.76	3.58	1	45.5
G2 - Thermal cycling	19.61	3.79	1	40.5
G3 - Mechanical cycling	21.13	3.60	1	54.6
G4 - Thermal cycling/ mechanical cycling	21.02	3.54	1	47.4

Table 2						
		Dy	e Penetrati	on Scores		
Groups	0	1	2	3	4	Total
G1	2	16	4	6	2	30
G2	3	14	3	8	2	30
G3	2	19	2	2	5	30
G4	5	17	3	2	3	30

used, independent of the material and force employed. Further studies should be developed to evaluate the different number of cycles and different materials.

Some groups have reported simulation of thermal stress for shear bond strength evaluation. Miyazaki and others (1998) and Yoshida and Atsuta (1999) reported no influence of thermal stress when using 10,000 and 20,000 cycles, respectively. Differences in bond strength, when subjected to thermocycling, are closely related to adhesion material/material than to material/substrate (dentin. enamel). Yoshida and others (2001) reported a decrease in bond strength between porcelain and resin after 50,000 thermal cycles. Leibrock and others (1999), evaluating the adhesion of porcelain repair systems, observed changes in shear bond strength with 2,400 thermal cycles and 480,000 mechanical cycles, depending on the materials employed. Miyazaki and others (1998), when comparing the different numbers of thermal cycles, observed a decrease in bond strength at 30,000 cycles for some adhesive systems. On the other hand, an increase in shear bond strength was observed after thermal cycling in enamel/material (Hosoya, 1994) and material/material (Atta, Smith & Brown, 1990; Yoshida & others, 2001). Considering the data of all studies cited and the current study, the authors of this study suggest that the influence of thermocycling is dependent on the number of cycles, restorative materials and dental substrates. In this study, thermal cycling was unable to affect bonding, which was most likely due to an insufficient number of cycles and high bond strength values.

The use of mechanical load cycling has been studied due to its potential for simulating mastication. In microleakage evaluations, load is applied with different tips, loading sets, number of cycles and load forces. This study used a cylindrical polyacetal tip that touched only the material aimed to fatigue the restoration. The force of 80 N was chosen as an average of the masticatory forces observed by Anderson (1956). It is difficult, if not impossible, to simulate the occlusal forces due to the variation in age, sex, type of tooth and food. These factors interfere with the force employed; however, the simulation of a mean force is necessary for further comparison between studies.

Abdalla and Davidson (1996), da cunha Mello and others (1997) and Ausiello and others (1999), using spherical tips touching only cusps, observed differences in microleakage values when applying 4,000 cycles at 125 N. In these studies, dye immersion was performed during loading as opposed to the current study. According to Jorgensen, Matono and Skimokobe (1976), during loading, temporary gaps are formed that allow for microleakage detection. However, other studies have shown results similar to the current

study when evaluating the effect of loading on microleakage. Prati and others (1994), using a spherical tip touching only a MOD restoration, did not observe any changes in microleakage using 17 Kgf for 1,440 cycles. In another study (Hakimeh & others, 2000), no effect on leakage was seen using 50,000 mechanical loading cycles (100N) on Class V restorations. The authors of this study suggest, according to the information obtained from the studies cited and the current study, that the restorative material, tip shape, location of force application, number of cycles and load force are responsible for the varying results.

The use of load cycling to analyze shear bond strength was studied by Leibrock and others (1999) and Williamson and others (1993). Both studies evaluated the adhesion between material/material. For the current study, a previous pilot study was conducted in order to evaluate the capacity of the specimen to support 80N for 50,000 cycles as proposed for the previous microleakage evaluation. The shape of the cylindrical resin cone (3 mm high and 3 mm in diameter) allowed for use of the load, and no specimen was lost during the mechanical loading. In this study, bond strength was not influenced by mechanical loading. Adhesive systems on the market today have been shown to produce high bond strength values and good sealing ability (Cardoso & others, 1998, 1999; Ogata & others, 2001). The ethanol-based adhesive Single Bond, employed in this study, is a representative one-bottle system that performs well in most studies. The authors suggest that the high quality of adhesion achieved by recent adhesive systems might limit the possible effect of mechanical cycling on bond strength and microleakage.

Further studies using different numbers of cycles and load need to be conducted in order to observe a possible influence of increased numbers of cycles on bond strength. In addition, the evaluation of microtensile and nanoleakage also need to be performed in order to evaluate the influence of thermal and mechanical load cycling.

#### CONCLUSIONS

According to the results, the null hypothesis was accepted. Thermocycling and mechanical loading showed no effect on microleakage and shear bond strength, either independently or in combination.

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## Support of Occlusal Enamel Provided by Bonded Restorations

LP Grisanti • KB Troendle • JB Summitt

#### Clinical Relevance

Bonded restorative materials provide some support to undermined enamel, but the amount of support is less than half that provided by sound dentin.

#### **SUMMARY**

In this in vitro study, the resistance to fracture of occlusal enamel supported by a bonded tooth-colored restorative material was compared to unsupported enamel and enamel supported by sound dentin. Eighty extracted human lower molars were sorted into five groups of 16 teeth each. Lingual cusps were removed. In Groups 2-5, dentin was removed from the facial cusps, leaving a shell of enamel. In Group 1, dentin was not removed. Group 2 remained unrestored. The groups in which a restorative material was inserted to replace missing dentin were as fol-Group 3-bonded resin (Scotchbond MP/Filtek Z250 [A2] in capsules, 3M); Group 4-resin-modified glass-ionomer (Fuji II LC [A2] in capsules, GC); Group 5-conventional

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\*Reprint request: Mail Code 7890, 7703 Floyd Curl Drive, San Antonio, TX 78229-3900, USA: e-mail: summitt@uthscsa.edu glass ionomer (Fuji IX [A2] in capsules, GC). Specimens were thermocycled (1500 cycles, 6°-60°C, dwell 30 seconds), then mounted in die stone with lingual inclines of facial cusps approximately horizontal. The cusp ridges of the lingual inclines were flattened slightly using a horizontally mounted separating disk. Specimens were loaded evenly on flattened inclines in an Instron with a flat rectangular rod at a crosshead speed of 5 mm/minute.

Data analysis was with one-way ANOVA and Student-Newman-Keul's test (F=50.30, p<0.0001). The bonded restorations provided significantly less enamel support than natural dentin and significantly more than when the enamel was left without support by dentin or a restorative material. There was no difference in support provided by the three restorative materials.

#### INTRODUCTION

The search for a direct restorative material that will serve to support occlusal enamel while still providing adequate esthetics has existed since the earliest days of dentistry. Throughout history, restorative materials were selected based on their best physical performance, hence, authors have established dental amalgam, zinc phosphate, zinc oxide eugenol and dental silicate cements as available materials (Wilson, 1991). As dental materials science continued to advance, goals shifted

toward esthetic appearance. As a result, numerous other restorative materials were developed, each having unique properties and characteristics. These materials include bonded resin composite, glassionomer and resin-modified glass ionomer.

Currently, it is common practice to remove occlusal enamel that is not supported by sound dentin in order to prevent the enamel from fracturing due to occlusal stresses. Major textbooks continue to advocate this ageold principle in operative dentistry (Roberson & Sturdevant, 2002; Hilton, 2001; Gilmore & others, 1982). The materials of choice for direct intracoronal restorations involving the occlusal surfaces of posterior teeth were traditionally amalgam and direct gold (foil).

Several studies have shown that bonded restorative materials reinforce the fracture resistance of teeth with cavity preparations (Eakle, 1985, 1986; Donly, Wild & Jensen, 1988; Reel & Mitchell, 1989; Joynt & others, 1987). One study showed that endodontically treated teeth restored with bonded resin composite/glass ionomer cement had a higher resistance to cusp fracture than sound, natural teeth. The study also showed no significant difference between the fracture resistance of sound, natural teeth and those restored with a bonded resin composite and a silver-filled glass ionomer (Wendt, Harris & Hunt, 1987). Other studies (Ausiello & others, 1997; Oliveira, Denehy & Boyer, 1987; Jagadish & Yogesh, 1990) have shown that resin composite, in combination with dentin bonding techniques, increases the fracture resistance of restored teeth so that it is greater than or equal to that of sound, natural teeth.

In the tunnel restoration used by some practitioners today, the marginal ridge is left intact and the carious dentin is accessed via a tunnel under the marginal ridge. This means that the enamel of the marginal ridge is left in place but is not supported by dentin. In one study (Hill & Halaseh, 1988), tunnel restorations, when the restorative material was glass ionomer, provided the strength of restored teeth that was not sig-

Figure 1



Figure 1A. Separating disk, with water coolant, being used to make the first cut in removal of the lingual cusps.



Figure 1B. #169L bur, being used to make the second cut for removal of the lingual cusps. An air/water spray was used when this cut was made.

nificantly weaker than sound teeth. When the restorative material used was dental amalgam, the strength of the marginal ridge of restored teeth was significantly less than sound teeth and not significantly greater than tunnel-prepared, unrestored teeth.

There would be significant advantages to having a restorative material that would reinforce occlusal enamel not supported by dentin. Dentists could preserve occlusal enamel left unsupported by removing the undermining carious dentin, which would preserve natural occlusal wear characteristics, esthetics and tooth strength.

This *in vitro* study evaluated and compared the resistance to enamel fracture imparted by natural dentin and three bonded restorative materials.

#### **METHODS AND MATERIALS**

Eighty extracted human mandibular molars with no restorations or fractures were selected and stored in water at room temperature. The molars were cleaned using a #14L carver (Thompson Dental Manufacturing Co, Inc, Missoula MT, USA) and a #4R/4L scaler (Hu-Friedy, Chicago, IL, USA). They were then measured in a mesiodistal direction and sorted into five groups of 16 teeth, each group having approximately the same average dimensions.

A separating disk (#5177, 7/8" x 0.025", Dedeco International, Inc, Long Eddy, NY, USA) in a handheld straight handpiece with water irrigation and a #169L friction-grip bur (Brasseler USA, Savannah, GA, USA) in a handheld high-speed handpiece with air/water spray, were used to remove the lingual cusps of each molar. The first cut, with the separating disk, was in a vertical direction parallel to the long axis of the tooth and extended 1 mm facial to the central groove in a mesiodistal direction through the marginal ridges (Figure 1A). That cut was made to a depth of approximately 4.5-mm in a pulpal direction from the occlusal surface. The second cut, using the #169L bur, began

approximately 2 mm apical to the CEJ on the lingual surface of each tooth and followed an obtuse angle to join the first cut (Figure 1B).

In Groups 2 to 5, a #2 friction-grip carbide bur (Brasseler) was used in a high-speed handpiece under water spray to remove all but a thin layer of the dentin in the facial cusps of each tooth. The remaining layer of dentin was

removed using a #7003 finishing bur (Brasseler) in a high-speed handpiece under water spray. A light brushing motion at stall-out speed was used so that there was less likelihood that the remaining enamel shell would be weakened. Visual and tactile evaluation were used to determine that dentin within the facial cusps was removed (Figure 2). A slight amount of enamel was also inevitably removed in the process of removing the dentin.

Group 1, which served as a control, consisted of 16 molars in which the dentin within the facial cusps was left intact.

Group 2, the second control, consisted of 16 molars, where the dentin within the facial cusps was removed but no restorative material was used to fill the cavity formed by removal of the dentin.

In Group 3, the cavity was filled with a bonded resin composite (Scotchbond Multi-Purpose dentin bonding system and Filtek Z250 resin composite in capsules, shade A2, 3M ESPE Dental Products, St Paul, MN, USA). Scotchbond etchant was applied for 20-30 seconds to the internal surface of enamel and to the dentin floor. The preparation was then lightly dried but was allowed to remain slightly moist, and Scotchbond Multi-Purpose primer was applied for five seconds to enamel and dentin of the area to be filled and dried. Scotchbond Multi-Purpose adhesive was then applied to the walls and light cured for 10 seconds. The Z250 composite was applied in 2-mm deep increments and each increment was light cured for 40 seconds. The restorative material was inserted and contoured so that its external surface was flush with the vertical cut in each molar.

In Group 4, the cavity was filled with a resin-modified glass ionomer (Fuji II LC in capsules, shade A2, GC America, Inc, Alsip, IL, USA). GC Cavity Conditioner was applied for 10 seconds to enamel and dentin and rinsed off. The preparation was then lightly dried. The material was mixed according to the manufacturer's directions and inserted using a layering technique, with each increment being light cured for 20 seconds. The last increment was inserted and contoured so that it was flush with the vertical cut in each molar prior to light curing. GC Fuji Varnish was applied to the surface of the restorative material for 10 seconds, then air dried.

In Group 5, the cavity was filled with conventional glass ionomer (Fuji IX in capsules, shade A2, GC America, Inc). GC Cavity Conditioner was applied to enamel and dentin for 10 seconds, rinsed and the preparation lightly dried. The material was mixed according to the manufacturer's directions and applied with its external surface flush with the vertical cut in each molar. GC Fuji Coat LC was applied and light cured for 10 seconds.



Figure 2. Dentin has been removed, leaving occlusal enamel unsupported.

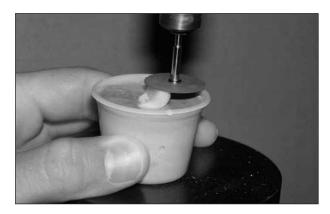


Figure 3. After teeth were mounted with lingual inclines of facial cusps approximately horizontal, a separating disk, mounted horizontally, was used to slightly flatten the cusp ridges.

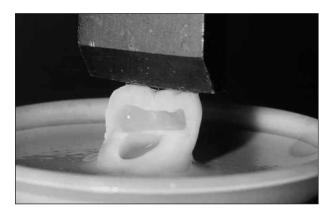


Figure 4. A rectangular, flat-ended rod was used to load the flattened inclines of the facial cusps of each specimen.

All specimens were thermocycled 1500 cycles, 6°C to 60°C, with a dwell time of 30 seconds, and had their roots notched. All specimens were then embedded in dental stone (Milestone Die Stone, blue, Modern Materials) to approximately 1 mm apical to the lingual extent of the second cut. The stone was contained in a one-ounce plastic soufflé cup (Georgia-Pacific Dixie Foodservice, Norwalk, CT, USA). A flat-ended rod was

held vertically by a Ney surveyor (JM Ney Company, Bloomfield, CT, USA) and was used to position the molar during mounting so that the lingual inclines of the facial cusps were approximately horizontal. To do this, the inclines were attached to the flat end of the rod with sticky wax. The cusp ridges of the lingual inclines of the facial cusps in each specimen

the facial cusps in each specimen
were then flattened slightly using a horizontally
mounted separating disk (Figure 3) to assure that the
cusps would be loaded simultaneously. The inclines
were then loaded to failure using an Instron testing
machine (model 1125, Instron). The rod used for
loading was rectangular in shape, 3 mm in depth and 8
mm in breadth. The rod was placed against the slightly
flattened lingual inclines of the facial-cusp enamel, con-
tacting the inclined ridge of each cusp (Figure 4).
Failure was defined as the first deflection on the stress-
strain chart recorder.

Throughout the study, when specimens were not being mounted, sectioned, treated or tested, they were stored in water at room temperature.

#### **RESULTS**

Results are shown in Table 1. A shattering of the enamel was observed in the majority of the specimens. Most of the fractures in Groups 3, 4 and 5 occurred at the restorative material/enamel interface. There were several instances in Group 5, where the restorative material shattered along with the enamel. In Group 1, one specimen fractured at the CEJ and another fractured within the root.

The mean failure load and standard deviation were calculated for each group. A one-way ANOVA showed f=50.30, p<0.0001, indicating a significant difference among the groups. A Student-Newman-Keul's test showed that the mean loads required to cause failure in Groups 1 and 2 were significantly different from each other, with each being significantly different from Groups 3, 4 and 5. There were no significant differences in mean failure loads among Groups 3, 4 and 5.

#### DISCUSSION

When enamel does not have sound dentin support, it is significantly weakened (Latino, Troendle & Summitt, 2001). During restorative treatment, it is common practice to remove occlusal enamel not supported by sound dentin. In some instances, however, unsupported enamel may be allowed to remain. For example, in occlusal enamel that will not be directly loaded, occlusal enamel may be left unsupported by dentin (Hill & Halaseh, 1988). This allows for the preservation of esthetics.

Table 1	Table 1: Results—Mean Failure Load in KN and Standard Deviation for Each Group			
Gp	Description	Mean Failure Load (KN)	SD	
1	Supported by dentin	2.880	0.965	
2	Unsupported	0.615	0.206	
3	Bonded resin composite	1.199	0.303	
4	Resin-Modified Glass-Ionomer	1.093	0.248	
5	Conventional Glass-Ionomer	1.054	0.298	
A line links the groups between which there was no significant difference (5.005)				

A line links the groups between which there was no significant difference (p=0.05).

Currently, allowing a restorative material to support occlusal enamel is not recommended in most situations. Unfortunately, dental materials science has not found a restorative material that will support and reinforce enamel as well as sound dentin. Although studies have shown that bonded restorative materials can provide strength and support for cusps that are supported by dentin, it is inadvisable to rely on these materials to support enamel from which the support of sound dentin has been lost (Boyer & Roth, 1994; Donly & others, 1988; Reel & Mitchell, 1989; Joynt & others, 1987; Eakle, 1985, 1986).

This study demonstrated that bonded resin composite, glass ionomer and resin-modified glass ionomer provided some support for occlusal enamel. This support was approximately intermediate between that provided by sound dentin and enamel with no support. These findings are consistent with similar studies (Latino & others, 2001; Boyer & Roth, 1994).

Based on the results of this study, bonded restorative materials should not be relied upon to support occlusal enamel to the extent that dentin supports overlying enamel. In order to reduce the possibility of fracture, any occlusal enamel that is subject to significant occlusal loading and is not supported by sound dentin should be removed and replaced by a strong restorative material. With further dental materials research, there is hope that a restorative material will someday be able to provide support for occlusal enamel equal to that provided by sound dentin.

#### **CONCLUSIONS**

Bonded resin composite, resin-modified glass ionomer and glass ionomer provided some support for occlusal enamel but it was significantly less than that provided by sound dentin.

#### Comment

These study results were presented by Mr Grisanti at the 2002 annual meeting of the International Association for Dental Research, San Diego, CA, USA; the abstract was published in the *Journal of Dental Research*, 2002, **81** A-175, Abstract #1258.

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# Effect of Different Application and Polymerization Techniques on the Microleakage of Proximal Resin Composite Restorations *In Vitro*

PA Brunton • A Kassir M Dashti • JC Setcos

#### **Clinical Relevance**

Conventional incremental build-up is recommended when proximal preparations are restored with resin composite.

#### **SUMMARY**

This *in vitro* study investigated cervical microleakage of proximal resin composite restorations placed with three application and polymerization techniques. Uniform mesio-occlusal-distal (MOD) preparations featuring cervical margins above (mesial) and below (distal) the CEJ suitable for restoration with resin composite were copy milled into 33 recently extracted permanent molars. The teeth were divided randomly into three groups of 11 teeth and restored using a conventional incremental technique

(Group A) and two novel curing devices (Groups B and C). After 24 hours, a dye penetration test was used to assess microleakage. Conventional placement in preparations with cervical margins in enamel had significantly lower interfacial leakage scores than those recorded for placement in preparations with margins in cementum regardless of the technique used to place the restorative material. Use of the two novel curing devices conferred no advantage in reducing microleakage irrespective of where preparation margins were placed.

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#### **INTRODUCTION**

In recent years, resin composite materials have become more widely used as alternatives to dental amalgam when restoring posterior teeth (Jordan & Suzuki, 1991; Leinfelder, 1989; Wilson, Dunne & Gainford, 1997). Despite concurrent developments in dentin bonding systems and associated application procedures, polymerization shrinkage continues to be a major problem associated with the use of resin composite restorative materials (Nordbo, Leirskar & von der Fehr, 1998). Polymerization contraction causes internal stresses that may become manifest at the restoration interface with the preparation walls. These stresses, if not correctly handled, will result in poor marginal adaptation at the

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cervical interface, which can lead to significant interfacial leakage, possibly resulting in early failure of the restoration in clinical service (Feilzer, de Gee & Davidson, 1987).

Clinical studies on proximal resin composite restorations have reported a high incidence (30-80%) of defective cervical margins (Kreulen & others, 1992; Opdam & others, 1998). It is not surprising, therefore, that resin composite restorations have been shown to have higher interfacial leakage at the cervical margins, whereas, interfacial leakage at the occlusal tooth/ restoration interface is relatively rare (Yap, Mok & Pearson, 1997). Defective cervical margins are related to certain factors, which include polymerization shrinkage of resin composite during curing, the difference in thermal expansion coefficient between resin composites and dental hard tissues, inaccessibility of the cervical area as well as problems with developing reliable adhesion to cervical enamel (Schuckar & Geurtsen, 1997). Lack of an optimal marginal seal further potentiates interfacial leakage at the cervical interface, which can induce staining, post-operative sensitivity and, in the long-term, pulpal inflammation leading to early failure of the restoration in clinical service (Crim & Chapman, 1994).

It is accepted that the extent to which interfacial leakage at the cervical interface is likely to occur depends on the technique used to restore the tooth (Nordbo & others, 1998). Many studies, therefore, have investigated new application techniques and or curing strategies designed to improve the marginal adaptation of restorative materials to the cervical margin of proximal preparations (Demarco & others, 2001; Dietschi & others, 1995; Dietschi & Herzfeld, 1998; Duncalf & Wilson 2001; Feilzer & others, 1995; Frankenberger & others, 1999; Manhart & others, 2001; Oberlander & others, 2001; Papathanasiou & Bardwell, 2001; Szep & others, 2001; Yoshikawa, Burrow & Tagami, 2001).

It has been reported that superior proximal contacts can be produced using a light-focusing tip coupled with the use of a pre-contoured, biplanar concave, sectional matrix (Lacy, 1996). Similarly, the use of a transparent cone to transmit light directly into the resin composite during curing appears to reduce cervical gap formation in proximal resin composite restorations (Ericson & Dérand, 1991).

The position of the cervical margin of the preparation in relation to cemento-enamel (CEJ) and the effect this has on the marginal adaptation of resin composite restorative materials has also been investigated. Preparations with margins in enamel, located 1.0 mm coronal to the CEJ, had excellent marginal adaptation, while preparation margins apical to the CEJ were found to have relatively poor marginal adaptation (Schuckar & Geurtsen, 1997).

The use of different matrices when preparation margins are in enamel had no effect on marginal adaptation at the cervical interface. This is not the case when preparation margins were placed in cementum. On cementum, composite inserted by oblique/horizontal increments then polymerized with a collimator cone had the least leakage, while resin composite placed with a transparent matrix band and a light-conducting reflective wedge produced the greatest interfacial leakage (Neiva & others, 1998).

#### **Hypothesis**

This *in vitro* study evaluated the cervical microleakage of proximal resin composites placed using three placement and polymerization techniques. The effect of the location of the cervical preparation margin on cervical microleakage was also investigated. The null hypothesis was that cervical microleakage is not affected by the technique used to place the resin composite restoration or the position of the cervical margin in relation to the cemento-enamel junction (CEJ).

#### **METHODS AND MATERIALS**

Thirty-four recently extracted, non-carious permanent molars free of visible cracks, defects or other deficiencies when trans-illuminated, were selected. One tooth was selected at random and a mesi-occlusal-distal (MOD) master cavity suitable for restoration with resin composite was prepared. The preparation featured cervical margins above (mesial) and below (distal) the CEJ. The prepared cavity was 4.5-mm wide at the isthmus and marginal ridge areas and 4- and 6-mm deep mesially and distally, respectively (Figure 1). An impression of this preparation was taken using an addition-cured silicone impression material (Dimension, 3M/ESPE, Seefeld, Germany), which was cast using an epoxy resin (Epoxy-Die, Ivoclar-Vivadent AG, Benderer-strasse 2, FL-9494, Schaan, Liechtenstein) to produce an ana-

logue master die for use in the Celay copymilling machine (Mikrona Ltd, Spreitenbach, Switzerland). The analogue die was mounted in the Celay machine and the preparations were copy milled into the 33 molars to give 33 proximal (MOD) preparations fea-



Figure 1. Master MOD preparation.

turing cervical margins above (mesial) and below (distal) the CEJ. The Celay machine was calibrated before, during and after the preparations were cut and all preparations were cut using the same bur type to improve consistency. Prior to any restorative procedure, the prepared molars were individually mounted in white plaster blocks and all teeth were stored in sterile water at room temperature. The molars were then randomly divided into three groups (A, B and C) using random number tables.

#### **Treatment Common to All Groups**

All molars were etched with 37% phosphoric acid (Dentsply DeTrey Gmbh, Konstanz, Germany). The etchant was placed on enamel for 15 seconds and extended to dentin for an additional 15 seconds, giving total etch times of 30 seconds and 15 seconds for enamel and dentin, respectively. Two coats of a dentin-bonding agent, One Coat Prime & Bond NT (Dentsply DeTrey Gmbh), were applied to the preparations and cured for 10 seconds according to the manufacturer's directions. The teeth were restored with Surefil (Dentsply DeTrey Gmbh) resin composite, shade A2. For restoration placement, a metal matrix band and Siguevaland (Ash Instruments, Dentsply Ltd, Plymouth, Devon) band retainer was used. All restorations were light cured in standard mode (Elipar, 3M/ESPE, Seefeld, Germany) from the occlusal aspect only using a standard curing time of 40 seconds. The output of the light was 800 mw/cm2, and the light was calibrated using the radiometer attached to the curing light which compensates for power fluctuations and the effects of bulb aging to give a constant light output. High speed, water cooled diamond finishing burs (Shofu Inc, Kyoto, Japan) were used to finish the occlusal surface and only interproximal finishing strips (Shofu Inc) with a grit size of 80-3 µm (coarse to fine) were used to finish the proximal part of the restoration.

Following restoration and after storage in water for 24 hours at room temperature, 11 molars from each group were selected at random and stored in a 4% solution of Rhodamine B for 24 hours at room temperature. The teeth were then washed, dried and mounted in cold-cured epoxy resin blocks and sectioned twice mesio-distally, 2 mm either side of the midline, with a diamond wheel. The cervical restoration/tooth interface was examined mesially and distally and the degree to which the dye had penetrated along the interface was scored using a protocol described by Chohayeb and Rupp (1989). Two readings were taken both mesially and distally and the highest score recorded. The data was entered into SPSS (SPSS Inc, Chicago, IL, USA) and analyzed using the Kruskal Wallis (nonparametric) one way analysis of variance statistical test to determine whether there was a statistical significance between the groups at the 5% level of significance (p<0.05). Individual differences between pairs of



Figure 2. Contact Pro and Opti Tip novel curing devices.

groups were investigated using the Mann Whitney U test; however, a Bonferroni correction was made to the p-value (p<0.003) as 15 comparisons were made.

#### **Treatment Specific to Each Group**

#### Group A

Teeth in this group were restored using a conventional incremental technique. Resin composite was applied in increments. The first increment (1 mm) was applied and adapted using hand instruments to the cervical floor and cured for 40 seconds. Subsequent oblique increments of 1-2 mm were applied and cured for 40 seconds.

#### Group B

Molars in this group were restored using the Contact Pro (CEJ Dental, San Juan Capistrano, CA, USA) novel curing and placement device (Figure 2). This light-conducting instrument is double-ended with mesial and distal heads that are believed to create tight and properly located interproximal contacts when proximal resin composites are placed.

