

# OPERATIVE DENTISTRY



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# Whatever Lola Wants...



**T**he customer is always right" has been an axiom of consumer merchandising for as long as I can remember (although we have all seen a decline in actual practice). I also teach my Graduate Operative Dentistry residents that the primary goal of cosmetic dental procedures is to make the appearance of the teeth/smile more pleasing to the patient, and that patient satisfaction is one of the most important keys to a successful practice. I am beginning to question, however, the extent to which patient demands are controlling treatment decisions and the ramifications of our own complicity in promoting cosmetic therapies that may not be in the best interests of oral health.

"Cosmetic" dentistry is a big business in today's dental marketplace. Manufacturer advertising, media articles and professional presenters all extol the virtues of enhancing personal appearance through esthetic dental therapies. The positive side is that patients are much more cognizant of the importance of an attractive, healthy smile in both their personal and professional relationships. They no longer seek dental care only to relieve discomfort or to treat obvious carious lesions or trauma, but to correct discoloration and other dental imperfections that limit their confidence and willingness to smile when interacting with others. They are much more receptive to saving teeth and are willing to work with us to optimize their dental health and appearance. I truly believe that the various cosmetic dental treatments have been a boon to the dental profession and our patient population.

Unfortunately, there is so much media hype and emphasis on youth and glamour that patients' desires for and expectations related to cosmetic dentistry are frequently unrealistic and the limitations and long-term ramifications of the available treatment modalities are not clearly understood. The idea that all dental restorations must be tooth-colored and that all smiles

must be as even and as white as possible has permeated the national dental consciousness and is reinforced by what our patients see on the screen, read in magazines and hear on talk-shows and "infomercials." Although beauty is certainly in the eye of the beholder, this "eye" is heavily influenced by peer, social and environmental factors. As practitioners, we are under tremendous pressure to provide the kinds of treatment that our patients perceive will fit them into the modern ideal of attractiveness. This perspective has resulted in dental practices that advertise themselves as "metal-free" (a gross misnomer since our resin composites, glass ionomers and ceramics all contain metal ions) and in "bleaching" shades of dental materials that are lighter than the natural color spectrum of the adult dentition. It has also evoked the frequently heard comment from colleagues that certain types of treatment are performed because "their patients demand" tooth-colored restorations.

Treatment rendered strictly because of patient demands automatically raises the question of appropriateness of care. Do these demanding patients understand all the ramifications of what they are asking? Do they realize that the reduction of tooth structure necessary for an all-ceramic restoration is considerably greater than that for gold and could adversely impact the vitality of the tooth? Are they aware that our amazing dental adhesives are extremely technique sensitive and may not provide a good seal at cemental margins or in situations where good isolation is difficult? If they are demanding veneers to give them whiter teeth, do they understand that bleaching may be a less aggressive alternative? Do they truly desire a particular treatment option or are their demands a reflection of our own biases in dental treatment? I have had occasion to ask many patients why they felt the need for an all-ceramic crown or large resin com-

posite on a second molar only to be told it was because their dentist recommended the treatment. Sometimes these restorations have failed, and I have often found that such patients choose a non-tooth-colored replacement when presented with all the available alternatives.

Although patient satisfaction must be an integral part of every practice, as health care providers we cannot subjugate our responsibility to maintain the health and function of the oral environment strictly to satisfy our patients' perceptions of attractiveness. Today's demanding patient is also discriminating and intelligent and deserves to be honestly informed of all the available treatment options as well as their advan-

tages and disadvantages. The alternatives we recommend should be based on sound evidence with as little bias as possible. Cosmetic treatments and tooth-colored restoratives have a definite place in dental practice as long as we don't allow them to become the only "choice" for our patients. "It's not how you feel, it's how you look" should not become a practice credo, and the words from the old song, "Whatever Lola wants, Lola gets" are not an appropriate foundation for dental care.

Michael A Cochran  
Editor



# pH Stabilizing Properties of a Posterior Light Cured Resin Composite: An *In Vivo* Study

Y Chacko • L Lakshminarayanan

## Clinical Relevance

Posterior composite restoratives that stabilize the pH of saliva can be beneficial in countering acid demineralization of enamel.

## SUMMARY

This study evaluated, *in vivo*, the pH stabilizing properties of a posterior resin composite (Ariston pHc, Vivadent Ets, Schaan/Liechtenstein). Fifteen human subjects with four or more active carious lesions were selected. Their salivary pH, in relation to the occlusal surface of these lesions, was recorded. The teeth were restored with resin composite, and the pH in relation to the restorations was recorded one day, one week, one month and two months post-operative. Results showed that the resin composite countered the acidic pH of saliva and maintained

it at levels where demineralization would not occur.

## INTRODUCTION

Caries initiation is a complex physico-chemical interactive process; one of its earliest manifestations is demineralization of enamel. The primary damaging action results from a highly localized drop in pH at the plaque-tooth interface. Once the pH falls below 5.5, minerals are dissolved and there is a release of ions ( $\text{Ca}^{++}$  and  $\text{PO}_4^{-}$ ) into the plaque. Unless this pH drop (seen especially after a cariogenic meal) is buffered, demineralization of enamel can result over time.

The volume and buffering capacity of saliva available to tooth surfaces play a major role in caries prevention. Salivary pH is slightly acidic prior to secretion and becomes slightly alkaline on expulsion from the gland. Normal salivary pH ranges from 6.5 to 7.2. Salivary buffers, namely sodium bicarbonate, phosphate, amphoteric proteins and urea, serve to stabilize any sudden drop in pH. Of this, sodium bicarbonate is the main buffering and neutralizing constituent as reported by Kreusser & others (1972).

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Table 1																	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	Mean	Std dev
pre operative	5.5	5.5	5	5.5	5.5	5	5	5.5	5	5.5	5.5	5	5	5.5	5	5.2667	0.258
post op 1 day	7	6.5	5.5	6.5	6.5	7	6	6.5	6	7.5	6.5	6.5	6	7	7	6.5333	0.516
post op 1 wk	6.5	6.5	6	6.5	6	6.5	6	6.5	6	6.5	6	6.5	6	6.5	7	6.3333	0.309
post op 1 mth	6.5	6	6	6.5	6	6.5	6	6	6	6	6	6.5	5.5	6.5	6.5	6.1667	0.309
post op 2 mth	6	6	6	6.5	6	6.5	6	6	5.5	6	6	6	5.5	6.5	6.5	6.0667	0.319

Table 2				
	Change in Mean	Change in SD	Paired <i>t</i> Value	<i>p</i> -Value
pre-op to 1 day	1.27	0.46	10.72	<i>p</i> <0.001
pre-op to 1 week	1.07	0.37	11.12	<i>p</i> <0.001
pre-op to 1 month	0.9	0.39	9	<i>p</i> <0.001
pre-op to 2 months	0.8	0.37	8.41	<i>p</i> <0.001

According to Harris & Suddick (1991), as the pH drops, the level of calcium and phosphate ions in the saliva also drops and the possibility of demineralization increases. The presence of fluoride at remineralizing sites can accelerate the process by a factor of four or five, as reported by Feagen & others (1976). Silverstone (1984) found that using 1 ppm of fluoride along with calcium in a remineralizing solution showed a 72% reduction of the body of the carious lesion as compared to 22% when the fluoride ion was omitted. Ariston pHc is a recently introduced light cured resin composite that can leach hydroxyl, calcium and fluoride ions in response to a drop in pH. Its use as a restorative enables it to act as a reservoir for providing ions to prevent demineralization near the restoration (Kraft & Hoyer, 1999). This study evaluated *in vivo* the pH stabilizing properties of the posterior light cured resin composite–Ariston pHc.

## METHODS AND MATERIALS

Fifty human subjects between the ages 18–20 years were selected and screened for four or more active carious lesions in the posterior region. Twenty subjects were selected, and the pH in relation to the lesions was recorded using a chromatic pH indicating paper (Fine Chemicals Division, Glaxo Laboratories, Bombay, India). Fifteen subjects were selected from the 20 whose pH ranged from 5 to 5.5. Each patient's teeth were isolated under a rubber dam and high volume suction. Conventional Class I cavities were prepared and the cut surfaces dried with an oil-free air spray. Ariston liner (Vivadent, Schaan/Liechtenstein, Lot #A20266) was brushed over the cut surfaces, air-thinned and cured for 20 seconds. Ariston pHc (Vivadent, Schaan/Liechtenstein, Lot #A21827) was then placed in increments and each increment was

cured for 40 seconds. Occlusal carving and finishing was performed and the patient was put on recall for one day, one week, one month and two months after placing the restorations. On each of the recall visits, the pH of saliva was tested over the occlusal surface of the restorations using a chromatic pH testing paper (Glaxo, India). All the testing was done between 2:15 and 2:45 PM. The results were tabulated and the mean and standard deviation was calculated for each set of values Table 1). A *t*-test and Friedman two-way ANOVA were carried out on the mean difference between pre- and post-operative results to determine the level of significance.

## RESULTS

1. Both the *t*-test and Friedman two-way ANOVA gave statistically significant results (*p*<0.001) (Table 2).
2. There was a spike in the one-day post-operative pH with the mean difference between pre-operative and one-day post-operative results showing a change of 1.26 (Figure 2).
3. The mean pH showed a decline of more than two months from the initial spike but stabilized around 0.80 above the pre-operative value (Figure 1).
4. The mean difference between the pre-operative and each of the post-operative values was statistically significant (*p*<0.001) despite the decline in the pH stabilizing properties.

## DISCUSSION

The complex physical and chemical composition of saliva enables it to perform many functions. Among the various activities, it serves to dilute and remove acid concentration in plaque as reported by Boackle & Suddick (1980). The chemical protection afforded by saliva minimizes the pH drop, increases the resistance of the tooth surface to acid attack, accelerates the return of pH to normal and provides the ionic environment that facilitates repair of the enamel following demineralization. Sodium bicarbonate, phosphates, amphoteric proteins and urea are the neutralizing constituents of saliva (Kreusser & others, 1972). The cations and anions of saliva mostly associated with increasing the resistance of enamel to acid attack are

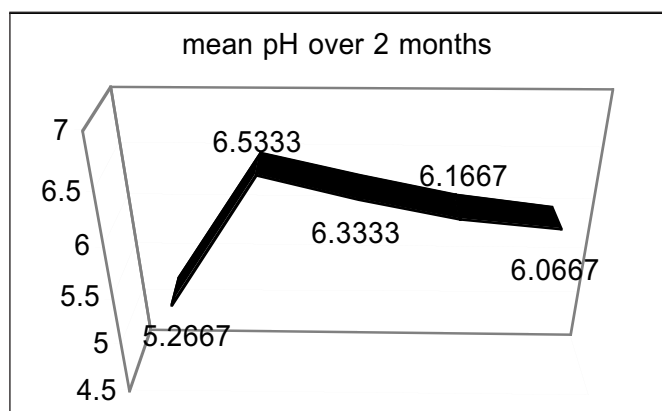


Figure 1.

calcium, phosphate and fluoride, according to Gron (1973). Plaque pH can drop to 4.0 after a glucose mouth rinse (Gibbons, 1989). Hence, after a sucrose meal, because of the pH drop, the possibility of demineralization increases. There is no exact pH at which demineralization of enamel begins, only a range from 5 to 5.5 identified as the “critical range.” This range occurs because demineralization is a function of both pH and duration of exposure of the enamel surface to the acid environment (Harris & Suddick, 1991). At this point, the calcium and phosphate ions that are released act as buffers and become available for reversing the demineralization process. This dynamic damage control process is set into action every time there is food intake, and homeostasis can only be maintained if:

- (1) the acidogenic challenge can be minimized in time and the pH drop controlled.
- (2) the saliva is normal in flow, ionic content and buffering capacity.

Salivary flow follows a circadian rhythm and is lowest around 4 AM and highest around mid-afternoon (Ferguson & Botchway, 1979). All the testing was done between 2:15 and 2:45 PM so that the results would reflect the best possible buffering capacity of the test subjects.

*In vitro* studies conducted by Johannsen (1965) have shown that the use of solutions containing calcium and phosphate ions induced remineralization in the presence of fluorides. Remineralizing solutions take effect depending on the time of interaction, extent of saturation, the rate of precipitation and the pH of the solution. When using a resin composite with similar characteristics, one eliminates the limit of time of interaction.

The three ions leached from Ariston pHc produce the following effects:

Fluoride ions: hamper demineralization, promote remineralization and inhibit bacterial growth.

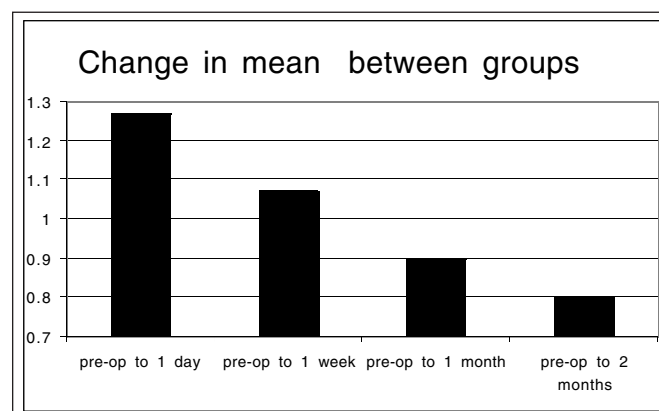


Figure 2.

Calcium ions: hamper demineralization and promote remineralization.

Hydroxyl ions: neutralize acids produced by cariogenic bacteria.

In a study done at the Dental Institute, University of Zurich, Switzerland, intraoral plaque telemetry was carried out on patients wearing test specimens of Ariston pHc on removable partial dentures to assess the buffering capacity of the resin composite (Imfeld, 1998). The results showed that the pH near the resin composite remained stable despite large variations of the surrounding pH.

One area of concern, while dealing with restoratives that have leaching characteristics, is the wear properties. In this regard, the abrasive behavior of Ariston pHc is comparable to that of modern fine-particle composites (Appert, 1998). The decrease in buffering capacity over the duration of the study also brings out another problem—the difficulty in controlling the rate of fluoride release (Skartveit & others, 1991). Further study is required, especially to correlate salivary flow to buffering capacity to get a more comprehensive view of this posterior resin composite.

## CONCLUSIONS

In light of this study, we can conclude that posterior restorative materials containing leachable ions ( $\text{Ca}^{++}$ ,  $\text{OH}^-$  and F) that are made available in response to a drop in pH, are beneficial in stabilizing the pH, thereby protecting the tooth from demineralizing acid attacks.

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# Diagnostic Accuracy of Intraoral Film and Direct Digital Images for Detection of Simulated Recurrent Decay

MK Nair • JB Ludlow • KN May  
UP Nair • MP Johnson • JM Close

## Clinical Relevance

PSP and CCD-based digital images provided a level of diagnostic performance comparable to Ektaspeed Plus film. Contrast and brightness enhancements improved digital image performance.

## SUMMARY

This study compared the diagnostic accuracy of bitewing images for detection of simulated recurrent caries using the following imaging modalities:

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ties: Ektaspeed Plus film and different digital imaging system technologies comprised of a charge-coupled device (CCD) based digital imaging unit, a photo-stimulable phosphor (PSP) based unit and contrast and brightness-enhanced PSP images.

**Study Design:** Twenty-four extracted posterior teeth with MOD inlay preparations were secured in models simulating a natural arrangement of teeth. Lesions were created in proximal boxes using dental burs of varying sizes. Defects were filled with wax and plaster and preparations were restored with composite or amalgam.

**Results:** Averages of receiver operating curve areas ( $A_z$ ) revealed diagnostic performances of  $A_z=0.74$  for film,  $A_z=0.80$  for CCD,  $A_z=0.73$  for unenhanced PSP and  $A_z=0.64$  for enhanced PSP. The differences between these means were significant (MANOVA  $p<0.0001$ ). Unenhanced PSP produced significantly poorer performance than other modalities. CCD performance was not significantly better than enhanced PSP. Lesions under radiopaque composite restorations were easier to detect, followed by those under amalgam and radiolucent composites across imaging modalities and lesion locations. Based on lesion

**location, those located at the buccal point angle were easiest to detect, followed by those at mid-gingival floor and lingual-point angle sites.**

**Conclusions: Contrast and brightness-enhanced digital images enabled better signal detection and a comparable performance with film for detection of artificially induced recurrent caries.**

## INTRODUCTION

Diagnosis of recurrent caries is challenging to the clinician. Recent studies compared the diagnostic efficacy of intraoral film with that of newer imaging techniques, such as tuned aperture computed tomography (TACT) (Nair et al, 1998b,c; Vandre & Webber, 1995; Webber et al, 1996; Webber et al, 1997). However, evaluation of photo-stimulable phosphor (PSP) sensors for this purpose has not been performed. This study compared the diagnostic efficacy of this type of imaging sensor with conventional intra-oral film and digital images acquired using a charge-coupled device (CCD).

Numerous factors are known to influence the detectability of recurrent dental caries in radiographs. These include proximity of the lesion to the adjacent restoration, size of the initial lesion, geometry of the radiographic projection and orientation of the lesion. Drawbacks of intraoral film image characteristics (Lundeen, McDavid & Barnwell, 1988; Espelid, 1986; Douglass et al, 1986), and lack of access to advanced imaging techniques such as tuned aperture computed tomography (TACT) may be cited as impediments to recurrent caries detection (Nair et al, 1998b,c). The prevalence of recurrent decay (Budtz-Jorgensen et al, 1996; Gilbert et al, 1996; Chestnut et al, 1995; Fitzgerald et al, 1994; McGuire et al, 1993; Mjör, 1996; Mjör & Qvist, 1997; Wilson et al, 1997) and the need for replacement restorations when this occurs (Wilson et al, 1997; Goldberg, 1990) emphasize the importance of radiographic evaluation (Espelid & Tveit, 1991) since most decision-making processes (Drake, Maryniuk & Bentley, 1990; Maryniuk, 1990; Merret & Elderton, 1984) rely greatly on radiographic images. Although film sensitivity and specificity (Espelid, 1986; Douglass et al, 1986) are relatively low, most clinicians use periapical and bitewing film for recurrent caries detection. Therefore, it is imperative that the diagnostic accuracy of recently introduced imaging options be compared to film to determine whether improvements may be made in the detection of recurrent caries. Previous studies have explored the application of PSP receptors in dental imaging, although none has evaluated recurrent caries detectability (Yoshiura et al, 1999; Brettle et al, 1996; Moystad et al, 1996; Conover, Hildebolt & Yokohama-Crothers, 1996; Kang et al 1998). These studies show promise of an imaging system capable of yielding a diagnostic performance comparable to that of film for various other diagnostic tasks.

The null hypotheses for this study was that the diagnostic efficacy for detection of mechanically simulated recurrent caries in proximal boxes of restored teeth is no different for conventional intra-oral film, enhanced digital CCD and enhanced and unenhanced PSP images.

## METHODS AND MATERIALS

This study used 24 maxillary (12) and mandibular molar teeth (12). Teeth were mounted four in a row in dental stone models with all the proximal surfaces in contact. Mesio-occluso-distal inlay preparations were created using a #271 carbide bur. This facilitated easy removal and replacement of the restorations. Lesions simulating recurrent decay were artificially induced using round burs sized #1/2, #1 or #2 to a depth of approximately half the diameter of the bur head (Nair et al, 1998b,c) and subsequently restored by an operative dentist. Lesions were randomly made in proximal boxes of 12 teeth, while the other proximal boxes on the remaining teeth served as controls. It was not necessary for the teeth to have lesions in one or both proximal boxes. There was a 50:50 chance of finding a lesion in any proximal box. Teeth were restored by randomized assignment of one of three commonly used restorative materials: amalgam (Dispersalloy, Dentsply Caulk, 38 West Clarke Ave, Milford, DE 19963), a radiolucent composite resin (Durafill, Heraeus Kulzer, Inc, 4315 S Lafayette Blvd, South Bend, IN 46614) and a radiopaque composite resin (Herculite, Kerr Dental Corporation, 1717 West Collins Orange, CA 92867). However, care was taken to include an equal number of surfaces restored with each type of material so that no single restoration type was used in excess.

The proportion of lesion sizes induced was equally distributed among the experimental teeth restored with different materials. Lesions were placed at one of two sites: the intersection of the lingual or facial wall, the axial wall and the gingival floor (buccal/lingual point angle) or along the gingival floor roughly midway between the facial and lingual walls of each proximal box. This constituted inclusion of two study sites in the two proximal boxes combined on each of the teeth. Thus, a total of 48 sites were evaluated using each imaging modality, of which any 24 boxes had lesions. This constituted a total of 192 sites for evaluation across all the imaging modalities. These surfaces were selected for lesion induction because previous studies have shown that the majority of recurrent decay occurs on the proximal surfaces of restored teeth on or near the gingival floor (Mjör, 1998). The mechanically induced lesions were filled with sticky wax (Sewerin, 1980) mixed with dental plaster to exclude air from the cavity, thereby, reducing the contrast at the edges of the artificial lesions. Previous studies have shown that artificially induced lesions simulating caries might have a

higher detectability based on the contrast at the margins when using film or digital images (Kang et al, 1998; Kang et al, 1996a,b).

A 2 cm-thick tissue equivalent material was used to simulate attenuation and scattering of the soft tissues of the face. Ground truth was known by recording the tooth surface and model number. Each restoration was removed, and the preparation was carefully inspected by each of the investigators to re-define the exact location and nature of the induced lesion. Ektaspeed Plus film (Eastman Kodak Company, 343 State St, Rochester, NY 14650) was used to acquire film-based images. Digital images were acquired using a #2 charge coupled device (CCD) sensor (Schick Technologies, Inc, 31-00 47<sup>th</sup> Ave, Long Island City, NY 11101). One of the authors enhanced the images produced with the CCD by manipulating the brightness and contrast controls of the software interface to ensure optimal visualization of the lesion (Moystad et al, 1996; Tyndall et al, 1998; Nair et al, 1998c). Pilot studies determined optimal enhancement settings for each image based on lesion visualization on the resulting images. Adequate contrast to tease out the signal of interest on teeth with lesions was the desired end-point of image enhancements. The images were then saved as uncompressed tagged image format (tif) files. Generation of PSP images involved the use of the Digora (Soredex Inc, 2150 Newmarket Parkway, Suite 110, Marietta, GA 30067) unit. Unenhanced images were saved as an independent modality. The images were subsequently enhanced by the same investigator using the brightness and contrast enhancement features of the Digora software and saved separately for viewing as the fourth imaging modality. This permitted evaluation of the contrast and brightness enhancement features of the Digora software, which was not previously evaluated. The enhancements were made so that the investigator could visualize the resulting images. A previous investigation revealed that observers using a new software interface did not employ the most optimal settings to detect the signal of interest (Tyndall et al, 1998; Nair et al, 1998a). One of the investigators conducted contrast and brightness enhancements based on visualization of the induced lesions on the images across all lesion sizes, locations and restorative materials.