An increment of resin composite 1-mm in depth was applied to the cervical floor of the preparation. The Contact Pro instrument was inserted into the resin composite and the instrument pushed against the matrix band, curing the material by delivering light through the placement device while the instrument was still in place. After curing, the instrument was removed and the remainder of the cavity was then restored incrementally as per the molars in Group A.

#### Group C

Teeth in this group were restored used a novel curing device Opti-Tip (Demetron, Orange, CA, USA) that was applied to the light curing tip (Figure 2). It was considered to recreate tight proximal contacts and allow light from the curing unit to concentrate in the tip of its cone placed within the resin composite, thus, improving curing of the resin composite in deeper parts of the preparation.

An increment of resin composite 1-mm in depth was applied to the cervical floor of the preparation. The

Opti-Tip instrument was inserted into the resin composite and the instrument pushed against the matrix band curing the material while the instrument was in place. After curing, the instrument was removed and the remainder of the cavity was then restored incrementally as per the molars in Group A.

#### **RESULTS**

Table 1 details the microleakage scores that are also summarized in Figure 3. The mean rank scores for microleakage for margins in enamel were 11.82, 31.41 and 30.59 for Groups A, B and C, respectively. The mean rank scores for microleakage for cervical margins placed in dentin were 32.91, 48.05 and 46.23 for Groups A, B and C, respectively. There was a highly significant difference (p<0.001) between the interfacial leakage scores for all the groups. Pairwise comparisons showed that between the groups with preparation margins placed in enamel, there was no statistical difference (p-values of 0.004, 0.006 and 0.97) in interfacial leakage scores. This was also the case for the groups with preparation margins in dentin with p-values of 0.0281, 0.047 and 0.80, respectively. Conventional placement in preparations with cervical margins in enamel had significantly (p-values of 0.002, 0.001 and 0.001) lower interfacial leakage scores than those recorded for placement in preparations with margins in cementum, irrespective of the technique used to place the restorative material.

#### DISCUSSION

The results of this study indicate that the marginal adaptation of proximal resin composite restorations placed in posterior teeth still remain problematical. Whatever the technique used to place a posterior proximal resin composite restoration in premolars and molars, there appears to be a tendency for cervical interfacial leakage to occur. This tendency is irrespective of the position of the margin in relation to the CEJ;

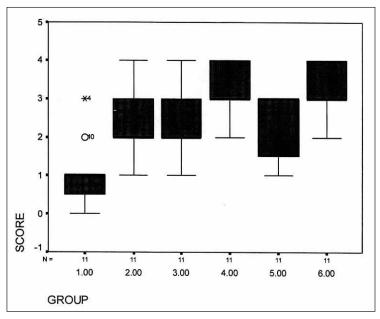


Figure 3. Box and whisker plot of microleakage score by group.

			Gro	oup		
	Α			В		С
	1	2	3	4	5	6
Specimen Number	Conventional Margins in Enamel	Conventional Margins in Cementum	Contact Pro Margins in Enamel	Contact Pro Margins in Cementum	Opti-tip Margins in Enamel	Opti-tip Margins in Cementum
1	1	3	2	3	3	3
2	1	3	1	4	2	3
3	1	2	4	3	2	2
4	3	4	2	3	3	4
5	0	2	2	2	3	3
6	1	2	1	3	1	3
7	0	1	3	4	3	4
8	1	2	3	4	3	4
9	1	2	4	4	1	4
10	2	3	2	3	3	2
11	0	3	2	3	1	3
Mean Rank	11.82	32.91	31.41	48.05	30.59	46.23

<sup>0.</sup> No dye penetration

<sup>1.</sup> Dye penetration up to half the length of the cervical floor

<sup>2.</sup> Dye penetration up to the full length of the cervical floor

<sup>3.</sup> Dye penetration up to half the axial wall

<sup>4.</sup> Dye penetration with involvement of the dentinal tubules

however, this study confirms that more cervical interfacial leakage is to be expected when the cervical margin is situated in dentin and cementum, that is, below the CEJ.

The difficulty of bonding resin composite restorative materials' reliably and reproducibly to the cervical aspects of proximal preparations, in particular to dentin and cementum, is well recognized. The bond strength of resin composite to dentin and cementum is less than to enamel, and the inability of this bond to resist the stresses generated during curing is likely to be responsible for marginal gap formation at the cervical interface of proximal preparations where the cervical margin is below the CEJ (Krejci & Lutz, 1990).

It has been suggested that bringing the curing light closer to the resin composite during curing of the initial increment of resin composite will improve cervical marginal adaptation of the restorative material (Ericson & Dérand, 1991). In this study, the use of two novel curing and placement devices designed specifically to improve the intensity of light immediately adjacent to this first increment was associated with poorer marginal adaptation and increased cervical interfacial leakage. Bringing the light closer to the curing increment of resin composite may be associated with an increased degree of conversion that would increase polymerization stress. This may explain the increased cervical interfacial leakage observed in this study. It is, therefore, difficult to justify the use of curing and placement devices of the type tested in this study if their primary aim is to improve marginal adaptation and reduce microleakage of resin composites. It is accepted, however, that one instrument tested in this study was designed to improve contact point formation as well, which can be difficult with low viscosity resin composites. This was not tested specifically in this study, but it is suggested that given the increased cervical interfacial leakage associated with this instrument, its routine use cannot be encouraged, and it is recommended that clinicians consider alternative methods of contact point formation when posterior proximal resin composite restorations are placed.

The results of this study contrast that of a previous study that reported that cervical gap formation was reduced by half when a light-transmitting cone was used during resin composite placement (Ericson & Dérand, 1991). This difference is probably explained by a dye immersion time of five minutes, which contrasts with a dye immersion time of 24 hours commonly used in microleakage studies such as this study. Equally, the teeth in the study were not etched and the proximal surface of the restorations polished with discs after placement, which casts some doubt on the clinical relevance of the results. A similar finishing technique was used by Neiva and others, (1998) but no

details of the dye immersion time was given, consequently, it is difficult to compare the results of these two previous studies with the data from this study.

This study could be considered as having certain limitations in that it was an *in vitro* study of microleakage and there is much debate about the clinical relevance of microleakage studies. This type of study design is, however, very useful for ranking and comparing techniques. While the effect of microleakage on restoration longevity is somewhat unknown, it is suggested that interfacial leakage at the cervical interface can induce staining, post-operative sensitivity and, in the longterm, can potentially cause pulpal inflammation leading to early failure of the restoration in clinical service (Crim & Chapman, 1994). Further research is needed to establish the clinical relevance of microleakage and its effect on the longevity of restorations. Meanwhile, an incremental technique should be used for placement of resin composite irrespective of whether the cervical margin is above or below the CEJ. Alternatively, the open sandwich or bonded-base technique should be used when the cervical preparation margin is below the CEJ given the reported success rates for this technique (van Dijken, Kieri & Carlén, 1999). Further research is necessary, however, to determine the effect that the use of a sandwich or bonded-base technique has on cervical interfacial leakage.

#### CONCLUSIONS

Within the limits of this study, it was concluded that:

- 1. Proximal preparations in molars restored with resin composite, placed incrementally, have significantly less cervical interfacial leakage when the cervical margins of the preparation are in enamel.
- 2. Use of the novel curing devices tested in this study conferred no advantage in reducing microleakage when the cervical margins of the preparation were above or below the cemento-enamel junction.
- 3. Conventional incremental build-up is recommended when proximal preparations are restored with resin composite.

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# Effect of Surface Treatment of Prefabricated Posts on Bonding of Resin Cement

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#### Clinical Relevance

Several surface treatments improved the bonding of resin cements to prefabricated posts of titanium alloy and zirconia.

#### **SUMMARY**

This in vitro study evaluated the effect of various surface treatments of prefabricated posts of titanium alloy (ParaPost XH), glass fiber (ParaPost Fiber White) and zirconia (Cerapost) on the bonding of two resin cements: ParaPost Cement and Panavia F by a diametral tensile strength (DTS) test. The posts received surface treatments in three categories: 1) roughening by sandblasting and hydrofluoric acid etching; 2) application of primer by coating with Alloy Primer, Metalprimer II and Silane and 3) a combination treatment in the form of roughening (sand-

blasting or etching) supplemented by the application of a primer or in the form of the Cojet system. After surface treatment, the post was embedded in a cylinder of resin cement (diameter = 4.0 mm, height = 4.0 mm). The surface-treated post was centered in the resin cement-filled mold with the aid of fixation apparatus. Fifteen minutes from the start of mixing the resin cement, the specimen was freed from the mold and stored in water at 37°C for seven days. Following water storage, the specimen was wet-ground to a final length of approximately 3 mm. The DTS of specimens was determined in a Universal Testing Machine. The bonding of resin cement to titanium alloy posts was increased by several surface treatments of the post. However, coating with primers as sole treatment had no effect on bonding. With the DTS method applied, none of the surface treatments had an effect on the bonding to glass fiber posts. The bonding of both resin cements to zirconia posts was improved by Cojet treatment, while sandblasting, followed by silane application, improved bonding of Panavia F.

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#### INTRODUCTION

Posts are frequently inserted into endodontically treated teeth to provide retention for a core in the restorative treatment of the teeth (Manning & others, 1995; Robbins, 1990). Two types of post-core systems exist: custom-made posts and cores and prefabricated posts. Custom-made posts and cores are traditionally cast from alloys of noble metals. Prefabricated posts of various materials, types and designs exist on the market.

Several studies have investigated the failure of teeth restored with posts and cores. The causes of these failures are loss of retention and fracture of the root, post or core (Hatzikyriakos, Reisis & Tsingos, 1992; Bergman & others, 1989; Sorensen & Martinoff, 1984; Turner, 1982). Retentive failure of posts has been reported to be the most frequent type of failure (Torbjörner, Karlsson & Ödman, 1995). Generally, retention of the post is affected by factors involving the post, cement and bonding of the cement to the post and the root canal.

Studies have investigated retentive factors related to the post and have found variables including length, diameter, design, surface structure and post material to affect post retention (Nergiz & others, 2002; O'Keefe, Miller & Powers, 2000; Ferrari & others, 2000; Isidor, Brøndum & Ravnholt, 1999; Mannocci, Ferrari & Watson, 1999; Miller & others, 1998; Isidor, Ödman & Brøndum, 1996; Torbjörner & others, 1995; Standlee & Caputo, 1993; Sorensen & Martinoff, 1984; Standlee, Caputo & Hanson, 1978; Newburg & Pameijer, 1976; Colley, Hampson & Lehman, 1968). Two groups of prefabricated posts have been introduced to the market: metallic posts, such as titanium alloy and stainless steel, and non-metallic posts, such as glass fiber, carbon fiber and zirconia. Unlike metallic posts, non-metallic posts are intended to maximize retention in the root canal by bonding to resin cements. *In vitro*, the bonding of posts to the root canal has also been found to reinforce the tooth and prevent root fracture (Mendoza & others, 1997).

As far as cement is concerned, the retention of endodontic posts is affected by parameters such as strength of the cement and its bonding to the post and dentin. Thus, studies on the retention of posts have shown superior retention of posts luted with resin cement compared to posts luted with zinc phosphate cement (Utter, Wong & Miller, 1997; O'Keefe & others, 1992). Two types of resin cements exist: conventional BisGMA-based resin cements and so-called adhesive resin cements that contain functional monomers such as MDP (10-methacryloyloxydecyl dihydrogen phosphate) or 4-META (4-methacryloyloxyethyl trimellitate anhydride).

In attempts to maximize the bonding of resin cement to prefabricated posts, several surface treatments of posts have been suggested. These surface treatments may fall within three categories: 1) treatments that result in roughening of the surface (sandblasting and etching with hydrofluoric acid) (Dérand & Dérand, 2000; Cobb & others, 2000; Nakagawa & others, 1999; Kern & Wegner, 1998; Kern & Thompson, 1994; Miller & others, 1998); 2) treatments that intend to create chemical bonding between cement and post (coating with primers) (Yanagida & others, 2002; Taira & others, 2000; Watanabe, Powers & Lorey, 1988) and 3) treatments that have both a roughening and chemical component either by a combination of two of the above-mentioned treatments or by the unique Cojet system (Cobb & others, 2000; Frankenberger, Krämer & Sindel, 2000; Kern & Thompson, 1994; O'Keefe & others, 1992). Sandblasting with alumina particles results in increased roughness of the surface and increased surface area. Etching with hydrofluoric acid is intended to create a roughening of the surface that allows for micromechanical interlocking of resin to the restoration (Wolf, Powers & O'Keefe, 1993). Primers such as silane and so-called metal primers contain functional monomers that create chemical adhesion between resin cement and dental restorations (Taira & others, 2000; Söderholm & Shang, 1993). The Cojet system for intraoral use is a modification of the Rocatec system introduced in 1989 for laboratory use. By sandblasting with silicate-coated alumina particles (Cojet, 3M ESPE, St Paul, MN, USA), a silicate layer is welded onto the surface by high spot heat produced by blasting pressure in a process referred to as tribochemical coating. Sandblasting is followed by silanization, and the Cojet system thus combines micro-mechanical retention produced by sandblasting and chemical bonding resulting from silanization of the silicated surface.

Several studies have investigated the bonding of resin cements to posts of titanium alloy, zirconia and carbon fiber and the effect of numerous surface treatments (Sahafi & others, 2003; Yanagida & others, 2002; Yanagida, Matsumura & Atsuta, 2001; O'Keefe & others, 2000; Taira & others, 2000; Wegner & Kern, 2000; Miller & others, 1998; Kern & Wegner, 1998; Fujishima, Fujishima & Ferracane, 1995; Kern & Thompson, 1994). However, these studies have been limited to investigating the bond strength of resin cement to posts that had been ground and therefore deprived of their original surface. Moreover, there is sparse information on the adhesion of resin cement to glass fiber posts.

It was hypothesized that the bonding of resin cement to prefabricated posts is influenced by the post material, type of resin cement and surface treatment of the post. The aim of this *in vitro* study was to investigate the influence of surface treatment of titanium alloy, glass fiber and zirconia posts on the bonding of resin cement (a Bis-GMA-based cement and an adhesive cement). To

allow for the bonding of resin cement to the original surface of the post, a diametral tensile strength test was applied.

#### **METHODS AND MATERIALS**

Three prefabricated posts: a titanium alloy post (ParaPost XH), a glass fiber post (ParaPost Fiber White) and a zirconia post (Cerapost) and two resin cements: a conventional BisGMA-based resin cement (ParaPost Cement) and an MDP-containing resin cement (Panavia F) were used. Table 1 lists the composition of the investigated posts and resin cements and their respective manufacturers. The surface treatments investigated are listed in Table 2.

#### Validation of Method

To assess the validity of the method, diametral tensile strength (DTS) of the two resin cements with and without a centrally placed cylindrical cavity and Vaseline-treated post were tested. A mold with a cylindrical cavity (diameter = 4.0 mm, height = 4.0 mm) was used to prepare the cylinders of resin cement. The cement was mixed according to the manufacturer's recommended procedure and applied into the cavity. The central cavity in the resin cement was produced by embedding the Vaseline-treated, parallel-sided part of a zirconia post (Cerapost 050, diameter = 1.4 mm). The post was centered in the resin cement by fixation apparatus (Figure 1) and removed after the setting time of the resin cement (15 minutes), leaving a cavity in the center of the resin cement specimen. The specimens, including the Vaseline-treated posts (ParaPost XH size 5, d = 1.25 mm; ParaPost Fiber White size 5, d = 1.25 mm; Cerapost No. 050, d = 1.4 mm), were produced in a similar manner except that the posts were not removed from the specimens after setting the resin cement. After 15 minutes, the specimens were freed from the mold and stored in water at 37°C for seven days. The specimens were then wet-ground (carborundum paper #220) to a final length of approximately 3 mm. For the

Table 1: List of Investigated Prefabricated Posts and Resin Cements		
Post	Composition According to Manufacturer	Manufacturer
ParaPost XH	90% titanium, 6% aluminum, 4% vanadium	Coltène/Whaledent, USA
ParaPost Fiber White	42% glass fiber, 29% resin, 29% filler	Coltène/Whaledent, USA
Cerapost	94.9% ZrO <sub>2</sub> , 5.1% Y <sub>2</sub> O <sub>3</sub>	Gebr. Brasseler, Germany
Resin Cement	Composition According to Manufacturer	Manufacturer
ParaPost Cement	BisGMA, BisEMA, TEGDMA, BPO, silanized barium glass, amorphous silica	Coltène/Whaledent, USA
Panavia F	Silanated barium glass, silanated silica, sodium fluoride, BPO, photosensitizer, 10-methacryloyloxydecyl dihydrogen phosphate (MDP), hydrophobic and hydrophilic dimethacrylates, Bis-phenol A polyethoxy dimethacrylatebic and hydrophilic dimethacrylates, Bis-phenol A polyethoxy dimethacrylate	Kuraray, Japan

Surface Treatment	Composition	Manufacturer	Type of Post
None			XH, FW, CP
Sandblasting	Alumina particles of 50 μm	BEGO, Germany	XH, FW, CP
Etching with hydrofluoric acid	9.6% hydrofluoric acid	Pulpdent, USA	XH, FW, CP
Cojet	Silicate-coated particles of 30 µm, silane	3M ESPE, Germany	XH, FW, CP
Alloy Primer	6-[N-(4-vinylbenzyl)propylamino]-1,3,5-triazine 2,4-dithione (VBATDT), 10-methacryloyloxydecyl dihydrogen phosphate (MDP), acetone	Kuraray, Japan	XH
Metalprimer II	Thiophosphoric methacrylate (MEPS)	GC Dental, Japan	XH
Sandblasting and Alloy Primer	Alumina particles of 50 µm and Alloy Primer		XH
Sandblasting and Metalprimer II	Alumina particles of 50 µm and Metalprimer II		XH
Etching and Alloy Primer	9.6% hydrofluoric acid and Alloy Primer		XH
Etching and Metalprimer II	9.6% hydrofluoric acid and Metalprimer II		XH
Silane	Silane	Pulpdent, USA	FW, CP
Sandblasting and silane	Alumina particles of 50 µm and silane		FW, CP
Etching and silane	9.6% hydrofluoric acid and silane		FW

specimens that included a post, the non-embedded part of the post was removed before grinding with a diamond-coated cutting wheel in a handpiece under water spray. The DTS of specimens was determined in a Universal Testing Machine (Instron, High Wycombe, UK) at a crosshead speed of 10 mm/minute (Figure 2). The DTS was calculated according to Specification No 27 of the American Dental Association (ANSI/ADA Specification No 27 for direct filling resins, 1977) as  $T_{\rm s}$  (MPa) = 2P/ $\pi$ dl, in which  $T_{\rm s}$  = tensile strength (MPa), P = load at fracture (N), d = diameter of specimen (mm) and l = length of specimen (mm). The number of specimens in each group was 10.

#### **Effect of Surface Treatments of Posts**

The DTS method described above was subsequently used to evaluate the effect of surface treatment of the posts on the bonding of resin cement. ParaPost XH posts (Size 5, d = 1.25 mm), ParaPost Fiber White posts (Size 5, d = 1.25 mm) and Cerapost posts (No 050, d = 1.4mm) were surface-treated according to one of the treatments in Table 2. The number of specimens in each group was 10. The surface treatments were of three different categories. The first category comprised surface treatments that resulted in roughening the surface, sandblasting with alumina particles and etching with hydrofluoric acid. Sandblasting was performed at 4 bar for 15 seconds using alumina particles of 50 µm in an extraoral sandblasting device (Basic duo, Renfert GmbH & Com, Germany) held perpendicular to the post surface at a distance of 20 mm. Following sandblasting, the post was ultrasonically cleaned in deionized water for two minutes. Etching with 9.6% hydrofluoric acid (Porcelain Etch Gel) was carried out for two minutes. The post was subsequently rinsed with deionized water for two minutes.

The second category of surface treatments comprised coating the posts with primers (Metalprimer II, Alloy Primer and Silane). These primers are intended to create chemical bonding between the post and resin cement. The primers were applied to the post surface according to the manufacturers' instructions. The third category of surface treatments comprised combinations of a roughening treatment and application of a primer or use of the Cojet system. Cojet treatment consisted of sandblasting with an intraoral sandblasting device (Dento-prep, Rønvig A/S, Denmark) at 4 bar for 15 seconds using 30 µm silicate-coated particles followed by silane coating with ESPE-sil according to the manufacturer's instructions.

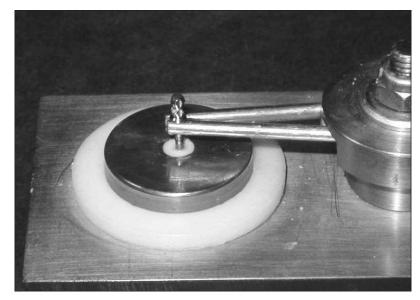


Figure 1. The apparatus used to center the post in resin cement.

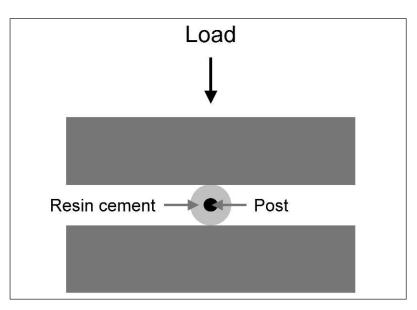


Figure 2. Schematic diagram of the diametral tensile strength test.

After surface treatment, the post was embedded in a cylinder of resin cement. A mold with a cylindrical cavity (diameter = 4.0 mm, height = 4.0 mm) was used to prepare the cylinder of resin cement. The cement was mixed according to the manufacturer's recommended procedure and applied into the cavity. The surface-treated post was placed in the center of resin cement with the aid of fixation apparatus as described above and shown in Figure 1. After 15 minutes, the specimen was freed from the mold and stored in water at 37°C for seven days. Following water storage, the specimen was wet-ground (carborundum paper #220) to a final length of approximately 3 mm by first removing the non-embedded part of the post. The DTS of the specimen was determined as described above.

#### **Statistical Analysis**

Results of the validation tests with ParaPost Cement were analyzed by one-way ANOVA and Newman-Keuls' multiple range test (Bruning & Kintz, 1997; Hald, 1952). Because of a lack of homogeneity of the standard deviations, the results obtained with Panavia F were analyzed by Mann-Whitney U-test (Bruning & Kintz, 1997). Results of the surface treatment tests were analyzed by three-way factorial ANOVA (SAS 8e software, SAS Institute, Cary, NC, USA) to identify differences among posts, resin cements and surface treatments, followed by Newman-Keuls' multiple range tests. For each post and resin cement, the surface treatments that gave a significant improvement in DTS of the resin cement containing posts were compared with the DTS of the resin cement itself by one-way ANOVA and Newman-Keuls' multiple range test. For each post and resin cement, the result obtained with the Vaselinetreated post of the validation tests was compared with the result obtained with the corresponding non-treated post by a Student's t-test (Hald, 1952). The level of significance for all analyses was set at  $\alpha$ =0.05.

#### **RESULTS**

#### Validation of Method

The results are presented in Table 3. The DTS of both resin cements decreased significantly by the presence of a Vaseline-treated post and even more by the presence of a central cavity.

#### **Effect of Surface Treatment of Posts**

Tables 4-6 show the results obtained with the three posts, and the results of the three-way factorial ANOVA is featured in Table 7. The factors post and surface treatment had a significant effect (p<0.0001). There was significant interaction between post and resin cement (p<0.0001) and interaction between post and surface treatment (p<0.0001).

With respect to ParaPost XH (Table 4), ParaPost Cement generally yielded higher DTS values than Panavia F. In the ParaPost Cement group, sandblasting, hydrofluoric acid etching, Cojet treatment and etching supplemented by coating with Alloy Primer resulted in an improvement in DTS compared to no treatment of the embedded posts. In the Panavia F group, six of the surface treatments of embedded posts (sandblasting, Cojet treatment, sandblasting supplemented by Alloy Primer or Metalprimer II coating and hydrofluoric acid etching supplemented by Alloy Primer or Metalprimer II coating) resulted in a significant improvement in DTS compared to no treatment of the embedded posts and to the same degree. For both resin cements, all surface treatments of the embedded posts which significantly improved the DTS gave DTS results that did not differ from the DTS of resin cements themselves, that is, without a central cavity or

a Vaseline-treated post ( $40.9 \pm 4.1$  MPa and  $38.5 \pm 6.5$  MPa, respectively). For both resin cements, the DTS of the resin cement containing non-treated posts was significantly higher than the DTS of the resin cement containing Vaseline-treated posts.

With ParaPost Fiber White, ParaPost Cement generally gave higher values of DTS than Panavia F (Table 5). For both cements, none of the surface treatments of embedded post significantly changed the DTS from the resin cements containing non-treated posts. For both resin cements, the DTS of resin cement containing non-treated posts was significantly higher than the resin cement containing Vaseline-treated posts.

For Cerapost, Panavia F yielded higher DTS values than ParaPost Cement (Table 6). When the embedded posts were Cojet-treated, the DTS of ParaPost Cement was significantly higher than ParaPost Cement containing non-treated posts and as high as the DTS of ParaPost Cement itself ( $40.9 \pm 4.1 \text{ MPa}$ ). Two surface treatments of the embedded posts (Cojet treatment and sandblasting supplemented by silane coating) significantly increased the DTS of Panavia F. While sandblasting followed by a silane coating of the embedded posts increased the DTS of Panavia F to the same DTS as Panavia F (38.5  $\pm$  6.5 MPa), Cojet treatment of the embedded posts resulted in a DTS of Panavia F that was significantly higher than Panavia F, itself. For both resin cements, the DTS of resin cement containing nontreated posts was significantly higher than the DTS of resin cement containing Vaseline-treated posts.

#### DISCUSSION

The type of resin cement was found to have a significant effect on the DTS that varied with the type of post. Examination of the results revealed that DTS values of ParaPost Cement generally were higher when used with ParaPost XH and ParaPost Fiber White than with Cerapost. On the other hand, the DTS values of Panavia F were generally higher when used with Cerapost. These differences may tentatively be explained by differences in surface energy characteristics between the posts and between the resin cements, and by speculation that the surface energy characteristics of a certain post match better with one of the resin cements than with the other (Asmussen, Attal & Degrange, 1995). Thus, when surface energy characteristics of the post and resin cement match each other, bonding is enhanced.

This study evaluated the effect of various surface treatments of prefabricated posts of titanium alloy, glass fiber and zirconia on the bonding of two resin cements by a diametral tensile strength test. Several studies have investigated the bonding between resin cement and posts by bond strength tests (Sahafi & others, 2003; O'Keefe & others, 2000; Dérand & Dérand,

2000; Miller & others, 1998) or by axial tensile tests (so-called retention tests) (Drummond, 2000; O'Keefe & others, 1992; Chapman, Worley & von Fraunhofer, 1985). To achieve a well-defined bonding area, bond strength testing usually requires grinding of the substrate; consequently, bonding is obtained to the core of the substrate, not to the original surface, and for posts, this implies a risk of irrelevant or skewed results. In retention tests, the original surfaces of the posts are intact. However, the resulting values of retentive strength are expressions of the multitude of facthat influence tors macro-retention, one factor being bonding.

In an attempt to eliminate the weak points of the above-mentioned tests and focus on bonding to the original post surface, a DTS test was applied in this study. Experiments were conducted to study the validity of the method. The presence of a central cavity in the cylinder of resin cement significantly decreased DTS of the resin cement. This reduced DTS was increased by the presence of a non-bonded (Vaseline-treated) post and was increased even further by the presence of a post with its original (non-treated) surface. The results also showed that certain surface treatments of the subsequently embedded posts improved the DTS to dif-

statistically different (p>0.05).