Each image receptor was mounted on the lingual surface of the tooth to be imaged and was maintained in position with a sensor-positioning device (Rinn Corporation, 1212 Abbott Drive, Elgin, IL 60123). In order to simulate clinical conditions while bitewing images were acquired, projection geometry was not strictly controlled. A standard source-to-object distance of 26 inches, however, was used for all imaging purposes. Optimal exposures for each modality were determined during a pilot study. Exposure parameters included the kilovoltage used (70 kVp), current in milliamperes (15

mA) and time in fractions of seconds (18 impulses) for film, CCD (6 impulses) and PSP (8 impulses) using the GX 1000 (Dentsply Gendex, 901 West Oakton Street, Des Plaines, IL 60018) x-ray source.

A panel of eight observers representing operative dentistry, periodontics, oral diagnosis and general dentistry were instructed as to the nature of the experiment and trained in the interpretation of all the images presented. All observers completed a test session to ensure inter-reader consistency, and one of the authors was available to address any problems that arose during the reading sessions. The digital images were electronically masked so that the viewer could only visualize one proximal surface of a tooth at a time. This reduced the chance of learning effects from observers recognizing a surface from the context of adjacent teeth and restorations. Each observer was made aware of a 50:50 probability of lesion occurrence and was required to express subjective certainty as to the presence or absence of a lesion using a 1-5 confidence rating scale (Nair et al, 1998b,c; Tyndall et al, 1998).

1	2	3	4	5
Lesion definitely not present	Lesion probably not present	Uncertain if lesion present or absent	Lesion probably present	Lesion definitely present

To avoid generation of degenerate data for ROC analysis, the readers used the entire scale of 1-5 to express their confidence in the diagnoses. The images were sequenced according to a pre-computed, balanced and randomized distribution so that no modality was seen more frequently than another and no sequences were alike. All observer sessions were conducted in a distraction-free environment with subdued ambient lighting. Film images were displayed on a viewbox with masks and a 2x magnifier. All digital images were viewed on a 17-inch color monitor (Trinitron Multiscan, Sony Electronics, 550 Madison Avenue, 9th Floor, New York, NY 10022-3211) using TACT workbench as the software interface. The a-priori alpha was preset at 0.05.

To test for inter- and intra-examiner variability, observers re-read 20% of images from each modality following a time lapse of at least two weeks between initial and repeat readings. Images were again randomly sequenced so that the observer viewed a surface only once with one imaging modality during the repeat session.

ROC curves (ROCFIT) (Metz, 1978) were generated and areas under the curves (Az) were compared using repeated measures analysis of variance (ANOVA). The ANOVA model evaluated the effects of imaging modalities and readers. Where ANOVA suggested significant differences, pairings of each modality were assessed



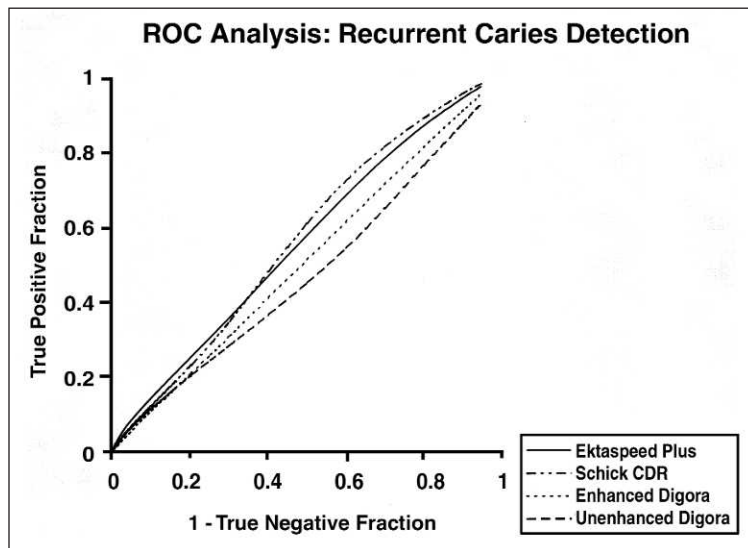


Figure 1. Representative ROC curves based on imaging modality.

using Tukey's HSD (Honestly Significant Difference) statistic.

## RESULTS

Areas under the ROC curves (Az) were computed for each imaging modality (Table 1). Data from the first readings were used to generate representative ROC curves for each imaging modality (Figure 1).

MANOVA was done using the score indicated on the confidence rating scale that was then correlated with

Table 1: Average Az Based on First readings for All Imaging Modalities

Imaging Modality	Average Az
Film	0.58
CCD	0.62
Enhanced PSP	0.55
Unenhanced PSP	0.53

Table 2: MANOVA: Dependent Variable: Scores Correlated with Ground Truth

Source	Sum-of-Squares	Df*	Mean-Square	F-Ratio	P
Modality	04.43	3	1.48	07.50	<0.0001
Restoration	10.47	2	5.23	26.57	<0.0001
Location	07.77	3	2.59	13.15	<0.0001
Modality * Restoration	01.21	6	0.20	01.02	0.4100
Modality * Location	35.75	9	3.97	20.16	<0.0001
Restoration * Location	19.43	6	3.24	16.44	<0.0001
Modality*Restoration*Location	09.58	18	0.53	02.70	<0.0001
Model	83.38	47	1.77	09.00	<0.0001
Total	376.55	1535	0.25		

\*Df: Degrees of freedom

the ground truth and revealed a statistically significant effect due to: imaging modality, restoration type, lesion location and interactions between restoration and location, modality and location, modality vs restoration vs location (Table 2). Table 3 shows the results of the post-hoc tests using least-squared means and a Tukey's comparison analysis of the four imaging modalities used. Tukey's statistic of the first readings demonstrates significant differences between the unenhanced PSP images and all other modalities. There are no other significant differences. Unenhanced PSP images were significantly inferior in their diagnostic performance to film and the contrast and brightness enhanced CCD based images. CCD images provided the highest Az value but were not significantly different from film and enhanced PSP images.

Paired *t*-tests were done based on the first and second reading session data and Pearson's correlation coefficient was computed (0.958).

Tables 4-6 depict the mean percent correct score for each of the different interactions. The highest percentage of correct responses was noted when radiopaque composites were used, followed by amalgam and radiolucent composites. Even the absence of lesions (control sites) was correctly identified more frequently with radiopaque composites compared to the other restorative materials. Similarly, while analyzing detection efficacy based on lesion location across all restorations, it was noted that those lesions induced along the buccal point angle recorded the highest score followed by those on the gingival floor.

Sensitivity and specificity values were computed for each of the imaging modalities (Table 7).

## DISCUSSION

Results of the detectability of defects designed to simulate recurrent carious lesions in this study suggest a substantial drop in diagnostic yield with unenhanced PSP images at the exposures chosen, compared to Ektaspeed Plus and enhanced CCD and PSP images. Performance of film, enhanced PSP and CCD images were not significantly different. This is consistent with the observations of Yoshiura and colleagues regarding PSP performance, in which an aluminum step wedge containing a series of shallow holes was imaged using different sensors to assess percepti-

Table 3: *Post Hoc Test of Scores Comparing the Four Modalities Using Least Squared Means and Tukey's HSD Multiple Comparisons: Matrices of Pair-Wise Mean Differences and Comparison Probabilities*

Imaging Modality	Film	CCD	Enhanced PSP	Unenhanced PSP
Film	0 (1.000)			
CCD	0.059 (0.212)	0 (1.000)		
Enhanced PSP	-0.018 (0.921)	-0.077 (0.066)	0 (1.000)	
Unenhanced PSP	-0.104 (0.009)*	-0.163 (0.0001)*	-0.086 (0.035)*	0 (1.000)

\* Pairs of means that are significantly different.

Table 4: *Percentage Mean Correct Responses (PMCR) Across Imaging Modalities for Different Types of Restorations*

Imaging Modality	Restorative Materials		
	Amalgam	Radiolucent Composite	Radiopaque Composite
Film	61.60	51.00	61.81
CCD	63.28	57.03	65.63
Enhanced PSP	61.70	56.06	62.50
Unenhanced PSP	51.56	50.78	58.59

\* Pairs of means that are significantly different.

Table 5: *Percentage Mean Correct Responses (PMCR) Across Restorations for Different Lesion Locations*

Restoration	PMCR Location-Wise			
	Control Sites	Mid-Gingival	Buccal Point Angle	Lingual Point Angle
Amalgam	65.44	45.31	72.00	40.00
R/L Composites	55.42	37.50	38.00	26.00
R/O Composite	72.40	78.13	72.92	63.28

Table 6: *Percentage Mean Correct Responses (PMCR) for Different Restorations and Lesion Locations Across All Imaging Modalities Pooled Together*

Restoration Type	PMCR
Amalgam	60
Radiolucent composite	49
Radiopaque composite	62
Lesion Location	PMCR
Control sites	64
Mid-gingival floor	47
Buccal point angle	61
Lingual point angle	45

bility differences (Yoshiura et al, 1999). Observers were unable to detect as many holes in the thicker steps of the wedge (6 mm – 12 mm) using unenhanced PSP images in comparison with film. When enhanced digital images were assessed over the same

range of steps, PSP performance was indistinguishable from Ektaspeed Plus film, while CDR images performed slightly better (Yoshiura et al, 1999). This probably explains why unenhanced raw PSP image data failed to perform as well as the other imaging modalities.

Brettelle et al also investigated the properties of PSP receptors for their physical qualities and inferred that these receptors can be used with exposures up to 80% lower than that required to expose Ektaspeed film without losing image quality (Brettelle et al, 1996). However, photo-

stimulable phosphor systems are sensitive to changes in beam quality and can behave differently when a specific diagnostic task, such as recurrent decay detection, involves generation of low-contrast, low-intensity signals. The results of this study may appear somewhat contrary to another study that reported similar performance of unenhanced PSP and film images and superior performance of enhanced PSP images for proximal caries detection (Moystad et al, 1996). The earlier study utilized Ektaspeed film, which has been shown to be less efficacious for detection of small proximal caries than the current formulation of Ektaspeed Plus film (Ludlow et al, 1997). The densities of the 9 mm step of the aluminum wedge reported by Yoshiura suggest that a sub-optimal exposure was employed for Ektaspeed film. In addition, film and PSP plates were exposed simultaneously resulting in greater exposure

Table 7: Sensitivity and Specificity of Imaging Modalities

Imaging Modality	Sensitivity	Specificity
Film	75%	73%
CCD	86%	76%
Enhanced PSP	81%	63%
Unenhanced PSP	65%	61%

of the PSP sensor than is typical in routine clinical practice.

A significant advantage of the digital images that cannot be discounted is the reduction in radiation exposure, which is in accordance with the ALARA concept (National Council on Radiation Protection, 2000). A previous report on dose reduction involving PSP plates determined that exposure could be reduced by about 50-75%, compared to Ultra-Speed film (Eastman Kodak, Rochester, NY), without compromising image quality (Conover et al, 1996). Assessment of radiation-related risk of developing somatic and genetic changes has been described in a previous report (White, 1992). It assumes significance because the current standard of care in private practice in the United States continues to be Ultra-Speed film with round collimation (Suleiman & others, 1999). Use of Ektaspeed Plus (50% reduction in radiation exposure compared to UltraSpeed film with round collimation) and the newer Insight intra-oral film (further 50% reduction in exposure from Ektaspeed Plus film) significantly reduces the radiation exposure compared to the standard of care (Farman & Farman, 2000).

This is a major advantage considering that CCD images and enhanced PSP images revealed no significant difference in lesion detection with film. With respect to CCD-based images, studies have shown that the diagnostic efficacy of these systems does not vary much between similar systems (White & Yoon, 1997; Price & Ergul, 1997). Therefore, it can be inferred that the diagnostic yield from such systems roughly parallels that of intra-oral film for purposes of recurrent decay detection as was seen in this investigation. Studies evaluating digital sensors based on solid state and PSP systems indicate a higher resolving power for the former, owing to smaller pixel sizes and a shorter gray scale display, while the performance of the latter improved with enhancements as was seen in this study (Borg, 1999). Numerous studies have compared the cost-effectiveness of all these types of imaging modalities (Miles & Razzano, 2000; Sanderink & Miles, 2000; Wenzel, 2000; White et al, 1999).

Previous reports have discussed concerns regarding the use of different caries models in diagnostic efficacy studies (Benn, 1994; Silverstone, 1968). The rationale for using the caries model and lesion sizes employed here has also been explained elsewhere (Nair et al, 1998b). Acid gel models have been known to demon-

strate a higher rate of demineralization than is naturally seen with induced lesions that do not progress parallel to the incremental lines (Silverstone, 1968). Natural caries appear to have an irregularly advancing front with about 25% mineral loss and tend to appear oblong (Benn, 1994). Besides, the acid gel model is not recommended for system evaluation studies since a natural carious lesion would be considerably larger and deeper than would be radiographically detectable or inducible with acid gels (Benn, 1994). Benn had noted in his study that no ideal mechanism for artificial caries induction devoid of all drawbacks exists.

Carious lesions may be simulated using software, or alternatively intact restored teeth with recurrent decay that have been extracted may be used, although this does not incorporate flexibility in the types of restoration used or lesion size. Besides, this will be very difficult and time-consuming to accomplish. Teeth with natural recurrent decay beneath restorations seldom get extracted. Farman (Farman et al, 1996) noted that a significant difference in lesion detection based on the nature of lesion induction exists. Studies comparing artificial mechanically-induced lesions with natural caries concluded that the differences between modalities were not large for lesions requiring clinical intervention, when evaluating the Ektaspeed Plus film, the CDR digital images and the PSP images (Kang et al, 1996ab, 1998). The mechanical defects, however, had more contrast. The enhanced contrast at the margins was probably reduced in this study by the use of wax mixed with plaster within the defect itself (Nair et al, 1998c). This would have provided a diagnostic challenge that more closely approximated detecting subtle changes in radiographic density associated with carious changes. Different lesion sizes were included to represent the varying degrees of demineralization that may occur during evolution of the natural lesion. The investigators realize that simulated lesions are different from actual lesions; however, it is no less of a challenging diagnostic task that is representative of most of those aspects of the detection/decision-making process which makes recurrent caries detection unique from primary caries detection. Under the current circumstances, the authors spared no effort to ensure use of a most optimal methodology to study detection efficacy of most commonly employed imaging modalities with respect to lesions simulating recurrent decay, bearing in mind the limitations of such a study *in vitro*. Without any *in vitro* data, an *in vivo* study cannot be justified. Furthermore, in a recent study Hintze & Wenzel opined that the diagnostic accuracy of radiographs taken *in vitro* is not significantly different from that for *in vivo* radiographs in spite of the variations in density and projection geometry (Hintze & Wenzel, 1996), and therefore, the authors think it reasonable to extrapolate the findings of this investigation to the actual clinical situation.



## CONCLUSIONS

Enhanced digital images acquired using the photo-stimulable phosphor-based PSP and the CCD-based Schick CDR systems provided a level of diagnostic performance comparable to that of intra-oral Ektaspeed Plus film for detecting defects designed to simulate recurrent caries in proximal boxes of restored molar teeth. Evaluation of these sensors with respect to recurrent caries detection in the clinical setting is, therefore, in order. The results of this study also suggest that contrast and brightness enhancements improved digital image performance.

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# Effects of APF Gel on the Physical Structure of Compomers and Glass Ionomer Cements

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## Clinical Relevance

The erosive effect of APF gel could increase restoration wear and bacterial adhesion and may reduce the longevity of restorations.

## SUMMARY

This study assessed the effect of an acidulated phosphate fluoride (APF) gel on the surfaces of eight modern esthetic restorative materials. Five specimens each of three high powder: liquid ratio conventional glass ionomer cements (ChemFlex, Fuji IX GP, Ionofil Molar), four poly-acid-modified resin composites (compomers) (Compoglass F, Dyract AP, Freedom, F2000) and an alkaline glass filled resin composite (Ariston pHc) were prepared and immersed at 37°C in 2 mL of artificial saliva for six weeks. The aged specimens were then coated with 1.23% APF gel for four minutes, rinsed and again immersed in

artificial saliva for another six weeks. The immersed, fresh specimens for each material were then examined with SEM and surface profilometry. After APF gel application, mean surface roughness ( $R_a$ ) measurements and SEMs showed that roughness increased significantly, generally from the resin composite and compomers to the conventional glass ionomer cements ( $p < 0.05$ ).

## INTRODUCTION

An increase in surface roughness has been used as a criterion to assess and predict the clinical deterioration of restorations of different types of materials (Smales, Webster & Leppard, 1992).

The surface roughness of conventional glass polyalkenoate (ionomer) cements (GICs) is related to the size and type of filler particles contained and to voids present, which are inevitable during mixing and placement of the cements. Smith (1988) has shown that the GIC surface is significantly roughened by one minute of phosphoric acid etching and that individual particles protrude from the surface or are lost after acidulated phosphate fluoride (APF) gel application for four minutes (Neuman & García-Godoy, 1991). APF gels have also been shown to damage the surfaces of resin-modified GICs, although to a lesser extent (Triana & others, 1994; El-Badrawy & McComb, 1998).

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However, materials containing a photocurable unit that allows polymerization to take place upon light activation exhibit reduced solubility. Recent laboratory reports (Rothwell, Anstice & Pearson, 1998; García-Godoy & Hosoya, 1998; Millar, Abiden & Nicholson, 1998) and clinical trials (Roeters & others, 1998) have reported favorably on polyacid-modified resin composites (compomers) as restorative materials. Other clinical trials have reported that marginal staining was of concern when using these restorative materials (Tyas, 1998). Although the matrix of compomers is generally more resistant to erosion than conventional GICs, damage to earlier products may still occur (Yip, Lam & Smales, 1999).

Little has been reported on the effects of APF gels on any of the newer esthetic restorative materials used in restorative dentistry (Smales & Yip, 2000). Therefore, this study investigates the effects of 1.23% APF gel on the physical structure of several high powder: liquid ratio GICs, compomers and a resin composite.

## METHODS AND MATERIALS

### Specimen Preparation and Immersion

Five specimens each of three high powder: liquid ratio esthetic conventional GICs (ChemFlex, Fuji IX GP, Ionofil Molar), four polyacid-modified resin composites (Compoglass F, Dyract AP, Freedom, F2000) and an alkaline glass filled ion-releasing resin composite (Ariston pHc) were evaluated (Table 1). The specimens were prepared according to the manufacturers' instructions, placed in disposable cylindrical Teflon molds (3.0 mm diameter x 2.7 mm height), then pressed between two Mylar strips sandwiched with two glass slides. The resin-containing materials were

light cured from both ends of the molds for 40 seconds (VCL 200—Demetron Research Corporation, Danbury, CT 06810). After about one hour of storage in a humidifier, each specimen was removed from its mold and placed in a polypropylene vial with 2 mL of artificial saliva (0.05 M acetate buffer with 2.2 mM  $\text{CaHPO}_4$ , adjusted with glacial acetic acid to pH 5.0) and stored at 37°C. The solution was replaced at six hours, one day and two days, then weekly for six weeks.

After aging for six weeks, each specimen was coated with 2 mL of 1.23% APF gel (John O-Butler Company, Chicago, IL, 60630) and left in place for four minutes. The specimens were then rinsed with deionized water to remove any visible remnants of gel. This was done with a gentle spray to avoid any surface damage. Each specimen was again placed in a fresh polypropylene vial with 2 mL of artificial saliva and stored at 37°C. The solutions were replaced using the same time schedules as before for another six weeks.

### Surface Profilometry

After 12 weeks, one fresh new specimen for each material was prepared as before. The specimens were kept moist at all times and blotted dry upon profilometry measurement. The mean surface roughness ( $R_a$ ) of this and of each of two randomly selected immersed specimens for each material was measured with a profilometer (Talysurf 10 Surface Roughness Tester—Rank Taylor-Hobson Laboratory, Leicester LE2 0SP, England). Two graphical printouts and digital readings of the  $R_a$  were made across the diameter of each circular specimen surface, and then another two measurements were made perpendicular to the first. The procedure was repeated for the opposite surface, for a total of eight measurements being made for each specimen.

Table 1: *Manufacturers' Data for Materials Used*

Product	Manufacturer	Shade	Presentations: Lot #	Expiry
ChemFlex	Dentsply DeTrey GmbH, D-78467 Konstanz, Germany	A4	Powder: UK 20-142-1 Liquid: 9710000432 (auto cure)	98-05-31 98-05-31
Fuji IX GP	GC Corporation, Tokyo, 174-8585 Japan	A2	Capsule: 120467 (auto cure)	1998-04
Ionofil Molar	Voco D-27457 Cuxhaven, Germany	A3	Powder A3: 73664, Liquid: 76579 (auto cure)	2000-04 2000-08
Compoglass F	Vivadent Ets, FL-9494 Schaan/Liechtenstein	A3	Compule 210 A3 U: 819798 (light cure)	not available
Dyract AP	Dentsply DeTrey GmbH, D-78467 Konstanz, Germany	A3	Compule: 9707000760 (light cure)	1999-05
Freedom	Southern Dental Industries, Bayswater 3153, Victoria, Australia	A3	Compule: 2346 (light cure)	2000-11
F2000	3M Dental Products, St Paul, MN 55144-1000, USA	A3	Compule: 19970905 (light cure)	1999-07
Ariston pHc	Vivadent Ets, FL-9494 Schaan/Liechtenstein	Universal White	Compule: A09227 (light cure)	2000-03

### Scanning Electron Microscopy (SEM)

Again, one fresh specimen of each material was prepared as before. The specimens were soaked in pure HMDS (hexamethyldisilane; Sigma Chemical Company, St Louis, MO 63118) for 10 minutes and placed on a piece of filter paper inside a covered-glass vial and dried inside a fume chamber at room temperature. This and the three remaining immersed specimens were sputter coated with gold, then examined using a SEM (Philips XL30CP, Philips Electron Optics, Eindhoven, The Netherlands). Photomicrographs at x400 and x1,000 magnification were taken.

### Statistical Methods

The raw data were input via a spreadsheet (Excel 7.0—Microsoft Corporation, Redmond, WA 98052). Any surface roughness changes in surface profilometry between the fresh and APF-treated specimens for each material were assessed using a *t*-test with Welch correction. The Kruskal-Wallis non-parametric ANOVA test was used to compare surface roughness among the materials, and Dunn's multiple comparison post-tests were used where appropriate. A statistical software package was used (InStat 3.0—GraphPad Software Inc, San Diego, CA 92121). Statistical significance was set at the 5% probability level.