Table 3: Diametral Tensile Strength (MPa) of Resin Cements with and without a Central Cavity or a Vaseline-treated Post\*

	Resin Cement		
Resin Cylinder	ParaPost Cement	Panavia F	
Without central cavity	40.9 ± 4.1°	38.5 ± 6.5°	
With central cavity	$11.6 \pm 3.6^{a}$	$6.1 \pm 1.4^{a}$	
With Vaseline-treated ParaPost XH	16.7 ± 2.3°	16.1 ± 3.2 <sup>b</sup>	
With Vaseline-treated ParaPost Fiber White	17.3 ± 5.2 <sup>b</sup>	13.7 ± 3.4 <sup>b</sup>	
With Vaseline-treated Cerapost	16.8 ± 4.8 <sup>b</sup>	$17.0 \pm 4.4^{\circ}$	

Table 4: Diametral Tensile Strength (MPa) of Resin Cements with Embedded ParaPost XH

Posts\*

	Resin Cement		
Surface Treatment	ParaPost Cement	Panavia F	
None	$26.6 \pm 5.2^{a,b}$	$23.4 \pm 4.2^{a}$	
Sandblasting	$36.2 \pm 5.4^{d,e}$	$30.2 \pm 5.1^{b,c}$	
Etching with hydrofluoric acid	$34.2 \pm 5.8^{c,d,e}$	$27.9 \pm 4.6^{a,b}$	
Alloy Primer	$28.9 \pm 5.8^{a,b,c}$	$25.0 \pm 3.7^{a}$	
Metalprimer II	$23.5 \pm 6.0^{a}$	$24.5 \pm 3.0^{a}$	
Cojet treatment	$40.1 \pm 4.7^{\circ}$	$35.3 \pm 3.3^{\circ}$	
Sandblasting + coating with Alloy Primer	$33.3 \pm 7.5^{b,c,d,e}$	$32.6 \pm 3.0^{b,c}$	
Sandblasting + coating with Metalprimer II	$30.5 \pm 3.9^{b,c,d}$	$32.2 \pm 5.7^{b,c}$	
Etching with hydrofluoric acid + coating with Alloy Primer	$36.3 \pm 5.4^{d,e}$	$32.3 \pm 4.6^{b,c}$	
Etching with hydrofluoric acid + coating with Metalprimer II	$27.1 \pm 5.1^{a,b}$	$32.2 \pm 4.6^{b,c}$	

\*Diametral tensile strength shown as mean ± SD (n=10). For each cement, mean values designated with the same superscript letter are not statistically different (p>0.05).

Table 5: Diametral Tensile Strength (MPa) of Resin Cements with Embedded ParaPost Fiber White Posts\*

	Resin Cement			
Surface Treatment	ParaPost Cement	Panavia F		
None	24.7 ± 5.8°	24.0 ± 3.1 <sup>a</sup>		
Sandblasting	$25.3 \pm 3.3^{a}$	$24.7 \pm 3.5^{a}$		
Etching with hydrofluoric acid	$23.2 \pm 2.7^{a}$	$24.0 \pm 3.9^{a}$		
Silane	$24.2 \pm 4.4^{a}$	$21.0 \pm 3.6^{a}$		
Cojet treatment	$26.7 \pm 6.0^{a}$	$23.2 \pm 4.7^{a}$		
Sandblasting + coating with silane	$24.0 \pm 2.7^{a}$	$23.8 \pm 4.8^{a}$		
Etching with hydrofluoric acid + coating with silane	$24.6 \pm 3.8^{a}$	$19.3 \pm 4.3^{a}$		
*Diametral tensile strength shown as mean ± SD (n=10). For each cement, mean values designated with the same superscript letter are not				

statistically different (p>0.05)

Table 6: Diametral Tensile Strength (MPa) of Resin Cements with Embedded Cerapost Posts\*

	Resin Cement		
ParaPost Cement	Panavia F		
27.5 ± 4.0°	28.8 ± 6.2 <sup>a,b</sup>		
$28.5 \pm 3.9^{a}$	$33.5 \pm 5.1$ <sup>b,c</sup>		
$26.5 \pm 3.5^{a}$	$27.3 \pm 6.1^{a}$		
$25.7 \pm 5.3^{a}$	$31.3 \pm 7.2^{a,b}$		
$39.3 \pm 6.9^{\circ}$	$43.7 \pm 8.4^{d}$		
$28.0 \pm 6.7^{a}$	$36.6 \pm 5.5^{\circ}$		
	$27.5 \pm 4.0^{\circ}$ $28.5 \pm 3.9^{\circ}$ $26.5 \pm 3.5^{\circ}$ $25.7 \pm 5.3^{\circ}$ $39.3 \pm 6.9^{\circ}$		

ferent degrees compared to no surface treatment. Based on this logic rank of results, the authors deemed the method able to express the quality of bonding between post and resin cement.

The zirconia posts used were 1.4 mm in diameter, while the titanium alloy and glass fiber posts were 1.25 mm in diameter. As the presence of a non-bonded post had a reducing effect on the DTS of resin cements, it may be presumed that the zirconia posts reduced the DTS of cements more than the other posts and the DTS values obtained with zirconia posts would have been higher if the 1.25-mm posts had been used. Although no such effect is evident from the results, care should be taken if comparisons are made among the different posts. The posts also differed with respect to surface morphology in that the zirconia posts are smooth, while the surface of the titanium alloy and glass fiber posts have retention patterns. However, no differences were observed among the DTS values obtained when the three types of posts had not been surface treated. This implies that surface morphology did not significantly influence the DTS values.

The results obtained with the embedded ParaPost XH titanium post showed that the two roughening treatments (sandblasting and hydrofluoric acid etching) and two of the combined treatments (Cojet treatment and hydrofluoric acid etching supplemented by Alloy Primer coating) improved the DTS in the ParaPost Cement group, while sandblasting and all combined treatments of the subsequently embedded posts improved the DTS in the Panavia F group. Several shear bond strength studies support the positive result obtained with sandblasting (Sahafi & others, 2003; O'Keefe & others, 2000; Miller & others, 1998). Hydrofluoric acid etching may have resulted in roughening of the titanium-based post by destroying the protective oxide film that causes corrosion of titanium (Nakagawa & others, 1999) and possibly pitting of the surface (Zavanelli & others, 2000; Reclaru & Meyer, 1998). Mechanical interlocking of resin cement in the resulting irregularities may explain the significant improvement in DTS of ParaPost Cement. The lack of significant improvement in DTS of Panavia F, when embedded posts had previously been etched with hydrofluoric acid, may be explained by a

negative effect on this specific resin cement of remnants of hydrofluoric acid (Sahafi & others, 2003; As mussen, 1997). In agreement with the results of Sahafi and others

(2003), Cobb and others (2000), Kern and Wegner (1998) and Kern and Thompson (1995), Cojet treatment of subsequently embedded posts was a very effective method for improving the DTS of resin cements. No additive effect was obtained when sandblasting or hydrofluoric acid etching was combined with application of a primer.

In this study, none of the surface treatments of subsequently embedded ParaPost Fiber White affected the DTS of resin cements. This finding disagrees with the previous study by Sahafi and others (2003), who found significant improvements in shear bond strength of resin cements to ParaPost Fiber White as a result of a number of surface treatments. The discrepancy between the results of the two studies may be explained by the different methods applied: The ParaPost Fiber White post is composed of glass fiber embedded longitudinally in a matrix of resin and filler. This implies that the mechanical properties depend upon the direction of the applied load. In the DTS test, the load was applied in a direction perpendicular to the orientation of the fibers and resulted in splitting of the fiber post, indicating that the diametral strength of the post in this direction was surpassed. Thus, the load at fracture was limited by the diametral strength of the post itself, implying that any differences between the surface treatments were obscured by premature fracture of the post. In the shear bond strength test, the force applied to the bonded cylinder of resin composite was transferred to the post in such a way that stress components were formed not only perpendicularly but also parallel to the orientation of the fibers. Since the fiber post is much stronger in the parallel direction, the force at fracture of the bond was not limited by the strength of the post. Thus, the DTS method did not allow determination of the effect of surface treatment on the bonding of resin cement to the glass fiber post. Sandblasting, including Cojet treatment, resulted in perceptible volume loss of the glass fiber posts. As this observation may be seen as having a weakening effect, sandblasting and Cojet treatment may not be recommended for glass fiber posts.

Whereas sandblasting and hydrofluoric acid etching of subsequently embedded ParaPost XH had a positive

Table 7: Results of the Three-way Factorial ANOVA, Dependent Variable: Diametral Tensile Strength						
Effect	SS	df	ms	F	р	
Post	4593.57	2	2296.79	88.51	0.0001	
Resin cement	12.58	1	12.58	0.48	0.4866	
Surface treatment	3661.34	5	732.27	28.22	0.0001	
Post * Resin cement	1123.68	2	561.84	21.65	0.0001	
Post * Surface treatment	2013.16	10	201.32	7.76	0.0001	
Resin cement * Surface treatment	165.11	5	33.02	1.27	0.2747	
Post * Resin cement * Surface treatment	379.76	10	37.98	1.46	0.1504	
Error	11236.48	433	25.95	-	-	

effect on the DTS of ParaPost Cement, these surface treatments had no effect on the DTS of either of the two resin cements in the case of Cerapost. The fact that sandblasting of subsequently embedded Cerapost did not significantly improve the DTS of the resin cements is in line with a study by Kern and Wegner (1998) that showed a significant reduction in bond strength of a Bis-GMA-based resin cement to zirconia, which had been sandblasted with alumina particles of 110 µm. The authors suggested that the decreased bond strength was due to the production of limited or minimal undercuts compared to undercuts produced when sandblasting metals. The roughening effect of sandblasting depends on the size of the alumina particles and the mechanical properties of the material. The previous study by Sahafi and others (2003) showed a significant improvement in the bond strength of resin cements to the sandblasted core of ground Cerapost. The lack of effect of sandblasting in the current study may be explained by the fact that sandblasting was performed on the original surface of the Cerapost and not on the core of the post, the original surface being rougher than the ground surface.

By applying hydrofluoric acid to ceramics, selective etching of certain phases of the ceramic is obtained, and hydrofluoric acid etching has been found to improve the bond strength of resin to conventional silicate-based ceramics (Wolf & others, 1993; Stangel, Nathanson & Hsu, 1987). Hydrofluoric acid etching of subsequently embedded Cerapost had no effect on the DTS of resin cements. This finding is in accordance with studies by Sahafi and others (2003), Dérand and Dérand (2000) and Kern and Wegner (1998), which demonstrated no effect or even a negative effect of hydrofluoric acid etching on the bond strength of resin cement to zirconia ceramics.

Cojet treatment of subsequently embedded Cerapost was an effective method for improving the DTS of resin cements. The increased bonding of resin cement to silicate-coated (Cojet-treated) zirconia has also been reported in studies by Sahafi and others (2003) and Kern and Wegner (1998). Since the combination of sandblasting and silane coating of subsequently embedded posts for both resin cements resulted in the same DTS as sandblasting alone, application of silane had no additive effect. The inefficiency of silane treatment of subsequently embedded Cerapost on bonding may be explained by a very weak or absent bond of the functional group of silane (silanol) to non-silicate based materials (Kern & Wegner, 1998; Söderholm & Shang, 1993).

#### **CONCLUSIONS**

The hypothesis of this study regarding titanium alloy and zirconia posts was confirmed in that the results showed that the bonding of resin cement to posts was affected by the surface treatment of the post, the post material and the type of resin cement. Based on DTS measurements, the following conclusions can be drawn:

- 1. Generally, ParaPost Cement bonded better to titanium alloy posts (ParaPost XH) and glass fiber posts (ParaPost Fiber White), while Panavia F bonded better to zirconia posts (Cerapost).
- 2. This method did not allow for the determination of the effect of surface treatment on the bonding of resin cement to glass fiber posts.
- 3. For the titanium alloy post, several surface treatments improved bonding of both resin cements.
- 4. For the zirconia post, Cojet treatment improved the bonding of both resin cements, while sandblasting followed by silane application improved the bonding of Panavia F.

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# Tensile Bond Strength of Composite to Air-abraded Dentin

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#### Clinical Relevance

Acid etching is necessary for bonding composite to air-abraded dentin.

#### **SUMMARY**

This study evaluated the influence of air abrasive treatment of dentin surfaces on the tensile bond strength between dentin and two different composite-adhesive-systems (Scotchbond Multi-Purpose/Z100 and OptiBond FL/Herculite XR). The crowns of 200 maxillary central incisors were embedded in resin and then ground to expose a dentin surface 5 mm in diameter. The surfaces were etched or abraded by using a KCP 1000 device with different treatment conditions. Adhesive systems were applied according to the

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#### **INTRODUCTION**

Since the beginning of the 1990s, the air abrasive technique has again become the subject of dental research. This technique was invented in the 1940s (Black, 1945) and has been further developed and adapted to today's demands.

It has been speculated that this technique might have some advantages compared to rotary instruments; for example, it causes no vibrations and, therefore, is less painful to the patient. Moreover, this technique helps to reduce the time required for patient treatment (Christensen, 1998; Goldstein & Parkins, 1994; Goldstein & Parkins, 1995). The air abrasive treatment

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changes enamel and dentin surfaces in a way (Geitel & others, 2000; Gwinnett & Berry, 1996; Jahn & others, 1999; Los & Barkmeier, 1994) that can lead to an improvement of bond strength to tooth structures. Therefore, it has been assumed that even acid etching might become unnecessary (Keen, von Fraunhofer & Parkins, 1994; Manhart & others, 1999). However, according to other studies, acid etching is superior to the abrasive technique (Eakle, Wong & Huang, 1995; Rinaudo, Cochran & Moore, 1997; Roeder & others, 1995; van Waveren Hogervorst, Feilzer & Prahl Andersen, 2000). Roeder and others (1995) found higher tensile bond strengths between dentin and the composite Herculite when dentin was pre-treated with etching as opposed to air abrasion. Rinaudo and others (1997) achieved similar results by applying Scotchbond Multi-Purpose or One-Step and the resin composite Herculite by means of shear bond strength tests. However, Manhart and others (1999) reported that pretreatment with air abrasion caused an increase in shear bond strengths when compared to untreated



Figure 1. Embedded crown with exposed dentin area.

specimens.
Additional acid etching and the application of adhesives on dry versus wet dentin had no influence on bond strength values.

Coli and others (1999) could not find significant differences between etching

vs air abrasion when using All Bond2/Z100. Even the negative control (untreated dentin) did not differ significantly from the test groups.

This study examined the influence of air abrasive treatment on dentin surfaces with respect to tensile bond strength using two different composites.

#### **METHODS AND MATERIALS**

Two hundred extracted, caries-free maxillary central incisors were selected for this *in vitro* study. The periodontal ligament and other debris were removed. The roots were then separated from the crowns and discarded. The crowns were embedded in acrylic resin (Paladur, Kulzer GmbH, Wehrheim, Germany) using Teflon forms. Buccal enamel was removed by wet grinding with silicon carbide paper (600 grit, Struers, Copenhagen, Denmark) until an area of dentin approximately 5 mm in diameter was exposed to accommodate the restorative material (Figure 1).

The dentin surfaces were conditioned by acid etching (Scotchbond Phosphoric acid etchant, 3M/ESPE, St Paul, MN, USA) or by the air abrasive technique (KCP1000, American Dental Technologies Inc, Corpus Christi, TX, USA) with different treatment conditions (Figures 2 and 3).

For air abrasion, the nozzle of the KCP device was held perpendicular to the dentin surface at a distance of 2 mm. It was moved in two directions in serpent form for five seconds each to achieve a surface free of grooves and pits.

Subsequently, the adhesive systems were applied to the conditioned dentin surface (Scotchbond Multi-Purpose and Z100, 3M/ESPE, and OptiBond FL and Herculite XR, Kerr, Karlsruhe, Germany, respectively) according to the manufacturer's instructions. Then,

58µ 88psi (580×) 108µm

Figure 2. Dentin after acid etching.

Dentin 15s geätzt 500x

Figure 3. Dentin after air abrading (arrows: opened tubules).

cylinders of the corresponding resin composite were created with a bonding surface 4-mm in diameter using a custom made split mold. The resin composite was applied in 1-mm increments and light-cured for 40 seconds with a plasma light-curing unit (ADT1000, American Dental Technologies). A screw was then polymerized perpendicular to the dentinal surface into the resin using a special fixing device (Figure 4). With the screw, the specimens could be mounted to the testing machine for the tensile bond strength measurements.

Tensile bond strength was determined using an Instron Dynamic Testing System with a 5 kN load cell (Instron Corporation, Canton, MA, USA) at a crosshead speed of 0.5 mm/minute and a sampling rate of 0.02 kHz (Figure 5). The failure sites of the debonded specimens were examined under a light microscope (Carl-Zeiss-Jena, Jena, Germany) at 12x magnification. The data were analyzed with the Program SPSS using the Kruskal-Wallis-test and the Kolmogoroff-Smirnoff test. In Figure 6, an overview of the method is given.

### **RESULTS**

Irrespective of the applied treatment parameters in the groups of the air abraded specimens, the tensile bond strength of Scotchbond Multi-Purpose/Z100 and OptiBond FL/Herculite XR was significantly lower than in the acid etched groups (Figures 7 and 8).

The size of the abrasive particles had no significant impact on the tensile strength of the bonded composites. A higher particle pressure resulted in significantly lower tensile bond strength when a particle size of  $50~\mu m$  was used during abrasion and when the combination of Scotchbond Multi-Purpose and Z100 was applied.

The tensile bond strength of OptiBond FL/Herculite XR to dentin was significantly higher than to Scotchbond Multi-Purpose/Z100, independent of the conditioning method.

### **Investigation of the Failure Sites**

Adhesive fractures between dentin and composite and mixed fractures (a mixture of adhesive fractures between

dentin and composite and cohesive fractures within the composite) were found more often than with cohesive fractures within the resin. Cohesive fractures within the dentin did not occur. There were more adhesive fractures after air abrasion than after acid etching (Figure 9).

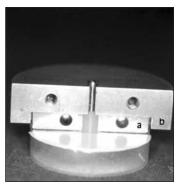


Figure 4. Specimen with a shared form (a) and a fixing device (b) for the screw that was polymerized into the resin.

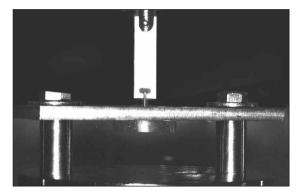


Figure 5. Specimen mounted on the Instron testing machine.

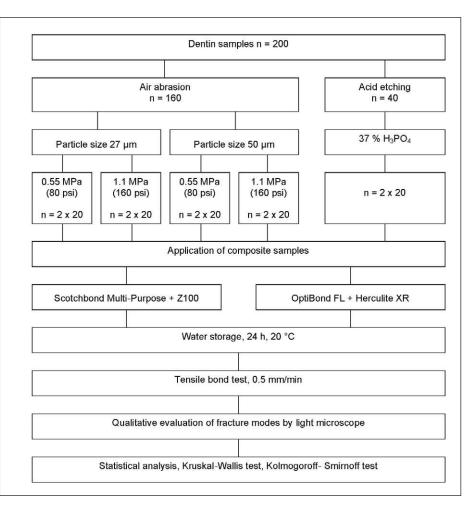


Figure 6. Overview of the method (1 psi corresponds to 6.9 x 103 MPa).

### DISCUSSION

The air pressure and particle size used in this study were selected because of the results of preliminary investigations (Geitel & others, 2000; Jahn & others, 1999).

Both composite-adhesive combinations resulted in higher tensile bond strengths between dentin and composite when the dentin was pre-treated with etching as opposed to air abrasion. This is in accordance with investigations by Roeder and others (1995) and Rinaudo and others (1997). Nikaido and others (1996) did not find significantly different tensile bond strength values between the treatment of dentin with 50 µm-particles at a pressure of 41.8 psi (=0.29 MPa) or with 600 grit-paper as a control. It is conceivable that low air pressure induced insufficient roughness of the dentin surface. In this investigation, higher pressure had significant relevance in one group, which resulted in a negative impact on the tensile strength. This is in disagreement with Manhart and others (1999), who achieved significantly higher bond strength values with 160 psi (=1.1 MPa) pressure compared to 120 psi (=0.83 MPa).

The particle size used in this study seems to have no influence on bond strength. Roeder and others (1995) and Manhart and others (1999) compared the particle sizes of  $27~\mu m$  and  $50~\mu m$  with regard to their influence on bond strength and found no significant differences either. This may be due to a lack of purity in the powder because both kinds of powders contain particles between 1.6 and 173.8  $\mu m$  and only the amount of particles and the mass distributions differ as the preliminary investigations have shown.

In several studies by various authors, it is often insufficiently explained how air abrasion was performed (Coli & others, 1999; Hannig & Femerling, 1998; Los & Barkmeier, 1994; Nikaido & others, 1996; Rinaudo & others, 1997; Roeder & others, 1995; van

Waveren Hogervorst & others, 2000). For example, often, in regard to information about nozzle distance and angulation, movement of the nozzle or duration of treatment is missing and it is not clear whether the abraded surface is planar and rough or if it has pits or grooves. It has not yet been investigated whether macroretentions, pits, grooves or a uniform roughness of the dentin surface caused by air abrasion have an influence on the bond strength between dentin and composite (Santos Pinto & others, 2001).

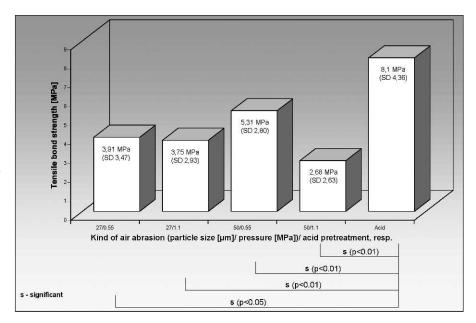


Figure 7. Tensile bond strength values of Scotchbond Multi-Purpose and Z100 dependent on different treatment conditions.

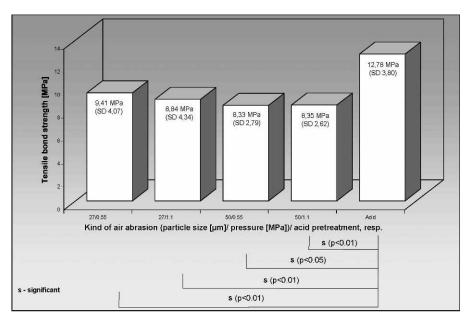


Figure 8. Tensile bond strength values of OptiBond FL and Herculite XR dependent on different treatment conditions.

Los and Barkmeier (1994) examined the shear bond strength values between dentin and composite when six different adhesive systems were applied with or without air abrasion. Significantly different bond strength values have occurred, depending on the adhesive system used, but no differences between the airabraded groups and corresponding control groups were detected. It can be concluded that the choice of adhesive system has more influence on the bond between dentin and composite than the method of pre-conditioning.

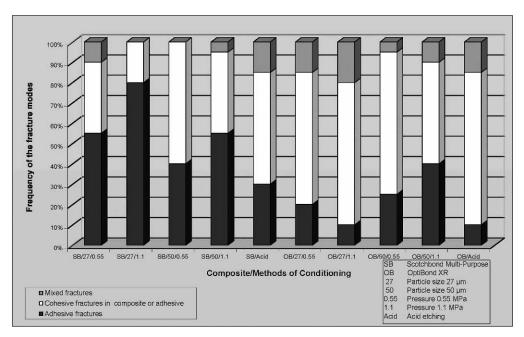


Figure 8. Tensile bond strength values of OptiBond FL and Herculite XR dependent on different treatment conditions

Certainly, air abrasion does not contribute to an increase in bond strength.

In this investigation, no cohesive fractures occurred in dentin after tensile strength testing, instead, predominantly mixed and adhesive fractures were found. Los and Barkmeier (1994) observed in their Scotchbond Multi-Purpose groups 70% cohesive fractures within dentin of the abraded group and 30% within the control group (ground but not etched dentin). However, Manhart and others (1999) found cohesive fractures only in the air-abraded groups and 80-100% adhesive fractures in untreated and etched specimens, respectively. Nikaido and others (1996) detected fractures within the bonding resin as well, which they described as cohesive fractures. The differences might be attributed to the different tests used (tensile and shear bond strength, respectively) or to different definitions of the fracture modes.

Margin quality investigations by other authors also confirmed that dentin acid etching is essential for bonding to composite (Eakle & others, 1995; Hannig & Femerling, 1998; von Fraunhofer & others, 2000).

Even though the comparability of different studies is difficult due to different study designs (tensile or shear bond strength) and the application of different adhesive systems and resin composites, it may be concluded from most of the results that the exclusive conditioning by air abrasion is inferior to acid etching. A combination of both techniques could possibly lead to an increase in bond strength between dentin and composite (Roeder & others, 1995).

### CONCLUSIONS

Pre-treatment of the dentin surface with acid etching showed higher tensile bond strengths than pre-treatment with air abrasion. The air abrasive technique is not an alternative to conventional acid treatment. Therefore, additional acid etching is recommended when the air abrasive technique is applied.

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### Effect of an "All-in-one" Adhesive on Pulp Blood Vessels: A Vitalmicroscopic Study of Rat's Teeth

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### **Clinical Relevance**

All-in-one adhesive applied on a very thin layer of dentin may influence pulpal circulation during clinical restorative procedures.

### **SUMMARY**

The newly developed self-etching, self-priming all-in-one adhesives are appealing to clinicians because they are simple and efficient to use. These single-application bonding systems contain chemically active compounds that can alter pulpal blood circulation when applied to deep dentin surfaces. Since adequate microcirculation and oxygenation are the basic requirements for tissue survival, the aim of this study was to investigate the immediate vascular effect of a new self-etching adhesive, Prompt L-Pop/composite and compomer version/(test group).

The technique of vitalmicroscopy was used to record the changes in vessel diameter of the first lower incisor of 20 (10-10 in each group) male Sprague-Dawley rats (weighing 315 ± 74/SE/g) prior to, and at 5, 15, 30 and 60 minutes after the investigated materials were applied to dentin. The application of saline served as the untreated control.

The systemic arterial pressure remained unchanged throughout the experiments both in the control (110  $\pm$  8 mmHg) and in test animals (114  $\pm$  4 mmHg). In control rats, the diameter of the vessels was stable during the experiment. In the presence of Prompt L-Pop, the diameters decreased significantly during the experimental period (5 minutes: -11.15  $\pm$  5.03%; 15 minutes: -14.66  $\pm$  7.71%; 30 minutes: -13.35  $\pm$  5.79%; 60 minutes: -11.82  $\pm$  5.63% p<0.05 in each cases). In this group, stasis developed in pulpal circulation was 1 out of 10 rats.

The results from the rat model used in this study suggest that Prompt L-Pop may result in compromised pulpal microcirculation.

### INTRODUCTION

In dental practice, there is an increasing demand for aesthetic restorations that leads to the extensive use of adhesive dental techniques. Many adhesive systems have been developed since Buonocore (1955) first

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described the acid etching technique on enamel in 1955. The newly introduced systems—all-in-one adhesives—can fulfill two duties simultaneously: they etch and infiltrate resin monomers into the dentin layer (Frey, 2000). Since the introduction of this material, numerous *in-vitro* and cell-culture examinations were conducted (Frankenberger & others, 2001; Ernst & others, 2002; Bouillaguet & others, 2001; Pashley & Tay, 2001; Imazato & others, 2000). Some studies show that self-etching primers on enamel are a useful alternative to the multi-step, multi-bottle system (Rosa & Perdigão, 2000; Hannig, Reinhardt & Bott, 1999). However, in an investigation by Hara and others (1999), bond strength to enamel is lower than the values of other adhesive techniques. Prompt L-Pop has been reported to result in dentin bond strengths and marginal sealing/integrity that is lower than those obtained with total-etch multi-step adhesives (Frankenberger & others, 2001; Ernst & others, 2002; Bouillaguet & others, 2001). Imazato and others (2000) showed the cytotoxic effect of composite restorations employing self-etching primers on human cells in vitro.

Similar to previous bonding-systems, all-in-one adhesives cannot effectively eliminate the difficulties originating from the inhomogeneous tubular structure and intrinsic wetness of dentin (Frankenberger & others, 2001; Ernst & others, 2002; Bouillaguet & others, 2001). The polymerization shrinkage of composite and, consequently, the development of microleakage, causes severe postoperative complications such as sensitivity, recurrent caries, pulpal inflammation, degradation or necrosis (Bergenholtz & others, 1982).

Restorative dentistry should endeavor to minimize

complex traumatic injuries of pulp during clinical procedures. Thus, the manufacturers make tremendous efforts to study the long-term (weeks to months) reactions of pulp in response to restorative materials (Avery, 1975; Stanley, Bowen & Folio, 1979). Experiments investigating the early, acute, in vivo (minutes to hours) responses so far are inconclusive. The rate of inward diffusion of some chemically active materials applied to exposed dentin is higher in vitro than vivo(Vongsavan inMatthews, 1991; Vongsavan, Matthews & Matthews, 2000). The outward flow of dentinal fluid through the tubules reduces the penetration of some materials. This defense mechanism of the living, healthy dentin-pulp complex decreases the concentration and toxicity of dental materials near the pulp (Pashley & Matthews, 1993). Thus, to get reliable information about the biocompatibility of chemicals, *in vivo* investigations should be performed.

Prompt L-Pop applied on the exposed dentin surface contains chemically-active compounds that can alter pulpal blood circulation due to their close proximity to microvessels. Since adequate microcirculation and oxygenation are basic requirements for tissue survival, this study examined the acute *in vivo* effect of an all-inone adhesive on rat pulpal microcirculation with the vitalmicroscopic technique. The result of this investigation can provide additional information on the biocompatibility of this material.

### **METHODS AND MATERIALS**

### **Animal Preparation**

All observations were made on the mandibular left incisor of two groups (test and control) of 10 male Sprague-Dawley rats (weighing  $315 \pm 74$  /SE/g) with the vitalmicroscopic technique (Figure 1). The rats were anaesthetized with pentobarbitone sodium (Nembutal 35 mg/kg, ip, supplemented as required). Breathing was supported by tracheal cannulation. The right femoral artery was cannulated with a heparinized (1500 IU/ml) polyethylene catheter and connected to an electromanometer for measurement and registration of the systemic blood pressure. The body temperature was kept constant at 37°C using a heating lamp (150 W). The skin was removed from the lower jaw of the rat. From the left part of the lower jaw, the mucous mem-

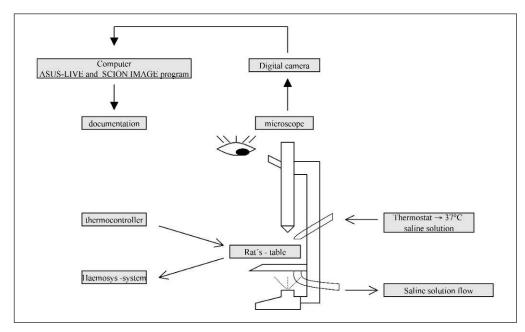


Figure 1. Experimental set-up of vitalmicroscopy.

brane was retracted and the jaws were separated by transsecting the connective ligaments between the left and right halves of the lower jaws. The left part of the lower jaw was fixed with a circular polyester strip to position the incisor for further preparation. With the teeth firmly secured, the mesial and distal surfaces of the incisor and part of the alveolar bone were ground away with a dental bur. The excavation was made on the mesial and distal surfaces from the labial to the lingual margins and from the alveolar bone to the incisal edge. Grinding was done at about 15,000 rpm with a diamond fissure bar and minimally applied pressure. The tooth was sprayed with physiological saline solution (37°C) to avoid heat-damage and desiccation of the tooth during preparation. After removing the enamel and alveolar bone, the authors continued the preparation under a dissecting binocular microscope (1.6 x 6.3) Nikon, Japan) until the pulpal vessels became clearly visible through the dentinal wall and only a very thin plate of dentin covered the intact pulp tissue.

After preparation, the rat was placed on its right side on a suitable animal board attached to the stage of a Nikon stereo light microscope  $(10x0.255 \pm 1x16)$ equipped with a digital camera (Nikon Coolpix 990) connected to a computer (IBM-compatible). The prepared tooth was kept wet with saline solution (37°C) (by means of a peristaltic pump) so as to guarantee a standard, permanent temperature. After a one-hour equilibration time, a suitable arteriole was chosen for ease of measurement of its inner diameter. The vessel diameter was measured before (baseline value) and after applying the test materials on the monitor by means of image analyzing software (Scion Image, Scion Corporation, Frederick, Maryland, USA). The placement of test materials on the prepared surface was apical from the observed pulp-area so that the investigated materials would not interfere with visualization of the microvessels.

Test Group (10 animals): With a small piece of absorbent paper, the tooth was dried only until it remained "visibly moist." Then, the test material—Prompt L-Pop/compomer and composit version/ (ESPE Dental AG, D-82229 Seefeld, Germany)—was applied to the prepared surface with an applicator tip according to the manufacturer's instructions. The layer was dried with a gentle air steam and cured for 10 seconds with a halogen lamp (750 mW/cm²) at a 1 mm distance from the prepared surface (Astralis 3, Vivadent Ets, FL-9494 Schaan/Liechtenstein).

Control Group: The dentin surface was dried and an application of physiologic saline (0.9% NaCl) served as the non-treated control. The light curing steps were missed in that group.

The diameter changes that were compared to their own baseline diameters were calculated at 5, 15, 30 and

60 minutes after application of the test materials or saline (control). For statistical analysis two-way ANOVA was applied using treatment and time as factors. Normal distribution was tested by Kolmogorov-Smirnov test. A significant level less than or equal to 0.05 was chosen to indicate statistical significance. After the measurements were recorded, the rats were killed by injection of pentobarbitone.

### **RESULTS**

The systemic arterial pressure remained unchanged throughout the experiments both in the control ( $110 \pm 8$  mmHg) and in the test animals (114 + 4 mmHg).

In the control groups, no significant changes were observed in the mean vessel diameter. The average of the control measurements at different time periods was used for comparison with the experimental data. The baseline value of the arteriole diameter was  $49 \pm 5.46$  µm.

In the presence of Prompt L-Pop (Figure 2), the vessel diameter was decreased (5 minutes: -11.15  $\pm$  5.03%; 15 minutes: -14.66  $\pm$  7.71%; 30 minutes: -13.35  $\pm$  5.79%; 60 minutes: -11.82  $\pm$  5.63%). These changes were significant in 5, 15, 30 and 60 minutes when compared to the control values (ANOVA, p<0.05). In one case, stasis developed in the pulpal circulation 30 minutes after applying Prompt L-Pop and continued until the end of the experiment.

### **DISCUSSION**

According to the American Dental Association (ADA) and the Fédération Dentaire Internationale (FDI) specification and certification programs, investigations are focused on the physical and chemical properties of dental materials (ISO/TC, 1992). In view of increasing concern for safety and biocompatibility, in this study, the authors investigated the acute vascular response of a newly developed all-in-one adhesive on a rat's incisor using the vitalmicroscopic technique. Prompt L-Pop compomer and composite version—is a solution consisting of methacrylate phosphates and water in a unique application unit. This self-etching primer conditions and primes enamel and dentin simultaneously without rinsing. This is a major step towards simplification of the clinical procedure in order to eliminate several bonding steps and errors during the adhesion of restorative materials (Swift, Perdigão & Heymann, 1995; Barkmeier, Shaffer & Gwinnett, 1986).

There is hardly any reference in the literature to *in vivo* testing of this all-in-one adhesive. The main difficulty is that pulp, which is enclosed by a rigid dentin wall, has specific haemodynamic parameters. *In vivo* examination of living dentin and pulp is a delicate procedure, because any injury affecting the dentin layer will trigger changes in the interstitial pressure within

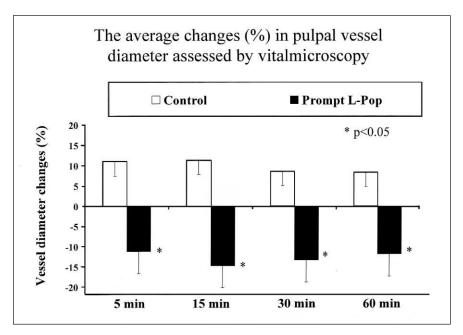


Figure 2. Each column represents a mean value (+ SE) expressed as % changes of the baseline vessel diameter.

the closed chamber of pulp and, consequently, the blood flow in it. Thus, *in vivo* knowledge can only be obtained from the intact, living dentin-pulp complex. When using the vital microscopic technique, the authors had to be fully aware of the above concerns.

After preparation, the initial reaction of pulp is the outward flow of dentinal fluid resulting from high interstitial (fluid) pressure (Mjör & Odont, 2001). In this vital microscopic study, a one-hour time interval was used prior to adhesive application to compensate for the changes caused in pulpal circulation after tooth preparation (Meyer, 1980). During the entire procedure—preparation and observation (approximately four hours), bacterial infection is reduced by continuous administration of sterile physiologic saline (37°C) on the exposed dentinal surface.

Prompt L-Pop was applied to the prepared tooth surface after a one hour equilibration time and the vascular reaction was recorded immediately, because the intensity of pulp response and the toxicity of this material is initially high and reduces with time (Hamid & Hume, 1996; Palasz, Gerzina & Hume, 1994; Stanley, 1994).

In the experimental conditions of this study, through a very thin dentin layer, Prompt L-Pop causes vasoconstriction and stasis. This result suggests that administration of this investigated material may alter pulpal microcirculation, resulting in an acute failure in one case. This chemically active and cytotoxic (Imazato & others, 2000) material, placed directly on the exposed dentin tubules, can penetrate into the pulp and, thus, can influence pulpal circulation.

Tay and Pashley (2001) compared several self-etching primers and found that Prompt L-Pop was the most aggressive, probably because it solubilizes smear plugs and penetrates deeper into the smear layer.

According to Mjör, there are three major factors that determine the nature of pulpal reactions, dentin permeability, remaining dentin thickness and the penetration-ability of the chemically active substance (Mjör & Odont, 2001; Heyeraas, Sveen & Mjör, 2001). This water-based, all-in-one adhesive has enhanced hydrophilic properties that guarantee better penetration into dentin (Pashley & others, 1995) and cause higher concentrations in dentinal fluid. The concentration of adhesive into pulp is further increased by rubbing this material into exposed dentin for 15 seconds prior to light curing according to the manufacturer's instructions.

The use of a high concentration adhesive combined with metacrylated phosphoric acid mono- and diesters of pH 1.0 (Pashley & Tay, 2001) causes more changes to the pulp complex microcirculation compared to 36% o-phosphoric acid for 15 seconds (Conditioner 36, DeTrey, Dentsply pH<0.5) with rinsing or NRC Non-Rinse Conditioner (DeTrey, Dentsply; contains itaconic acid, maleic acid pH≈1.1) for 20 seconds. Thirty-six percent o-phosphoric acid applied for 15 seconds with rinsing and Non-Rinse Conditioner for 20 seconds cause vasodilatation (Iványi & others, 2001). This vasodilatation is a protective function of the pulp. It must be noted that the experiment was carried out in extreme conditions and did not test the contingent effect of light curing on pulp tissue. Additional work should be done in this area.

### **CONCLUSIONS**

These results of this study suggest that applying Prompt-L-Pop may result in compromised dental pulp microcirculation of rat teeth.

### Acknowledgements

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### Finishing Tooth-Colored Restorations *In Vitro*: An Index of Surface Alteration and Finish-line Destruction

PR Schmidlin • TN Göhring

### Clinical Relevance

Among the instruments tested, diamond burs with a particle size of 8 µm and 40 fluted tungsten carbide finishing burs produced the smoothest surfaces while producing the least finishing-line destruction; the authors used these results to create a new index to evaluate and select appropriate instruments.

### **SUMMARY**

Many studies have evaluated the surface characteristics of finishing and polishing instruments on different restorative materials using two- and three-dimensional models based on mechanical and optical techniques. However, only limited data are available regarding the problem of marginal causing destruction. **Instruments** detectable surface alterations such as scratches or grooves may also cause marginal damage. This study aimed to correlate the smooth-surface polishing efficacy of different instruments with their potential for destructive effects on restoration margins and enamel finish lines. An index was created that will help to evaluate future pol-

A planar inlay system with a 100 µm wide defined gap was simulated in vitro. Pre-fabricated ceramic (n=40) and composite blocks (n=40) were connected to bovine enamel without luting material. After standardized pre-polishing, mean surface roughness and marginal quality were assessed using a profilometer and scanning electron microscopy (SEM). Enamel and restorative surfaces were colored, and subsequently prepared using one of 10 different finishing and polishing instruments. Four specimens per instrument and material were evaluated, resulting in eight interfaces for each test group. Surface roughness (Ra) and marginal quality (expressed as the percentage fracture-free margin) were measured and compared statistically using unpaired t-tests and two-way ANOVA, respectively. The level of significance was set at 0.05

Eight-micrometer diamond burs and 40-fluted tungsten carbide finishers produced smoother surfaces and less finishing-line destructions than the other instruments under evaluation. The index values developed will prove helpful in evaluating and selecting appropriate instruments.

ishing instruments and select suitable ones for different clinical situations.

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### INTRODUCTION

Tooth-colored adhesive restorations have to be polished to minimize possible gingival irritation, surface staining, patient discomfort and the development of secondary caries (Larato, 1972; Weitmann & Eames, 1975; Chan, Fuller & Hormati, 1980). After restoration placement, a slightly overfilled cavity or luting interface has to be finished and polished to provide an oxygen-inhibiting surface layer. Remnants of this layer present surfaces that can easily be contaminated (Yatabe & others, 1999) and are possibly cytotoxic (Tang & others, 1999).

Finishing has been defined as the gross contouring of a restoration to obtain the ideal anatomical shape. Polishing refers to the reduction of roughness and scratches created by finishing instruments (Yap, Lye & Sau, 1997). Much in vitro research has been performed on initial contouring, finishing and polishing to provide clinical concepts and protocols (Lutz, Setcos & Phillips, 1983; Schmid, Krejci & Lutz, 1991; Fehér & Mörmann, 1995; Kaplan & others, 1996; Hondrum & Fernandez, 1997; Jung, 1997). Ideally, a filler-rich, enamel-like, glossy polished surface should be achieved. In addition, a perfect marginal adaptation and seal is desired. Repolishing within a few days or weeks after placement of the restoration allows for optimal initial restoration quality, and long-term quality is ensured by consistent professional maintenance care (Goldstein, 1989; Goldstein & others, 1992; Baillod, Krejci & Lutz, 1994).

Although it has been suggested that all adhesive restorations require ongoing maintenance, including

periodic re-polishing (Goldstein, 1989), little information on these procedures is available (Miller, 1990). Dentists have a plethora of finishing instruments at their disposal, which are used for refinishing and polishing, eliminating excess material, worn out feathered edges and marginal imperfections, as well as marginal and superficial discolorations. However, polishing procedures themselves may lead to crevice formation and marginal disintegration. Enamel and/or restoration fractures may occur and result in white margins and subsequent discoloration (Fukushima, Setcos & Phillips, 1988; Bryant, Marzbani & Hodge, 1992). Microleakage may thus occur (Ferracane & Condon, 1999). Also, there is an increased risk of developing secondary caries (Han, Okamoto & Iwaku, 1992; Wilder & others, 2000).

Only limited data are available on surface roughness produced by finishing and/or polishing instruments and their influence on marginal quality. This study evaluated the ability of different finishing and polishing instruments to produce smooth surfaces and rank these instruments in relation to their destructive potential on restoration margins and enamel finish lines.

### **METHODS AND MATERIALS**

### **Specimen Preparation and Instrumentation**

A "worst case scenario" was devised by adapting and modifying Guzman, Moore and Andres (1997) in order to standardize *in vitro* evaluation and make a combined study of both surface alterations and marginal destructive potential produced by rotating, polishing and finishing burs in a single set-up.

Forty ceramic (Vita MK II, Vita Zahnfabrik, Bad Säckingen, Germany) and 40 fine hybrid composite (Tetric Ceram, Ivoclar Vivadent AG, Schaan, Liechtenstein) blocks were reduced to a height of 8 mm and finished to a uniform flat surface using 1000 grit silicon carbide abrasive paper (Struers, Copenhagen, Denmark). In addition, 80 clinical crowns from bovine second incisors were cut in half along the bucco-lingual axis, finished and leveled under water-cooling using silicon carbide abrasive paper to a final grade of 1000 grit. Restorative material blocks and bovine tooth sections (Figure 1) were separated using a 100 µm copper band (h&s Präzisionsfolien GmbH, Pirk, Germany) fixed with cyanoacrylate cement (Cementit, Merz & Benteli sa, Niederwangen, Switzerland). The latter were embedded with resin (Paladur, Heraeus Kulzer GmbH, Wehrheim, Germany) to form resin blocks, each with two standardized restorative interfaces. These specimens were glued to scanning electron microscopy (SEM) mounts (Balzers Union AG, Liechtenstein) using superglue (Renfert Sekundenkleber No 1733, Kaladent

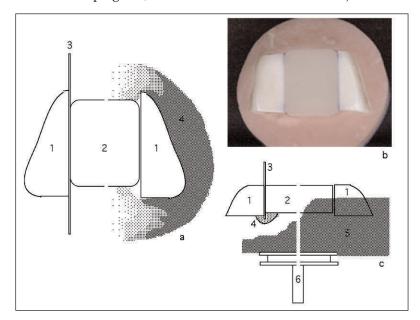


Figure 1 a) Test specimen with two half-bovine incisors (1) and a central restorative material block (2) b) Graphic cross-section: The tested materials were separated using a 100 µm thick copper band (3) that was fixed with a cyanoacrylate cement (4) and then embedded in resin (5) on an SEM mount (6).

AG, Zurich, Switzerland). The copper band was removed and the surface was again finished with silicon carbide abrasive papers at 1200 and 2400 grit.

Before instrumentation, enamel and restorative surfaces were colored with a waterproof felt pen (Lumocolor 317 permanent, Staedtler, Nürnberg, Germany). Ten different finishing and polishing systems were used on four specimens per instrument, resulting in eight interfaces for evaluation (Table 1). The same slow- and high-speed contra angles (Mikro Mega, Sirius, Besançon, France) were used with watercooling for all instruments except the felt wheel. The handpiece was used with a constantly moving repetitive stroking action along the longitudinal axis of the restorative interface. A standardized pressure of 2.5 N was applied and verified on an 8600 digital multimeter (Kontron Electronic AG, Zürich, Switzerland). The polishing process continued until the marked area was visibly free of color.

### **Surface Roughness Measurements**

The average surface roughness (Ra) of the standard machine pre-polished restorative materials was evaluated using five horizontal and five vertical measurements that covered an area 1 x 3 mm (Talysurf 50, Rank Taylor Hobson, Leicester, England). These measurements were repeated after each instrumentation. The results were compared statistically with baseline results (machine finishing procedure) using unpaired t-tests, with the level of significance set at 0.05.

### **Marginal Quality Assessment**

Before and after instrumentation, impressions were made using an addition-type polyvinylsiloxane (President light body, Coltène AG, Altstätten, Switzerland). After 24 hours at room temperature,

replicas were cast using epoxy resin 12666, (Stycast Emerson & Cunning, Westlo, Belgium) and gold-sputtered (SCD 030, Balzers). Tooth restoration interfaces were analyzed using SEM (Amray 1810T, 15 kV,  $12 \pm 2 \text{ mm}$ working distance; Amray Inc, Bedford, MA, USA). All samples were examined quantitatively for "perfect" (fracture-free gap) or "imperfect" margins (fracture of restorative material or fracture of enamel)

at 200x magnification. The results were compared statistically using two-way ANOVA and unpaired *t*-tests (StatView 4.02, Abacus Concepts, Berkeley, CA, USA).

### **Index**

In addition, linear regression was performed in order to reveal any possible relationship between surface roughness and marginal fractures. An index was introduced, emphasizing the above-mentioned criteria to a "synoptic" roughness and marginal destruction value in the following marginal finishing index (MFI):

$$MFI = \frac{Ra \times 100,000}{FfMe \times FfMr}$$

where FfMe designates the percentage of total length categorized as fracture-free enamel finish line, FfMr is the percentage of total length categorized as fracture-free restoration margin and Ra is the surface roughness. The factor 100,000 in the nominator is a correction to achieve lower, more plausible index values. For example, perfect margins without any fractures (FfMe and FfMr each 100%) and a baseline surface roughness of 0.1 would result in an index of 1.

### **RESULTS**

### **Surface Roughness Measurements**

The average surface roughness (Ra) after standardized machine polishing was  $0.21 \pm 0.08$  on ceramic and  $0.13 \pm 0.02$  on composite material.

After instrumentation, all instruments created significantly higher surface roughness on both materials compared with the baseline (Figure 2). Diamond burs with a particle size of 40, 25 and 15  $\mu$ m and green and white stones showed a significantly rougher surface on ceramic

Table 1		
Finishing Quality	Abrasive Instruments (LT = latch-type, operational speed-10,000 rpm) (FG = friction-grip, operational speed-20,000 rpm)	# of Measurements per Restorations/ Interface
Control	1900 AB Automed + abrasive paper disks, silicone- carbide, grit 1000, 1500 and 2500	80/8
	Diamond Burs (Intensiv, Grancia, Switzerland)	
Coarse	Contouring diamond bur, FG, diamond particle size 40 µm Finishing diamond bur, FG, diamond particle size 25 µm	80/8 80/8
Fine	Fine finishing bur, FG, diamond particle size 15 µm Ultrafine finishing bur, FG, diamond particle size 8 µm	80/8 80/8
	Tungsten Carbide Burs (Jota, Rüthi, Switzerland)	
Coarse	Tungsten carbide bur : 8 flutes, FG FineTungsten carbide bur : 40 flutes, FG	80/8 80/8
	Stones (Jota, Rüthi, Switzerland)	
Coarse	Green stone FineWhite stone	80/8 80/8
	Polishing Instruments	
Ultrafine	Nylon bristle brush/6 μm diamond paste (Hawe, Bioggio, Switzerand) UltrafineFelt wheel/1 μm diamond paste (Renfert, Singen, Germany)	80/8 80/8

compared to composite material. In contrast, the 40-fluted tungsten carbide bur showed higher Ra values on the composite blocks. In general, the mean surface roughness values after instrumentation corresponded to instrument textures.

### **Marginal Quality Assessment**

At baseline, the percentage of total length categorized as a "fracture-free" enamel finish line was  $97.34 \pm 0.59\%$  for ceramic and  $90.8 \pm 1.89\%$  for composite materials. At the restorative interface, fracture-free margins of  $96.91 \pm 1.10\%$  and  $96.98 \pm 5.14\%$  were achieved for the ceramic and composite group, respectively.

On enamel (Figure 3A), diamond burs and tungsten carbide burs showed no statistically significant differences between the two materials, however, variations in results shown as standard deviation composite restorations increased. All instruments caused more enamel alterations compared with the baseline except for the nylon-bristle brush in combination with a 6  $\mu$ m diamond polishing paste and the impregnated felt wheel. The 40  $\mu$ m diamond bur showed the most pronounced destructive pattern (p<0.05).

For the restoration interface (Figure 3B), ceramic showed significantly more fractures when coarse 40 and 25  $\mu$ m diamond burs were used compared with composite restoration interfaces (p<0.05). In addition, these two instruments produced more enamel fractures when compared with other instruments used on ceramic and they compared with the baseline. Nevertheless, compared to enamel finish lines, marginal alterations at the restoration sites were less evident.

### **SEM Assessment**

Figure 3

SEM microphotographs at 200x revealed surface roughness and enamel fractures produced by some instruments on composite (Figure 4A) and ceramic (Figure 4B) specimens. Typical striations were observed in the direction of the working action for diamond burs and white

stone. Smooth surfaces were seen when the 8-µm diamond bur and 40-fluted tungsten carbide bur were used.

### Index

The relationship between marginal quality (y-axis) and mean surface roughness (x-axis) is illustrated for ceramic (Figure 5A) and composite specimens (Figure 5B). Linear regression showed a statistically significant correlation ( $p \le 0.01$ ) between the two tested parameters at all interfaces with r²-values ranging from 0.601 to 0.743.

On ceramic specimens, regressions between the enamel and restoration interface showed comparable gradients and arrangements for the evaluated mean values (Figure 5A). On composite specimens, instruments produced smoother surfaces. A tendency toward a more destructive potential at the enamel interface was evident, indicated by a steeper regression line (Figure 5B).

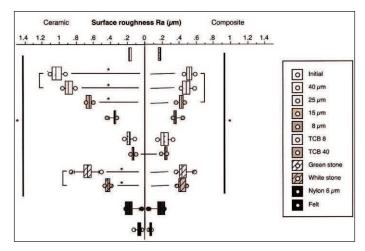
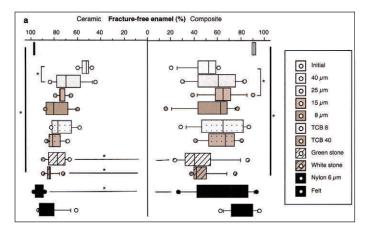


Figure 2. Mean surface roughness on ceramic and composite restorations. Instruments under the bold line showed statistically significant higher Ravalues compared with baseline (\*p≤ 0.05).



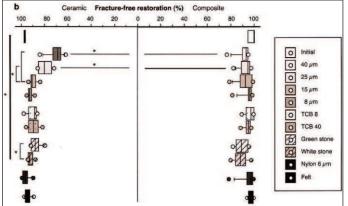


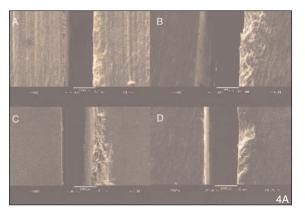
Figure 3A and B: Marginal quality assessment (\*p≤ 0.05). Instruments under the bold line produced a significantly decreased percentage of fracture-free margins compared with baseline. 3A) Fracture-free enamel interface on ceramic and composite specimens. 3B) Fracture-free restoration interface.

There was an attempt to create a measure to assess the overall restoration quality by creating a marginal finishing index (MFI, see Materials and Methods). Based on the correlation between surface roughness and marginal quality, instruments with higher destruction potentials exhibited higher index values (Figure 6). In accordance with SEM observations, it was determined that surface values lower than 0.5 and fracture-free margins not lower than 75% were tolerable, which corresponded to an MFI of 10.