## RESULTS

### Surface Profilometry

All materials showed a statistically significant increase in their surface roughness after storage for 12 weeks in buffered artificial saliva, with exposure to a single, four minute APF gel application after six weeks (Table 2). Significant differences in surface roughness were present among the eight materials both before and after the experiment. The roughest surfaces were found with the three conventional GICs. Initially, the smoothest sur-

faces were for Ariston pHc, but after the 12-week experiment, the smoothest surfaces were for Compoglass F. The percentage increase in surface roughness was least for Fuji IX GP (143%) and most for Dyract AP (1,343%) and Ariston pHc (1,390%). However, in absolute terms, the smallest increase in surface roughness was 50  $\mu\text{m}$

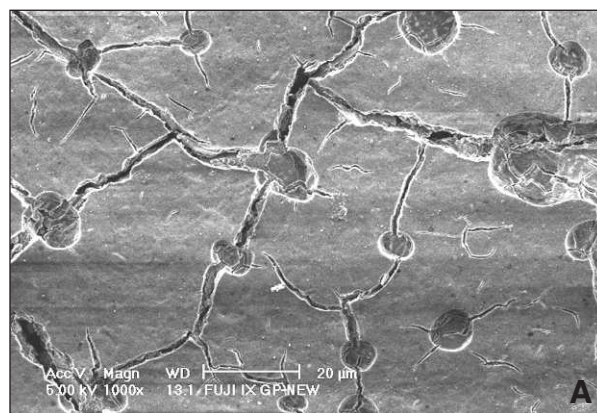


Figure 1A. Fuji IX GP before APF exposure. (Magnification x1000)

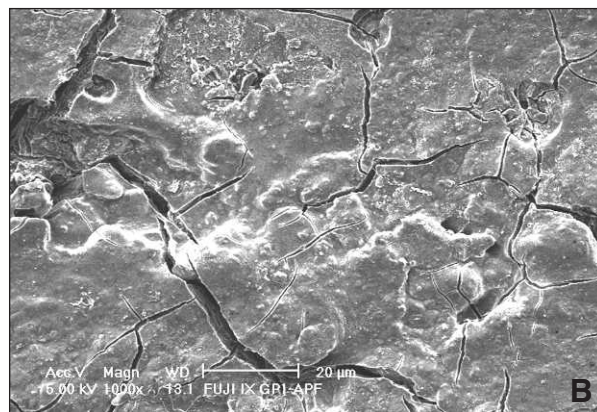


Figure 1B. Fuji IX GP after APF exposure. (Magnification x1000)

Table 2: Statistical Summary for Surface Roughness of Eight Restorative Materials Before, and After Artificial Saliva Immersion for 12 weeks (including APF gel application)

Material	*Mean $R_a(\mu\text{m})$ (before) 95% Confidence Interval	**Mean $R_a(\mu\text{m})$ (after) 95% Confidence Interval	% Change	P-Values (Welch t-Test)
Ionofil Molar	0.195 (0.158 - 0.231)	0.665 (0.539 - 0.770)	241	<0.0001
Fuji IX GP	0.193 (0.002 - 0.314)	0.469 (0.238 - 0.699)	143	0.0323
ChemFlex	0.177 (0.120 - 0.234)	0.520 (0.260 - 0.781)	194	0.0141
F2000	0.043 (0.033 - 0.052)	0.135 (0.124 - 0.147)	214	<0.0001
Freedom	0.036 (0.024 - 0.048)	0.162 (0.117 - 0.207)	350	<0.0001
Compoglass F	0.028 (0.011 - 0.044)	0.078 (0.060 - 0.095)	179	<0.0002
Dyract AP	0.014 (0.010 - 0.018)	0.202 (0.103 - 0.302)	1343	0.0011
Ariston pHc	0.010 (0.009 - 0.010)	0.149 (0.095 - 0.203)	1390	<0.0001

\*Kruskal-Wallis statistic = 54.871 (corrected for ties),  $p < 0.001$

\*\*Kruskal-Wallis statistic = 57.856 (corrected for ties),  $p < 0.001$

Values connected by the same vertical lines are not significantly different from each other, except for Compoglass F, which is significantly different from all three GICs after APF gel application.



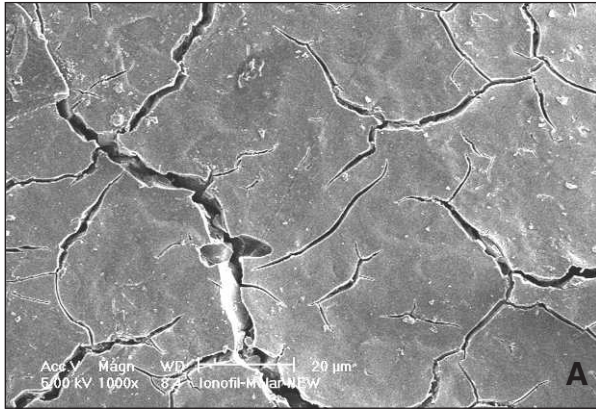


Figure 2A. Ionofil before APF exposure. (Magnification x1000)

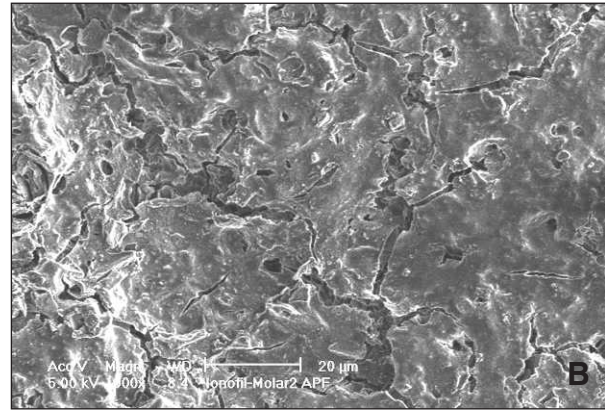


Figure 2B. Ionofil after APF exposure. (Magnification x1000)

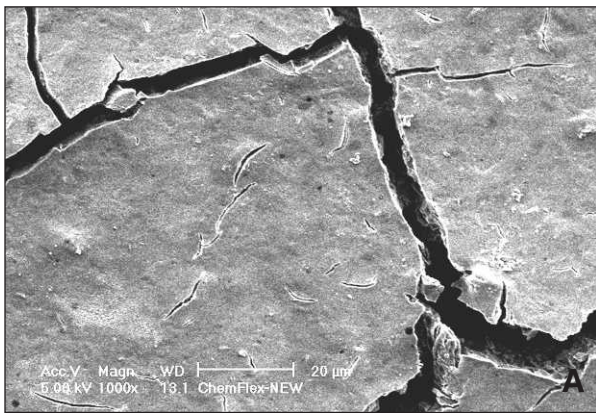


Figure 3A. ChemFlex before (3a) APF exposure. (Magnification x 1000)

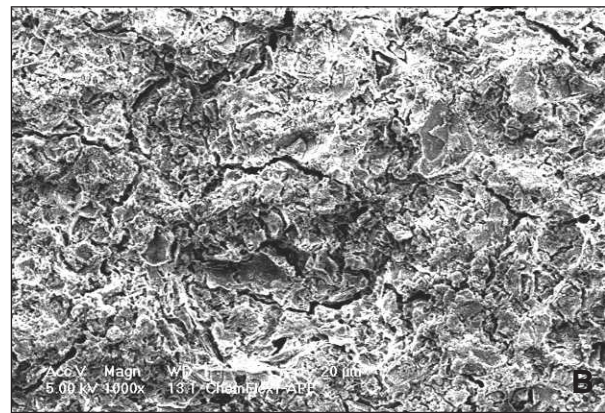


Figure 3B. ChemFlex after APF exposure. (Magnification x 1000)

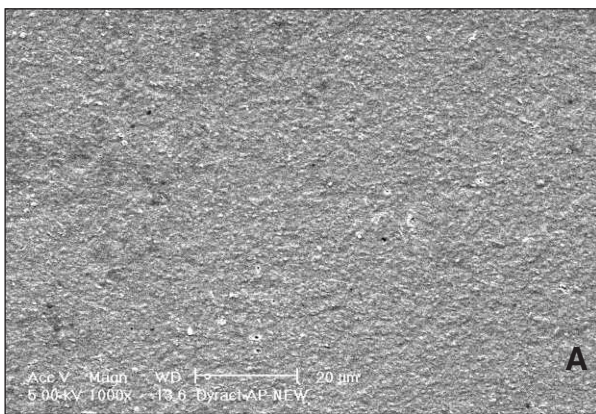


Figure 4A. Dyract AP before APF exposure. (Magnification x1000)

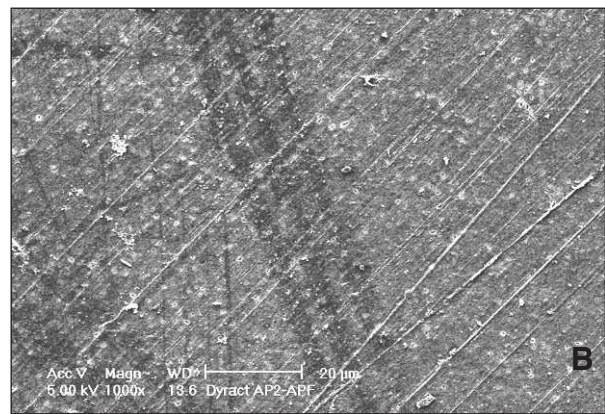


Figure 4B. Dyract AP after APF exposure. (Magnification x1000)

for Compoglass F, and the largest was 470  $\mu\text{m}$  for Ionofil Molar.

### Scanning Electron Microscopy (SEM)

The conventional GICs showed obvious surface voids and large cracks as a result of the SEM dehydration. Following treatment over 12 weeks, the surfaces of the

cements became very eroded. The encapsulated Fuji IX GP (Figure 1) appeared to be slightly less affected than the other two hand-mixed GICs (Figures 2, 3). Protruding glass particles were observed on the APF gel-treated surfaces.

The surfaces of Dyract AP, Freedom, F2000 and Ariston pHc showed fewer and smaller voids and finer



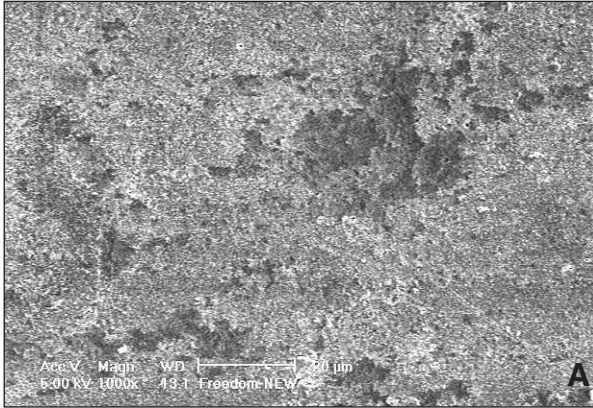


Figure 5A. Freedom before APF exposure. (Magnification x1000)

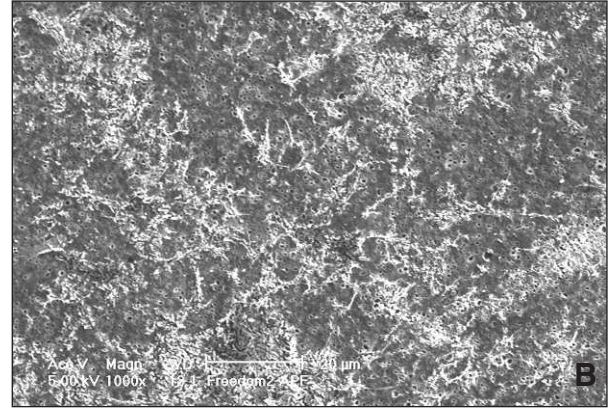


Figure 5B. Freedom after APF exposure. (Magnification x1000)

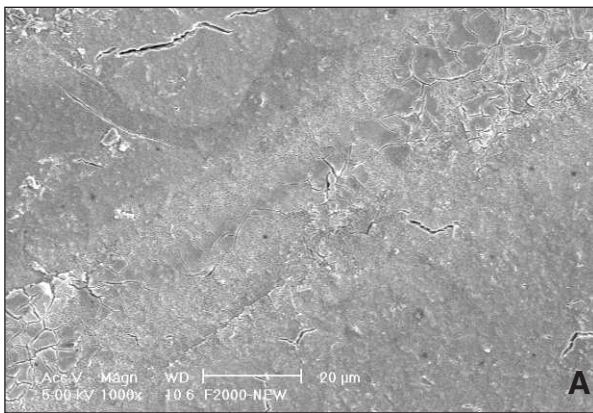


Figure 6A. F2000 before APF exposure. (Magnification x1000)

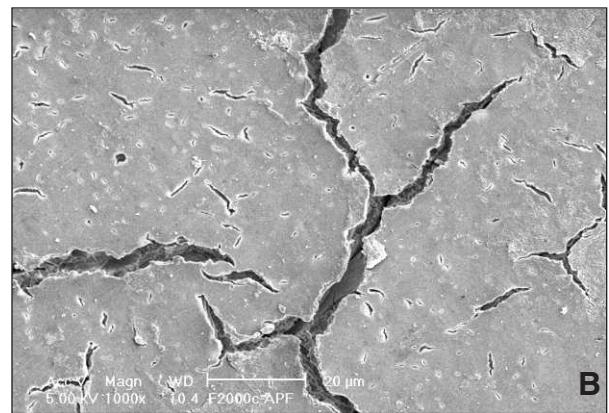


Figure 6B. F2000 after APF exposure. (Magnification x1000)

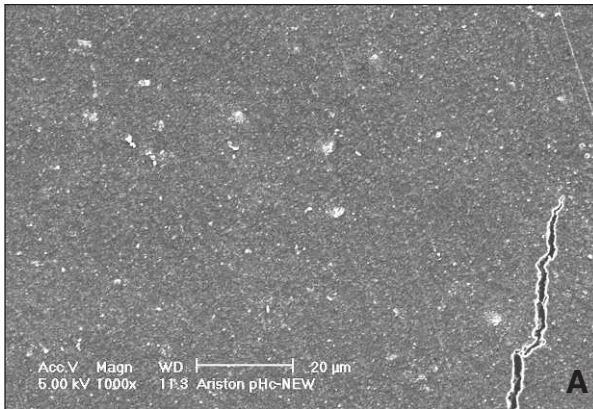


Figure 7A. Ariston pHc before APF exposure. (Magnification x1000)



Figure 7B. Ariston pHc after APF exposure. (Magnification x1000)

cracks than the conventional GICs (Figures 4-7). The cracks were more obvious following APF treatment, especially for F2000. There were numerous fine cracks present for Freedom following treatment. All four materials showed some surface erosion which was less obvious for F2000. The small initial voids and the later enlarged voids after APF treatment could be readily differentiated from the photomicrographs.

Very few small voids and cracks were visible for Compoglass F (Figure 8). Although slightly eroded, the surfaces appeared less affected by the treatment than were the other materials.

## DISCUSSION

Although the order of surface roughness for the fresh specimens was: resin composite < compomer < poly-



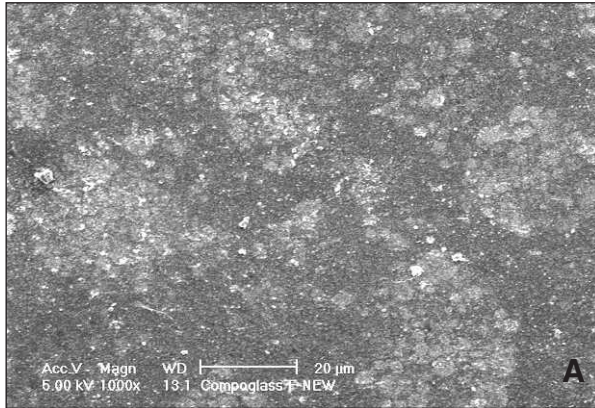


Figure 8A. *Compoglass F* before APF exposure. (Magnification  $\times 1000$ )

acid-modified resin composite < conventional GICs, statistical significance was limited to between only a few materials (Table 2). The general order was similar to that reported for other products of the same type (Yip & others, 1999). However, one compomer (Compoglass F) had smoother surfaces than the resin composite (Ariston pHc) after acid gel treatment. This finding has not been cited in the literature.

The critical mean surface roughness ( $R_a$ ) value for bacterial colonization of various dental materials has been considered to be  $0.2 \mu\text{m}$  (Bollen, Lambrechts & Quirynen, 1999). Surface roughness more than the critical roughness value is likely to cause significant increased bacterial adhesion, dental plaque build-up and acids to act on the dental material's surface.

Conventional GICs, whose surfaces were not eroded to the same extent, are known to be acid soluble, in contrast to the more acid insoluble compomers and resin composites examined in this study. Before application of APF gel, the surface roughness of the GICs was just below the critical roughness value; any surface damage will potentially lead to significantly increased bacterial adhesion. Although not using the same specimens, the initial relatively smooth surfaces of the three conventional GICs were absent after treatment. The SEM findings showed very rough surfaces, with voids present (Figures 1-3), confirmed by profilometry (Table 2). Smith (1988) showed that the glass ionomer surface integrity was essentially destroyed after one minute of phosphoric acid etching and that individual particles dissociated from each other as the gel matrix dissolved. Neuman & García-Godoy (1991) also showed that glass particles were left protruding from the cement surface after APF gel application. The specimens in this study were placed in buffered artificial saliva for an additional six weeks after APF gel application and any loose glass particles present were probably dislodged.

It was suggested that the application of APF gel could recharge the fluoride content of exhausted glass

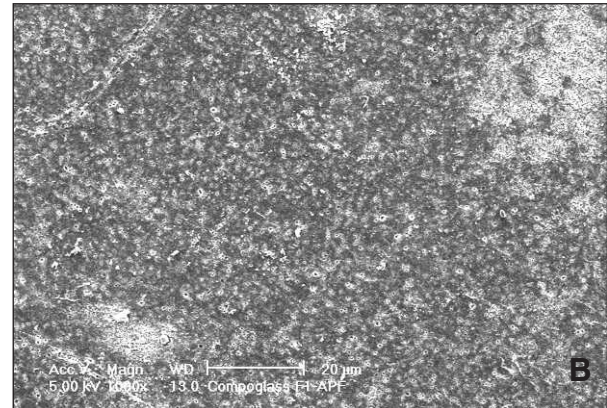


Figure 8B. *Compoglass F* after APF exposure. (Magnification  $\times 1000$ )

ionomer cements (Yip & Smales, 1999). However, APF gel contains hydrofluoric acid and phosphoric acid (El-Badrawy, McComb & Wood, 1993). Phosphoric acid has the ability to etch glass particles (Council on Dental Materials, Instruments, and Equipment, 1988). Hydrofluoric acid is more destructive than phosphoric acid because it can etch glass at lower temperatures (Kula et al, 1986). The pH of APF gel is about 5. The pH affects the chemical erosion of the cement by acid etching the surface and leaching the principle matrix-forming cations, Na, Ca, Al and Sr ions, and will also increase its fluoride release (Crisp, Lewis & Wilson, 1980). However, erosion of glass ionomer cements depends not only on the pH of the gel, but also on the complex formation ability of the particular acid present (Matsuya & others, 1984). El-Badrawy & others (1993) suggested that phosphoric acid was capable of forming stable complexes with metal ions in the ionomer, resulting in greater surface erosion. Diaz-Arnold & others (1995) found that APF gel caused the greatest amount of fluoride release followed by neutral sodium fluoride gel. Stannous fluoride gel, which had the lowest pH, was similar to deionized water in its effects. The buffered artificial mouth also has a pH of 5, but does not exert the acid erosion effects due to the presence of buffering.

The lesser surface roughness of the four compomers and resin composite after exposure to APF gel detected qualitatively from SEM observations (Figures 4-8) was also confirmed by profilometry (Table 2). Voids were also smaller and less frequent than those observed with the three conventional GICs. The application of 1.23% APF gel for four minutes has also been shown to reduce the surface hardness of compomer and resin composite materials (Santos-Pintos & others, 1997). When Dyract specimens were immersed in the same buffered artificial saliva at pH 5, as used in this study, an extremely small weight gain of the specimens was observed at 12 weeks and the light-cured surfaces were little affected (Lavis & others, 1997). These findings were in contrast to the significant weight losses found for Dyract speci-

mens following a four-minute application of 1.23% APF gel, that correlated well ( $r=0.98$ ) with the greatly-increased cumulative fluoride ion release measured (Yip & others, 1999).

Compomers were introduced to help overcome problems of handling, appearance, moisture sensitivity and early low-mechanical strengths associated with conventional GICs, while at the same time maintaining some of their clinical advantages. However, the surfaces of all materials were eroded to varying extents by the APF gel treatment and, apart from Compoglass F (Figure 8), cracking was also apparent for all materials to varying extents. As this cracking was less obvious for the fresh specimens of F2000, Freedom, Dyract AP and Ariston pHc (Figures 4-7), it may not be entirely an artifact caused by the SEM procedures. The materials with  $R_a$  values below the  $0.2\text{ }\mu\text{m}$  threshold following APF gel application were F2000, Freedom, Compoglass F and Ariston pHc (Table 2). The surface roughness of Compoglass F was even less than that of Ariston pHc, which is a single-shade opaque posterior resin composite substitute for amalgam. It has been called an alkaline glass-filled restorative by the manufacturer and releases fluoride, hydroxyl and calcium ions in response to a local decrease in pH. All the other materials are more likely to show increased bacterial adhesion following APF gel treatment.

In this study, Dyract AP and Ariston pHc showed relatively larger percentage increases in their surface roughness following APF gel application than did the other materials, but the increases in absolute terms were still very small when compared with those for the three GICs. It appears that the surface degradation of resin composites exposed to APF gel is partly related to the filler particles present (Kula & others, 1986), the strontium fluorosilicate glasses present in Dyract AP and Freedom and the alkaline glass present in Ariston pHc, which may be more susceptible to acid erosion than the aluminum fluorosilicate glasses present in the other products.

The surfaces of all the materials were etched and eroded by the application of 1.23% APF gel for four minutes after storage for 12 weeks in artificial saliva. McIntyre investigated the alternative use of a 13% tartaric acid gel for acidulation, but this also damaged cement surfaces (McIntyre JM—personal communication, 1997). Neutral topical fluoride application is preferred (Triana & others, 1994; Diaz-Arnold & others, 1995).