### DISCUSSION

The long-term aesthetic quality of restorations is hampered by a number of processes. Water sorption can induce color changes in restorative materials. Marginal micro-cracks can result in marginal discolorations and

Figure 4



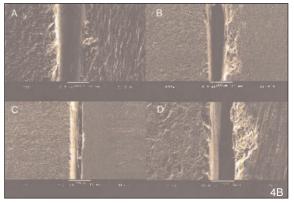
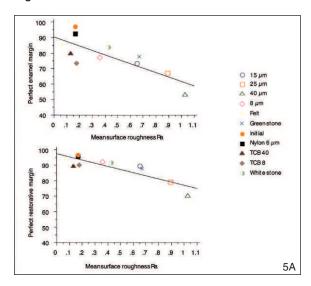


Figure 4A and 4B: Typical SEM aspects of composite (A) and ceramic (B) specimens show the outcomes produced by the following instruments: A, 40 µm diamond bur; B, 8 µm diamond bur; C, 40-fluted tungsten carbide bur and D, white stone. The 100-µm gap divides restorative material (left) from enamel (right). Enamel fractures and striations can be noted (200x).

Figure 5



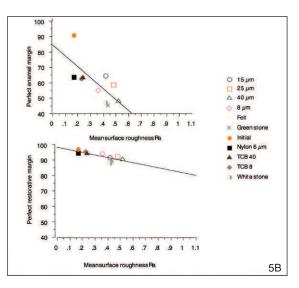


Figure 5A: Linear regression performed for ceramic specimens (mean values). Figure 5B: Linear regression performed for composite specimens (mean values).

increase the risk of developing secondary caries. Increasing surface roughness enhances plaque accumulation and staining. Oral fluids and patient habits related to smoking, eating and/or drinking influence the quality and durability of composite restorations (Roulet & Walti, 1984). Therefore, it is not sufficient to temporarily re-establish secondary oral health. Dentists are responsible for the patient's care and need to discern and avoid new caries and periodontal disease and ensure the longterm quality of tooth restorations. The longevity of the latter varies significantly (Downer & others, 1999), and the reported mean survival time for resin restorations is five years or less. The primary reasons for replacement are tooth or restoration fractures and secondary caries diagnosed visually or on radiographs. A high quality restoration at placement does not guarantee longevity;

> it merely improves the chances of complying with professional standards, despite a progressive loss of quality.

> For surface roughness, the results of this study were comparable to previous investigations. Berastegui and others (1992)recommended tungsten bide burs to finish composite restorations. Jung (1997)tested diamond burs and tungsten carbide burs for the production of surface roughness and efficiency. Finishing with diamonds was characterized by high cutting efficiency and relatively rough corresponding surfaces, whereas, tungsten carbide burs led

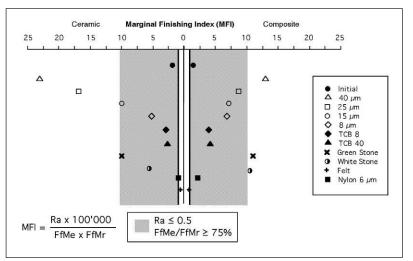


Figure 6. Marginal Finishing Index (MFI). A fracture-free margin of 100% in combination with a baseline surface roughness of 0.1 would result in an index value of 1 (bold line). More finish line destruction and an increase in the mean surface roughness, therefore, results in an increase in the index, indicating greater destructive potential. The dotted area represents a tolerable index range.

to smoother surfaces. Joniot and others (2000) stated that finishing with burs alone left a rougher surface and therefore recommended using subsequent finishing systems to improve surface quality. Neme and others (2002) stated that prophylactic polishing protocols might be used to restore a smooth surface. From the results of this study, the authors suggest that even fine and ultra-fine instruments may negatively influence the integrity of enamel and, to a lesser extent, restoration margins. Therefore, every additional step in a polishing sequence may increase the risk of further finishline destruction. It was evident that the enamel on ceramic specimens was better protected from the destructive influence of finishing instruments compared with composite restorations. Using the marginal finishing index (MFI), 8 µm finishing and 40-fluted tungsten carbide burs were concluded to be the best suited among the instruments assessed in this study for finishing and polishing the tested materials.

More *in vitro* tests are needed to investigate the influence on surface and margin characteristics in order to improve the future management of adhesive restorations after placement and during maintenance. The use of finishing and polishing instruments and the application sequence on adhesive restorations should be minimized to avoid marginal destruction, while guaranteeing good surface performance.

### CONCLUSIONS

This study evaluated the ability of different finishing and polishing instruments to produce smooth surfaces and rank them in relation to their destructive potential on restoration margins and enamel finishing lines. Based on the results of this study, an index was created that will help to evaluate future polishing instruments and select a suitable polishing instrument for different clinical situations. Diamond burs with a particle size of 8 µm and tungsten carbide finishers produced smoother surfaces and less finishing-line destruction. In composite—enamel gaps, more enamel fractures were noted than in ceramic—enamel gaps. In ceramic and composite specimens, a relationship between surface alterations expressed as mean surface roughness and marginal quality was noted.

The MFI index may be helpful in evaluating and selecting appropriate instruments. This study made an effort to implement two important factors that influence the performance and longevity of adhesive restorations. Additional research is required to evaluate the influence of luting material and gap width to simulate clinical situations that apply different treatment sequences and physical treatments and confirm the results in clinical trials.

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# Surface Finish of Resin-Modified and Highly Viscous Glass Ionomer Cements Produced by New One-step Systems

AUJ Yap • JJ Ng SH Yap • CK Teo

### Clinical Relevance

The effectiveness of one-step finishing/polishing systems is product dependent. While Pogo and Sof-Lex Brush produced superior or comparable surface finish to two-step rubber abrasive and graded abrasive disk systems, the surface finish obtained with One Gloss was significantly rougher.

### **SUMMARY**

This study investigated the surface finish of resin-modified (Fuji II LC, GC) and highly viscous (Fuji IX GP Fast, GC) glass ionomer cements after treatment with three one-step finishing/polishing systems (One-Gloss [OG], Shofu; Pogo [PG], Dentsply; Sof-Lex Brush [SB], 3M-ESPE). The surface roughness obtained was compared to that using a matrix strip [MS], a two-step rubber abrasive (CompoSite [CS], Shofu) and a graded abrasive disk (Super Snap [SS], Shofu) system. Eight specimens (3-mm long x 3-mm wide x 2-mm deep) of each material were made for the various

treatment groups. With the exception of the MS group, all groups were roughened with 320 grit grinding paper using a lapping device prior to finishing/polishing with the different systems. The mean surface roughness (µm) was measured with a profilometer. Data was subjected to ANOVA/Scheffe's tests at significance level 0.05. Mean Ra ranged from 0.13 to 1.04 um for Fuji II LC and 0.14 to 0.81 for Fuji IX GP. For both materials, the smoothest surface was obtained with MS and the roughest with OG. Depending on the materials, the surface finish produced by PG and SB was superior or comparable to that obtained with CS and SS. The effectiveness of one-step systems, when used to finish/polish resin-modified and highly viscous glass ionomer cements, is product dependent.

### INTRODUCTION

The favorable adhesive and fluoride-releasing properties of glass ionomer cements (GICs) have led to their widespread use as luting, lining and restorative materials. Resin-modified and highly viscous GICs were developed to overcome early moisture sensitivity and low mechanical properties associated with conventional materials. In resin-modified GICs, a second light-initiated curing process supplements the fundamental acid-

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base curing reaction. In their simplest form, they are glass ionomers with a small quantity of resin component, such as HEMA (hydroxytheyl methacrylate) or BisGMA (Bisphenol A-glycidyl methacrylate). More complex materials have been developed by modifying polyacid with side-chains that can be polymerized by light curing. Highly viscous GICs were designed as an alternative to amalgam for posterior preventive restorations (Saito, Tosaki & Hirota, 1999). These high powder: liquid ratio cements provide a condensable feel and are particularly useful for restoring Class V cavities, where access/isolation are compromised and aesthetics is of secondary importance, the Atraumatic Restorative Technique (ART) introduced by the World Health Organization for use in developing countries (Frencken & others, 1996) and the open-sandwich technique. The latter, also known as the "composite-laminated GIC" restoration, was introduced to counter sealing and stress problems associated in Class II cavities (Woolford, 1993). In these restorations, GIC is placed so that the cement covers most or all the exposed dentin and extends to the periphery of the proximal box to form the cervical seal.

The aesthetics and longevity of tooth-colored restorative materials, including resin-modified and highly viscous GICs, are dependent on the quality of surface finish, as the presence of surface irregularities may influence appearance, surface discoloration, plaque retention and gingival irritation (Shintani & others, 1985; Dunkin & Chambers, 1983; Chan, Fuller & Hormati, 1980; Weitman & Eames, 1975; Larato, 1972). Smoother restorations are also more easily maintained (Strassler & Bauman, 1993; Weitman & Eames, 1975). Restoratives cured against a matrix represent the smoothest surface possible (Yap, Lye & Sau, 1997). However, despite careful placement of matrixes, some degree of finishing (gross contouring) and polishing (reduction of roughness and scratches caused by finishing instruments) of restorations is often required clinically. Although the surface finish of resin-modified glass ionomer cements has been widely investigated (Yap & others, 2002: Wilder & others, 2000: Hoelscher & others, 1998), few studies have researched the quality of surface finish of highly viscous GICs. In addition, the effectiveness of new one-step finishing/polishing systems, when used with resin-modified and highly viscous GICs, has not been widely explored.

This study determined the surface finish of resinmodified and highly viscous glass ionomer cements after treatment with three one-step finishing/polishing systems. The surface roughness obtained was compared to using a matrix strip and other conventional multi-step systems.

### **METHODS AND MATERIALS**

Table 1 shows the technical profiles of the glass ionomers investigated. Both cements were in capsulated form and activated/mixed according to manufacturer's instructions. The cements were injected into the square recesses (3-mm long x 3-mm wide x 2-mm deep) of customized acrylic molds and covered with matrix strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide was then placed over the molds and pressure applied to extrude the excess material. The resin-modified cement was light-polymerized for 20 seconds, while the highly viscous cement was allowed to set for 10 minutes. The intensity of the light source (Trilight, 3M-ESPE, Seefeld, Germany) was determined with the built-in radiometer and a constant output of 800 mW/cm<sup>2</sup> was established. Forty-eight specimens of each material were made and divided into six groups of eight specimens. Specimens in Group 1 were left alone (matrix strip [MS] finish), while the remaining groups were subjected to gross finishing with ANSI (American National Standards Institute) 320 grit grinding paper (Carbimet disks; Wirtz-Buehler, Dusseldorf, Germany) using a lapping device (Phoenix Beta; Wirtz-Buehler, Dusseldorf, Germany) at 300 rpm for one minute. The groups were then finished/polished with the following systems: Group 2—Super Snap [SS]; Group 3— CompoSite [CS]; Group 4—One Gloss [OG]; Group 5— Pogo [PG] and Group 6—Sof-Lex Brush [SB]. Table 2 reflects the manufacturers and details of the finishing/polishing sequences that were based upon manufacturers' instructions. SS is a multi-step graded abrasive disk system, while CS is a two-step rubber abrasive system. OG, PG and SB are all one-step finishing/polishing systems. To minimize the effect of operator vari-

Table 1: The Glass Ionomer Cements Investigated						
Material	Manufacturer	Components	Mean Particle size (μm)	Lot #		
Fuji II LC (Resin-modified)	GC Corporation, Tokyo, Japan	Powder: Alumino silicate glass, pigments	4.5	0107105		
		Liquid: Polyacrylic acid, distilled water, HEMA (17%), dimethacrylate monomer, camphoroquinone				
Fuji IX GP Fast (Highly viscous)	GC Corporation, Tokyo, Japan	Powder: Alumino silicate glass, pigments Liquid: Polyacrylic acid, distilled water	7.0	0109083		

Table 2: Finishing/Polishing Systems and Sequences							
Product Usage Handpiece Speed Manufacturer							
Super-Snap							
Coarse	Dry, 6 strokes	12,000 rpm	Shofu Inc,				
Medium	Dry, 6 strokes	12,000 rpm	Kyoto, Japan				
Fine	Dry, 6 strokes	12,000 rpm					
Extra-Fine	Dry, 6 strokes	12,000 rpm					
CompoSite Polishers							
CompoSite	Wet, 12 strokes	12,000 rpm	Shofu Inc,				
CompoSite Fine	Dry, 12 Strokes	12,000 rpm	Kyoto, Japan				
One-Gloss	Wet, 12 heavy strokes	10,000 rpm	Shofu Inc,				
	Wet, 12 light strokes	10,000 rpm	Kyoto, Japan				
Pogo	Dry, 24 light	12,000 rpm	Dentsply, Konstanz,				
	intermittent strokes	12,000 rpm	Germany				
Sof-Lex Brush	Dry, 24 strokes	12,000 rpm	3M ESPE,				
			St Paul, MN, USA				

Table 3: Mean Surface Roughness Observed with the Different Finishing/Polishing Systems

	-	
Finishing/Polishing System	Fuji II LC	Fuji IX GP Fast
Matrix strip [MS]	0.13 (0.02)	0.14 (0.03)
Super-Snap [SS]	0.68 (0.10)	0.62 (0.10)
CompoSite [CS]	0.58 (0.16)	0.69 (0.12)
One-Gloss [OG]	1.04 (0.17)	0.81 (0.12)
Pogo [PG]	0.45 (0.09)	0.34 (0.12)
Sof-Lex Brush [SB]	0.51 (0.30)	0.63 (0.13)
Standard deviations in parentheses.	•	•

Table 4: Comparison of Mean Surface Roughness Between Finishing/Polishing Systems

1 11110111119/1 0	Timering, Telering Cycleria				
Materials	Differences				
Fuji II LC	MS < PG, SB, CS, SS < OG PG < SS				
Fuji IX GP Fast	MS < PG < SS, SB, CS, OG SS < OG				
<denotes of<="" significant="" statistically="" td=""><td>difference. Results of one-way ANOVA/Scheffe's test (p&lt;0.05).</td><td></td></denotes>	difference. Results of one-way ANOVA/Scheffe's test (p<0.05).				

Table 5: Comparison of Mean Surface Roughness Among Materials

table of Companion of Mount Canado Floagimese Funding Materials					
Finishing/Polishing Systems	Differences				
Matrix strip [MS]	NS				
Super-Snap [SS]	NS				
CompoSite [CS]	NS				
One-Gloss [OG]	Fuji II LC > Fuji IX GP Fast				
Pogo [PG]	NS				
Sof-Lex Brush [SB]	NS				
> donotos statistically significant	difference and NS indicates no statistical significance. Posults of Independent				

>denotes statistically significant difference and NS indicates no statistical significance. Results of Independent Samples t-test (p<0.05).

ability, all finishing/polishing procedures were carried out by a single researcher.

After finishing/polishing, the mean surface roughness (µm) of the specimens was measured using a pro-

filometer (Surftest SV-400; Mitutoyo, Kanagawa, Japan). Readings were taken at the center of each specimen and four sampling lengths of 0.25 mm were used, giving a total evaluation length of 1 mm. All statistical analysis was carried out at significance 0.05. level Two-way ANOVA was used to determine significant interactions between the independent variables (materials and finishing/polishing techniques). One-way

ANOVA and Scheffe's post-hoc tests were used to compare the surface roughness obtained with the different finishing/polishing systems, while independent samples *t*-tests were employed to evaluate differences between the two materials.

### **RESULTS**

Table 3 shows the mean surface roughness observed with the different finishing/polishing systems. Results of statistical analysis are shown in Tables 4 and 5. Mean Ra ranged from 0.13 to 1.04 µm for Fuji II LC and 0.14 to 0.81 for Fuji IX GP Fast. For both materials the smoothest surface was obtained with MS and the roughest with OG.

Two-way ANOVA revealed significant interaction between the materials and finishing/polishing techniques. The effect of finishing/polishing systems on surface finish was therefore material dependent. For both materials, the use of MS resulted in significantly lower Ra values than all other finishing/polishing systems. For Fuji II LC, treatment with OG resulted in significantly rougher surfaces compared to treatment with PG, SB, CS and SS. In addition, Ra values obtained with PG were significantly lower than with SS. For Fuji IX GP, specimens treated with PG were significantly smoother than specimens treated with all other systems. Specimens treated with SS were significantly smoother than specimens treated with OG. Although no significant difference in Ra values was observed between Fuji II LC and Fuji IX GP when treated with MS, SS, CS, PG and SB, significant differences

were observed when the materials were polished with OG. When finished/polished with the latter, Fuji II LC was significantly rougher than Fuji IX GP.

### **DISCUSSION**

Rough surfaces are made smooth by a process known as abrasion, in which hard, sharp particles of abrasive are moved over the surface. Each particle of abrasive acts as a fine tool, cutting a groove in the surface of the dental restoration/material. The surface is smoothened, but a series of scratches are left, the dimension of which is dependent on the particle size of the abrasive. With the exception of PG, which uses fine diamond powder, aluminum oxide was the abrasive utilized in all systems. In the polishing process, a series of abrasives of increasing fineness were used. Disorderly application of different grades of abrasives instead of descending order could markedly reduce smoothness of the finished surface (Kanter, Koski & Bogdan, 1983). To reduce cost and clinical time, multi-step (graded abrasive disks) systems were replaced with three- and twostep systems (rubber abrasives with/without polishing pastes). One-step systems were recently introduced but their effectiveness has not been widely researched. Instead of using abrasives of increasing fineness, these systems generally employ the use of varying and intermittent pressure for finishing and polishing.

In this study, the smoothest surfaces were obtained by curing both cements against a matrix strip. This finding agreed with previous studies on conventional and resinmodified glass ionomer cements (Paulillo & others, 1997; Yap & others, 1997; Hondrum & Fernandez, 1997). All finishing/polishing procedures decreased the smoothness obtained with matrix strips. Glass ionomers are heterogeneous and biphasic in nature. The set material consists of unreacted glass particles embedded in a polysalt/resin matrix. During finishing and polishing, the softer matrix phases are preferentially removed, leaving the harder, unreacted glass particles to protrude from the surface. This accounts for the significant increase in Ra values observed with all finishing/ polishing procedures. Bollen, Lambrechts and Quirynen (1997) reported a critical threshold surface roughness for bacteria adhesion of 0.2 µm. While no further reduction in bacterial accumulation is expected below this threshold value, any increase in surface roughness above 0.2 µm results in a simultaneous increase in plaque accumulation and increases the risk of caries and periodontal inflammation (Bollen & others, 1997). As all treated surfaces had Ra values greater than 0.2 um, the effect of finishing/polishing systems on surface finish of resin-modified and highly viscous GICs is clinically relevant.

The effect of finishing/polishing systems on surface finish was material dependent and the effectiveness of one-step systems was product dependent. For both cements, the roughest surface was observed after finishing and polishing with OG. These results corroborated those of previous studies involving OG and a similar system (Yap & others, 2002; Hoelscher & others, 1998; Yap

& others, 1997; Tate & Powers, 1996). The surface finish obtained with OG was significantly rougher than all the other systems for Fuji II LC and was significantly rougher than PG and SS for Fuji IX GP Fast. As the abrasive used was generally the same, differences in performance between OG and the other systems may be attributed to variations in abrasive particle shape/size, configuration (wheel, points and cup) and abrasive delivery medium. The aluminum oxide and silicon dioxide abrasives used in OG were embedded in polyvinysiloxane. Due to its high elasticity, polyvinylsiloxane delivery medium may be resistant to wear by the relatively soft polysalt matrix and fluorosilicate glasses used in GICs, leading to reduced efficiency of the abrasives. The aforementioned may explain the enhanced performance of these systems when used to finish/polish composites and compomers (Yap & others, 1997).

Previous studies have shown that the use of graded abrasive disks generally gave the best surface finish with glass ionomer cements (Yap & others, 2002; Hoelscher & others, 1998; Yap & others, 1997; Hondrum & Fernandez, 1997). As no significant difference in Ra values was observed between SS and PG and SB and CS, use of the latter finishing/polishing systems is clinically viable for resin-modified glass ionomers. For the highly viscous GIC, the use of PG resulted in a significantly smoother finish compared to the other systems, including the graded abrasive disks SS. The superior performance of PG for both materials may be attributed in part to the use of fine diamond powders instead of aluminum oxide and the cured urethane dimethacrylate resin delivery medium. PG is, however, only available in disk configuration. Its clinical use for finishing/polishing cervical restorations with margins located at or below the gingival crest may not be feasible, as it can result in severe gingival trauma. The use of SB and CS are viable alternatives where disk configurations are not feasible. SB was developed for polishing the concave and convex anatomy found on posterior composite restorations. The relatively large standard deviation observed when SB was used to finish/polish Fuii II LC may be due to the better wear resistance of Fuji II LC compared to Fuji IX (Shabanian & Richards, 2002) and the irregular contacts between the thermoplastic polyester elastomer bristles and the surface of the material. The effectiveness of PG and SB for finishing/polishing composites and componers are currently being evaluated.

The surface finish of glass ionomers is dependent in part on their particle size range. The latter can be estimated by the mean particle size of the fluorosilicate glasses used. Glass ionomers with larger particles are expected to be rougher after finishing and polishing. The mean particle size of Fuji II LC is 4.5 µm, while that of Fuji IX GP Fast is approximately 7 µm (Yap, Pek & Cheang, 2003). The mean particle size of regular set Fuji

IX GP is much larger (13.5 µm). With the exception of OG, no significant difference in Ra values were observed between the two cements for all finishing/polishing systems. This may account for the similarity in mean particle size of the two cements. When finished/polished with OG, Fuji II LC was significantly rougher than Fuji IX GP Fast. The exact reasons for this are not known. The low efficiency of OG when used with resin-modified GICs and the higher wear resistance of resin-modified GICs may be contributing factors.

### CONCLUSIONS

Within the limitations of this study:

- 1. The use of matrix strips resulted in the best surface finish for resin-modified and highly viscous glass ionomer cements.
- 2. All finishing/polishing procedures decreased the smoothness obtained with matrix strips and resulted in Ra values above the critical threshold value of 0.2 um.
- The effectiveness of one-step systems for finishing/polishing glass ionomers is product dependent.
- Pogo disk and Sof-Lex Brush produced superior or comparable surface finish to two-step rubber abrasive (CompoSite Polishers) and graded abrasive disk systems (Super-Snap).
- 5. The use of One Gloss resulted in the poorest surface finish.

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## Composite Cure and Pulp-cell Cytotoxicity Associated with LED Curing Lights

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### **Clinical Relevance**

Composites cured with LED lights were more cytotoxic than composites cured with conventional halogen lights.

### **SUMMARY**

This study compared the cure and pulp-cell cytotoxicity of composites polymerized with lightemitting diode (LED) and halogen-based light curing units. A mini-filled resin composite (Tetric Ceram, Vivadent), two LED (E-light [EL], GC and Freelight [FL], 3M-ESPE), a conventional halogen (Max [MX], Dentsply) and a high-intensity halogen light (Astralis 10 [AS], Vivadent) were evaluated. Cure associated with the different lights was determined by measuring the top and bottom surface hardness (KHN: n = 5) of 2-mm thick specimens using a digital microhardness tester (load = 500 gf; dwell time = 15 seconds). Pulp-cell cytotoxicity was assessed using a direct contact method involving incisor tooth slices dissected from 28-day old Wistar rats maintained in Dulbecco's Modified Eagle's Medium (DMEM) and 1% agarose. The bottom surfaces of the cured composite specimens (7-mm diameter and 2-mm deep) were placed in contact with the openings of each tooth slice. After incubation in 5% CO<sub>2</sub> atmosphere at 37°C for 48 hours, the tooth slices were fixed, demineralized and processed for histological examination. Pulp fibroblasts and odontoblasts were counted histomorphometrically at 400x magnification within a 1500 μm<sup>2</sup> area using a computerized micro-imaging system. Eighteen readings were obtained for each curing light. Data was subjected to ANOVA/Scheffe's test and Pearson's correlation at significance level 0.05 and 0.01, respectively. At the top surfaces, the cure with AS was significantly greater than the other curing lights, with MX and FL being significantly greater than EL. At the bottom surfaces, MX, AS and FL had significantly better cure than EL. Specimens cured with MX were less cytotoxic than those polymerized with other curing lights.

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Specimens cured with AS and EL were significantly less cytotoxic than FL. Composite cure and cytotoxicity associated with LED lights is device dependent. Composite cure was not correlated to pulp-cell cytotoxicity. The response of pulpal fibroblasts to unreacted/leached components of composites differs somewhat from odontoblasts.

### INTRODUCTION

The use of light-cured composite restoratives has increased substantially over the last few years due to improvements in formulation, simplification of bonding techniques and increased aesthetic demands by patients. Methods and devices used to prepare and light cure composites have also evolved concomitantly. Halogen curing lights remain the more widely employed light source for curing composites. Halogen bulbs generate light by electrical heating of a tungsten filament to extremely high temperatures. Mostly heat radiation, which is in infrared of the electromagnetic spectrum, is generated (Althoff & Hartung, 2000) and only a small percent of the light output is in the visible part (including the blue range) desired for polymerization. Despite the use of dielectric filters, halogen curing lights emit a considerable number of other wavelengths beyond the absorption spectrum of the camphorquinone photoinitiator employed in many lightcured composites. These spectral impurities are highly absorbed by dental materials, inducing heating of the tooth and composite during the curing process (Masutani & others, 1988; Hannig & Bott, 1999). The high operating temperatures and large quantity of heat produced during the duty cycles degrade the bulb and reflector, reducing the curing effectiveness over time (Barghi, Berry & Hatton, 1994).

To overcome the problems inherent with halogen lights, new devices based on solid state light-emitting diode (LED) technology have been developed for polymerizing composites. Rather than a hot filament, LEDs use junctions of doped semiconductors (p-n junctions) to generate light (Nakamura, Mukai & Senoh, 1994). As the spectral output of gallium nitride blue LEDs fall within the absorption spectrum of camphorquinone (450–490 nm) (Nomoto, 1997), no filters are required. In addition, LEDs also have an expected lifetime of several thousand hours without significant degradation of

light emission over time (Haitz, Craford & Weissman, 1994). Although some research had been conducted on the use of LED lights on composite hardness, modulus, depth of cure, compressive and flexural strengths (Jandt & oth-

ers, 2000; Kurachi & others, 2001; Stahl & others 2000; Mills, Jandt & Ashworth, 1999), the cytotoxicity of composites cured with LED lights has not been investigated. The latter is particularly important in view of recent studies reporting the lower polymerization efficiency of LED lights when compared to halogen lights (Mills & others, 1999; Leonard & others, 2002; Dunn & Bush, 2002).

This study examined the effectiveness of cure and composite pulp-cell cytotoxicity associated with use of LED curing lights. Composite cure and the cytotoxicity of LED lights were compared with conventional and high-intensity halogen lights. The correlation between composite cure and pulp-cell cytotoxicity was also investigated.

### **METHODS AND MATERIALS**

An A2 shade mini-filled resin composite (Tetric Ceram, Vivadent, Schaan, Liechtenstein), two LED (E-light [EL], GC Europe, Leuven, Belgium; Freelight [FL], 3M-ESPE, Seefeld, Germany), a conventional halogen (Max [MX], Dentsply) and a high-intensity halogen light (Astralis 10 [AS], Vivadent, Schaan, Liechtenstein) were evaluated. Light intensities of the curing lights were determined with commercial radiometers (Hilux, Benlioglu Dental Inc, Ankara, Turkey; CureRite, EFOS Inc, Ontario, Canada) prior to starting the experiment. Two radiometers were used, as one was designed for both LED and halogen lights (Hilux), while the other was intended for only halogen lights (CureRite). Five readings were made for each curing light and the results are reflected in Table 1, together with the manufacturers' stated light intensities. The emission spectrum of curing lights was determined with a photometer (662, Schmidt Scientific, Metrohm, Switzerland) equipped with a light guide measuring cell with attached reflector. Three direct measurements were obtained over a wavelength range of 430 to 510 nm. Figure 1 shows the mean spectral output of the curing lights.