The clinical significance of the increased surface roughness of the materials is several fold. The roughness could increase restoration wear and become an area to harbor the colonization of *Streptococcus mutans* (Neuman & García-Godoy, 1991) as increased plaque formation has been found on the rough surfaces of conventional GICs *in situ* (Smales, 1981; Forss, Seppä &

Alakuijala, 1991). An average surface roughness ( $R_a$ ) value of  $0.2\text{ }\mu\text{m}$  has been found to be critical for a dramatic increase in the colonization of cariogenic microorganisms, and an extensive review of the literature on this topic has been recently published (Bollen & others, 1999). In this study, the GICs approached this critical  $R_a$  value after setting in contact with Mylar strips (Table 2). When placed with free-hand contouring, GICs will not meet the critical  $R_a$  value and will tend to promote bacterial adhesion. For the other materials, although most approach the critical  $0.2\text{ }\mu\text{m}$  value after the APF gel, only Dyract AP reached it.

Another factor that may reduce bacterial adhesion on restorative material is the level of fluoride release (Yip & Smales, 2000). Measurement of intracellular fluoride in *Streptococcus mutans* shows that freshly prepared conventional GICs have antibacterial effects, but this seems to disappear within a few weeks (Seppä, Smalankivi & Forss, 1992). Fluoride in plaque growing on GICs is higher than on composite resin restorations and reduced the level of accumulation of *Streptococcus mutans* (Forss et al, 1991). An 80% reduction in bacterial accumulations on enamel surfaces has been reported when freshly prepared GICs were used (Palenik & others, 1992). The fluoride level also stayed high in plaque formed on 28-day old material when the release of fluoride has already leveled off after initial high release (Swartz, Philips & Clark, 1984; Forss & Seppä, 1990). Although aged compomer restorative materials have increased fluoride release upon APF gel application, the level of fluoride release is not sustained (Yip & Smales, 1999) and the surface roughness ( $R_a$ ) increased substantially for bacterial adhesion (Yip & others, 1999).

Increased surface roughness is a reflection of the deterioration of restorations (Smales & others, 1992; Larsen & Bruun, 1994). The obvious re-surfacing of restorations, particularly conventional GICs by adding fresh materials or resin composite overlays (as in the sandwich or laminate technique), may be considered if the restorations are otherwise satisfactory.

## CONCLUSIONS

The single application of 1.23% APF gel to all materials for four minutes resulted in significantly increased surface roughness that was particularly obvious for the three conventional GICs. The effects were less pronounced for the four compomers and the alkaline glass-filled resin composite. APF gel should not be applied routinely to the newer esthetic restorative materials investigated in this study. Precontoured polyester matrices are desirable when conventional GICs are placed, followed by minimal trimming, to reduce bacterial colonization. Smoother surfaces leading to the potential for a reduced susceptibility to bacterial adhesion favor compomers and resin composites as being more suitable restorative materials.



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# Characterization of Dentin-Bonding-Amalgam Interfaces

SB Geiger • Y Mazor  
E Klein • H Judes

## Clinical Relevance

The lowest degree of gap formation and microleakage between restoration and tooth surface in bonded amalgam restorations is achieved by applying a bonding agent over the etched cavity surface, followed by a layer of resinous adhesive material and immediate condensation of amalgam.

## SUMMARY

Applying a bonding agent and a resinous adhesive layer before amalgam condensation has become a common clinical procedure. However, interactions between the different interfaces formed, and the extent of sealing obtained, have not been extensively studied. This study characterized the interfaces formed in the bonded amalgam restoration. Specifically, the individual contributions of the bonding agent (One-Step) and the adhesive resin (Resinomer) were examined, along with their mode of application on the prevention of microleakage and the formation of a tight, continuous adhesion to amalgam. To this

end, a dye penetration assay and scanning electron microscopy (SEM) were used, including high resolution elemental analysis, for the characterization of the sealing properties and the interface structure obtained following various procedures of applying amalgam adhesives. Results indicated that placing bonding material under the amalgam restoration is essential to preventing microleakage. When condensed against uncured or cured adhesive material, the adhesive resinous glass layer creates a thick interface with protrusions and inclusions in the amalgam, though microleakage studies indicate that condensation over the uncured adhesive results in a better seal than that of the cured adhesive. SEM combined with elemental analysis indicates that the adhesion between amalgam and adhesive material is mainly of mechanical character and is formed by interdigitations of the adhesive material protruding into the amalgam. Gaps formed at the various interfaces in the different modalities could be localized. In addition, resinous glass composite alone, without bonding, was found to provide an unacceptable degree of sealing between the tooth and amalgam. The clinical significance of these findings is further discussed.

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## INTRODUCTION

The advent of adhesive materials capable of bonding to enamel, dentin and metallic surfaces has an intriguing effect on the clinical approach to amalgam restorations (Setcos, Staninec & Wilson, 2000). The potential advantages of applying adhesive material to the dental surface include the reduction of microleakage (Staninec & Holt, 1988; Turner, Germain & Meiers, 1995; Chang & others, 1996; Royse, Ott & Mathieu, 1996; Meiers & Turner, 1998) and the consequent prevention of recurrent caries, thermal insulation and the reduction of post-operative pain (Gordan & others, 1999, Nakabayashi, Ashizawa M & Nakamura, 1992).

Another major goal is strengthening the mechanical bond between the restoration and the cavity surface, thus reducing the need for excessive removal of tooth structure to ensure retention and lessening the need for retentive pins and preventing post-operative cuspal fracture (Belcher & Stewart, 1997; Ianzano, Mastrodomenico & Gwinnett, 1993; Burgess, Alvarez, & Summitt, 1997; Setcos, Staninec & Wilson, 1999).

A wide variety of amalgam adhesive products are currently available for clinical use (for example) Amalgambond Plus by Parkell, Panavia by Kuraray and RelyX ARC by 3M). One approach to optimal restoration is based on the use of a bonding material such as One-Step, (BISCO, Inc, Itasca, IL) combined with a resinous glass-filled composite (Resinomer, BISCO, Inc, Itasca, IL) as an additional adhesive agent for amalgam. This system, thus, entails four major interfaces: the adhesive-amalgam interface, the bonding-adhesive interface, the bonding-dentin interface and the bonding-enamel interface, each of which differ in their physical and chemical properties. The enamel-bonding and dentin-bonding interfaces have been previously characterized (Swift, Perdigão & Heymann, 1995; Van Meerbeek & others, 1993; Perdigão & Lopes, 1999), while the adhesive-amalgam interface has been analyzed to a lesser extent (Ramos & Perdigão, 1997). Application of an additional resinous material over the bonding agent and the condensation of amalgam over the resin offer different working procedures which should be studied and characterized. According to the manufacturer's recommendations, Resinomer should be placed on the cavity surface and amalgam may be condensed immediately over the uncured resin. However, the placement of amalgam over uncured resin may be associated with some problems, including displacement of the resin into the amalgam (Mahler, 1996) and displacement of resin towards the matrix band, potentially leaving voids and irregularities at the surface of the restoration. To test whether condensation of amalgam over the uncured resin leads to a tighter seal than condensation over cured resin and to assess the role of the bonding agent in this system, the authors compared the interface between the tooth and

the restoration following application of Resinomer by two different placement procedures (cured and uncured) before amalgam condensation in the presence or absence of bonding agent. It was concluded that a bonding agent applied over the etched cavity surface is essential for the prevention of microleakage in the bonded amalgam restoration, and that condensation of amalgam over the uncured resinous adhesive provides a better seal compared to the delayed condensation over cured adhesive. The properties of the various interfaces in three layer restorations, such as these, are described.

## METHODS AND MATERIALS

### Tooth Preparation

A total of 36 caries-free premolar teeth extracted for orthodontic reasons were used in this study. Class V cavities were prepared using a 330-carbide bur mounted on a high-speed hand piece. The cavities were prepared on the lingual and buccal side of the teeth and confined to the coronal part of the tooth. All cavity surfaces, both enamel and dentin, were etched for 15 seconds using Uni-Etch (32% phosphoric acid + benzalkonium chloride, BISCO, Inc, Itasca, IL 60143), rinsed with a water-air spray for 10 seconds and air dried with two gentle blows of air, keeping the dentinal surface damp.

### Experimental Design

The teeth were randomly divided into six groups and subjected to the following treatment procedures:

- A) One Step + Resinomer (uncured) + amalgam
- B) One Step + Resinomer (cured) + amalgam
- C) Resinomer (uncured) + amalgam
- D) Resinomer (cured) + amalgam
- E) Amalgam
- F) One Step + amalgam

### Restorations

Two layers of the bonding material—One-Step (BISCO, Inc, Itasca, IL 60143, Lot 129036) were applied (groups A,B,F) and light cured for 20 seconds using an Optilux 400 (Demetron Research Corporation, Danbury, CT 06810) light source. Resinomer (BISCO, Inc, Itasca, IL 60143, Lot #039065/609314) was mixed according to the manufacturer's instructions and applied to the entire cavity surface with a brush (groups A-D). Pre-capsulated dispersed amalgam alloy (Valiant-PhD, Caulk/Dentsply, Milford, DE 19963-0359) was triturated for 14 seconds in a high-speed triturator (G-C'S HIGH MIX VS-II GC, Japan). Condensation was performed using a mechanical condenser (Dentatus, Sweden). In groups A and C, amalgam was condensed immediately after applying Resinomer. In groups B and D, Resinomer was allowed to cure for 20 minutes before condensation of amalgam.



Table 1: The Scoring System for Microleakage

Microleakage Score	Degree of Dye Penetration
0	No dye penetration
1	Penetration of dye to mid dentinal wall
3	Penetration of dye through entire dentinal wall
4	Penetration of dye through entire dentinal walls and into tubuli towards the pulp

### Thermocycling and Microleakage

The teeth were left at 4°C for 48 hours before further processing. The roots were then coated with varnish and subjected to automatic thermocycling (TC-2 I Manes, Tel-Aviv Israel). This treatment consisted of 500 cycles between water baths maintained at 50±3°C and 550±3°C (10 seconds at each temperature and 10 seconds dwell time, Liberman & others, 1985). The teeth were then immersed for seven days in a 0.5% aqueous basic fuchsin solution at 37°C, after which they were rinsed in water and dried. Teeth were then sectioned longitudinally through both restorations using a diamond disc. The cut surfaces were inspected using a dissecting microscope (Wild t-327733, Wild Leitz Ltd, Heerbrugg, Switzerland). The degree of dye penetration at the occlusal and gingival margins on both sides of the sectioned restorations were independently scored by two evaluators according to the criteria described in Table 1, and the mean value of dye penetration was determined. Photographs of the different samples were taken.

### Electron Microscopy (EM)

Samples for EM examination were sectioned using a diamond disc; the cut surfaces were mounted, ground and polished to 1µm fineness according to standard metallographic procedures in a Buhler Minimet Polisher. A thin, transparent carbon film was applied to the polished surface (~150 Å, with an SPI fiber coater) to provide the electrical conductivity required for examination in the EM.

The samples were examined in a JEOL JSM 6400 scanning electron microscope (SEM) provided with: 1) a detector for secondary electrons (SE), 2) a detector for back scattered electrons (BSE) and 3) a Link-Isis energy dispersive X-ray spectrometer (EDS) that permits *in situ* elemental chemical analysis.

The SE signal gives information about topography and is also most intense for good electrical conductors (that is, metals). The BSE reveals

differences in atomic density, with brighter regions corresponding to heavier atoms. The X-ray spectra are characteristic of each element and can be used as a fingerprint to identify every element, its location and relative abundance.

### Statistical Analysis

Comparison of microleakage data obtained following the various restorative procedures was carried out using the analysis of variance test (ANOVA), followed by Multiple comparisons using Tukey's method. Differences with *p* values smaller than 0.01 were considered significant.

## RESULTS

### Effect of Restorative Procedure on Microleakage

Six distinct procedures for application of the amalgam adhesive were compared. They included direct condensation of amalgam into the etched cavity, etched cavity lined with bonding agent (One-step) and etched cavity lined with bonding agent (One-Step) followed by an adhesive layer (Resinomer), which was followed by immediate or delayed (20 minutes) condensation. Two additional samples were etched and lined directly with Resinomer followed by immediate or delayed condensation of amalgam. To quantitatively compare the various treatments, the dye penetration in each sample was scored. As shown in Figure 1(A-F), dye penetration was usually higher along the gingival walls of the cavity, compared to the occlusal walls. The degree of microleakage was significantly reduced with the use of One-Step as bonding agent (Figure 1 A,B,F), compared to samples in which One-Step was not applied (Figure 1

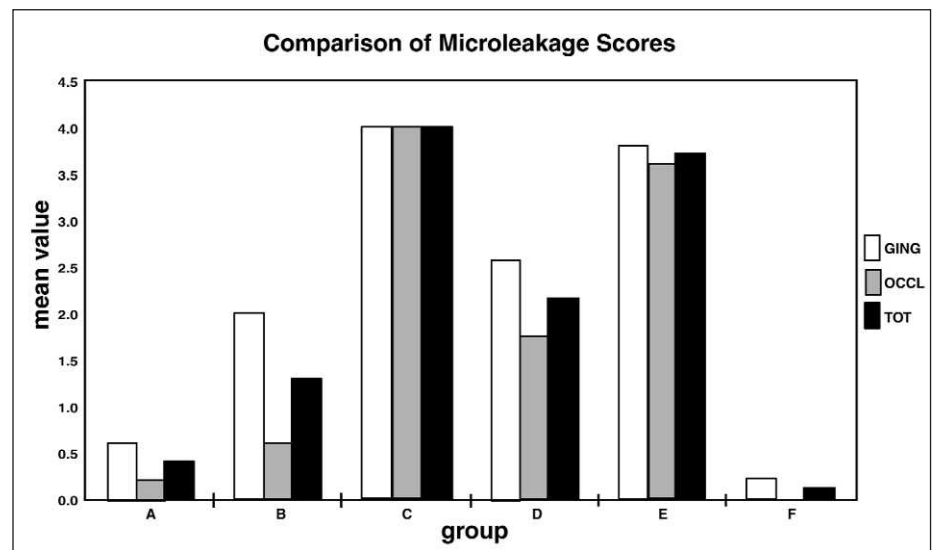


Figure 1: Comparison of microleakage scores. Gingival, occlusal and total microleakage scores measured in the different specimens with the following constituents at the interface between amalgam and cavity surface: A) One-Step + uncured Resinomer; B) One-Step + cured Resinomer; C) Uncured Resinomer; D) Cured Resinomer; E) None; F) One-Step.

C,D,E) ( $p < 0.001$ ). Condensation of amalgam over the uncured resin (Figure 1A) presented lower levels of microleakage as compared to samples with cured resin (Figure 1B) ( $p < 0.01$ ). Resinomer, alone, did not prevent microleakage (Figure 1C, D), especially when the amalgam was packed over a fresh (uncured) Resinomer (Figure 1C). Leakage was somewhat reduced when the Resinomer was left to set for 20 minutes before amalgam condensation (Figure 1D) ( $p < 0.01$ ). Interestingly, the lowest level of microleakage was observed when One-Step was used as the only bonding agent (Figure 1F). On the other hand, massive leakage occurred when the cavity was only etched and no intermediary material was used prior to amalgam condensation (Figure 1E). It should be emphasized that condensation of amalgam over etched dentin was used as a negative experimental control for the bonding and adhesive resins and does not represent an acceptable treatment procedure. Among the samples treated with One-Step, highest levels of microleakage were noted when adhesive was applied on top of the bonding agent and allowed to set before amalgam conden-

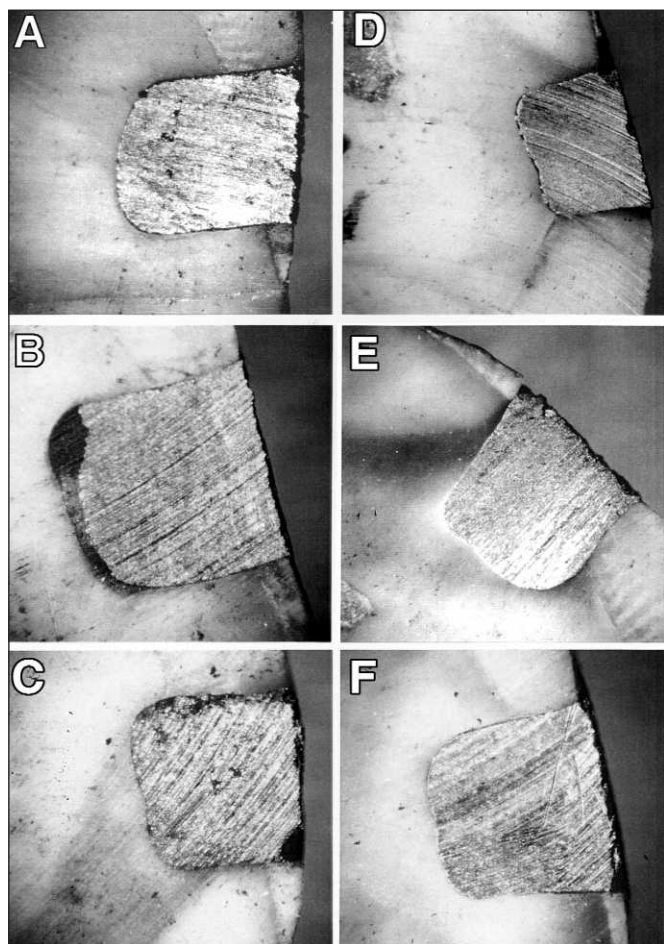


Figure 2: Photographic presentation of microleakage patterns in the different restorative models A) One-Step + uncured Resinomer; B) One-Step + cured Resinomer; C) Uncured Resinomer; D) Cured Resinomer; E) None; F) One-Step.

sation (Figure 2B compared to A). Standard deviations were within 30% of the mean value.

### Light Microscopic Examination

Light microscopic examination revealed limited dye penetration in samples treated with the bonding agent prior to further restoration/filling (Figure 2A,B,F), compared to massive dye penetration along the dentinal tubules in samples with no bonding (Figure 2C,D,E). Interestingly, the limited dye penetration was restricted to the interface between the restoration and resin (Figure 2B) without spreading into the dentinal tubules. Some additional features should be noted: when bonding agent and adhesive agents were not applied, a sharp and continuous border between the amalgam and the cavity wall was observed (Figure 2E). Cavities lined with only the bonding agent had a similar appearance (Figure 2F). On the other hand, application of adhesive created significant irregularities along the restoration-tooth interface, manifested by deep protrusions of adhesive agent into the amalgam (Figure 3A,B,C,D). In addition, in all samples lined with Resinomer, either cured or uncured before condensation of amalgam, inclusions of

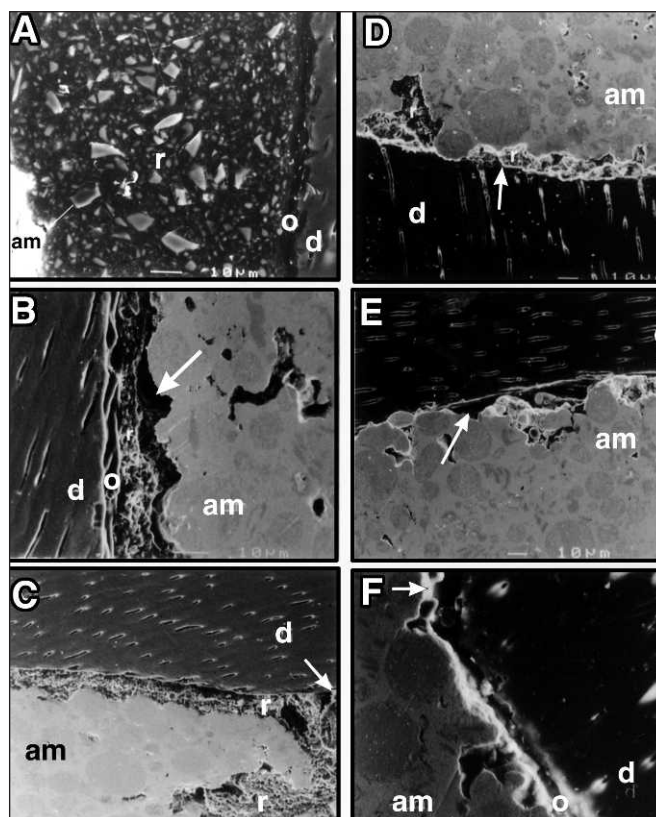


Figure 3: Representative SEM photographs of the different interfaces in the different restorative models, gaps are shown by arrowheads between interfaces (Am-amalgam, r- Resinomer, o- One-Step, d- dentin), A) One-Step + uncured Resinomer; B) One-Step + cured Resinomer; C) Uncured Resinomer; D) Cured Resinomer; E) None; F) One-Step.



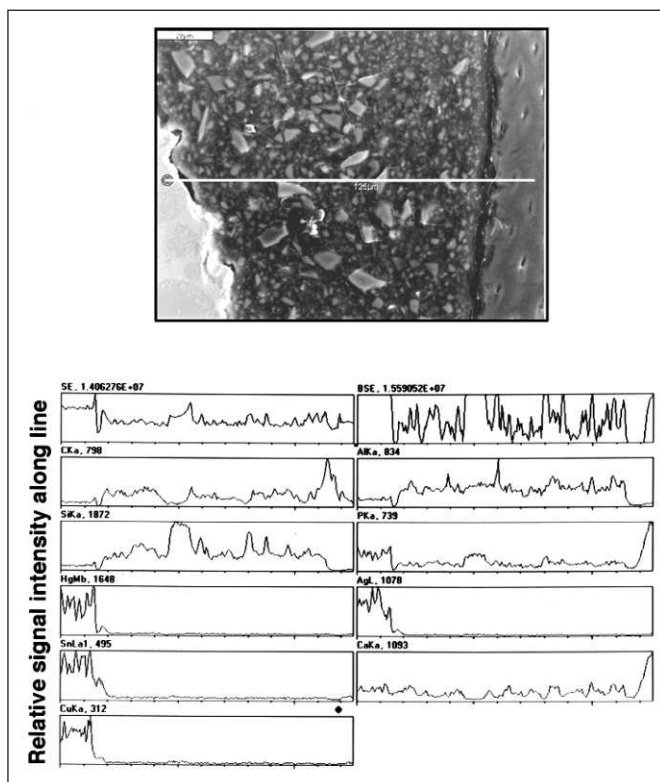


Figure 4: Elemental analysis based on EDS carried out on a sample with One-Step + uncured Resinomer at the interface (Group A). SE profile presents the relative light intensity emitted. The BSE presents the difference in atomic density. The EDS profiles present the following elements: C-Carbon; Si-silicate; Hg-Mercury; Sn-Tin; Cu-Copper; Al-Aluminum; P-Potassium; Ag-Silver; Ca-Calcium. The linear ratio between the image and the chart is 1:1.25.

resin were noticed within the amalgam mass. It appeared that in the samples with uncured resin, inclusions were larger and more numerous (Figure 3A,C). These inclusions were later analyzed and identified as consisting of Resinomer (not shown).

### Scanning Electron Microscopic Examination

Visualization of the treated samples at higher magnification revealed the level of penetration of the adhesive and bonding agent into dentin. Gaps could be visualized and localized and the tightness of the bonding could be evaluated.

In samples treated with One-Step followed by uncured Resinomer, the four layers of the restoration could be distinguished (Figure 3A). The One-Step bonding agent formed a layer of uniform thickness, apparently penetrating into dentinal tubules, with formation of a hybrid layer at the dentinal surface. The interface between the Resinomer and amalgam was typically rough, with occasional gaps. The samples in which amalgam condensation was performed over cured Resinomer exhibited a similar penetration of the One-Step bonding agent into dentin (Figure 3B), whereas the interface between

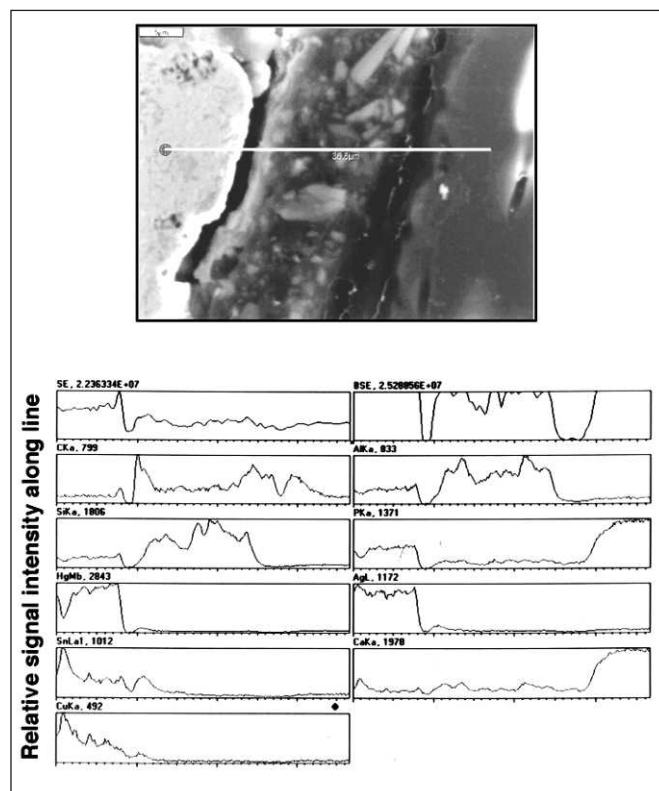


Figure 5: Elemental analysis based on EDS carried out on a sample with One-Step + cured Resinomer at the interface (Group B). Details about SE, BSE and EDS profiles are as described in the legend for Figure 4. The linear ratio between the image and the chart is 1:1.22.

Resinomer and amalgam showed multiple apparent gaps and irregular inclusions of material within the amalgam. These inclusions were further analyzed (by EDS) and identified as consisting of Resinomer. Direct application of Resinomer to the etched dentin resulted in a different morphology of the dentin-restoration interface: a gap was formed at the dentin-adhesive interface with no apparent hybrid layer (Figure 3D). The interface between amalgam and Resinomer showed an irregular surface with deep and wide inclusions of Resinomer within the amalgam, with apparent gaps between dentin and the adhesive layer (Figure 3C, arrowhead). Condensation of amalgam over the set Resinomer (Figure 3D) showed irregularities at the amalgam-Resinomer interface with penetration of amalgam creating a convoluted engagement with a tight junction between the two phases.

Direct condensation over etched surfaces (Figure 3E) resulted in the formation of a gap of 2-10 microns between dentin and amalgam phases (Figure 3E, arrowhead). Control specimens in which amalgam was condensed over the set One-Step layer (Figure 3F) showed a tight adhesion of the One-Step to dentin, with the formation of a hybrid layer sealing the dentinal surface. However, poor adhesion was observed at the amalgam-



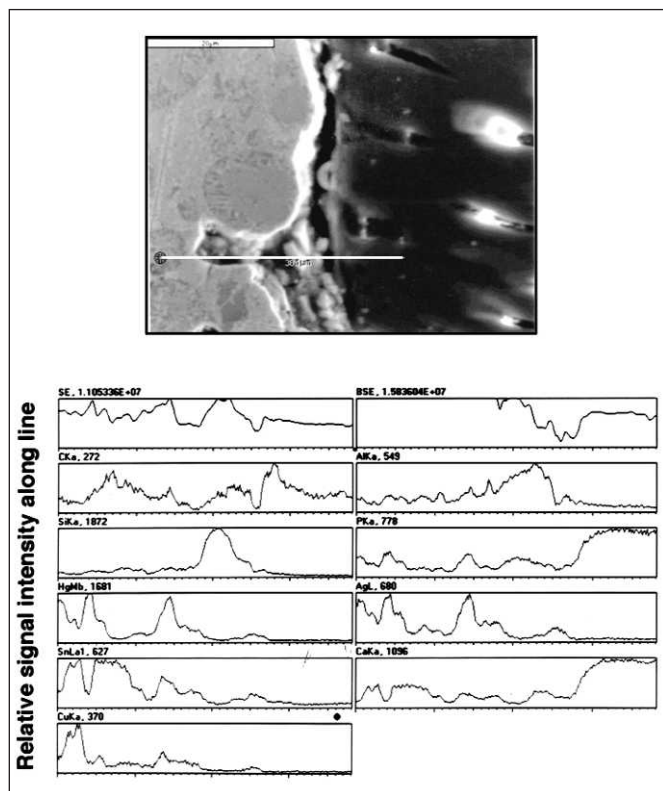


Figure 6: Elemental analysis based on EDS carried out on a sample with uncured Resinomer at the interface (Group C). Details about SE, BSE and EDS profiles are as described in the legend for Figure 4. The linear ratio between the image and the chart is 1:0.85.

One-Step interface, displaying extensive gaps ranging in width from a few microns to more than 10 microns.

### Elemental Analysis

To closely examine the various interfaces between dentin, bonding, adhesive and amalgam, SEM equipped with EDS was used. This approach enabled the authors to perform elemental analysis, which provided unequivocal information about the chemical nature of each layer and the gaps between them. In this analysis, amalgam was characterized by a signal of secondary electrons (metals) and back-scattered electrons (heavy elements), and the high content of mercury (Hg) and silver (Ag), tin (Sn) and copper (Cu), as demonstrated by EDS elemental analysis. The Resinomer used was characterized by a “noisy” profile of secondary and back-scattered electrons and was marked by high levels of aluminum (Al) and silicate (Si). The bonding agent was characterized by a high carbon (C) peak and a low level of back-scattering (due to its high content of light atoms). The dentin displayed high signals for calcium (Ca) and phosphate (P) and high back-scattering (though, lower than the amalgam). Examination of specimens containing both One-Step and Resinomer (uncured or cured, in Figure 4 and 5, respectively) exhibited a continuous and tight adhesion, with distinct transition between the dentin and the

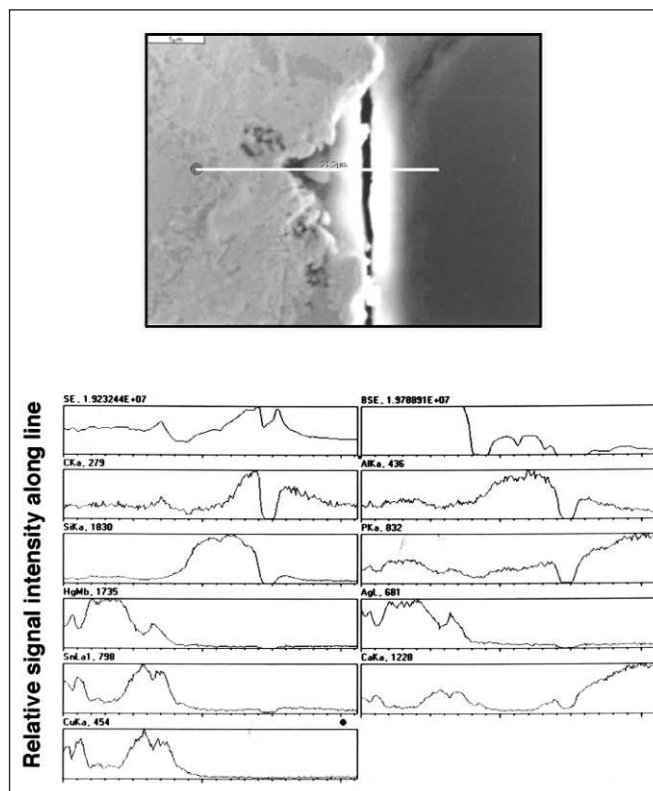


Figure 7: Elemental analysis based on EDS carried out on a sample with cured Resinomer at the interface (Group D). Details about SE, BSE and EDS profiles are as described in the legend for Figure 4. The linear ratio between the image and the chart 1:0.84.

One-Step layer, as well as between One-Step and the Resinomer. In these specimens, penetration of resin into the dentinal tubules is observed (5-10 µm), and the bonding layer itself is 10-20 µm thick. The interface with the amalgam bulk, on the other hand, was characterized by multiple gaps ranging between 2-5 microns. The gaps formed between amalgam and cured Resinomer in Figure 5 seemed continuous compared to samples with uncured Resinomer in which intermittent gap was observed (Figure 4). This was consistent with the higher values of microleakage observed in the same samples. In line with the SEM data, the elemental analysis indicated that these gaps were empty and contained none of the components of the restoration. In addition, specific examination of apparently non-metallic inclusions detected within the amalgam phase (Figure 3B, C) confirmed that they were derived from the Resinomer phase (not shown). Such inclusions, similar in size and frequency, were formed in samples where amalgam was condensed over either cured or uncured Resinomer. In specimens with no bonding agent (Figure 6 and 7), Resinomer was applied directly on the dentinal surface and was either cured or uncured before condensation of amalgam. In these specimens, a gap of 2-5 µm is formed between dentin and Resinomer, while the

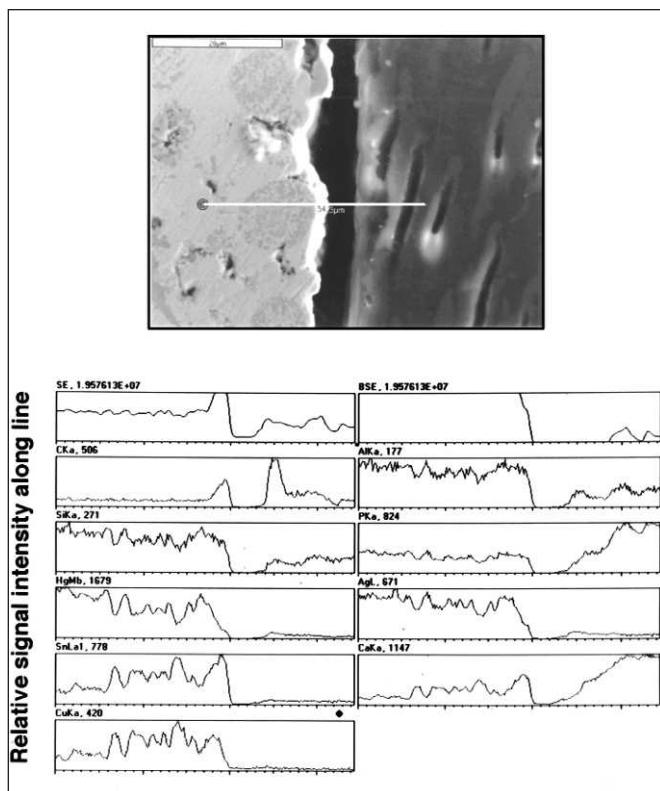


Figure 8: Elemental analysis based on EDS carried out on a sample with no intermediate substance at the interface (Group E). Details about SE, BSE and EDS profiles are as described in the legend for Figure 4. The linear ratio between the image and the chart is 1:0.80.

gap between amalgam and Resinomer is hardly detectable. In specimens where no intermediate bonding or adhesive was used (Figure 8), a prominent gap of 5-10  $\mu\text{m}$  was formed between dentin and amalgam. The elemental analysis shows no evidence of any organic material in this gap. In specimens with One-Step only (Figure 9), penetration of resin into dentin to a depth of about 10  $\mu\text{m}$  could be detected, and an obvious gap between this bonding layer and amalgam could be noted.

Spectra of amalgam and dentin and Resinomer were taken separately (not shown). These spectra showed that the P window superimposes partially with the Hg peak (2.00-2.40), and the Ca window (3.5-3.8) with Sn peaks, so that some of the intensity of the Hg in the amalgam shows as an apparent concentration of P, which is an artifact.

## DISCUSSION

Application of adhesive resins as an intermediate layer between dental surfaces and amalgam restorations has become a common procedure (Winkler & others, 1997, Mahler, 1996), yet the optimal mode of sequential application of adhesive and amalgam was not rigorously tested. Various amalgam adhesives were developed for this

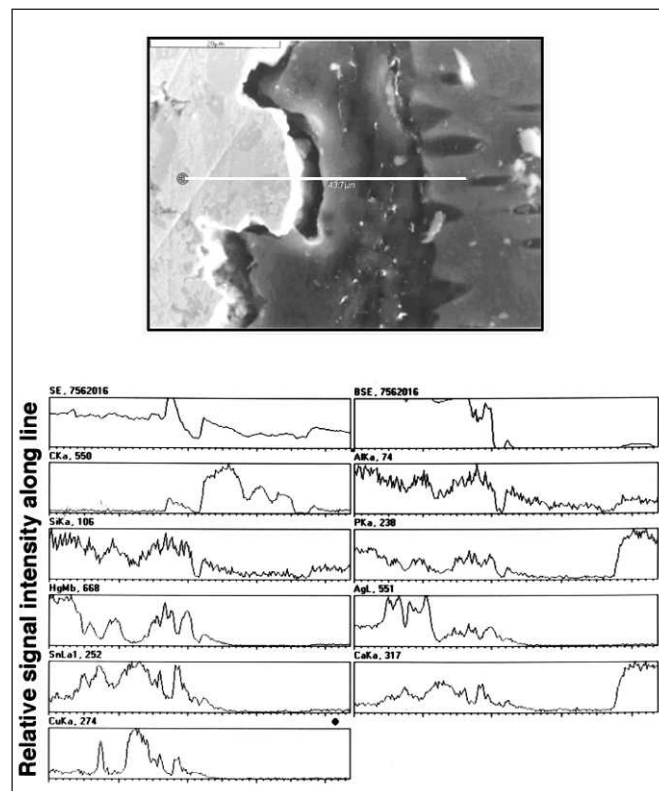


Figure 9: Elemental analysis based on EDS carried out on a sample with One-Step at the interface (Group F). Details about SE, BSE and EDS profiles are as described in the legend for Figure 4. The linear ratio between the image and the chart is 1:1.00.

purpose. Resinomer was chosen for this study due to its wide use, and the information presented here might be applicable to similar or equivalent materials. According to manufacturer's recommendations, "Condensation of amalgam into the uncured (oxygen inhibited) Resinomer layer creates a tenacious mechanical bond to amalgam." Resinomer is composed of Bis-GMA and Hydroxyalkyl Methacrylate resin filled with glass particles, which also comprise the typical filling particles of glass ionomer materials. Questions regarding the type of bonding created between this material have been raised, and in this study, the authors tried to elucidate some of these issues by combining conventional microleakage assay and ultra-structural examination of the same samples using SEM. According to the manufacturer's instructions, a bonding agent (One-Step) should be applied prior to application of the adhesive Resinomer. This study evaluated the relationship and specific contribution of the two components to the tightness of the adhesion. As shown here, application of the bonding agent is indeed crucial for the formation of a tight seal with little microleakage. Thus, all samples treated with One-Step exhibited low levels of microleakage. This reduction in microleakage is related to the tight sealing of the dentinal surface and forma-

tion of a hybrid layer (Nakabayashi, Ashizawa & Nakamura, 1992) resulting from the penetration of the bonding agent into the dentinal tubules. On the other hand, restorations in which direct application of Resinomer to the etched surface without an intermediate layer resulted in a high degree of microleakage. Some penetration of Resinomer to the opened tubules was detected, but this presumably occurred with no interaction with the collagenous meshwork at the intertubular dentin. Partial inhibition of microleakage is observed when Resinomer is allowed to set before condensation of amalgam (D). This may be due to the fact that the thicker set layer provides a somewhat better seal, though not necessarily tight enough, and high levels of microleakage are also exhibited by these samples. This observation might have an interesting implication on the potential route of dye penetration. Examination of restorations under SEM often revealed multiple gaps between the different phases of the restorations. In One-Step-containing restorations, such gaps were detected mainly along the Resinomer-amalgam interface, and the dye penetration observed was probably at this location. In restorations in which the Resinomer was applied directly on the dentinal surface, gaps were observed at the Resinomer-dentin interface and dye penetration was detected in the dentinal tubules, similar to the effect observed in control specimens lacking any intermediate layer. This may be consistent with the view that Resinomer tends to shrink during setting, and thus detaches from the surface with which it forms weaker bonds. Apparently, interactions between Resinomer and One-Step are tight and stable, and Resinomer tends to detach from the amalgam phase, whereas, in the absence of One-Step, Resinomer tends to bind more strongly to amalgam and detaches from dentin. Turner, Germain & Meiers (1995) have postulated that the shrinkage of resin at the interface is a major cause for gap formation. This observation also suggests that dentin bonding plays a crucial role in blocking microleakage. Another aspect observed in this study was the state of Resinomer at the time of amalgam condensation. Displacement of the adhesive, formation of inclusions in the amalgam and formation of extensions and protrusions at the surface amalgam were observed both in specimens with immediate condensation over uncured Resinomer, or delayed condensation over cured Resinomer, indicating that the "oxygen-inhibited-layer" plays an active role in adhesion.

The clinical implications of this study are that for the prevention of microleakage, applying a bonding agent such as One-Step is obligatory, though, as shown by the ultra-structural evaluation, One-Step by itself is insufficient for bonding to amalgam. Thus, in order to create a tight bond between the tooth and amalgam, the combination of One-Step and Resinomer is essential. From the different combinations studied, it seems that the

best bonding of amalgam to tooth surfaces is provided by applying a bonding agent followed by a resinous adhesive material and immediate condensation of amalgam.

## CONCLUSIONS

1. Bonding gel (such as One-Step) applied over the etched cavity surface is essential for the prevention of microleakage in the bonded amalgam restoration.
2. Adhesion to amalgam by the resinous adhesive layer is mainly formed by interdigitations and mechanical interaction.
3. The "oxygen-inhibited layer," which remains at the surface of the resinous adhesive layer after polymerization, may play a significant role in the adhesion to amalgam, though quality of adhesion may be reduced.
4. The best procedure for bonding amalgam to tooth surface would be by application of a bonding agent over the etched surface, followed by a resinous adhesive material over it and immediate condensation of amalgam.

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# The Effect of Acid Etching on Vascular Diameter of Pulp-Vessels in Rat Incisor (Vitalmicroscopic Study)

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## Clinical Relevance

In the dental pulp of rats, acid etching applied for 15 or 20 seconds in very deep cavities has no deleterious effect; however, prolonged etching time results in failure in microcirculation of the rat dental pulp.

## SUMMARY

Conditioning agents used on dentin with composite materials are biologically active and may have deleterious effects on the pulpal microcirculation. No data are available on the immediate vascular effect of etching materials applied on a constant thin pulpal dentin. In this study the authors examined whether the application of 36% phosphoric acid (Conditioner 36, 15 seconds) or itakonic acid with 10% maleic acid (NRC Non-Rinse Conditioner, 20 seconds), as recommended by the manufacturers, alters the blood circulation in the pulp of the rat's lower incisors. The effect of pro-

longed etching time (60 seconds) was also assessed (Conditioner 36). The application of saline served as the untreated control. The technique of vitalmicroscopy was used on the first lower incisor of 40 (10-10 in each group) male Sprague-Dawley rats (weighing  $350 \pm 8$  g SE) to record the changes in vessel diameter prior to and at 5, 15, 30 and 60 minutes after the test materials were administered on the dentin. In the control rats, the vessel diameter was stable during the entire experiment. Acid conditioning as recommended by the manufacturers tended to cause vasodilatation, though these alterations were statistically not significant when compared to the control group (ANOVA,  $p > 0.05$ ). After prolonged etching time (Conditioner 36, 60 seconds) significant vasoconstriction ( $-14.4 \pm 6.13$ ;  $-10.59 \pm 4.2$ ;  $-11.96 \pm 6.75$ ;  $-5.49 \pm 5.78\%$ ) was observed (ANOVA,  $p < 0.05$ ). In this group, stasis developed in pulpal blood circulation in 40% of rats (Cochran's-Q test,  $p < 0.05$ ), gas-bubble formation was observed in 30% and the disappearance of the pulpal wall occurred in 20%. These results suggest that exposition time with acid is crucial to the pulpal microcirculation. That is, acid conditioning applied as indicated (for 15-20 seconds) onto a very thin layer of dentin only slightly affects the blood supply to the dental pulp; however, prolonged etching time (for 60 seconds) results in

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## immediate failure of microcirculation in the dental pulp of rats.

### INTRODUCTION

Remarkable advances in adhesive dentistry have led to the extensive utilization of polymeric materials. Recent developments, such as the introduction of total-etch bonding systems, have affected some basic concepts of restorative dentistry. To enhance bonding of a composite filling material, the entire adherent surface of the cavity—including the pulpal dentin—is etched, providing the possibility (with bonding systems) of improved microretention. Etching materials—being chemically active and cytotoxic (Chan & others, 1999)—placed directly on the dentin could adversely influence the pulp. One of the first signs of the resulting pathological changes is the impaired pulpal microcirculation (Kim, 1985).