### **Composite Cure**

A micro-indentation technique was used to assess composite cure (Leonard & others, 2002; Dunn & Bush, 2002; Yap, Soh & Siow, 2002). The composite was placed in stainless steel molds with cylindrical cavities 7-mm in diameter and 2-mm in depth confined between

Table 1: Light Intensities of the Different Curing Units							
Curing Light Mode Manufacturer Hilux CureRite (mW/cm²) (mW/cm²) (mW/cm²)							
Max	Standard	400	423 (3)	358 (4)			
Astralis 10	Hi Power	1200	1282 (9)	1296 (2)			
E-light	Standard	350	311 (3)	260 (3)			
Freelight	Standard	400	287 (2)	260 (3)			
Standard deviations in pa	Standard deviations in parentheses.						

two opposing acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide (1-mm thick) was 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 curing lights. With the exception of AS, the curing time for all lights was 40 seconds. Based on manufacturer's instructions, a 10-second curing time was used for AS. Immediately after light polymerization, the acetate strips were removed and the specimens were positioned centrally in their molds just beneath the indenter of a digital microhardness tester (FM7, Future-Tech Corp, Tokyo, Japan) to assess the

Knoop's 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 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 curing light and mean KHN was determined. The mean hardness ratio for each light was then computed using the following formula: Hardness ratio = KHN of bottom surface/KHN of top surface.

### **Pulp-Cell Cytotoxicity**

Maxillary and mandibular incisors were acquired from three 28-day old Wistar rats. Anaesthetic comprising of 1:1:2 ratio hyponorm, domicum and water, respectively, was injected into the peritoneal cavity of the rodents. The surgical site was cleansed with iodine and ethanol prior to removing the incisors. Each extracted incisor was sterilized with 70% ethanol solution, rinsed with phosphate buffer saline [PBS] and maintained in Dulbecco's Modified Eagle's Medium [DMEM] (Sigma, Missouri, USA). Between 10 and 12 transverse incisor sections 2-mm thick were obtained from each rat. These transverse sections involved only the middle intact portion of each incisor, with apical/coronal segments being discarded. A diasectioning mond instrument

(Renfert GmbHm, Hilzingen, Germany) was used with PBS coolant to prepare the incisor slices. Flawed specimens were removed and five incisor slices were randomly selected from the remaining acceptable specimens of each rat.

Each incisor slice was individually placed into six-well plates and partially submerged with 2 ml of DMEM complex medium comprised of 4 mM L-glutamine, 4500 mg/L glucose and incorporated additives: -0.15 mg/ml Vitamin C (Sigma, Missouri, USA), 10% heat inactivated fetal calf serum (Biological Industries, Kibbutz Beit Haemak, Israel), 1% of 5 mg/ml streptomycin (Gibco BRL, Maryland, USA), 1% of 5,000 units/ml penicillin

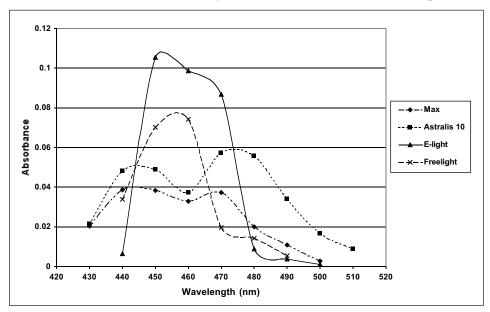


Figure 1. Mean emission spectral of the curing lights.

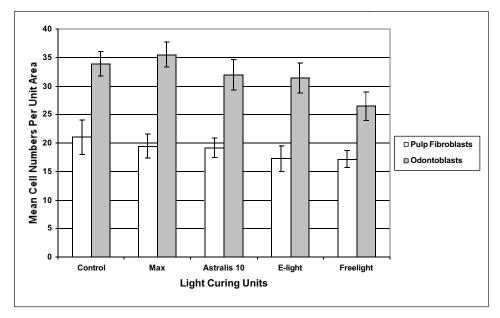


Figure 2. Mean cell numbers per unit area for the various curing lights.

(Sigma, Missouri, USA) and  $3.7~\rm g/L$  sodium bicarbonate (E Merck, Darmstadt, Germany). The plates were then incubated at  $37^{\circ}\rm C~in~5\%~\rm CO_2$  atmosphere in a humidified incubator. Medium was replenished on a daily basis for five days.

After the five-day culture period, the incisor slices were transferred to new sterile six-well plates with an overlay of about 3 ml of culture gel (DMEM complex medium with addition of 1% agarose). The five tooth slices were then randomly assigned to the following groups: Group 1control group (not exposed to composite); Group 2—exposure to composite cured with MX; Group 3—exposure to composite cured with AS; Group 4—exposure to composite with EL and Group 5—exposure to composite cured with FL. The incisor slices were positioned at the center of each culture gel and towards the bottom surfaces of two composite disks (7-mm diameter and 2-mm thick) in direct contact with the two open ends of each incisor slice. The composite disks were prepared as described above and placed using agarose gel support. Approximately 1 ml of DMEM complex medium was added to partially submerge

Table 2: Mean KHN, Hardness Ratio, Average Pulp Cell Numbers and Percentage Cytotoxicity for the Various Treatment Groups						
Variables	Control	Max (MX)	Astralis 10 (AS)	E-light (EL)	Freelight (FL)	
Top KHN	NA	22.34 (2.22)	33.32 (3.05)	15.88 (1.01)	22.50 (0.87)	
Bottom KHN	NA	21.10 (2.23)	20.04 (1.41)	13.18 (0.95)	19.98 (1.80)	
Hardness ratio	NA	0.94 (0.01)	0.61 (0.07)	0.83 (0.09)	0.89 (0.07)	
Average # of cells	27.47 (1.92)	27.47 (1.72)	25.58 (1.70)	24.31 (1.90)	21.83 (1.37)	
Percentage cytotoxicity (fibroblasts)	0%	7.7%	9.0%	18.2%	18.5%	
Percentage cytotoxicity (odontoblasts)	0%	0%	5.6%	7.4%	21.8%	
Percentage cytotoxicity (average # of cells)	0%	0%	6.9%	11.5%	20.5%	

**Figure 3:** Pulpal fibroblasts and odontoblasts in the control and FL group (haematoxylin and eosin stain). Although morphology of the cells was similar between FL and the control group, fibroblasts/odontoblasts were more dispersed and cell numbers were lower in the FL group (Figures 3.1A and 3.2A).

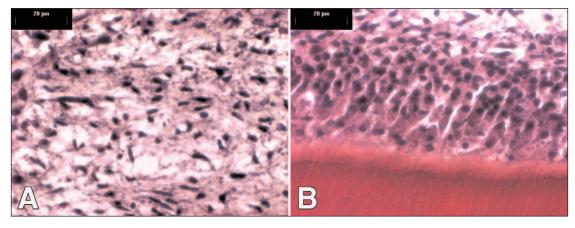


Figure 3.1. Pulpal fibroblasts (A) and odontoblasts (B) in a control tooth slice.

Standard deviations in parentheses. NA denotes not applicable.

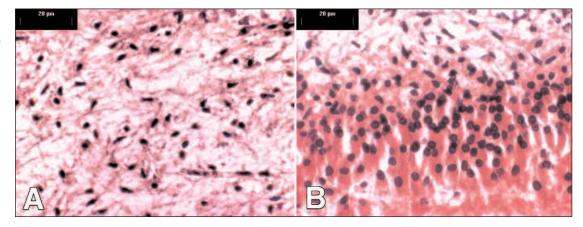


Figure 3.2. Pulpal fibroblasts (A) and odontoblasts (B) of a tooth slice exposed to composite cured with FL.

the incisor slices. The plates were then incubated at  $37^{\circ}\mathrm{C}$  in 5%  $\mathrm{CO}_2$  atmosphere for two days, replenishing the medium daily.

After the two-day contact testing period, the incisor slices were fixed in 10% formalin for 24 hours, demineralized in 10% formic acid for 48 hours and processed and embedded in paraffin wax for histological examination. Two longitudinal sections, 5 µm apart, were obtained from the center of each incisor slice and stained with haematoxylin and eosin. Pulp fibroblasts and odontoblasts in each section were counted histomorphometrically at 400x magnification within an area of 1500 mm<sup>2</sup> using a computerized micro-imaging system (Microimage Version 4.0, Olympus, Tokyo, Japan). To ensure accurate analysis of the cell counts in each section, three arbitrary areas of pulp fibroblasts and odontoblasts were evaluated. Thus, for each experimental group, a total of 18 readings were obtained from the three rats. The percentage of cytotoxicity for each cell type was subsequently determined using the formula: 100%—(Mean cell number of test group/Mean cell number of control group X 100%).

Microhardness and pulp-cell cytotoxicity data were subjected to analysis using one-way ANOVA and Scheffe's post-hoc test at significance level 0.05. The correlation between composite cure and cytotoxicity was assessed using Pearson's correlation at significance level 0.01.

### **RESULTS**

The mean KHN, hardness ratio, pulp-cell numbers and percentage of cytotoxicity are shown in Table 2 and Figure 2. Results of statistical analysis are shown in Tables 3 and 4. At the top surfaces, specimens cured with AS were significantly harder than those cured with the other curing lights. The top KHN observed with MX and FL was significantly greater than that observed with EL. At the bottom sur-

faces, KHN obtained for MX, AS and FL were significantly greater than EL. The hardness ratio of MX, EL and FL was significantly greater than for AS.

Ranking of percentage cytotoxicity was as follows: Pulp fibroblasts—Control < MX < AS < EL < FL; Odontoblasts—Control and MX <

AS < EL < FL and Average number of cells—Control and MX < AS < EL < FL. Micrographs depicting the survival of pulp cell populations for Control and FL are shown in Figures 3.1 and 3.2, respectively. No significant difference in the number of pulp fibroblasts was observed between specimens in the control group and for those cured with MX and AS. Specimens cured with both LED lights were cytotoxic to pulp fibroblasts (Table 3). For odontoblasts, specimens cured with FL were significantly more cytotoxic than those cured with MX, AS, EL and the control group. Specimens cured with MX were less cytotoxic than those cured with the other curing lights. When the average number of cells was assessed, only MX was not significantly different from the control group. Specimens in the control group and those cured with MX were significantly less cytotoxic than those cured with AS, EL and FL. AS and EL were significantly less cytotoxic than FL.

The correlation between hardness ratio, top and bottom surface hardness and pulp-cell cytotoxicity was very weak and statistically insignificant. The correlation between top and bottom surface hardness was positive and significant, with a correlation coefficient r=0.61. The correlation between the number of vital pulp fibroblasts and odontoblasts was also positive and significant. A correlation coefficient of r=0.39 was observed for the latter.

Table 3: Comparison of Means Between Experimental Groups						
Tests	Tests Variables Differences					
Cure	Top KHN	AS > MX, EL, FL MX, FL > EL				
	Bottom KHN	MX, AS, FL > EL				
	Hardness ratio	MX, EL, FL > AS				
Cytotoxicity	# of pulp fibroblast	Control > EL, FL				
# of odontoblast MX > AS, EL, FL Control, AS, EL > FL						
Average # of cells  Control, MX > AS, EL, FL  AS, EL > FL						
>denotes statistically sign	ı nificant differences. Results of one-way A	NOVA/Scheffe's test (p<0.05)				

Table 4: Pearson's Coefficient for the Various Variables							
Variables	Top KHN	Bottom KHN	Hardness Ratio	Pulp Fibroblast	Odontoblast	Average # of Cells	
Top KHN	1	0.61*	-0.70*	0.03	-0.50	-0.04	
Bottom KHN	0.61*	1	0.12	0.16	0.09	0.14	
Hardness Ratio	-0.70*	0.12	1	0.13	0.17	0.19	
Pulp Fibroblast	0.03	0.16	0.13	1	0.39*	0.71*	
Odontoblast	-0.50	0.09	0.17	0.39*	1	0.93*	
Average # of cells	-0.04	0.14	0.19	0.71*	0.93*	1	
>denotes statistically sig	gnificant correlation	n. Results of Pearso	on's correlation (p<0.0	1)			

### DISCUSSION

Effective composite cure is critical, not only to ensure optimum physical properties (Asmussen, 1982a), but also to ensure that clinical problems do not arise due to cytotoxicity of inadequately polymerized materials (Caughman & others, 1991). In addition to material factors, polymerization is dependent on the effectiveness of the radiation source, including spectral distribution, intensity, exposure time and position of the light-cure tip (Harrington, Wilson & Shortall, 1996). As material factors were standardized by using one composite and fixing the position of the light-cure tip at 1 mm, any difference in cure or cytotoxicity can be attributed to the radiation source. The composite cure may be assessed directly or indirectly. Direct methods, such infrared spectroscopy and laser Raman spectroscopy, are not used routinely, as they are complex, expensive and time consuming (Rueggeberg & Craig, 1988). Indirect methods have included visual, scrapping and hardness testing. Incremental surface hardness using micro-indentation techniques has been shown to be an indicator of the degree of conversion (Asmussen, 1982b) and a good correlation between Knoop's hardness and infrared spectroscopy has been reported (DeWald & Ferracane, 1987).

For 2-mm thick composite specimens, radiation source intensity and duration have been shown to be the more important factors influencing composite cure (Rueggeberg & others, 1993). Since the light energy density (intensity x duration) of AS was similar or lower than the other curing lights, the significantly greater top KHN observed may be attributed to additional cross-linking offered by its high thermal emission (Yap & Soh, 2003; Wendt, 1987). The significantly higher top KHN of MX and FL, compared to EL, was consistent with the manufacturer's light intensity values. These values varied somewhat from those obtained with the two commercial radiometers that were calibrated to read a wide spectral range (400 to 500 nm wavelength). With commercial radiometers, the intensity readings of LED lights were generally lower, due to the narrower spectral output of LEDs. The output intensity of EL was greater than FL when assessed with the Hilux radiometer. This can be accounted for by wider spectral output of EL (440 to 500 nm) when compared to FL (440 to 490 nm) (Figure 1). At the bottom surfaces, specimens cured with MX, AS and FL were significantly harder than those cured with EL. The latter observation may be partially explained by differences in light energy densities. Based on top and bottom KHN, it can be concluded that the cure of LED lights is device dependent and some LED curing lights are not as effective as conventional and high-intensity halogen lights. This finding corroborates those of recent studies on the polymerization efficiency of LED curing lights (Leonard & others, 2002; Dunn & Bush, 2002).

Ideally, the degree of cure of the composite should be the same throughout its depth and the hardness ratio should be very close or equal to 1. As light passes through the composite, light intensity is reduced due to light scattering, thus, decreasing cure at the bottom surfaces (Ruyter & Øysæd, 1982). A hardness ratio of 0.8 has been used to indicate satisfactory cure (Leonard & others, 2002; Yap & others, 2002; Pilo & Cardash, 1992). All curing lights evaluated had a hardness ratio greater than 0.8 except for AS. The low hardness ratio of AS can be attributed to the high top KHN associated with its use. The thermal effect on composite cure is expected only at the top surfaces as composites are poor conductors of heat (Civian & others, 1972). The bottom KHN of AS was comparable to MX and FL, and significantly greater than EL. Hence, hardness ratio alone cannot be used to assess composite cure.

The term "cytotoxicity" is used to describe the cascade of molecular events that interfere with macromolecular synthesis, causing unequivocal cellular functional and structural damage (Murray & others, 2000). The choice of cell line for *in-vitro* cytotoxicity testing remains controversial and a vast number of cells have been used. They range from immortalized mouse cell lines obtained from commercial suppliers (L-929 and 3T3) (Sletten & Dahl, 1999; Geurtsen & others, 1998) to the diploid culture of primary cells from dental pulps (Geurtsen & others, 1998; van Wyk, Olivier & Maritz, 2001). Primary cell cultures derived from oral tissues are arguably more relevant to the clinical situation. Pulp cell lines (which are more sensitive indicators of cytotoxicity) are, however, difficult to culture and have poor survival rates (van Wyk & others, 2001; Heywood & Appleton, 1984). A tooth slice organ model was thus used in this study. The use of cultured human tooth slices can be problematic, as disease-free, extracted teeth are in short supply. Variations in human tooth pathology and history between individuals could affect cell responses, creating test conditions that are difficult to replicate (Murray & others, 2000). By using the incisors of 28-day old Wistar rats, it was possible to treat all teeth identically by controlling their history, care, extraction and processing. Variations between teeth were minimized by using only the middle portion of upper and lower incisors. This provided a near physiological and pathologically similar tooth population that can be directly compared. Two different kinds of pulp cells (fibroblasts and odontoblasts) were assessed in order to minimize the effect of inter-cell-line differences (van Wyk & others, 2001).

The response of pulpal fibroblasts to unreacted/leached components of composites differed somewhat from odontoblasts (Tables 2 and 3). The percentage cytotoxicity of pulpal fibroblasts was generally higher than odontoblasts. Pulpal fibroblasts may, therefore, be a more sensitive cell type for cytotoxicity testing. When

cytotoxicity data was pooled and averaged, the control and composite cured with MX was found to be significantly less cytotoxic than those cured with AS, EL and FL. Composite cured with AS and EL were, in turn, significantly less cytotoxic than those cured with FL. Unlike earlier studies (Caughman & others, 1991; Quinlan & others, 2002), the results of cytotoxicity testing did not correlate with composite cure. The apparent discrepancy can be explained by the use of a single light source in past studies. Although cure with EL was lower than FL, the resultant composite was more biocompatible. A recent study (Asmussen & Peutzfeld, 2001) showed that differences in radiation procedures (with the same lights) led to dissimilar structures of the resulting polymer despite the same degree of cure. Therefore, it is conceivable that differences in light sources could result in different polymer structures despite having similar cures. For example, a bifunctional monomer that reacts only in one end of each molecule leads to a completely linear polymer structure with 50% remaining double bonds. The double bond in the other end of each molecule may react with either a neighboring double bond on the same polymer chain or with double bonds on another polymer chain. The two situations give rise to the same quantity of remaining double bonds (and indirectly hardness) but would have diverse polymer structures after several reactions (Asmussen & Peutzfeldt, 2001). Composites with linear polymer structures and fewer crosslinks may release more polymeric materials (unreacted monomer, oligomers and liner polymer), resulting in greater cytotoxicity. Disparity in spectral outputs between the two LED lights is offered as a hypothesis for the difference in biocompatibility and possible variation in polymer structure. From Figure 1 it can be seen that spectral output of FL lies mainly within 440 and 470 nm. For halogen lights, spectral outputs were wider ranging from 430 to 500 nm. Although EL is based on LED technology, its spectral output is larger than FL and somewhat similar to the halogen lights.

### CONCLUSIONS

The results of this study suggest that composite cure and cytotoxicity associated with LED lights is device dependent. Since the correlation between composite cure and cytotoxicity was insignificant and weak, an effective cure cannot be equated to good biocompatibility. Composite cured with LED lights was more cytotoxic than composite cured with conventional halogen lights. The response of pulpal fibroblasts to unreacted/leached components of composites differed somewhat from odontoblasts. The effect of radiation source on polymeric structure and elution of leachable components from composites warrants further investigation.

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### Comparison of Surface Finish of New Aesthetic Restorative Materials

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### Clinical Relevance

The surface finish of glass ionomers and compomers is poorer than composites. Among composites, materials based on ormocer and nanomer technology have the smoothest surface finish.

### **SUMMARY**

This study compared the surface finish of eight different types of aesthetic restorative materials. The materials included resin-modified (Fuji II LC [FL], GC) and highly viscous (Fuji IX GP Fast [FN], GC) glass ionomer cements, a compomer (F2000 [FT], 3M-ESPE), minifilled (Z100 [ZO], 3M-ESPE) and microfilled (A110 [AO], 3M-ESPE) composites and materials based on recently introduced ormocer (Admira [AM], Voco), nanomer (Filtek Supreme Translucent [FST], 3M-ESPE) and nanocluster technology (Filtek Supreme [FS], 3M-ESPE). Sixteen specimens (3-

mm long x 3-mm wide x 2-mm deep) of each material were divided into two equal groups. Specimens in Group 1 received no further treatment after polymerization against a matrix strip, while the specimens in Group 2 were roughened with 320 grit grinding paper using a lapping device and were finished/polished with a graded abrasive disk system (Super-Snap, Shofu). The mean roughness (Ra, µm) of materials was determined using a surface profilometry. Data was analyzed by ANOVA/Scheffe's test at significance level 0.05. Mean Ra values ranged from 0.04 to 0.16 µm for Group 1 specimens and 0.15 to 0.68 um for Group 2 specimens. Results of statistical analysis were as follows: Group 1-FS, FST, FL, FN, AM > FT, AO, ZO; Group 2—FN, FT, FL > AO, FS, ZO, AM, FST (> indicates significantly greater Ra values). For the finished/polished composite materials, Ra values observed for AM and FST were significantly lower than for AO and FS. The surface finish of glass ionomers and compomer was significantly poorer than composites. Composite materials based on ormocer and nanomer technology were significantly smoother than those based on microfillers and nanoclusters.

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### INTRODUCTION

The demand for aesthetic restorations has increased substantially in recent years. On the extreme ends of the continuum of direct tooth-colored restorative materials are conventional glass ionomer cements (GICs) and resin composites. While GICs consist of basic glasses and acidic polymers set by acid-base reactions, resin composites are ceramic-filled polymers that set by visible light and/or chemically activated free-radical polymerization. To combine the major advantages of GICs (chemical bonding to tooth structure, fluoride release and good biocompatibility) with the easy handling and aesthetic properties of composites, various hybrid materials have been developed. These include resin-modified GICs and compomers (polyacid-modified resin composites). Resin-modified GICs are hybrid materials that retain a significant acid-base reaction as part of their overall curing process. In contrast, compomers are materials that may contain either or both of the essential components of GICs but at a level insufficient to promote acid-base cure reaction in the dark (McLean, Nicholson & Wilson, 1994). Concurrent with the development of hybrid materials, advances have also been made in dental composite technology. Composite restoratives based on ormocer (organically modified ceramics) and nanofiller technologies have been recently introduced to the dental profession. Among several other advantages, these new materials claim to have improved surface finish and polish retention.

The quality of surface finish influences the aesthetics and life span of tooth-colored restoratives as the presence of irregularities on the surface of materials may influence appearance, staining, plaque retention, secondary caries risk and gingival irritation (Shintani & others, 1985; Dunkin & Chambers, 1983; Chan, Fuller & Hormati, 1980; Weitman & Eames, 1975; Larato,

1972). In addition, smoother restorations are also more easily maintained (Strassler & Bauman, 1993; Weitman & Eames, 1975). Although the surface finish of composites, compomers, conventional and resin-modified GICs has been widely investigated both *in-vitro* (Yap & others, 2002; Hoelscher & others, 1998; Yap, Lye & Sau, 1997; Hondrum & Fernandez, 1997) and *in-vivo* (Folwaczny & others, 2001; Gladys & others, 1999; Duke & Trevino, 1998), the quality of surface finish of ormocers and nanofilled composites has not been reported.

This study compared the surface finish of an ormocer and nanomer and nanocluster composites to the continuum of direct tooth-colored restorative materials after polymerization against a matrix strip and treatment with a graded abrasive disk system. The results were contrasted against the critical threshold surface roughness for bacterial adhesion of 0.2 µm reported by Bollen, Lambrechts and Quirynen (1997).

### **METHODS AND MATERIALS**

Table 1 lists the spectrum of restorative materials evaluated in this study. They include resin-modified (Fuji II LC [FL]) and highly viscous (Fuji IX GP Fast [FN]) GICs, a compomer (F2000 [FT]), minifilled (Z100 [ZO]) and microfilled (A110 [AO]) composites and materials based on ormocer (Admira [AM]), nanomer (Filtek Supreme Translucent [FST]) and nanocluster technology (Filtek Supreme [FS]). Both glass ionomer cements were in capsulated form and were activated/mixed according to manufacturer's instructions. The restorative materials were injected or placed in the square recesses (3 -mm long x 3-mm wide x 2-mm deep) of customized acrylic molds and covered with matrix strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide was placed on the molds and pressure was applied to

Table 1: Aesthetic Restorative Materials Evaluated in the Study						
Material	Category	Manufacturer	Batch #	Shade Cure Time		
Fuji II LC	Resin-modified GIC	GC Corporation, Tokyo, Japan	0107105	A2 20 seconds		
Fuji IX GP Fast	Highly viscous GIC	GC Corporation, Tokyo, Japan	0109083	A2 Not applicable		
F2000	Compomer	3M-ESPE, St Paul, MN, USA	20010122	A2 40 seconds		
Z100	Composite (Minifill)	3M-ESPE, St Paul, MN, USA	20001128	A2 40 seconds		
Filtek A110	Composite (Microfill)	3M-ESPE, St Paul, MN, USA	20010208	A2 40 seconds		
Admira	Composite (Ormocer)	Voco, Cuxhaven, Germany	025801	A2 40 seconds		
Filtek Supreme Translucent	Composite (Nanomer)	3M-ESPE, St Paul, MN, USA	EXM#612	A2 20 seconds		
Filtek Supreme Body	Composite (Nanocluster)	3M-ESPE, St Paul, MN, USA	EXM#612	A2 20 seconds		

Table 2: Mean Surface Roughness of the Different Materials						
Materials	Group 1 (Matrix)			oup 2 er-Snap)		
	Mean	SD	Mean	SD		
Fuji II LC (FL)	0.14	0.02	0.62	0.10		
Fuji IX GP Fast (FN)	0.13	0.02	0.68	0.10		
F2000 (FT)	0.05	0.01	0.63	0.09		
Z100 (ZO)	0.04	0.01	0.32	0.13		
Filtek A110 (AO)	0.05	0.01	0.41	0.10		
Admira (AM)	0.11	0.04	0.15	0.05		
Filtek Supreme Translucent (FST)	0.15	0.04	0.15	0.02		
Filtek Supreme Body (FS)	0.16	0.04	0.33	0.06		
SD = standard deviations						

Table 3: Results of Statistical Analysis	
Treatment Group	Differences
Group 1	FS, FST, FL, FN, AM > FT, AO, ZO
Group 2	FN, FT, FL > AO, FS, ZO, AM, FST AO, FS > AM, FST
>denotes statistically significant difference in Ra values. Results of one-way ANOVA/Scheffe's post-hoc test (p<0.05).	

extrude the excess material. The light-cured materials were then polymerized according to manufacturers' cure times (Table 1) through the glass slide with a halogen light curing unit (Trilight; 3M-ESPE, Seefeld, Germany) while the highly viscous glass ionomer was allowed to set for 10 minutes. The intensity of the light source was determined with the in-built radiometer and a constant output of 800 mW/cm² was established.