The thickness of the remaining dentin over the pulp is crucial in protecting the pulp from toxic materials, however, in clinical situations and in the recommended histopathological biocompatibility-tests (ISO, 1984), the distance of the material tested from the pulp is not known. Histopathological tests are recommended (pre-clinical pulp and dentin usage test) to evaluate the biocompatibility of restorative materials *in vivo* (Stanley, 1994; ISO, 1992). However, there are some shortcomings with these tests (Kanca, 1990; Quist, Stolze & Quist, 1989) as follows: not specified remaining dentin thickness (RDT), the disturbing effects of microleakage (Quist, 1993; Bergenholtz, Cox & Loesche, 1982; Felton, Bergenholtz & Cox, 1989), confounding variable of bacterial contamination (Mjör, 1974; White & others, 1994) and problems concerning the Zinc Oxide Eugenol control groups (Brännström, Nordenvall & Torstenson, 1981; Kanca, 1990; Cox & others, 1987). To provide supplemental information to the findings of the recommended *in vivo* usage tests and to continuously study the immediate vascular changes on the living pulp, investigations have been performed using pulpal vitalmicroscopy in rats. This study aimed to find out whether acid etching applied (without any liner) directly onto a constant, thin pulpal dentin alters the blood circulation of the pulp of the rat's lower incisors.

### METHODS AND MATERIALS

#### Materials

Conditioner 36 (DeTrey, contains 36% o-phosphoric acid, pH<05) was applied on the prepared surface for 15 seconds (Condi-15sec); NRC Non-Rinse Conditioner (DeTrey, contains itaconic acid, maleic acid pH $\approx$ 1.1) was applied on the prepared surface for 20 seconds (NRC-20sec). The manufacturer's instructions (DeTrey, Dentsply, D-78467 Konstanz) were followed. The effect of prolonged etching time (for 60 seconds) was assessed with Conditioner 36 (Condi-60sec). The application of saline served as sham-treated control (control).

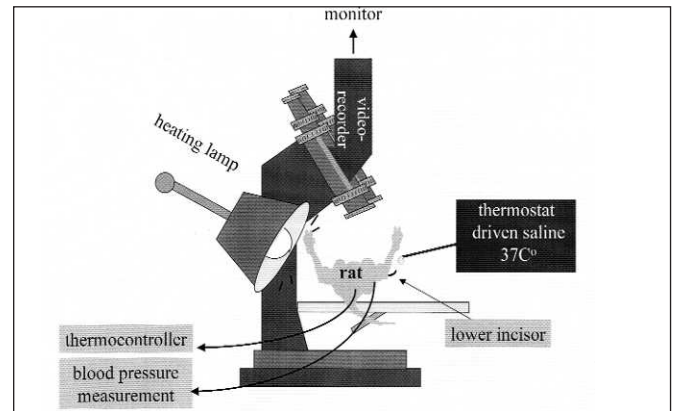


Figure 1. Experimental setup for vitalmicroscopy.

#### Animal Preparation

The technique of vitalmicroscopy was used on the first lower incisor of 40 (10-10 in each group) male Sprague-Dawley rats (weighing  $350 \pm 8$  g) anesthetized with pentobarbitone sodium (Nembutal 35 mg/kg, ip, supplemented as required). Figure 1 shows the experimental setup. Breathing was facilitated by tracheal cannulation, and arterial blood pressure was monitored through a cannula inserted into the left femoral artery. A heating lamp was used to keep the body temperature at a constant 37°C. The lower jaws were separated by transecting the connective ligaments between them and the mucous membrane was retracted from the left part. This jaw was fixed with a circular polyester strip to establish a firm grip for further preparation. The distal and mesial surfaces of the left lower incisor and some part of the alveolar bone were ground away with a diamond fissure bur at about 15,000 rpm. The excavation was made from the labial to the buccal margins and from the alveolar bone to the incisal edge. After removing the enamel and part of the alveolar process, grinding was continued under a dissecting binocular microscope (1.6x6.3 Nikon, Japan) until the pulp vessels became clearly visible through the remaining dentin. Special care was taken to ensure uniform controlled preparation. The applied pressure was kept to a minimum and the exposed dentin was rinsed with a constant temperature (37°C) saline solution. When grinding was completed, a thin plate of dentin (approximately 15-25  $\mu$ m in thickness) still remained intact over the delicate pulpal tissue.

Measurements were performed under a Nikon stereo light microscope (10x0,255+1x16) equipped with a video camera and an image amplifier (500x, Sony CCD-IRIS). The exposed dentin was kept wet at a constant temperature by a thermostat-driven continuous saline rinsing. After one-hour equilibration, a suitable arteriole ( $40 \pm 2$   $\mu$ m) was chosen for the measurement of its inner diameter, and this baseline vessel diameter was measured on the monitor (on the screen 1 mm  $\approx$  2  $\mu$ m in the pulp).



Subsequently, etching materials or saline solution (control group) were administered on the prepared surface. Alterations in vascular luminal diameter of a pulpal arteriole were measured prior to and at 5, 15, 30 and 60 minutes after the etching materials or saline (control) were administered on dentin.

Changes of arterial diameter were compared to those registered prior to the application. The alterations were expressed as a measurement of the percent of increase or decrease in diameter of the vessel (mean  $\pm$  SE) in each examined time. For statistical analysis, two-way ANOVA was applied with treatment and time as factors. Normal distribution was tested by Kolmogorov-Smirnov test. Treatments were compared to control by LSD method. Differences in the frequency of pulpal stasis were evaluated by Cochran's-Q test (extension of McNemar's Chi-square test). A significant level less than or equal to 0.05 was chosen to indicate statistical significance.

## RESULTS

The systemic arterial pressure measured in the femoral artery ( $114.3 \pm 5.8$  mmHg) remained unchanged during the experiment, both in control and in test animals. In control rats, no significant changes were observed in the vessel diameter (mean diameter during the period tested:  $37.78 \pm 2.83\mu\text{m}$ ).

Acid conditioning with Conditioner 36 for 15 seconds (Figure 2) tends to cause vasodilatation ( $7.96 \pm 4.34$ ;  $5.76 \pm 5.31$ ;  $-0.28 \pm 3.61$ ;  $0.12 \pm 3.74\%$ , respectively), as did the application of NRC Non-Rinse Conditioner for

20 seconds ( $5.1 \pm 7.15$ ;  $9.23 \pm 8.87$ ;  $6.15 \pm 6.49$ ;  $5.33 \pm 7.25\%$ ); however, statistical comparisons to the control values did not reveal any significant difference (ANOVA,  $p > 0.05$ ). There was no sign of stasis, prestasis or any other alterations in the dental pulp of these groups.

After acid etching for 60 seconds, significant vasoconstriction ( $-14.4 \pm 6.13$ ;  $-10.59 \pm 4.2$ ;  $-11.96 \pm 6.75$ ;  $-5.49 \pm 5.78\%$ ) was found when compared to the control. Changes over time, however, were statistically non-significant. On the other hand, obvious and serious changes in pulpal microcirculation developed; namely, stasis in 40% of the rats (Cochran's-Q test,  $p < 0.01$ ). This was observed in three specimens immediately, and in one specimen 15 minutes after application of the acid. Gas-bubble formation was observed in 30%, a total disappearance of the pulpal dentin occurred in 20% of the rats.

## DISCUSSION

Several laboratories have investigated the biocompatibility of dental etching materials but these studies have yielded conflicting results (Kanca, 1990). Considerable studies have indicated that different factors affect pulpal responses to dental materials (Quist & others, 1989; Al-Dawood & Wennberg, 1993). These factors include cavity preparation, toxicity of the materials applied, incomplete marginal sealing, bacteria and bacterial products along cavity walls. The growing use of resin restorative materials with various cavity treatments makes it essential to distinguish among relevant factors determining the pulpal reactions to resin restorations.

In this study efforts were made to standardize the thickness of remaining dentin. Direct measurement of RDT was not achievable within this experiment; however, the optical density of dentin made it possible to precisely judge RDT over the pulp. Many studies have shown (Pohto & Scheinin, 1958; Kim & others, 1984) that the microscopical observation of the pulpal circulation is only feasible through constant ( $15\text{--}25\mu\text{m}$ ) thin pulpal dentin layer. More than  $25\mu\text{m}$  dentin thickness hinders the visibility of the moving red cells. In this study preparation was always carefully performed under a dissecting binocular microscope by the same investigator until the circulation in the pulp vessels became clearly visible through the remaining dentin. The availability of the preparation can be judged by

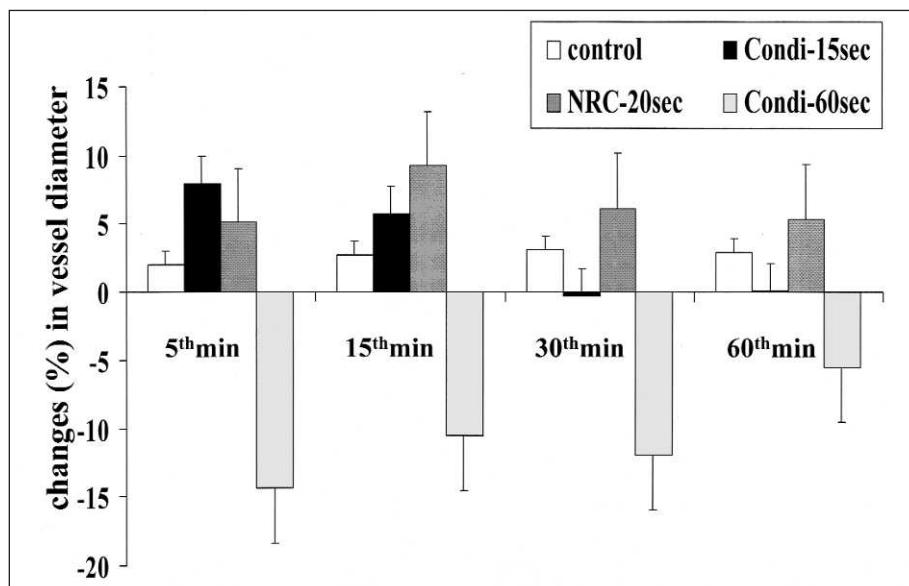


Figure 2. The average of changes (%) in arteriole diameters in rat dental pulp treated with various etching procedures (black bars = Condi-15 sec: Conditioner 36 for 15 seconds; gray bars = NRC-20 sec: NRC for 20 seconds; dotted bars = Condi-60sec: Conditioner 36 for 60 seconds) or with saline (empty bars = control group). Each column represents a mean value ( $\pm$  SE) expressed as % changes of the baseline vessel diameter.

close observation of the microcirculation during the one-hour equilibration time.

Problems concerning the bacterial contamination (Cox & others, 1987; White & others, 1994) are excluded in this model via the short-term observation. Pulpal reactions seen in many dental materials usage studies seem to partly result from the bacterial colonization of gaps between restorative materials and tooth structures. The penetration of bacteria and bacterial products from these colonies cause continual immunological irritation of the pulp. The time necessary for bacterial colonization, diffusion and immunological pulpal reactions seems to exceed the duration of this experiment (Meryon, Jakeman & Browne, 1986). The possibility for bacterial irritation was further reduced by the continuous irrigation with sterile physiological saline of the tooth fixed outside the oral cavity. The chances for bacterial irritation and bacterial pulpal reaction were the same in the control and test groups. Thus, when changes were observed in the pulp circulation (as compared to the sham-treated control group), they were due to the effect of the investigated material per se.

As irreversible circulatory changes (such as stasis or prestasis) have not been observed in group Condi-15sec and group NRC-20sec, the thin layer of dentin seems sufficient to protect the dental pulp (Hume, 1994) from the effect of acids when applied as recommended by the manufacturer. A vasodilatation appears immediately after application that can be considered as a protective response (Pashley, 1996). Because of the enhanced pulpal blood flow, the rate of outward flow of fluid through the dentinal tubules increases (Vongsavan & Matthews, 1994), reducing the rate of diffusion of acid from the cavity surface to the pulp (Pashley & Matthews, 1993). On the other hand, the increased pulpal circulation can enhance the washing-out of acids penetrating into the pulp (Pashley & Pashley, 1991). Thus, in this study, the noticed tendency for vasodilatation can be a protective mechanism against the diffusion of toxic material to the pulp.

These results support the assumption that acid etching, when applied as recommended by the manufacturer, has no deleterious effect on pulpal circulation (even in deep cavities); however, it may have a minimal protective acute effect on the pulpal tissue. The vascular responses evoked by group Condi-15sec and NRC-20sec materials were nearly identical.

Phosphoric acid (35%) etching for 60 seconds demineralizes the underlying dentin up to 25µm (Uno & Finger, 1996). Thus, in group Condi-60sec during the prolonged etching time, phosphoric acid can demineralize the full remaining dentin thickness, getting into the pulp in large quantities, causing pulpal vasoconstriction and acute pulpal necrosis. This result is consistent with the findings of Nakanishi, Gu & Momma (1996),

who showed that acids coming in direct contact with blood vessels cause vasoconstriction in rabbit mesenteric artery. Pihan & others (1986) have shown vascular stasis caused by topical administration of acids with the method of vitalmicroscopy on gastric mucosal capillaries.

Vitalmicroscopy allows persistent observation of pulpal circulation, thus the vascular response—evoked by dental materials—is continuously assessable. The stable vessel-diameters in control animals serves as a good comparison during the period tested. The protocol used in this study does not allow for performing the observation over 60-minutes. Using this technique, the prompt reaction of pulpal microcirculation to the etching procedure can be noticed immediately after application. These results indicate that etching procedure is followed by the pulpal circulatory reaction within 15 minutes after application. On the other hand, changes in vessel diameter showed no apparent tendency for a progressively-increasing alteration and there is no reason to expect a delayed significant response after 60 minutes.

## CONCLUSIONS

Under present experimental conditions, the initial effect of etching per se seems tolerable to the pulp when applied as recommended by the manufacturers. Thus, it might be concluded that the pulpal enfeeblement after acid etching, previously shown in many histopathological studies, could be the consequence of other factors such as improper seal of the treated cavity that resulted in bacterial invasion to the pulp.

On the other hand, on the basis of the authors' results, it may be concluded that prolonged etching time on a thin pulpal dentin is a serious hazard to the dental pulp. Therefore, extra care should be exercised in the treatment of deep cavities in order to adhere to the maximal etching time, which can be as long as 15-20 seconds (in agreement with the recommendation of the manufacturers). The results presented in this study supplement the histopathological findings provided by the investigations carried out under the ADA, FDI and ISO *in vivo* usage testing guidelines.

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# Influence of Different Restorative Techniques on Microleakage in Class II Cavities with Gingival Wall in Cementum

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## Clinical Relevance

The combination of amalgam/composite in Class II cavities with gingival margin in cementum could be an alternative to reducing microleakage.

## SUMMARY

This study compared marginal leakage of Class II cavities with gingival margin in cementum using different techniques. Twenty-four recently extracted third molars were used. Proximal standard box cavities were prepared in both mesial and distal surfaces. The gingival margin was located apical to the cemento-enamel junction. All the preparations and restorations were performed by the same operator. Standard cavities were randomly divided into three groups (n=16) and restored as follows: Group 1—light-cured composite resin; Group 2—self-cured composite resin + light-cured

composite resin and Group 3—amalgam + light-cured composite resin. After polishing, the teeth were thermocycled and their gingival margins exposed to dye. Specimens were sectioned and leakage scores observed in accordance with a standard ranking. Data were subjected to statistical analysis (Kruskal-Wallis). Results showed that the amalgam/resin composite combination demonstrated the least leakage.

## INTRODUCTION

Polymerization shrinkage is probably the greatest problem associated with resin composites (Lösche, 1999). The polymerization shrinkage of a composite resin can create contraction forces that may disrupt the bond to cavity walls, which cause marginal failure and subsequent microleakage (Davidson, de Gee & Feilzer, 1984). Secondary caries as a consequence of microleakage is the predominant reason for restoration replacement (Mjör & Qvist, 1997). One of the weakest links of Class II composite resin restorations is microleakage at the gingival margin of the proximal box. The absence of enamel at the gingival margin leads to the adhesion of composite materials to cementum/dentin, an unstable substrate (Coli & Brännström, 1993; Carvalho & others, 1996). While enamel is almost exclusively an inorganic tissue, dentin is less mineralized and contains more moisture, which can cause variations in adhesion (Eick & others, 1997).

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In cavities with margins limited to the coronal enamel, the contraction force during setting is counteracted by bonding to the beveled and etched enamel. In Class II cavities where the cervical margin is located on the root dentin apical to the cemento-enamel junction, contraction forces may exceed the adhesive strength to dentin of the bonding agents, producing a gap. Gap formation allows access to bacteria and their products to the pulp, and may lead to hypersensitivity (Opdam & others, 1998), pulpal inflammation and secondary caries (Cox, 1987).

The newer generation of dentin bonding agents exhibit bond strengths to dentin which are higher than 20 MPa (Eick & others, 1997). That is sufficient to resist the contraction stress (13-17 MPa) generated by polymerization shrinkage (Davidson, De Gee & Feilzer, 1984). However, despite the use of hydrophilic monomers, microleakage at cementum/dentin margins is still present (Coli & Brännström, 1993; Hilton, Schwartz & Ferracane, 1997).

Several different approaches to minimizing or eliminating microleakage have been proposed. One method is the layering technique with clear matrix/reflective wedge combination, which allows light curing through the wedge to produce shrinkage toward the gingival margin (Lutz & others, 1992). However, the clear matrix bands are more difficult to handle (Miller & others, 1996) and the ability of clear wedges to ensure the polymerization of the composites is limited (Ciamponi, Del Portillo Lujan & Ferreira Santos, 1994). Recent reports have indicated that microleakage is similar between the clear and metal matrix (Hilton, Schwartz & Ferracane, 1997; Neiva & others, 1998).

A first layer of chemically cured resin composite for the gingival aspect of a proximal cavity has been suggested (Bertolotti, 1991; Fusayama, 1992) in order to improve marginal adaptation. The outer portion is then restored with light cured composite for color match and stability and to minimize air bubbles (Garberoglio, Coli & Brännström, 1995). The rationale for this is based on the supposition that the contraction stress of the chemically cured resin will be directed towards the heat of the cavity walls instead of the center of the mass, avoiding the formation of a gap in the gingival area (Fusayama, 1992). However, the results of such techniques are controversial (Miller & others, 1996; Garberoglio, Coli & Brännström, 1995; Hilton, Schwartz & Ferracane, 1997).

The combined amalgam/resin composite restoration could be another alternative to limit microleakage in the gingival area (Cardash & others, 1990; Eidelman & others, 1990; Hovav & others, 1995; Holan, Chosack & Eidelman, 1996). Amalgam is a unique material since products of

corrosion will deposit in the tooth/restoration interface, sealing this interface in time (Berry, Nicholson & Troendle, 1994). However, initially, the seal may not be ideal for amalgam restorations due to initial contraction of the material. To avoid initial leakage, the bonded amalgam technique has been developed (Berry & Tjan, 1994).

This study was designed to assess and compare *in vitro* marginal leakage at the gingival wall of Class II composite restorations with margins in cementum, using different restorative techniques.

METHODS AND MATERIALS

Sample and Storage Medium

Twenty-four recently extracted human third molars were selected. After extraction the teeth were scaled, polished and stored in distilled water at 37°C till the test.

Cavity Preparation

Slot Class II cavities were prepared in both mesial and distal surfaces with a #1090 diamond bur in an air/water-cooled high-speed turbine. A new bur was used for every five preparations to ensure cutting efficacy. Only one operator prepared the standard cavities. The buccal-lingual extension of the cavities was 3 mm. Axial walls were prepared to a standard depth of 1 mm in dentin from the dentino-enamel junction.

The gingival wall was located approximately 1.5 mm apical to the cemento-enamel junction. The internal angles were rounded and cavosurface margins were sharp without bevel and finished with gingival margin trimmers.

Restorative Procedures

Table 1 lists materials, batch numbers and their respective manufacturers. The prepared teeth were mounted in impression putty (Express, 3M Dental Products, St Paul, MN 55144) to simulate as closely as possible clinical conditions and supporting structures.

Table 1: Materials Used in This Study		
Materials	Lot Number	Manufacturer
Composite Bisfil 2B	#089147	BISCO, Inc Itasca, IL 60143, USA
Z100	#950427	3M Dental Products St Paul, MN 55144, USA
Amalgam Logic C	#950801/950908	SDI Bayswater, Victoria, Australia 3153
Bonding Agent Scotchbond Multipurpose Plus	#23641	3M Dental Products St Paul, MN 55144, USA

The teeth were mounted three at a time to simulate proximal contact. An individual metal matrix (T matrix) was prepared for each tooth. Wooden wedges were used to stabilize the matrixes.

Teeth were randomly divided into three groups of eight teeth each (16 cavities). Scotchbond Multi-Purpose (SBM) Adhesive System (3M Dental Products, St Paul, MN 55144) was used according to manufacturer's instructions to treat all the preparations. The cavities were filled as follows:

**GROUP 1:** Light-cured–Enamel and dentin were etched for 15 seconds with 37% phosphoric acid gel; then a 15-second water rinse, followed by gently drying. This was followed by the application of one coat of primer with drying; one coat of adhesive resin composite and light curing for 10 seconds. Z-100 (3M Dental Products, St Paul, MN 55144) resin was used to incrementally fill the cavities. Three horizontal increments were placed, each layer polymerized from the occlusal aspect for 60 seconds.

**GROUP 2:** Self-cured–Cavities were treated with SBM system in a manner similar to Group 1. As the first increment for the gingival area, a 2 mm layer of Bisfil 2B (BISCO, Inc, Itasca, IL, USA 60143) was used. Equal quantities of base and catalyst were mixed and applied to the cavity and allowed to cure for four minutes. After that two increments of Z100 were added, each being light-cured for 60 seconds from the occlusal aspect.

**GROUP 3:** Combined amalgam/composite–In this group, SBM was used as a dual cure adhesive system. After acid etching, primer was applied and gently dried. Adhesive resin plus catalyst were mixed and applied to the cavity. Capsulated amalgam was used and condensed into the cavity as a 2 mm initial layer on the gingival wall over the unpolymerized adhesive. After five minutes of setting the activator + primer of SBM were applied before the application of catalyst and adhesive. This was followed by two increments of the resin composite to fill the cavities. Each increment was exposed to a 60-second light activation.

To light cure the adhesive material and the resin composite, a curing unit (XL 1500, 3M Dental Products, St Paul, MN 55144) was used. The output level was greater than 450 mW/cm<sup>2</sup>. A Teflon instrument was used to insert and condense the resin composite, and amalgam was carved with a Ward instrument.

**Finishing/Polishing**

After 24 hours, finishing and polishing was performed. There was a minimal need for finishing, which was carried out using 30 blade carbide burs. Polishing of the resin material was conducted with Sof-Lex disks (3M Dental Products) and Prisma Gloss paste (Dentsply/

Caulk, Petrópolis, RJ, Brazil). Amalgam was polished with abrasive points. After polishing, specimens were removed from the impression material and stored for one week in distilled water at 37°C.