Sixteen specimens of each material were fabricated and divided into two equal groups. Specimens in Group 1 received no further treatment after polymerization against a matrix. Group 2 specimens were roughened with ANSI (American National Standards Institute) 320 grit grinding paper (Carbimet disks; Wirtz-Buehler, Dusseldorf, Germany) using a lapping device (Phoenix Beta; Wirtz-Buehler, Dusseldorf, Germany) at 300 rpm for one minute and were finished/polished with a graded abrasive disk system (Super-Snap; Shofu Inc, Kyoto, Japan) after removal of the matrix strips. Four grades of abrasive disks (coarse, medium, fine and extra-fine) were used. Six strokes of each grade of abrasive disk were applied to the specimens and finishing/polishing procedures were performed dry with a slow handpiece running at 12,000 rpm in one direction.

The mean surface roughness (Ra; µm) of the specimens was measured with a profilometer (Surftest SV-400; Mitutoyo, Kanagawa, Japan). Readings were taken at the center of each specimen and four sampling lengths of 0.25 mm were used, giving a total evaluation length of 1 mm. All statistical analysis carried a significance level 0.05. Two-way ANOVA was used to deter-

mine significant interactions between materials and treatment groups. One-way ANOVA and Scheffe's post-hoc tests were used to compare the mean surface roughness between materials for each treatment group. Independent Samples t-test was used to compare the surface finish between treatment groups for each material. The results were

contrasted against the critical threshold surface roughness for bacterial adhesion of 0.2 µm reported by Bollen, Lambrechts and Quirynen (1997).

### **RESULTS**

Table 2 and Figure 1 shows the mean surface roughness observed for the different materials. The results of statistical analysis are shown in Table 3. Mean Ra values ranged from 0.04 to 0.16  $\mu$ m for specimens in Group 1 and 0.15 to 0.68  $\mu$ m for Group 2 specimens. The Ra values for all specimens in Group 1 were below the critical threshold surface roughness for bacterial adhesion of 0.2  $\mu$ m. In Group 2, only AM and FST had a surface finish of less than 0.2  $\mu$ m. With the exception of AM and FST, the Ra values observed in Group 2 were significantly greater than those observed in Group 1.

Two-way ANOVA revealed significant interaction between materials and treatment groups. The surface finish of the materials was therefore treatment group dependent. In Group 1 (matrix strip), the surface finish of glass ionomers, ormocer and nanofilled composites was significantly poorer than the compomer, microfilled and minifilled composites. In Group 2 (Super-Snap), the surface finish of glass ionomers and compomer was significantly poorer than the composites evaluated. Among the composites, ormocer and nanomer composite had significantly lower Ra values than the microfill and nanocluster composites.

### DISCUSSION

Although restoratives cured against a matrix are not devoid of surface imperfections (Ra=0), they represent the smoothest surface possible for most direct tooth-colored restorative materials (Yap & others, 1997). These surface imperfections are the reproduction of flaws on the matrix strips (Van Noort, 1983). Matrix finished surfaces are polymer-rich (Kao, 1989) and this layer is relatively unstable (Hachiya & others, 1984; Shintani & others, 1985). Despite careful placement of matrixes,

removal of excess material and recontouring of restorations is often clinically necessary. This requires some degree of finishing and polishing that will violate the smoothness obtained with a matrix. The latter accounts for the significantly greater Ra values observed after finishing/polishing of mostmaterials. Finishing refers to the gross contouring or reduction of restorations to obtain the desired contour, while polishing refers to the reduction of roughness and scratches created by the finishing instruments. The finished/polished surface is filler/glassrich and more characteristic of the bulk material (Kao, 1989). Finishing and polishing devices can be broadly classified into four groups (Jefferies, Barkmeirer & Gwinnett, 1992): 1) coated abrasives (abrasive disks or strips); 2) cutting devices (carbide burs and stones); 3)

micron-sized burs and rubberized abrasives and 4) loose particulate abrasives (polishing pastes or powders). A graded abrasive disk system was selected as it has been shown to give the best surface finish among the different finishing/polishing systems for most direct tooth-colored restoratives (Marigo & others, 2001; Hoelscher & others, 1998; Yap & others, 1997; Hondrum & Fernandez, 1997). As improper application of these systems could lead to decreased effectiveness (Kanter, Koski & Bodgan, 1983). Strict adherence to the manufacturer's recommended procedure was observed.

Differences in surface finish between materials were treatment dependent. Although Ra values between materials were significantly different when specimens were finished with matrix strips, the differences may not be clinically relevant as values were all below the critical threshold value of 0.2 µm (Bollen & others, 1997). The significant differences in Ra values may be attributed to inherent material properties such as filler or glass particle sizes and their ability to form a homogeneous polymer-rich layer when the material is cured under pressure. While no further reduction in bacterial accumulation is expected below the threshold value of 0.2 µm, any increase in surface roughness above this value results in a simultaneous increase in plaque accumulation and increases the risk for caries and periodontal inflammation. With the exception of the ormocer (AM) and nanomer composite (FST), the finished/polished surfaces of all materials had Ra values greater than 0.2 um. The differences in surface finish between materials were therefore clinically relevant.

The resin-modified and highly viscous GICs and componer were significantly rougher than the composites after treatment with the Super-Snap system. This may be accounted for by the large fluoroalumino silicate par-

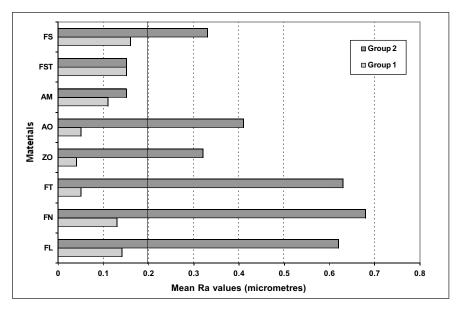


Figure 1. Mean surface roughness of the materials.

ticles used in glass ionomers and componers. The mean particle size of these materials (4 to 8 µm) is large when compared to composites (less than 1 µm). Among composites, the surface finish of microfilled and nanocluster composites was significantly poorer than ormocer and nanomer composite. Although the primary particle size of microfilled composite was 40 nm (0.04 µm), it was significantly rougher than the nanomer composite, which has a primary particle size of 75 nm. To increase filler content and improve properties, polymerized microfilled composites are ground into particles 10 to 20 um in diameter and used as reinforced filler particles along with colloidal silica. As these pre-polymerized reinforced filler particles are heat treated and form covalent chemical bonds with the resin matrix due to a lack of available methacrylate groups on their surfaces, disruption of the filler-matrix interface may occur during finishing/polishing procedures, resulting in the significantly greater Ra values observed. The effective particle size of the nanocluster composite was also smaller than the nanomer composite. The nanocluster composite contains a combination of non-agglomerated 20 nm nanosilica filler and aggregated zirconia/silica nanocluster (with primary particle size from 5 to 20 nm) filler. The cluster particle size range is 0.6 to 1.4 µm. Since composite contains predominantly zirconia/silica nanoclusters, the surface finish is expected to be poorer than nanomer composite. The nanomer composite contains mostly non-agglomerated 75-nm silica nanofiller. It also contains some nanocluster (with a primary particle size of 75 nm) filler but these are prepared only from silica.

The technology used in the ormocer material is somewhat different from that of other composites. While the latter are based on a purely organic matrix, ormocer

consists of an inorganic-organic (inorganic backbone based on  ${\rm SiO_2}$  functionalized with polymerizable organic units) network matrix formed through polycondensation. The filler particles are imbedded into this crosslinked inorganic and organic network matrix. The average particle size of AM is 0.7 µm, which is comparable to the minifilled composite ZO. It may be a combination of the novel network matrix and small filler particles that give AM its superior surface finish. AM and FST were the only two materials where the smoothness obtained with matrix strips was not violated by finishing and polishing. This attests to the superior surface finish offered by ormocer and nanomer composite. Only these two materials had Ra values below the critical threshold value of 0.2 µm after finishing and polishing.

### CONCLUSIONS

Under the limitations of this *in-vitro* study:

- 1. The surface finish of Fuji II LC, Fuji IX GP Fast and F2000 was significantly poorer than composites.
- Among the composites evaluated, the surface finish of Filtek A110 and Filtek Supreme was significantly poorer than Admira and Filtek Supreme Translucent.
- 3. With the exception of Admira and Filtek Supreme Translucent, all materials had Ra values above the critical threshold value of 0.2 µm after finishing and polishing.

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# Polymerization Efficiency of Curing Lamps: A Universal Energy Conversion Relationship Predictive of Conversion of Resin-Based Composite

RH Halvorson • RL Erickson • CL Davidson

### Clinical Relevance

On an equal energy basis, a LED during lamp was found to be more efficient than a tungsten-halogen curing lamp. Ultimate curing potential, however, must also consider the lamp's maximum power output.

### **SUMMARY**

A universal energy-conversion relationship (ECR $_{\rm u}$ ) predictive of conversion of a resin-based composite (RBC) polymerized with any light source has been described. This relationship was derived from an energy conversion relationship for RBC polymerized with a tungsten-halogen lamp and the lamp's efficiency relative to a hypothetical standard lamp. The ECR $_{\rm u}$  was then used to predict conversion throughout RBC polymerized with an LED lamp using the lamp's relative efficiency compared to the standard lamp. The universal energy scale has also been described as predictive of scrape-back lengths for this RBC family when polymerized with any light source. Despite a 31% greater relative efficiency, scrape-

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back lengths from RBC polymerized using the LED lamp were predicted to be only 6% greater than those polymerized with the tungstenhalogen lamp when RBC is polymerized on an equal energy basis. This result was experimentally verified.

### INTRODUCTION

Studies exploring the photopolymerization of resinbased composite (RBC) have shown that conversion and depth of cure is dependent upon the energy of exposure (Cook, 1980; Nomoto, Uchida & Hirasawa, 1994; Halvorson, Erickson & Davidson, 2002). In recent work, an energy-conversion relationship (ECR) was defined for RBC using Fourier Transform Infrared Spectroscopic (FTIR) analysis of photopolymerized RBC and the light transmitting properties of RBC (Halvorson, Erickson & Davidson, 2003). This ECR described the energy associated with a specific conversion at any depth within a cylinder of the photopolymerized RBC. Furthermore, it was shown that there was reciprocity between time and incident power density so that the ECR uniquely defined the conversion

with depth for any incident exposure energy. Coupled with the transmission properties of various shades of the RBC, the ECR could also be used to reliably predict conversion as a function of depth (conversion profile) for any shade with this RBC family of materials. Depth of cure, as defined by the scrape-back method (International Organization for Standardization, 2000), has also been investigated (Halvorson & others, 2002) and an energy associated with the scrap-back length of a photopolymerized RBC was determined and defined as the critical scrape-back energy  $(E_c)$ . This  $E_c$  was used to successfully predict the scrape-back length of cylinders of RBC for various incident energies and shades of RBC. While the ECR, as described, has been shown to have predictive capability that makes it useful, it has the limitation that it is valid only for the light source used to generate it. It would be beneficial to have an ECR that can be used with any light source to describe the conversion characteristics of RBC.

During photopolymerization, the spectral dependence of the energy absorbed by the RBC photosensitizer is a function of the product of the spectral absorbance of the photosensitizer and the spectral irradiance of the light source. This relationship will be different for each light source and could be used to define the relative energy efficiencies of different light sources. A universal ECR that could be used to describe RBC conversion characteristics for any light source by using the light source efficiency as an energy conversion factor might then be defined. Similarly, conversion characteristics obtained with one light source could be used to predict the characteristics for another light source by an energy conversion using the ratio of light source efficiencies.

The light source/photosensitizer combination most typically used to photopolymerize RBC is the tungstenhalogen incandescent lamp and camphorquinone (CPQ). The emission of tungsten-halogen lamps designed for dental use is filtered to pass radiation of wavelengths between 400 and 500 nm, corresponding to the carbonyl absorbance of CPQ. There is, however, a significant variation in the spectral distribution of such lamps (Cook, 1982), presumably due to filter composition. Recently, lamps based on light emitting diodes (LED) have become available for dental use. This technology eliminates the need for filtration since the emission is electronically, not thermally, generated. The resulting spectral bandwidth of LED lamps is relatively narrow, with peak intensity typically occurring near the maximum absorbance of CPQ at 470 nm (Mills, Jandt & Ashworth, 1999; Kurachi & others, 2001).

The objectives of this investigation were to derive a universal energy-conversion relationship ( $ECR_u$ ) predictive of RBC conversion characteristics and suitable for use with any light source by means of an efficiency factor for the light source; to use this universal energy scale to predict scrape-back lengths for RBC polymer-

ized with both a tungsten-halogen and light-emitting diode (LED) light source and to experimentally verify those predictions; and to predict and experimentally verify a conversion profile for RBC polymerized with the LED light source.

### **METHODS AND MATERIALS**

### Energy Conversion Relationship for Photopolymerization with a Tungsten-halogen Lamp

Construction of ECR for a tungsten-halogen lamp was described previously (Halvorson & others, 2002) and will be summarized below. A small particle hybrid resin-based composite (RBC) (3M ESPE Z100 Restorative, shade A3.5, 3M ESPE, St Paul, MN, USA) was used throughout this study. The RBC was packed into a split stainless-steel mold producing a cylindrically shaped sample approximately 6 mm in diameter by 16 mm in length. Two stainless steel wedges positioned on opposite sides and along the full length of the mold permitted splitting of the cured sample down its length. Transparent polyester film was placed over the openings of the mold and a light guide from a tungstenhalogen lamp (XL 3000 Curing Lamp, 3M ESPE) was placed over one of the ends. The sample was exposed for 60 seconds with the lamp adjusted, using a variable transformer to match the nominal power density of 250 mWcm<sup>-2</sup> (13,300 mJcm<sup>-2</sup> actual energy density) for the LED lamp (Freelight, 3M ESPE). The radiation energy density of the curing lamp was determined using a power meter (Power Max 500D Laser Power Meter, Molectron Detector Inc, Portland, OR, USA) integrating the radiant power density over the exposure time. Power density was determined by dividing the measured power by the cross sectional area of the light guide. After storing at room temperature for 24 hours, the sample was split and the specimens dissected along their length for analysis using transmission FTIR microspectroscopy. Specimens were placed on a KBr disc and measured in transmission with a Nic-Plan Microscope combined with a Magna-IR 750 spectrometer (Nicolet, Madison, WI, USA) co-adding 90 scans at a resolution of 4 cm<sup>-1</sup>. Three cylinders were prepared and analyzed for each group with three to five specimens measured at each depth. Conversion was determined by measuring the decreasing absorbance of the methacrylate carbon double-bond vibration at 1638 cm<sup>-1</sup> using the aromatic skeletal absorbance from Bis-GMA at 1582 cm<sup>-1</sup> as an internal reference. Integrated areas of both peaks were determined using a standard baseline technique.

Transmittance ( $T=P/P_0$ ) was determined by polymerizing the RBC in 6-mm diameter stainless steel molds of various lengths. The polymerized sample, together with its mold, was placed on the detector of a power meter (351 Power Meter, UDT Instruments, Baltimore, MD, USA), centering the light guide of the curing lamp

over the mold and in contact with the sample. The power measured in this fashion (P) was divided by the unattenuated power ( $P_{o}$ ) obtained by placing the light guide in direct contact with the detector head. A minimum of three replications was done for each condition and a mean value was determined. Transmission as a function of thickness was determined by regression analysis of the data.

The energy exposure at depths  $(E_d),$  where FTIR specimens were dissected, was determined from the incident energy  $(E_o)$  and the transmittance  $(E_d = \%T_d \; x \; E_o).$  This permitted the conversion to be related to energy and thereby define an ECR for the test material.

#### **Lamp Efficiency**

The relative efficiencies of the tungsten-halogen and LED lamps were expressed relative to a hypothetical standard light source having uniform spectral output of unity over the range of interest. The output of this light source was multiplied by the CPQ spectral absorbance normalized to unity at its peak absorption. The area of this process defined a standard relative energy absorption and the standard light source, which, by definition, would have an efficiency of 1.0. The spectral emission curves of the tungsten-halogen and LED lamps were also obtained and normalized to unity. These curves were similarly multiplied by the normalized CPQ curve, yielding areas that, when divided by the standard area, gave the relative lamp efficiencies. The spectral emission curves of the tungsten-halogen and LED lamps were determined using a spectralradiometer (Model S2000, Ocean Optics, Dunedin, FL, USA). The spectral absorbance of CPQ was determined in ethanol using a UV-VIS spectrometer (Model 8452A, Hewlett Packard, Palo Alto, CA, USA).

#### **Universal ECR**

A universal ECR (ECR $_{\rm u}$ ) was constructed by defining a new energy scale for the ECR previously described for the tungsten-halogen lamp. The new energy scale was obtained by multiplying the previous scale by the efficiency factor of the tungsten-halogen lamp. This universal ECR represents the energy-conversion relationship for the hypothetical standard lamp described earlier.

### LED Conversion Depth Profile: Prediction from Universal ECR

Conversion throughout the length of a cylinder of RBC polymerized with the LED lamp was predicted from the relative efficiency factor for the LED lamp and the universal ECR. First, the localized energy density through the length of RBC was determined using the transmission properties of the RBC (as described above) and the LED incident energy density. The product of the local energy density and relative efficiency factor for the LED scaled the energy density to the uni-

versal ECR, where the conversion value was determined. The prediction was experimentally verified using the FTIR microspectroscopic method described

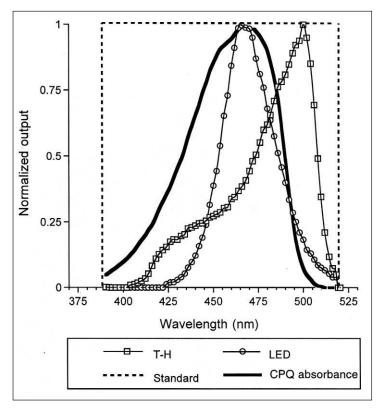


Figure 1. Normalized spectral emission of the tungsten-halogen and LED lamps together with the normalized spectral absorbance of CPQ. The standard light source with an output of unity is also shown.

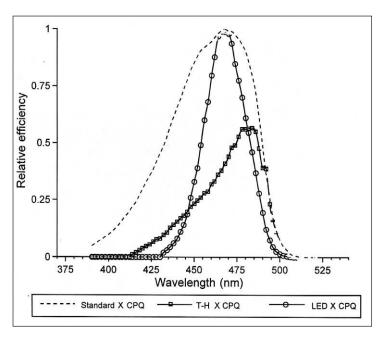


Figure 2. Spectral distribution of the relative energy absorbed by CPQ for each light source.

above. An incident exposure of 13,300 mJcm<sup>2</sup> was used (60-second exposure with the LED lamp), as measured with the power meter as described above.

#### Scrape-back Length: Prediction and Measurement

From previous measurements, a value of 32 mJcm<sup>2</sup> was found for the critical scrape-back energy for the test material when exposed using the tungsten-halogen lamp described above (Halvorson & others, 2003). This value was obtained by determining the local energy density at the scrape back length from the incident exposure and transmission data. Using the relative

efficiency factors and the critical scrapeback energy for the tungsten-halogen lamp, the critical scrape-back energy related to the LED lamp was predicted. This value was then used to predict scrape-back lengths for LED polymerized RBC at various energy densities. To experimentally verify the predicted scrape-back lengths, specimens were made by packing RBC into the cylindrical molds described above for FTIR sampling (without wedges). The RBC was exposed with the LED lamp at various energy densities achieved by using neutral density filters and modifying the exposure time. After 24 hours at room temperature, the molds were disassembled and the poorly polymerized material was gently scraped off with a rigid plastic spatula and the resulting cylinder length measured. Three replicates were prepared and measured to the nearest 0.01 mm. Scrape-back lengths were also predicted and experimentally measured for RBC polymerized with the tungsten-halogen lamp and were compared to the values obtained with the LED lamp.

#### **RESULTS**

Figure 1 shows the spectral distribution of the tungsten-halogen and LED lamps together with the spectral absorbance of CPQ, all on a normalized basis. The standard light source with an output of unity is also shown. Multiplying each light source by the CPQ absorbance yields the curves shown in Figure 2, which describe the spectral distribution of the relative energy absorbed by CPQ for each light source. The area corresponding to the standard light source provides the standard relative energy absorbed and defines an efficiency of unity. As expected, the areas for the LED and tungsten-halogen light sources are less than the hypothetical source, and division by the area of the standard source provides efficiency factors of 0.50 and 0.39, respectively.

Figure 3 shows the conversion profile of the RBC polymerized using the tungsten-halogen lamp with an incident energy density of 13,300 mJcm<sup>2</sup>. Transmission data was used to determine the energy density for each depth, allowing the ECR associated with the tungsten-halogen lamp to be generated as shown in Figure 4. Multiplying this ECR by the relative efficiency factor for the tungsten-halogen lamp (0.39) yields the universal ECR also shown in Figure 4. The predicted conversion profile of RBC polymerized using the LED lamp and the experimental values obtained with the same lamp

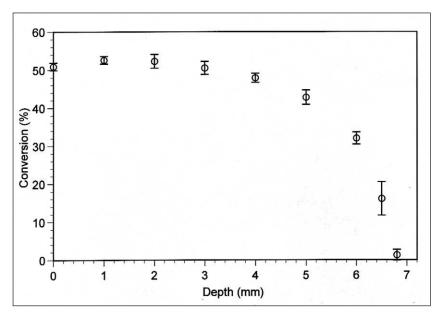


Figure 3. Conversion profile of the RBC polymerized using the tungsten-halogen lamp. Incident energy density was 13,300 mJcm<sup>2</sup>.

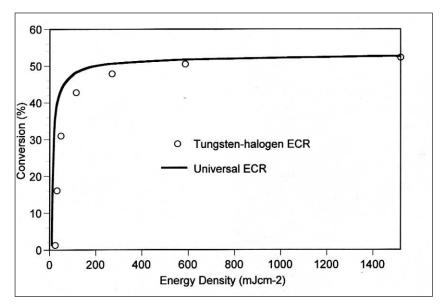


Figure 4. Energy conversion relationship (ECR) associated with the tungsten-halogen lamp. Multiplying this ECR by the relative efficiency factor for the tungsten-halogen lamp (0.39) yields the ECR for the standard lamp (universal ECR).

are shown in Figure 5. The conversion profile for tungsten-halogen polymerized RBC (Figure 3) is shown for comparison. The relative difference in lamp efficiency predicts the observed shift along the abscissa with equivalent energy exposures.

The relative difference in lamp efficiency also predicts a critical scrape-back energy of 24.5 mJcm<sup>-2</sup> for the LED lamp. Using this value, the predicted scrape-back curve for the LED lamp is shown in Figure 6a, together with the predicted curve for the tungsten-halogen lamp and experimentally measured scrape-back lengths for four different exposure energies. In Figure 6b, the scrape-back values are represented on the universal energy scale. The critical scrape-back energy for the universal energy scale corresponding to this curve is 16.3 mJcm<sup>-2</sup>.

#### DISCUSSION

The results of this investigation have shown that a universal energy-conversion relationship (ECR<sub>11</sub>) can be defined for a light-cured RBC that is predictive of conversion for any lamp by characterizing the spectral efficiencies of the curing lamps relative to a standard light source. The choice of standard light source is somewhat arbitrary, but the one chosen allows for light sources that could possibly approach an efficiency of 1.0 but cannot exceed that value. The area for the standard light source is, by definition, simply the area under the normalized CPQ absorbance curve. It is implicit under this process that the quantum yield for free radical generation is wavelength independent. Therefore, at low conversions, conversion versus wavelength should mimic the CPQ spectral absorbance curve when equal quanta per wavelength are incident on a sample. This has generally been observed for RBC

under exposure conditions yielding submaximal conversion (Nomoto, 1997).

The ECR produced in this experiment, using the tungsten-halogen lamp (Figure 4), was in very close agreement to the ECR described previously with the same lamp (Halvorson & others, 2003). While this ECR is sufficient to predict conversions for the LED lamp by comparing the LED lamp efficiency relative to the halogen lamp, it is more useful to express the ECR with respect to the standard lamp described. The reason being that others do not have access to this specific tungsten-halogen lamp, whereas, the standard light source proposed and the ECR<sub>11</sub> for this specific RBC family can be used by anyone. It is only necessary to determine the relative efficiency of any light source being used and the prediction of curing characteristics that can be calculated. From the relative efficiencies of the lamps, it is predicted that, with an equivalent exposure, the LED lamp will cure RBC to greater depths than the tungsten-halogen lamp (Figure 5). The experimental results verify this prediction. Due to the exponential decay of light through RBC, the 31% greater measured efficiency for the LED lamp does not result in a proportional increase in cure depths. This is observed from inspecting the conversion profiles in Figure 5 and comparing the scrape-back depths in Figure 6a, the latter showing only a 6% greater scrape-back length for the LED lamp. Similar differences in cure depths have been reported for RBC polymerized with approximately equivalent doses from an LED and tungsten-halogen lamp (Mills & others, 1999).

Although efficiency from the LED lamp is greater than the tungsten-halogen lamp, a benefit in cure depth is only realized when the tungsten-halogen lamp is operated at reduced power levels. Since the tungsten-halogen lamp was operated at roughly 50% of its maximum output, its cure times could be reduced by approximately half when operated at full output and it can achieve the corresponding cure depths reported here, which are consistent with the reciprocity previously determined (Halvorson & others, 2002). Therefore, lamp efficiency, as defined here, together with maximum irradiance, must be considered when evaluating the ultimate curing potential of different lamps. A measure of the photocuring efficiency inclusive of lamp irradiance has been derived from a kinetic expression for depth of cure (Cook, 1986).

Prediction scrape-back lengths found in this experiment for RBC polymerized with the tungsten-halogen lamp verifies previous results where a critical scrape-

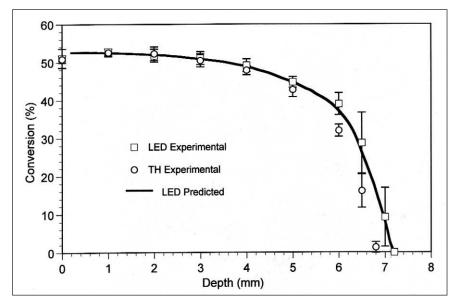


Figure 5. Predicted conversion profile of RBC polymerized using the LED lamp and the experimental values obtained with the same lamp. The conversion profile for tungsten-halogen (TH) polymerized RBC (Figure 3) is shown for comparison.

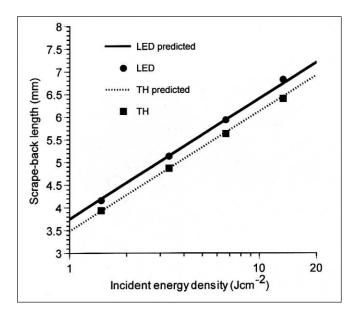


Figure 6a. Predicted scrape-back lengths for the tungsten-halogen (TH) and LED lamps together with experimentally derived values.

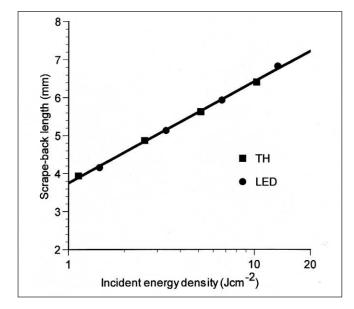


Figure 6b. Scrape-back lengths measured for tungsten-halogen and LED lamps plotted after converting the incident energies of the lamps to that of the standard lamp.

back energy of 32mJcm<sup>2</sup> was used (Halvorson & others, 2003). From relative lamp efficiencies, a critical scrape-back energy of 24.5mJcm<sup>2</sup> for LED predicts slightly greater scrape-back lengths for an equivalent exposure as shown in Figure 6a. For both lamps, the scrape-back length corresponds to approximately a 20% conversion as observed in Figure 4 and is consistent with previous results (Halvorson & others, 2003). Scaling the exposure energy with respect to the hypothetical lamp places the scrape-back values for both lamps on the universal energy scale and the scrape-

back lengths for both lamps converge on a single curve (Figure 6b). This curve represents a critical scrape-back value of 18mJcm<sup>2</sup>. Using this value, together with relative lamp efficiencies, provides a means for obtaining the critical scrape-back value for other lamps and, consequently, predicting associated scrape-back lengths.