**Thermocycling and Microleakage Test**

The apex of each tooth was sealed with a fast-set adhesive material (Araldite, Brascola Ltda, São Paulo, Brazil) and two coats of fingernail varnish were used to coat all tooth structure except the restorations and 2 mm around them. All specimens were subjected to 500 thermocycles between 5°C and 55°C, with 30-second dwell time at each temperature.

Specimens were immersed in 0.5% methylene blue for eight hours. After that specimens remained in tap water for 12 hours and were then cleaned to remove the fingernail varnish. All the specimens were sectioned longitudinally in a mesiodistal direction towards the center of the restorations using a diamond disk. The specimens were analyzed by two calibrated evaluators under magnification (X20) in a stereomicroscope. Leakage values were attributed according to a ranking: 0 = no leakage; 1 = leakage at the gingival wall; 2 = leakage at the axial wall.

**Statistical Analysis**

Data was subjected to statistical analysis using a non-parametric test (Kruskal-Wallis), at  $p<0.05$ .

**RESULTS**

The majority of specimens in Groups 1 and 2 exhibited the highest leakage values. The majority of specimens in Group 3 demonstrated no leakage. Leakage values for the different groups are shown in Table 2.

The statistical analysis using non-parametric Kruskal-Wallis test showed that Groups 1 and 2 had similar results and both exhibited statistically higher leakage than Group 3 ( $p<0.05$ ).

**DISCUSSION**

Posterior resin composite restorations have gained popularity in recent years because they are tooth-colored, mercury free, thermally non-conductive and bond to tooth structure with the use of adhesive agents

Table 2: *Cementum Leakage Score*

Groups	Leakage Scores			Total
	0	1	2	
1	5	1	10	16
2	4	1	11	16
3	12	-	4	16
<b>Total</b>	<b>21</b>	<b>2</b>	<b>25</b>	<b>48</b>

Kruskal –Wallis test detected similar results between Groups 1 and 2, and both exhibited statistically higher ( $p<0.05$ ) leakage than Group 3.



(Ferracane, 1995; Hilton, Schwartz & Ferracane, 1997).

The marginal seal can generally be preserved around cavity preparations when cavosurface margins are restricted to enamel. This is due to the strong adhesion achieved with this inorganic tissue (Carvalho & others, 1996).

For dentin, on the other hand, the values of the internal stress are often larger than the bond strength to dentin walls (Feilzer & others, 1987), and consequently a gap may form at the interface (Kinomoto & Torii, 1998). This polymerization shrinkage, which is inherent to composites, is considered mainly responsible for the gap formation. Modern posterior composites undergo volumetric contractions of between 2.6% to 4.8% (Lösche, 1999). The different coefficients of thermal expansion and Young's Modulus between composites and tooth structure tend to exacerbate the interfacial gap which leads to microleakage, the predominant reason for replacement of composite restorations (Mjör & Qvist, 1997; Yoshikawa & others, 1999).

Layering techniques of composite resin and the use of clear matrix and reflective edges were advocated as an efficient method to eliminate polymerization stress (Lutz & others, 1992). However, the ability of reflective wedges to cure resin composite has been contested (Ciamponi, Del Portillo Lujan & Ferreira Santos, 1994). In addition, a recent report (Neiva & others, 1998) has found that the incremental filling technique using a clear matrix and reflective wedges demonstrated the worst result in Class II resin composite restoration when the cervical wall was in cementum. The proximal contact is also more difficult to obtain using clear matrix (Miller & others, 1996). Consequently, in this study the authors used a metal matrix and wooden wedges.

Their results disclosed similar leakage patterns for both self-cured and light-cured composites and both techniques demonstrated statistically higher leakage than amalgam/composite combined restorations.

Similar to the results of this study, Hilton, Schwartz & Ferracane (1997) verified that microleakage occurred in almost every specimen in both light and self-cured composite composite restorations in gingival cementum/dentin margin. They also found that both techniques disclosed similar leakage values. Another finding observed in their work was the higher presence of voids in self-cured composite restorations.

In a clinical study, Van Dijken & Hörsted (1998) verified similar patterns of marginal adaptation between one self-cured composite (Bisfil 2B) and a light-cured composite (Aelitefill) in Class II cavities. Such findings suggested similar performances for both materials.

The leakage observed with light-cured technique could be explained because of the stress developed during

polymerization of the resin composites. Despite the results of recent papers contesting it (Versluis, Tantbirojn & Douglas, 1998), polymerization of the resin towards the light source remains the most accepted concept (Ferracane, 1995). Thus, the gingival wall located in cementum represents a longer distance to the light source that could increase polymerization stress, leading to greater leakage values. Kinomoto & Torii (1998) found a polymerization stress of 8-23 MPa in lateral walls and 11-23 MPa in pulpal wall. Since these values may be higher than the adhesion obtained to cementum margins in deep cavities, gaps could occur resulting in microleakage. It has been stated that bond strengths higher than 17 MPa will be sufficient to eliminate microleakage (Davidson, De Gee & Feilzer, 1984). According to Bayne & others (1994), under optimal conditions hydrophilic adhesive systems exhibited 18-22 MPa of shear strength to dentin, which is similar to those values obtained for enamel. The adhesive system used in this study has demonstrated bond strength values higher than 20 MPa (Carvalho & others, 1996; Eick & others, 1997). However, it is important to point out that these performances resulted from a specific testing methodology which may be more favorable than the clinical reality (Carvalho & others, 1996) and the validity of which is becoming controversial (Versluis, Tantbirojn & Douglas, 1997). When producing a bonded restoration in mouth, adhesion may be influenced by numerous parameters, most of which represent potential obstacles to the adhesion process. Also, recently, Hasegawa & others (1999) found that higher bond strengths cannot be used to ensure good marginal adaptation of composite restorations.

Another observation is that the authors used proximal box restorations with a design that increases the C-factor, thereby producing higher contraction stress (Carvalho & others, 1996). C-factor is the ratio between bonded walls over free walls (Feilzer & others, 1987). The design of the cavity may partly control the contraction stress. In 3-D cavities, the flow ability of the composite is reduced, increasing the stress. In such situations, lower adhesion may disrupt the adhesion to cementum, resulting in microleakage (Carvalho & others, 1996).

In this study the authors employed the incremental technique using horizontal layering. This technique increased the C-factor. If the oblique technique was applied, the C-factor could be reduced from 2 to 1.5. Yoshikawa & others (1999) reported that the higher the C-factor, the lower the adhesion when they used the same adhesives in cavities with C-factor 1 and C-factor 3. In the deep portion of a box cavity, bond strength is significantly reduced.

The rationale for the usage of a self-cured composite in the proximal box is that the adhesive chemical ini-

tiator will accelerate polymerization of the chemically-cured composite in contact with the adhesive itself. The composite curing will be directed toward the cavity wall (Bertolotti, 1991) and potentially counteract the tendency of composites to shrink toward the center of the mass. This curing "towards the tooth" is enhanced by the tendency of chemically-cured composites to begin polymerizing in the warmest area of the preparation, namely the tooth-restoration interface. Also, other factors might play a role in this attempt to control shrinkage direction: retardation of the curing allows better chances for dentin bond formation and stress relief, and a higher compliance of the core of the shrinkage could be due to the free surfaces offered by the porosity of chemical curing composites (Carvalho & others, 1996).

Despite these suppositions, using finite element analysis, Versluis, Tantbirojn & Douglas (1998) found no significant differences between light and self-cured composites in relation to the direction of stress contraction. Similar performances between both materials were found *in vivo* by Van Dijken & Hörssted (1998).

The combination of amalgam in the cervical wall and resin composite to fill the cavity demonstrated the best results in this study. The combination of amalgam and resin has been indicated to improve the esthetic aspect of the complex amalgam restorations (Roda & Zwicker, 1992) and to reinforce tooth structure that will receive an amalgam restoration (Franchi & others, 1994).

Amalgam remains the most used direct material in posterior teeth. The material presents several advantages: low cost, long-term clinical evaluation, good resistance to wear, decrease of leakage with aging, low technique-sensitivity, easy to place in a single appointment and non-abrasive to opposing teeth in function (Berry, Nicholson & Troendle, 1994). Nevertheless, some disadvantages are related to the material. Amalgam does not compare esthetically with composite or ceramics. Neither does it bond to the tooth, leading to removal of sound tooth structure to provide retention. These limitations have increased the application of composite restorations.

Amalgam is the only self-sealing material, but the sealing of the interface occurs in a slow rate and initially the material may present microleakage. The use of copal varnish is not an efficient method to avoid initial leakage. Dentin bonding agents can be used to reduce marginal leakage when compared to copal varnish under amalgam restorations (Berry & Tjan, 1994). In this study a dual curing adhesive system was used to reduce initial microleakage (Berry & Tjan, 1994).

The reduced microleakage found in combined amalgam and composite restorations could be related to the condensability of amalgam since it can be intimately adapted to the tooth structure at the gingival wall (Cardash & others, 1990). In contrast, the softer resin

composite tends to be drawn and adhere to the condenser during compression against the wall of the cavity. If condensable composites were employed, other results might be expected. This is currently being tested in the authors' laboratory.

Others laboratory studies using the combined amalgam and composite restorations are in agreement with the results of this study (Eidelman & others, 1990; Cardash & others, 1990). In a clinical evaluation of Class II restorations in primary molars, Holan, Chosack & Eidelman (1996) observed that combined amalgam/composite restorations performed in a similar manner as composite restorations. They found differences only in color match and in bubble formation. Bubbles appeared in the majority of composite restorations. Amalgam impaired the color match because it could be seen under the composite, but this could be prevented by the application of an opaque dentin-bonding agent (Holan & others, 1992).

In contrast, Hovav & others (1995) detected considerably less marginal leakage in composite restorations lined only with Superbond D than in those cavities restored with the combined amalgam/composite restoration. Nevertheless, the authors did not simulate the clinical situation, that is, restorations were not performed with adjacent teeth. Also, they used clear rather than metal matrix bands.

An advantage of the combined amalgam/composite technique is that the use of a thin layer of amalgam at the gingival wall of the box may eliminate the need for a transparent matrix, allowing the use of the metal matrix band with a wooden wedge (Holan & others, 1996). It is much more difficult to obtain good proximal contact and cervical adaptation with the clear matrix (Miller & others, 1996; Holan & others, 1996).

Leakage between amalgam and composite could be a problem in combined restorations since the materials do not have a chemical interaction, forming an interface. However, this would be a problem only if the leakage reaches to the tooth structure. Moreover, the composite/amalgam interface is less susceptible to recurrent caries than the cervical margin of the restoration (Cardash & others, 1990). While not within the scope of this study, the authors observed that in a few specimens with leakage between amalgam and resin, the dye never penetrated to the tooth. The excellent interface between amalgam and resin composite could be explained by the fact that the bonding penetrates into the irregularities and porosities of the amalgam surface, creating an intimate mechanical bond with the composite material (Eidelman & others, 1990). Acid etching of the roughened amalgam surface prior to application of the adhesive systems may decrease the bond strength between amalgam and resin (Hadavi, Hey & Ambrose, 1991 A) and also increase the microleakage

(Hadavi, Hey & Ambrose, 1991 B). Based on these findings, the authors did not perform acid etching in amalgam prior to the dentin bonding agent application.

Although the combined amalgam/composite restorations exhibited better performance than composite restoration, it represents a more time-consuming technique. In addition, the clinician will be working with two different materials making the technique more sensitive and complicated.

### CONCLUSIONS

Based on the methodology used, the combined amalgam/composite technique demonstrated less microleakage than the light-cured and the combined self-cured and light-cured composites technique which demonstrated similar results.

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# Soft-Start Polymerization: Influence on Effectiveness of Cure and Post-Gel Shrinkage

AUJ Yap • SC Ng • KS Siow

## Clinical Relevance

Soft-start polymerization involving step-wise modulation of light energy does not reduce the effectiveness of cure. No significant reduction in polymerization shrinkage was observed with the soft-start curing regimen.

## SUMMARY

This study investigated the influence of soft-start polymerization on the effectiveness of cure and post-gel shrinkage of a visible light cured resin composite (Z100). Three cure modes (LH-high intensity; LA-soft-start polymerization involving step-wise modulation of light intensity and LL-low intensity) of a commercial light-cure unit (Kavo PolyLUX II) were examined and compared to another light-cure unit (Spectrum). The effectiveness of cure with the different cure modes

was assessed by computing the hardness gradient between top and bottom surfaces of 2 mm composites specimens after different light exposure times. A strain-monitoring device was used to measure the linear polymerization shrinkage associated with the different cure modes and exposure times over 180 minutes. A sample size of five was used for both experiments. Data was analyzed using one-way ANOVA and Scheffe's post-hoc test at significance level 0.05. Results showed that effectiveness of cure generally increased with increase cure time. Although modulation of light energy intensity (LA 40/80 seconds) resulted in lower polymerization shrinkage compared to LH 40 seconds, no significant difference was observed between these three cure regimes. Curing with the Spectrum curing light resulted in the lowest polymerization shrinkage.

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## INTRODUCTION

Curing dental composite restoratives with visible light is the standard method of clinically polymerizing composites. An inherent disadvantage of resin composites,

including polyacid-modified composites, is that they shrink during light polymerization (Sakaguchi, Sasik & Bunczak, 1991, Yap & others, 2000). The shrinkage of resin composites can be divided into two phases: the pre-gel and post-gel phase. During pre-gel polymerization, the composite is able to flow and stress within the structure is relieved (Davidson & De Gee, 1984). After gelation, flow ceases and cannot compensate for shrinkage stresses. Post-gel polymerization consequently results in significant stresses in the surrounding tooth structure and composite-tooth bond (Feilzer, De Gee & Davidson, 1987). Stresses arising from post-gel polymerization shrinkage may produce defects in the composite-tooth bond, leading to bond failure with associated post-operative sensitivity, microleakage and recurrent caries (Eick & Welch, 1986). Such shrinkage stresses could also cause deformation of the surrounding tooth structure if the composite-tooth bond is good (Sheth, Fuller & Jensen, 1988), resulting in microcracks in the cervical enamel (Bowen, Nemoto & Rapson, 1983) that predisposes the tooth to fracture.

Relief of polymerization shrinkage stresses can be obtained in various ways. One approach involves the incremental placement of composites and allowing composites to contract freely to the adhesive surface. The less the restoration is bonded to the opposing wall, the less obstruction there is for shrinkage and the lower the resultant stresses (Davidson, 1986). A second approach to stress reduction is the application of liners (Kemp-Scholte & Davidson, 1990). The last and most recent approach, designed to allow the restoration some freedom of movement between the cavity walls and the center of contraction, consists of initially reduced conversion of the resin materials (Davidson & Feilzer, 1997). This soft-start polymerization involves a step-wise modulation of light energy and has been shown to result in smaller marginal gap and increased

marginal integrity (Uno & Asmussen, 1991; Goracci, Casa de Martinis & Mori, 1996). The effects of soft-start polymerization on the effectiveness of cure and polymerization shrinkage, however, have not been widely studied.

This study investigated the influence of soft-start polymerization on the effectiveness of cure and post-gel shrinkage of a visible-light-cured resin composite.

## METHODS AND MATERIALS

A mini-filled resin composite (Z100; 3M Dental Products, St Paul, MN 55144) of A2 shade and a commercial light-cure unit (PolyLUX II; Kavo Dental, Warthausen, Germany) with three cure modes were selected for this study. The cure modes were LH (high intensity), LL (low intensity) and LA (soft-start polymerization). The latter cure mode involved a two-step modulation of light energy (60% of cure time at lower intensity, 20% at LL and 20% at LH). Another commercial light-cure unit with only one cure mode (Spectrum Curing Light [SP]; Dentsply Inc, Milford, DE 19963) was used as the control light source. Before beginning the experiments, the light intensity of the two light-cure units (and different cure modes) was assessed with a radiometer (Cure Rite; EFOS Inc, Ontario, Canada). The output was determined through a 1 mm glass slide and an acetate strip; the mean light intensity is shown in Table 1.

For investigating the effectiveness of cure, the composite was placed in black delrin molds with square cavities 2 mm deep and 4 mm wide/long confined between two opposing acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). 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 for 40 seconds using the two light-cure units and different cure modes (LH, LL and LA).

Table 1: Mean Light Intensity, KHN and Hardness Ratio for the Different Cure Modes and Irradiation Times

Cure Mode	Light Intensity in mW/cm <sup>2</sup> (SD)	Cure Time in Seconds	Top KHN (SD)	Bottom KHN (SD)	Hardness Ratio (SD)
LH	540 (19.26)	40*	72.30 (2.76)	67.94 (2.00)	0.94 (0.06)
		80	76.04 (1.98)	74.90 (1.40)	0.98 (0.01)
		120	80.46 (3.31)	78.46 (2.16)	0.98 (0.04)
LA	289 (9.29)	40*	69.90 (0.83)	64.12 (1.29)	0.92 (0.01)
	354 (12.77)	80*	75.30 (1.29)	73.98 (0.97)	0.98 (0.02)
	433 (20.60)	120	74.68 (1.93)	74.16 (2.13)	0.99 (0.01)
LL	374 (4.55)	40	63.70 (4.85)	57.08 (3.93)	0.90 (0.06)
		80*	68.44 (3.16)	61.66 (4.86)	0.90 (0.07)
		120	75.96 (2.45)	63.10 (5.83)	0.83 (0.11)
SP	411 (5.89)	40*	70.42 (1.02)	59.74 (0.36)	0.85 (0.01)
		80	73.36 (1.25)	62.68 (3.64)	0.85 (0.05)
		120	76.00 (1.98)	69.80 (2.44)	0.92 (0.04)

\* denotes curing parameters selected for statistical analysis and assessment of polymerization shrinkage. Standard deviations are enclosed in brackets.



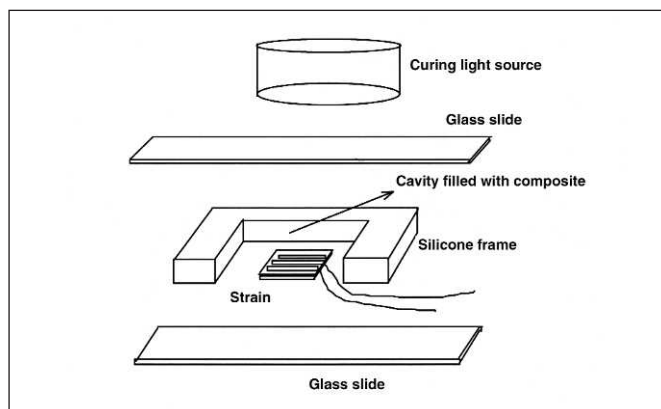


Figure 1. Schematic drawing of the experimental set-up for the assessment of polymerization shrinkage.

Immediately after light polymerization, the acetate strips were removed and the specimens in their molds were positioned centrally beneath the indenter of a digital microhardness tester (FM7, Future-Tech Corp, Tokyo, Japan) to assess Knoop's hardness (KHN) of the top and bottom surfaces. A 500g load was applied through the indenter with a dwell time of 15 seconds. Five specimens were made for each light-cure unit/cure mode. KHN readings after 40 seconds of light irradiation were recorded and the composite specimens were then further irradiated in 40-second increments up to 120 seconds through the glass slide and acetate strip. Hardness readings were taken at each time increment, and the mean KHN and hardness ratios were calculated and tabulated using the following formula:

Hardness ratio = KHN of bottom surface/KHN of top surface.

Table 1 reflects the mean KHN readings for the different cure modes and irradiation times. The results from this phase of the research were used to determine the curing parameters (cure mode and irradiation time) to be investigated in the second phase (that is, polymerization shrinkage). Selection was based on a minimal top KHN of  $70 \pm 2$  and hardness ratio of 0.8 (Pilo & Cardash, 1992; Yap & others, 2000) obtained with the control (SP40s—control light source (Spectrum) with 40 seconds irradiation as recommended by 3M). The qualifying curing parameters were LH with 40 seconds (LH40s); LA with 40 seconds (LA40s) and LL with 80 seconds (LL80s) irradiation. LA with 80 seconds (LA80s) irradiation was included in the second phase, as recommended by the manufacturer (Kavo Dental).

The test configuration for measuring polymerization shrinkage used a stiff black silicone frame (inner length 7.0 mm, width 4.0 mm and height 2.0 mm) which circumscribed the composite sample (Figure 1). A glass slide served as the base of the set-up. A foil electrical resistance strain gauge (Foil Strain Gauge, RS Components Ltd, Singapore) was attached to the flat glass surface. The gauge was 2 mm in length and had an electrical resistance 120  $\Omega$  and gauge factor 2.00. The resin composite was placed in the cavity of silicone frame with the strain gauge in place. Care was taken to ensure complete filling of the frame, and the excess composite material was extruded using pressure applied through a second glass slide and removed. The surface tack of the composite was adequate to ensure adhesion between the strain gauge and the composite materials. The leads from the strain gauge were connected to a strain-monitoring device (Strain Gauge Recorder, Cole Parmer Instruments, IL 60061) initially balanced at zero.

The composite specimens were light polymerized using the different cure modes and irradiation times mentioned above. The dimensional change during and post-light polymerization was monitored in air at room temperature ( $25 \pm 1^\circ\text{C}$ ). Polymerization shrinkage measurements were taken continuously every 10 seconds up to 40 seconds, and at one minute (60 seconds), 1.33 minutes (80 seconds) and two minutes. Readings were then taken at one-minute intervals up to 10 minutes. The change of resistance of the strain gauge was subsequently recorded at the following time intervals: 30, 60, 120 and 180 minutes. The percentage of linear shrinkage was derived from the following equation:

$$\text{Percentage linear shrinkage } (\Delta L/L \times 100) = (\Delta R/R)/K \times 100$$

Table 2: Results of Statistical Analysis of KHN and Hardness Ratio

Factor	Differences
KHN Top	LA80s > LA40s, LL80s and SP40s
KHN Bottom	LA80s > LH40s, LA40s, LL80s and SP40s LH40s > LL80s and SP40s
Hardness Ratio	LA80s > SP40s

Results of one-way ANOVA and Scheffe's test at significance level 0.05. > denotes statistical significance.