Predicting the conversion and depth of cure from ECRs and critical scrape-back lengths presented here are expected to be subject to similar restrictions described previously (Halvorson & others, 2003). These restrictions involve, primarily, the effect of different mold geometries and mold composition on depth of cure (Fan & others, 1984) and differences due to polymerizing at other temperatures (Maffezzoli & others, 1994).

#### **CONCLUSIONS**

A universal energy-conversion relationship has been described, which is predictive of conversion of a resinbonded composite (RBC) polymerized with any light source. The universal energy scale has also been described as predictive of scrape-back lengths for this RBC family when polymerized with any light source. Both predictions rely on characterization of lamp efficiencies in comparison to the described hypothetical light source.

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#### Clinical Technique/Case Report

## **Custom-Made Resin Post and Core**

N Velmurugan • A Parameswaran

#### **Clinical Relevance**

This paper describes a single sitting chairside procedure for fabrication of a custommade resin post and core.

#### **SUMMARY**

Resin posts are used more often than metal posts, as they are esthetically compatible and less damaging to remaining tooth structure. Many resin post systems are available, where a light transmitting post is used to build up the composite in layers. An easier and economical alternative method, a custom-made resin post, is described.

#### INTRODUCTION

Post and core is done to replace missing coronal tooth structure to provide the required retention and resistance form for final restoration. Posts are of two basic types—ready-made and custom-made and may be either metal or resin posts. Use of metal posts has declined, as they lead to unsalvageable fractures due to a wedging effect and their metal shadow is difficult to mask. Resin posts are esthetically compatible and, as they are bonded to the root dentin, the outcome is a better stress distribution over the root surface (Freedman, After preparing the post space, a conventional direct wax pattern is taken (Figure 1). A solid metal sprue of suitable size (tapered end) is selected and serrations are made for wax retention. Softened inlay wax is added in increments to the metal sprue, which is inserted into the post space preparation. This procedure is repeated until the size and shape of the entire post space preparation is duplicated. An elastomeric impression of this wax pattern is made (Figure 2). After removing the wax

2001). Ready-made resin posts systems use a light transmitting post to build composite in multiple layers

(Layered adhesion technique) (Glassman & Seroto,

1998; Freedman & others, 1994). A simple alternative

may be thought of by fabricating a custom-made resin

**PROCEDURE** 

post as described below.

material (Figure 4).

pattern, resin composite is packed into the mold space that has been created and is cured (Figure 3). Additional

curing can also be done after sectioning the impression

After trial fit, a suitable bonding agent is selected. As conventional etching and rinsing procedures might be difficult to perform within the root canal, a sixth generation dentin-bonding agent (Prompt-L-Pop), which has a self-etching primer, was used. The custom-made resin post was luted using a dual cure resin luting cement (Rely X-3M) (Figures 7 and 8).

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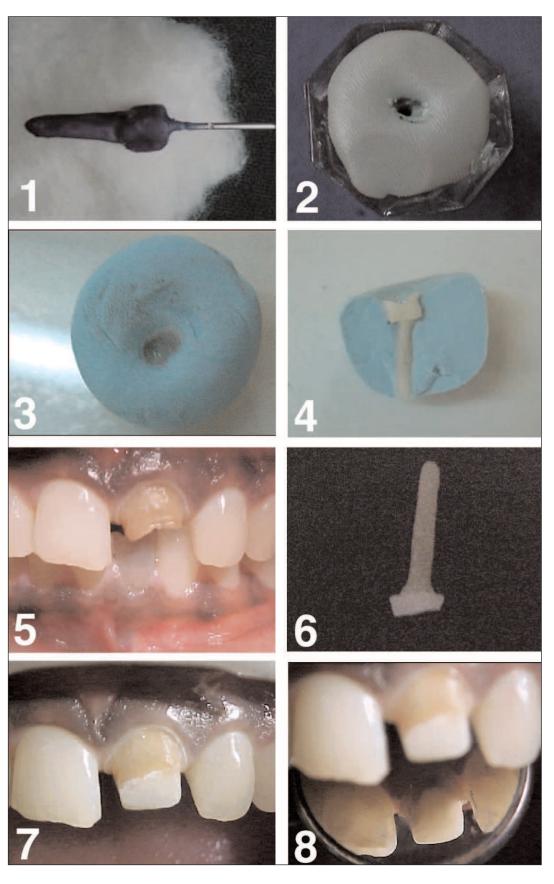


Figure 1 (left). Direct wax pattern of a post space preparation.

Figure 2 (right). Elastomeric impression of wax pattern.

Figure 3 (left). Resin composite packed and cured.

Figure 4 (right). Additional curing after sectioning of impression material.

Figure 5 (left). Tooth No 21 requiring post and core.

Figure 6 (right). Custom-made resin post and core made for tooth 21.

Figure 7 (left). Custom-made resin post and core bonded using dual cure resin luting cement.

Figure 8 (right). Palatal surface of resin post and core.

#### CONCLUSIONS

In ready-made resin post systems, a light transmitting post is used to build the composite in layers (Layered adhesion technique) (Freedman & others, 1994), resulting in multiple composite interfaces. After removthe ing Light Transmitting Post, a ready-made post selected and cemented. Fabricating a custommade resin post and core is easier, time saving and economical. The custommade resin post and core is esthetically compatible and is bonded to the root dentin, resulting in a single monobloc (Toepke & Grajower, 1986) that helps in better stress distribution. This resin post is a custom-made, single unit post and core that eliminates the number of composite interfaces that occurs during the layered adhesion technique.

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#### **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.

In vitro study of endodontic post cementation protocols that use resin cements. \*Varela SG, Rabade LB, Lombardero PR, Sixto JML, Bahillo JDG & Park SA (2003) Journal of Prosthetic Dentistry 89(2) 146-153.

(University of Santiago de Compostela, Faculty of Dentistry, Department of Pathology and Therapeutics, Galicia, Spain)

Successful bonding of intraradicular posts with resinbased cements depends upon adhesion produced by the hybrid layer, resin tags and adhesion obtained in the specific area of the dentin. Acid etched and deproteinized dentin are markedly different from solely acid etched dentin. Resin bond adhesion and tensile strength may be significantly affected by removal of demineralized collagen via NaOCl treatment. This *in vitro* study assessed the effect of NaOCl on the resindentin interface and compared the tensile bond strengths of posts cemented utilizing different resin bonding protocols with or without NaOCl treatment

A total of 120 single-rooted, caries free human teeth were decoronated and received endodontic treatment via the Profile system, 10% NaOCl, Top Seal and gutta percha. Post space was prepared with Gates Glidden drills leaving 2-5 millimeters of apical gutta percha remaining. The periconductual dentin of all 120 specimens was etched with 37% orthophosphoric acid for 60 seconds to expose collagen fibers and remove the smear layer plugs. The teeth were divided into two groups (n=60): Group I (no NaOCl treatment) and Group II (post-etch treatment with 10% NaOCl for 60 seconds followed by a 10 second distilled water wash prior to applying bonding agents.) Four different cementation protocols were evaluated for each group (n=15): Panavia 21 Ex cement alone; Dual Cement alone: Panavia 21 Ex cement with ED Primer dentin adhesive or Dual Cement with ED Primer dentin adhesive. In each specimen, the resin tags, hybrid layer over the periconductual dentin and post surface were analyzed by SEM. Then, the specimens were loaded uniaxially with an electromechanical testing machine until cement failure to determine bond adhesion and tensile strength. Statistical analysis of the data was performed with multiple analysis of variance to a significance level of p < 0.05 in all experiments.

Morphologic differences were noted via SEM as follows: Group I revealed tapered hollow tags and Group II showed cylindrical, solid tags. The diameter of the tubule orifice was increased after NaOCl treatment of the acid-etched dentin due to the loss of peritubular dentin and a resultant increase in intertubular dentin. The uniaxial tensile strength tests demonstrated that Panavia 21 Ex was strongest in Group I but weakest in Group II, where Dual Cement with adhesive was the strongest. In Group II, the decreased bond strength of Panavia 21 Ex was improved with the use of dentin adhesive.

#### **Conclusions**

ED Primer dentin adhesive improved the tensile bond strength of resin cements. No significant differences existed between Panavia 21 Ex and Dual Cement. The combination of NaOCl and ED Primer dentin adhesive increased the tensile bond strength of resin cements and dentin in endodontically treated teeth. However, the NaOCl and Panavia 21 Ex combination without ED Primer decreased the tensile bond strength. The tensile bond strength was improved by resin tag presence. NaOCl treatment created solid, cylindrical tags with minimal lateral branches due to odontoblast elimination, whereas, hollow, tapered tags with a multitude of lateral branches resulted when NaOCl was not used.

In vitro testing of xylitol as an anticariogenic agent \*Sahani PS, Gillespie MJ, Botto RW & Otsuka AS (2002) General Dentistry 50(4) 340-343.

(Indiana University in Indianapolis, IN, USA)

This study determined the concentration of xylitol required to inhibit the growth of S *mutans*, S *salivarius and* S *sanguis*.

Strains of S *mutans* (ATCC 35668), S *salivarius* (ATCC 13419) and S *sanguis* (ATCC 10556) were purchased from the American Type Culture Collection that was initially cultured from the oral cavity. The bacteria was subcultured and stored frozen at –7°C. An 0.3 mL inoculum of each bacteria culture was aseptically transferred to 10 mL of brain heart infusion (BHI) as a control. Additional 0.3 mL inoculums were transferred to BHI containing xylitol concentrations ranging from 0.78-50%. Growth was determined with

a Varian-Cary spectrophometer measuring optical dentistry at 600 nm. Each sample was performed twice and contamination was tested by using random culture samples. S *mutans* was the only bacterium that was inhibited by 1.56% xylitol concentrations. Statistically significant inhibition (p<0.05) was found between S *mutans* and the control at 1.56% xylitol concentrations. It required xylitol concentrations of 12.5% to significantly inhibit S sanguis and S salivarius to the same level as S mutans with 1.56% xylitol concentrations. The 25% xylitol concentration inhibited all bacteria sample growth by 30%. Increased inhibition with increasing concentrations was observed to 75% growth inhibition at 50% xylitol concentrations. The results indicate that normal oral streptococcal flora would not be adversely affected by low concentrations of xylitol and only the cariogenic bacteria have growth inhibition at low levels. Initial high concentrations of xylitol in the oral cavity would be lowered by saliva and swallowing. Since the lower xylitol concentrations only affect S mutans, it would benefit the oral cavity by possibly altering the oral flora to a less cariogenic bacteria. The low concentrations of xylitol would be able to inhibit the S mutans but would be less likely to cause intestinal distress in xylitol sensitive patients.

Shear bond strength of resin cements to both ceramic and dentin Stewart GP, Jain P & Hodges J (2002) *Journal of Prosthetic Dentistry* 88(3) 277-284.

(\*School of Dental Medicine, Southern Illinois University, Alton, IL and School of Dentistry, University of Minnesota, Minneapolis, MN, USA

This study evaluated shear bond strengths between a feldspathic ceramic and four resin cements using six different surface-conditioning treatments. Shear bond strengths of the four resin cements to dentin were also measured. Four hundred and eighty Ceramco II ceramic discs 10 mm in diameter were prepared. They were divided into six groups of 80. All groups were sanded with 600-grit silicone carbide paper (SiC). Group 1 was the control, sanding only (SiC). Group 2— Microetching (ME). Group 3—Silane solution (Sil). Group 4—Microetch and Silane (ME + Sil). Group 5— 9.6% Hydrofluoric acid (HF). Group 6—Hydrofluoric acid and Silane (HF + Sil). Ceramic-cement bond strengths were tested at 24 hours and six months for four different resin cements (Nexus, Panavia 21, RelyX ARC and Calibra) on each of the six ceramic surfaces. Dentin-cement bond strength testing was performed on 60 extracted molars divided into six groups. Prodigy resin composite was bonded to dentin utilizing the following protocols: Groups 1-4 used the four resin cements noted above with their associated dentin bonding agents. Group 5 used Calibra with Prime and Bond NT without auto-polymerizing activator. Group 6 used Nexus with Optibond Solo Plus light-polymerized dentin bonding agent. All shear bond strength testing was performed using an Instron universal testing machine at a crosshead speed of 5 mm/minute. The ceramic surfaces were examined under SEM to determine the effects of four surface treatments: 1) sanding with 600-grit SiC paper, 2) sanding followed by 36% phosphoric acid etch, 3) sanding followed by 50-µm alumina microetch and 4) sanding followed by 9.6% hydrofluoric acid etch. Post hoc comparisons were presented on the basis of two-way ANOVAs that compared the standard surface treatment (HF + Sil) to the other surface treatments separately for each cement. In most cases, the porcelain surfaces treated with HF + Sil yielded significantly greater ceramic-cement shear bond strength than the other surface treatments. Regardless of the resin cement used with HF + Sil treated porcelain, the ceramic-cement bond strengths were essentially similar at 24 hours and at six months. For dentin-cement bonding, auto-polymerizing agents vielded greater bond strength than light polymerized, which, in turn, were stronger than dual polymerized agents. SEM study showed HF + Sil porcelain surface treatment yielded the most surface relief with severe undercuts. HF + Sil treatment yielded the most effective and reliable bond strengths (16.0 MPa to 21.7 MPa at 24 hours, 15.9 to 21.8 MPa at six months) no matter what cement was used. At 24 hours silane, alone, gave higher bond strength than HF, alone. At six months, silane, alone, gave bond strengths close to HF + Sil. Single mode polymerization (light cure or auto cure) dentin bonding agents yielded significantly greater dentin-cement shear bond strength than dual-polymerizing agents. It may be concluded that the greatest bond strength for bonded ceramic restorations is found with a combination of hydrofluoric acid etch, silane and an auto or light-polymerized dentin bonding agent.

Effect of cyclical lateral forces on microleakage in cervical resin composite restorations \*Fruits TJ, VanBrunt CL, Khajotia SS & Duncanson MG (2002) *Quintessence International* 33 205-212.

(\*University of Oklahoma, College of Dentistry, Department of Operative Dentistry 1001 Stanton L Young Boulevard Oklahoma City, OK 73190, USA)

This study assessed the effect of cyclic lateral fatigue forces on microleakage in three different types of cervical resin composite restorations. Lee and Eakle were the first to suggest that lateral occlusal forces on teeth could be the causative factor in the development of non-carious cervical lesions. Alternating compressive and tensile stresses caused by the flexure of teeth in the cervical area can cause the enamel and dentin in the area to crack and slowly erode. Sixty extracted human premolars were used in the study. Notchshaped preps were made on the buccal surfaces using a 125 µm flame shaped-diamond bur with water coolant. The occlusal surface was prepared in enamel, the gingival aspect was placed 1.0 mm apical to the CEJ. All Bond 2 total etch adhesive bonding system was used. The resin was placed in three increments. The teeth were randomly divided into three groups of 20 teeth. The three types of resin composite used were: 1) hybrid resin composite (TPH), 2) microfilled (Silux Plus) and 3) Flowable (Aeliteflo). Ten teeth in each group of 20 were subjected to cyclic fatigue stresses. Four polyethylene bumpers placed on the perimeter of a drum were used to cyclically stress the teeth. A force with a displacement of 0.020 inch at 44N was applied in the buccal and lingual directions. Each specimen was subjected to 8,400 cycles. The specimens were stored in 50% silver nitrate for 48 hours at 24°C, rinsed and stored in x-ray developing solution for 12 hours while exposed to fluorescent light, then placed in photographic fixing solution for 60 minutes. The specimens were sectioned sagitally and examined under 10x magnification for leakage of silver nitrate stain between the resin-tooth interface. The specimens were rated on a scale used by Willer and others of zero to six.

Statistical analysis was performed with a Kruskal-Wallis analysis and Wilcoxon rank sums of scores. Results showed that the lower modulus of elasticity (MOE) of flowable and microfills allowed more flexibility. The lower MOE of a resin composite, the more it is able to absorb stresses induced by tooth flexure. The MOE of the three resins used were: Flowable (3.6 Gpa), Microfill (6.0 Gpa) and Hybrid (10.5 Gpa). Microleakage along the resin/enamel interface was not significant for the three groups. The non-fatigued control specimens showed some signs of leakage, perhaps due to polymerization shrinkage. There was a greater tendency for resin composite to pull away from the margin located in dentin and cementum whether or not the specimens were subjected to cyclic loading. For non-fatigued specimens, hybrid resin had significantly more leakage at the dentin-resin interface. In the fatigued group, the hybrid resin had significantly more leakage than the microfilled resin.

#### Conclusions

The hybrid resin composite generally exhibited more leakage, suggesting that the modulus of elasticity of resin materials used to restore cervical lesions may be related to the amount of microleakage observed.

Effect of A Fluoride Varnish on The Margin Leakage and Retention of Luted Provisional Crowns. Lewinstein I, Fuhrer N & Ganor Y (2003) The Journal of Prosthetic Dentistry 89(1) 70-75.

(The Maurice and Gabriela Goldschleger School of Dental Medicine, Tel Aviv University, Tel-Aviv, Israel)

Long-term provisional crowns are susceptible to marginal leakage and caries due to cement solubility and bacterial infiltration. Provisional cements require enough retention to keep the provisional crown in place and yet allow for easy removal. Due to the nature of the material and technique to fabricate provisional crowns, the marginal gap could be significant and predisposed to marginal leakage and insufficient retention. This study examines the effect of Duraphat varnish (contains 2.26% NaF and pH of 5.5) and two provisional cements on marginal leakage and retention of provisional crowns.

Eight human molars were prepared for full coronal coverage with shoulder margins and acrylic resin provisional crowns were fabricated. Testing was completed in three phases. Phase 1: The crowns (N=24) were individually cemented with Temp-Bond (TB), Freegenol (FG) or Duraphat (DU). Specimens were themocycled 500 times (5° and 60°C) with one-minute dwell time and stored in 100% humidity at 37°C for six days. After soaking in 0.5% Gentian violet solution for 24 hours, the shear retention test and the marginal leakage tests were performed. Phase 2: After dislodgement, the provisional crowns (N=16) were recemented with Duraphat varnish without removing the residual provisional cement from the crowns. Phase 3: Crowns were relined with 0.5 mm of acrylic (N=16) and cemented with TB and FG provisional cements mixed with Duraphat at a predetermined ratio. Crowns with no cement (N=8) served as control.

The retention forces for all luting cements used individually showed no statistically significant difference. Duraphat used with Temp-Bond increased the sevenday retention of provisional crowns by 69% to 145%. Marginal leakage was found in all groups. Temp-Bond showed less marginal leakage than Freegenol. Duraphat used with either provisional cement during recementation and cementation with the mixture groups resulted in reduced marginal leakage. Duraphat used with Temp-Bond reduced marginal leakage more than with Freegenol. Duraphat used alone resulted in the least marginal leakage.

#### **Conclusions**

This study suggests that Duraphat can be used as a provisional luting cement for single provisional crowns; however, clinical testing is needed.

Fracture resistance of endodontically treated teeth restored with composite posts \*Newman MP, Yaman P, Dennison J, Rafter M & Billy E (2003) *The Journal of Prosthetic Dentistry* 89(4) 360-367.

This study compares three types of composite post systems and a stainless steel control in narrow and flared canals.

Ninety maxillary central incisors were divided into eight experimental groups and one control group of 10 teeth each. Clinical crowns were sectioned 2 mm above the CEJ. Tooth roots were coated with a polyvinylsilicone coating to simulate the PDL and mounted in acrylic blocks at a 45° angle. The teeth were endodontically instrumented with conventional step-back technique to an ISO size 60 file, then obturated with gutta percha. To form a post space, 10 mm of gutta percha was removed with specific drills supplied with the individual kits. Forty sample teeth were prepped with wide post spaces 2 mm in diameter at the orifice. The canals were treated with 3M Scotchbond Multi-Purpose dentin bonding agent. All posts were 17-mm long. The specimens were divided into "Narrow" and "Wide" test groups. The "Narrow" canal groups consisted of: 1.1) FibreKor with Cement-It, 1.2) Luscent anchor with Cement-It, 1.3) Ribbond with Flow-It light-cured and 1.4) ParaPost XH (control) with Cement-It. "Flared" canal groups consisted of: 2.1) FibreKor with Flow-It auto-cured, 2.2) Luscent anchor with Flow-It auto-cured, and 2.3) Ribbond with Flow-It light-cured. The Ribbond samples in this study were fabricated with standardized coronal dimensions (1.6 mm diameter x 5 mms and 2.0 mm diameter x 5 mms for narrow and flared canals, respectively.) Twenty non-standardized Ribbond samples were excluded from the analysis, because varying core sizes yielded high standard deviations. An Instron universal testing machine loaded the sample at 0.05 cm/minute until failure occurred.

Mean failure for the narrow canal groups was 9.7 kg for FibreKor; 12.9 for Luscent Anchor; 4.55 for Ribbond standardized and 18.33 for ParaPost. Mean failure for the flared canal groups was 9.4 for FibreKor; 12.87 for Luscent Anchors and 12.87 for Ribbond standardized.

There was no statistically significant difference between narrow and flared canals for the FibreKor and Luscent Anchor posts; however, Ribbond was significantly weaker in narrow canals. There was a statistically significant difference between posts systems except for Luscent Anchors and Ribbond in flared canals. The mean failure range for the three fiber-reinforced composite posts in both narrow and flared canals were between 4.55 kg to 12.9 kg. The ParaPost failure averaged 18.33 kg.

Failure modes were also evaluated. No root fractures occurred in any of the fiber-reinforced composite posts. Three root fractures occurred in the cervical third of the stainless steel control.

The taper of the canals did not alter the outcome of fracture. Stainless steel posts were significantly stronger than composite posts. Composite posts did not fracture roots.

Three-dimensional analysis of dual-arch impression trays \*Cayouette MJ, Burgess JO, Jones RE & Yuan CH (2003) *Quintessence International* 34(3) 189-198.

(\*MUSC College of Dental Medicine, Department of Prosthodontics, 173 Ashley Avenue, BSB 546, Charleston, SC, USA)

The objective of this study was to determine the impression accuracy of four different impression trays using vinyl polysiloxane (VP) and polyether (PE). A master model Dentoform with ivorine teeth was prepared with 32 selected reference points in the shape of grooves and notches. Four teeth in one quadrant were prepared. The four impression trays employed were a plastic full arch stock tray, a triple tray (TT), a metal reinforced rigid dual-arch tray and a triad custom tray. For each tray, 13 impressions were made with VP and 13 with PE for a total of eight groups of 13 impressions each. Impressions were poured 48 hours after removal from the master model with vacuum mixed type IV stone and separated 45 minutes after they were poured. The reference points for each model were measured with a measuring microscope after dusting with tungsten powder to enhance topographical contours. Previous studies have suggested that dual-arch impression trays are as accurate or more accurate than custom trays. In this study, only the custom tray provided casts identical to the control. Casts made using a custom tray with PE or VP and the Triple Tray with PE showed no inaccuracies. PE resulted in positive error, that is, stone cast larger than the control, whereas, VP resulted in negative error.

#### **Conclusions**

Clinically acceptable casts can be obtained from VP or PE, and any combination of custom, stock or dual-arch impression trays. PE was the best overall impression material. The custom tray was more accurate than other impression techniques.

#### **Announcements**



## 33<sup>rd</sup> ANNUAL MEETING of the ACADEMY OF OPERATIVE DENTISTRY

18-20 February 2004, Fairmont Hotel, Chicago, IL

The Academy of Operative Dentistry's 33<sup>rd</sup> Annual Meeting once again offers an incredible group of essayists, an outstanding table clinic session and a wonderful social program.

SCIENTIFIC SESSION: Thursday's program begins with Dr Debra Gander speaking on "Panoramic Radiography—Enhancing Your Interpretation Skills' followed by Dr J William Robbins discussing the concept of "Biological Width." This year's Buonocore Memorial Lecturer is Dr Reinhard Hickel, who will offer an interesting look at "Classification and Clinical Results of Filling Materials." Thursday afternoon features Dr John Osborne's presentation on "Mercury and Other Illusions of Life" and Dr Werner Geursten offers answers to the question "Restorative Resins: Can These Wonderful Materials Generate Adverse Effects?" The afternoon session will close with a Panel Discussion Program with Drs Osborne and Geursten answering your questions.

Dr Henry Gremillion leads off on Friday morning with "Diagnostic Dilemmas: The Many Faces of Orofacial Pain" and Dr Gordon D Douglass concludes the essay program with "Occlusion: The Foundation of Patient Care." Friday afternoon's exceptional group of table clinics organized by Dr Frank Caughman will complete the 2003 Scientific Session.

COMPANION PROGRAM: The Companion Activities Program is particularly exciting this year. Thursday offers "A Day at Marshall Field's" (at the State Street Flagship Store), which includes make-up demonstrations, morning coffee and sweets, style show, a tour of Marshall Field's Trend House showcasing furniture and decoration ideas and a three-course lunch in the beautiful Walnut Room.

Friday morning features a "Continental Buffet Breakfast at the Fairmont" featuring Harris Kal, "The Game Show Master." Mr Kal is a professional actor who has studied at both the Goodman Theater and Chicago's Second City. You won't want to miss his unique and fun-filled game show extravaganza.

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 don't miss this fantastic opportunity for education, information exchange and fun. See you in Chicago in February! For more meeting information, please contact: Dr Gregory Smith, PO Box 14996, Gainesville, FL, USA 32604-2996; FAX (352) 371-4882.

## Operative Dentistry Home Page



We hope all our readers will take advantage of the information available by accessing our Internet home page. Our address is: http://www.jopdent.org/

The home page contains a search engine and buttons that, hopefully, will lead you to answers to any questions you may have related to Operative Dentistry. These are:

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#### **Exclusive Publication**

All material submitted for publication must be submitted exclusively to *Operative Dentistry*. Manuscripts not following the form outlined below may be returned for correction and resubmission.

#### **Manuscripts**

- □ Submit an original typed manuscript and three copies. The manuscript should include a short title for running headlines. Any identifying information (author's names, etc) should be on a separate page and not a part of the manuscript. Authors with English as a second language should consider having their manuscript reviewed for grammar, syntax and punctuation prior to submission.
- $\ \square$  Submit a computer disk and identify the operating system (Macintosh or IBM-compatible) and the word processing program used.
- □ Identify the corresponding author and provide a complete address, fax number and e-mail address.
- $\square$  Supply complete names, degrees, titles and affiliations for all authors (include addresses that are different from the corresponding author's).
- $\Box$  Proprietary names of equipment, instruments and materials should be followed in parenthesis by the name, company, city and state or country of the source or manufacturer.
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- □ Type double-spaced, including references, and leave margins of at least 3 cm (1 inch). Spelling should conform to the *American Heritage Dictionary of the English Language*. SI (Système International) units are preferred for scientific measurement, but traditional units are acceptable.
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