Table 3: Mean Linear Percent Shrinkage (standard deviation) at the Various Time Intervals

Time	LH40s	LA80s	LA40s	LL80s	SP40s
10 minutes	0.47 (0.04)	0.44 (0.02)	0.42 (0.01)	0.36 (0.02)	0.30 (0.06)
30 minutes	0.49 (0.06)	0.46 (0.02)	0.44 (0.02)	0.38 (0.02)	0.32 (0.07)
60 minutes	0.51 (0.08)	0.47 (0.02)	0.45 (0.02)	0.39 (0.02)	0.34 (0.07)
120 minutes	0.52 (0.08)	0.48 (0.02)	0.46 (0.02)	0.40 (0.02)	0.35 (0.02)
180 minutes	0.53 (0.08)	0.48 (0.02)	0.47 (0.02)	0.41 (0.02)	0.36 (0.07)

Table 4: Results of Statistical Analysis of Polymerization Shrinkage

Time	Differences
10 minutes	SP40s < LH40s, LA80s and LA40s LL80s < LH40s and LA80s
30 minutes	SP40s < LH40s, LA80s and LA40s LL80s < LH40s
60 minutes	As above
120 minutes	As above
180 minutes	As above

Results of one-way ANOVA and Scheffe's test at significance level 0.05. < denotes statistical significance.

Where

$\Delta L$  = Change in length

L = Original length

$\Delta R$  = Change of resistance

R = Original resistance

K = Gauge factor (that is, 2)

Five composite specimens were used for each curing parameter. The data was subjected to one-way ANOVA and Scheffe's multiple range tests at 0.05 significance level.

## RESULTS

The mean light intensity, KHN and hardness ratio for the different light-cure units/cure modes and irradiation times are shown in Table 1. Statistical analysis for KHN top, KHN bottom and hardness ratio between LH40s, LA80s, LA40s, LL80s and SP40s are shown in Table 2. Table 3 and Figures 2-3 show the mean linear percent shrinkage associated with the aforementioned curing parameters at the various time intervals. Results of statistical analysis of polymerization shrinkage are reflected in Table 4.

The hardness ratio generally increased with increased irradiation time. The hardness at the top surface obtained with LA80s was significantly higher than

that obtained with LA40s, LL80s and SP40s. No significant difference was observed between LA80s and LH40s at the top surface. At the bottom surfaces, KHN values obtained with LA80s were significantly greater than all other curing parameters (that is, LH40s, LA40s, LL80s and SP40s). In addition, the KHN bottom resulting from LH40s was significantly greater than that of LL80s and SP40s. A significant difference in hardness ratio only was observed between LA80s and SP40s.

The rate of shrinkage for all curing parameters was greatest during the light polymerization reaction and continued after the curing light was removed either at 40 or 80 seconds (Figure 2). Polymerization shrinkage at 10 minutes ranged from  $0.30 \pm 0.06\%$  for SP40s to  $0.47 \pm 0.04\%$  for LH40s. The ranking at 10 minutes was as follows: SP40s < LL80s < LA40s < LA80s < LH40s. Ranking of polymerization shrinkage at 30, 60, 120 and 180 minutes were similar. After 180 minutes the highest shrinkage was observed with LH40s and shrinkage was approximately one and one-half times that observed with the control (SP40s).

At 10 minutes, curing with SP40s resulted in significantly lower shrinkage compared to curing with LH40s, LA80s and LA40s. In addition, curing with LL80s resulted in significantly less shrinkage than curing with LH40s and LA80s. For the remaining time intervals, statistical analysis was similar with the exception that shrinkage associated with LL80s was only significantly lower than LH40s. No significant difference in shrinkage was observed between the control (SP40s) and LL80s at all time intervals.

## DISCUSSION

The effectiveness of cure may be assessed directly or indirectly. Direct methods that assess the degree of conversion, for example, laser Raman spectroscopy (Louden & Roberts, 1983) and infrared spectroscopy (Asmussen, 1982a), have not been accepted for routine

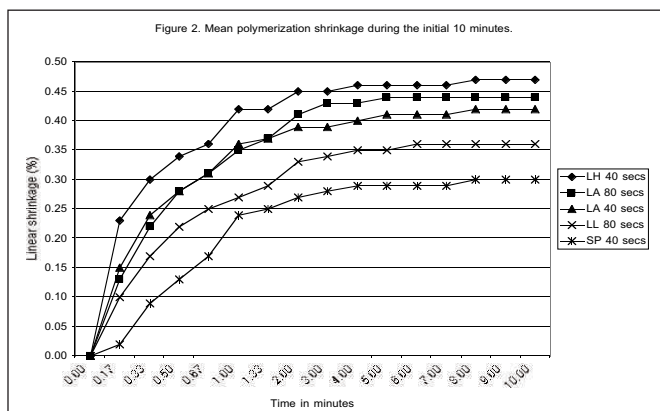


Figure 2. Mean polymerization shrinkage during the initial 10 minutes.

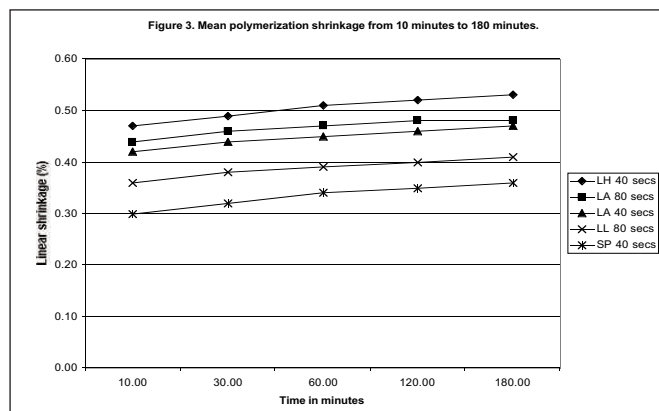


Figure 3. Mean polymerization shrinkage from 10 minutes to 180 minutes.

use as they are complex, expensive and time consuming. Indirect methods include scraping (Cook, 1980), visual (Murray, Yates & Newman, 1981) and surface hardness (Asmussen, 1982b). The latter (that is, surface hardness testing) has been used in many studies because of its relative simplicity and good correlation to the degree of conversion using infrared spectroscopy (Asmussen, 1982b; DeWald & Ferracane, 1987). If polymerization is completely effective, the hardness ratio should be 1, as the hardness of the bottom surface should be identical to that of the top surface. However, as light passes through the bulk of composite, the light intensity is greatly reduced due to light scattering decreasing the effectiveness of cure (Ruyter & Øysaet, 1982). This scattering of light accounts for the minor differences in hardness between top and bottom surfaces of the composite specimens. The hardness gradient should not exceed 10-20% (that is, hardness ratio 0.8) for photo-activated composites to be adequately polymerized (Pilo & Cardash, 1992). The Spectrum light-cure unit fulfilled this criterion although its hardness ratio was generally lower than that observed for the PolyLUX unit with its various cure modes. Although the hardness ratio after irradiation with LL for 40 seconds was 0.90, this curing parameter was not selected, as hardness of the top surface was much lower than that of the control (SP40s).

The effectiveness of cure generally increased with increase in light irradiation time except for the LL mode. After irradiation for 120 seconds in LL mode, a drop in hardness ratio was observed (Table 1). This can be attributed to an increase in top KHN and a disproportionate increase in bottom KHN due to the low light intensity. The KHN of the top surface after treatment with LA80s was significantly greater than LA40s, LL80s and SP40s. No significant difference in KHN was observed between LH40s and all the other curing parameters including in the control despite the higher light intensity (540 mW/cm<sup>2</sup>) used. These findings are in agreement with those of Rueggeberg & others (1994) and Sakaguchi & Berge (1998) who concluded that at the top surface of composites only duration of exposure or irradiation time is a significant factor contributing to monomer conversion. The use of LH40s, however, resulted in a significantly harder bottom surface compared to LL80s and SP40s. The higher light intensity thus allows for better cure at the bottom surface, but this was at the expense of polymerization shrinkage (Figures 2 and 3). The KHN bottom observed with LH40s was still significantly lower than that with LA80s. No significant difference in hardness ratio and indirect effectiveness of cure was observed between LH40s and the other curing parameters, including LL80s. Therefore, it is important that irradiation time should be doubled (that is, 80 seconds as opposed to 40 seconds with LH and SP) to ensure effective

cure when LL mode is used. The only significant difference in hardness ratio observed was between LA80s and the control (SP40s). As no significant difference in KHN top, KHN bottom and hardness ratio was observed between LH40s and LA40s, the use of LA40s is feasible.

Since not all curing parameters were clinically viable, cure modes with the minimal irradiation time necessary to achieve top KHN similar to the control (SP40s) and hardness ratio  $\geq 0.8$  were selected for investigation of polymerization shrinkage. The experimental set-up was identical to the one used by the authors in an earlier study (Yap & others, 2000). The rate of shrinkage for all curing parameters was the greatest during the light polymerization reaction and continued after the curing light was removed at either 40 or 80 seconds (Figure 2). The shrinkage observed after removal of the curing light may be attributed to the progressive cross-linking reaction that occurs following light polymerization initiation and thermal contraction due to loss of radiant heat (Watts, Amer & Combe, 1987; Yap & others, 2000). The shrinkage of the composite was not restricted by the silicone frame, as there was no bond between the two materials. The ranking at all time intervals (10 to 180 minutes) was as follows: SP40s < LL80s < LA40s < LA80s < LH40s. After 180 minutes the highest shrinkage was observed with LH40s, and shrinkage was approximately one and one-half times that observed with the control (SP40s).

At 10 minutes, curing with SP40s resulted in significantly lower shrinkage compared to curing with LH40s, LA80s and LA 40s. In addition, curing with LL80s resulted in significantly less shrinkage than curing with LH40s and LA80s. Results can be explained by the maximum light intensity emitted by the light sources/different cure modes and irradiation time. Greater light intensity/curing time leads to greater cure and polymerization shrinkage. The light energy emitted by SP (411 mW/cm<sup>2</sup>) and PolyLUX in LL mode (374 mW/cm<sup>2</sup>) was lower than that of LH (540 mW/cm<sup>2</sup>) and the maximum output of LA (433 mW/cm<sup>2</sup>). For the remaining time intervals, statistical analysis was similar with the exception of shrinkage associated with LL80s was only significantly lower than LH40s.

It is interesting to note that the usage of step-wise polymerization (LA) resulted in lower mean linear polymerization shrinkage compared to the high light intensity mode LH, even if the irradiation time of LA was increased twice (that is, LA80s). There was, however, no statistical significance in polymerization shrinkage between LH40s and LA modes with the two irradiation times. The mean difference at 180 minutes between LH40s and LA40s was only 0.05%. Results do



not support that of Sakaguchi & Berge (1998) which stated that samples cured with two intensities showed a 21.8% reduction from contraction strain predicted from a light energy density calculation. The lower shrinkage with LA is perhaps related to a decrease in contraction strain rate that allows stress relaxation to occur. Lower light intensity has been shown to produce lower contraction strain in general (Sakaguchi, Douglas & Peters, 1992). In addition, slower polymerization resulting from exposure to lower light intensities yields longer molecular chains with higher flow characteristics (Miyazaki & others, 1996). The lower shrinkage observed with LA could not solely be attributed to the step-wise modulation of light intensity, as a lower light intensity was used. Although the manufacturer claims a two-step modulation of light energy with 60% of cure time at lower intensity, 20% at LL intensity and 20% at LH intensity, the light intensity at "LL" and "LH" levels for LA mode were only 354 and 433 mW/cm<sup>2</sup>, respectively. These intensity levels are lower than the LL (374 mW/cm<sup>2</sup>) and LH (540 mW/cm<sup>2</sup>) modes. The progressive shrinkage from 10 to 180 minutes did not differ greatly with the different curing parameters and were as follows: LH40s—0.06%; LA40s—0.05%; LA80s—0.04%; LL80s—0.05%; and SP40s—0.06%. Therefore, it can be inferred that the differences in polymerization shrinkage occurred mainly during the light polymerization reaction.

### CONCLUSIONS

The results of this study only apply to composite specimens that are 2 mm (or less) thick. Under the conditions of this *in vitro* study:

1. The effectiveness of cure generally increased with increase light exposure time.
2. The effectiveness of cure was not significantly affected by soft-start polymerization.
3. No significant reduction in polymerization shrinkage was observed with the soft-start curing regimen.

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# Influence of Different Transitional Restorations on the Fracture Resistance of Premolar Teeth

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## Clinical Relevance

Fracture resistance testing indicates that use of a resin-modified glass ionomer cement alone may offer advantages over alternative materials and techniques in the transitional restoration of teeth destined to be crowned.

## SUMMARY

Controversy exists over the most favorable material and type of restoration to be used to transitionally restore teeth destined to be crowned. This *in vitro* study uses fracture resistance testing to compare eight different transitional restorations in maxillary premolars. Ninety sound maxillary premolars were randomly selected and allocated to nine groups, each comprising 10 teeth. One group remained unrestored and was used as the control. Teeth in the remaining

groups were prepared to a standard cavity form using: a copy milling process removing the palatal cusp. Restorations were placed using amalgam with dentin pins and cavity varnish; amalgam with an amalgam bonding agent; resin composite with dentin pins and a dentin bonding agent; resin composite with a dentin bonding agent only; resin-modified glass ionomer with dentin pins; resin-modified glass ionomer cement alone and cermet with dentin pins and cermet alone. Each restored tooth was then subjected to axial loading via a bar contacting the buccal and restored palatal cusps until failure of the restored tooth occurred. The mean load-to-fracture values were statistically compared and the modes of failure recorded. It was found that the choice of restorative material and type of restoration had little effect on the fracture resistance of the restored tooth with the exception of those teeth restored with reinforced glass ionomer cement alone, which exhibited a significantly lower resistance to fracture than the other restored teeth. However, the choice of restorative material/technique did influence the *mode* of failure. Failure in teeth restored with resin-modified glass ionomer cement alone produced the least damage to the remaining tooth tissue when failure occurred. Consequently, this material

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**may offer the most favorable range of properties for the transitional restoration of extensively broken-down maxillary premolar teeth destined to be crowned. Furthermore, the findings of this study fail to support the use of dentin pins in the placement of bonded build-up restorations.**

## INTRODUCTION

Good practice requires badly broken-down teeth, including many endodontically treated teeth, to be restored by means of transitional restorations prior to crowning. Transitional restorations, which may remain in clinical service for several months, must fulfill a variety of functions, ranging from the interim restoration of the form and function of the tooth to forming part of the preparation to support the planned extra-coronal restoration.

In considering alternative forms of transitional restorations, mindful of the possible advantages of different materials (Combe & others, 1999), it is important to take account of the protection afforded to the remaining tooth tissues and the resistance to fracture of the transitionally restored tooth unit. The advantages of a material that provides a foundation for a crown may be outweighed by its disadvantages as a transitional restorative. The alternative—planning the replacement of a transitional restoration by a core build-up prior to crowning—is considered to be contraindicated in the interest of preserving supporting tooth tissue and, where appropriate, pulp vitality.

Given the above and with a growing interest in using a range of materials for transitional restorations, this study investigated aspects of the fracture resistance of restored maxillary premolar teeth, including large, compound transitional restorations placed using a range of materials and techniques.

## METHODS AND MATERIALS

Ninety sound maxillary premolar teeth with a buccolingual width of between 9.08-9.54 mm and free of visible cracks when viewed by transillumination at 2.5x magnification were carefully selected. The teeth were allocated at random to one of nine sample groups of 10 teeth each. One group remained unrestored to serve as the control. All sample teeth were stored at 20°C in formal saline for 48 hours after extraction, then placed in water. At no stage in the investigation were the teeth allowed to dehydrate.

One maxillary premolar was selected at random for preparation and production of an acrylic resin replica. This tooth was mounted in a block of dental stone (British Gypsum, Mountfield, UK) to provide a stable support. The palatal cusp was removed using 543 and 556 medium diamond burs (Unident, UK) operating in a hand-held air turbine (KaVo Ltd, Amersham, UK) to a level 2 mm coronal to the amelocemental junction. The cavosurface angles were finished to a butt joint configuration. The occlusal floor was at 90° to the long axis of the tooth (Figure 1).

Two pin sites were selected in dentin along a mesiodistal axis parallel to the palatal surface of the tooth at a distance of 1 mm from the amelodentinal junction (Figure 2). Using the twist drill supplied with the selected pins (Stabilok, Fairfax Dental, London, UK), two pinholes were prepared. The floor and wall of the preparation were lightly smeared with Vaseline (Cheesebrough Ponds Ltd, London, UK). The twist drills were also smeared and replaced in the holes. Ribbon wax was placed around the crown of the tooth, forming a matrix. The enclosed preparation was filled with acrylic pattern resin (GC Pattern Resin, Tokyo, Japan). The acrylic pattern formed was subsequently used as a jig to produce identically located pinholes in

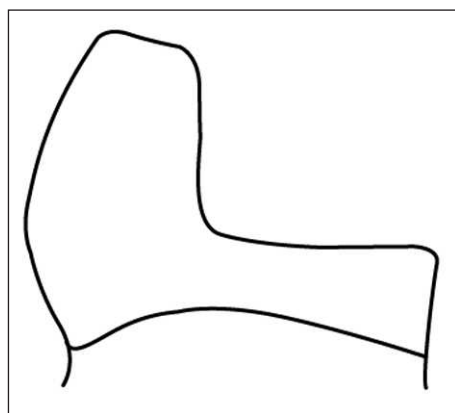


Figure 1. Preparation details. The palatal cusp was removed. The occlusal floor was at 90 degrees to the long axis of the tooth and the cavosurface angles were finished to a butt joint configuration.

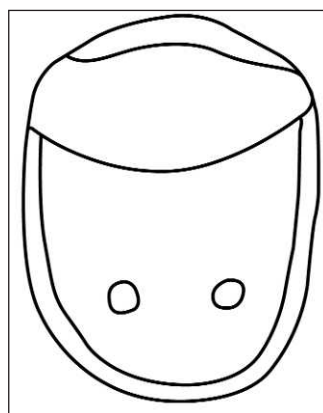


Figure 2. Pin sites. These were along the mesiodistal axis of the tooth, parallel to the palatal margin and at a distance of 1 mm from the amelodentinal junction.

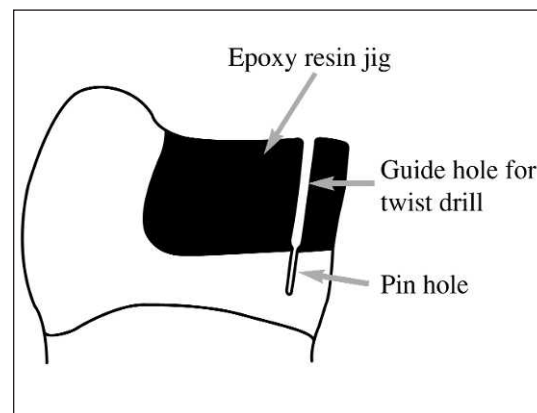


Figure 3. Pin jig formed by the acrylic pattern.

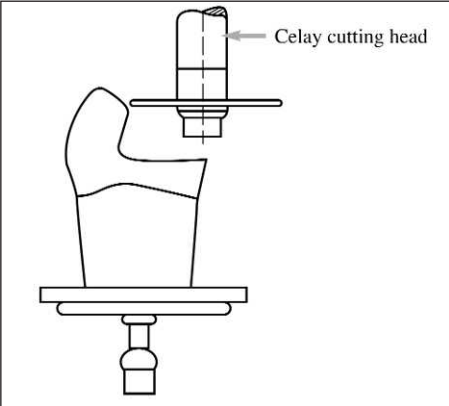


Figure 4. Tooth milling using the acrylic replica as a template on the scanning side of the Celay apparatus.

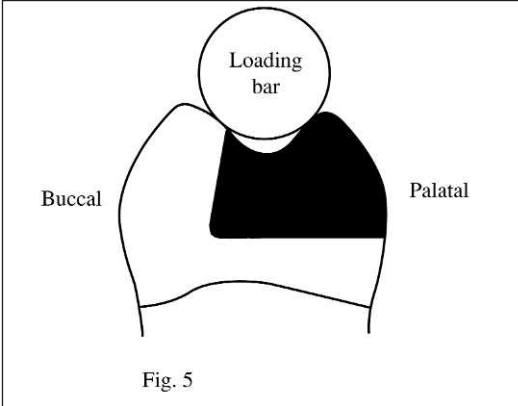


Figure 5. Tooth mounted on Universal Testing Machine, the roots being embedded in acrylic resin. A 4 mm diameter stainless steel bar is placed in contact with both tooth and restoration along the occlusal fissure.

milling instrument (Celay, Mikrona Technologie AG, Spreitenbach, Switzerland). Each sample tooth was sectioned at 90° to the long axis of the root at a point 13 mm from the top of the buccal cusp and mounted using cyanoacrylate adhesive (Bostik Superglue 4, Bostik Ltd, Leicester, UK) to a mounting index. The sample was placed on the cutting side of the milling instrument and aligned with a common point of reference to the acrylic replica. The preparation within the replica was then scanned to reproduce a replica prepara-

tion to the sample tooth (Figure 4). The teeth were restored using various combinations of restorative material and operative technique selected for investigation (Table 1). The mesial and distal walls were carved with minimal occlusal flare and the occlusal surface given a smooth, simplified contour. Relevant directions for use were strictly adhered to throughout the placement procedure. Each restored tooth was fixed crown uppermost with its long axis vertical in a 10 mm section of 22 mm copper pipe. The roots of the teeth were embedded in a chemically-cured acrylic resin (Special Tray, Dentsply, Weybridge, UK) and placed in the loading cell of a universal testing machine (Model 50TS, RDP Howden Ltd, Leamington Spa, UK) as shown in Figure 5. A 4 mm diameter stainless steel bar was placed along the occlusal fissure in contact with both the tooth and restoration and loaded at a cross-head speed of 1 mm per minute until fracture occurred. The load-to-fracture (KN) and the mode of failure (Table 4) were recorded and the results analyzed using an analysis of variance (ANOVA) and post hoc multiple comparison tests.

A chi-square test compared the nine groups with respect to mode 1 failure type (fracture of the buccal cusp). It was not possible to compare the other modes of failure in this way, as there was insufficient data.

RESULTS

The load-to-fracture values (KN) of the teeth in Groups 1-9 are summarized in Table 2 and the mode of failure by group is illustrated in Table 4. One-way analysis of variance (ANOVA) revealed statistically significant differences ( $p<0.001$ ) in mean load-to-fracture values among Groups 1-9 (Table 3). A Bonferroni post hoc multiple comparison test showed these differences existed between Groups 5 and 9 and Groups 1,2, 3 and 7.

Table 1: Combinations of Restorative Materials and Techniques Investigated	
Group	Materials and Technique
1	Unrestored controls
2	Two dentin pins <sup>1</sup> Cavity varnish Amalgam <sup>2</sup>
3	Amalgam bonding agent <sup>3</sup> Amalgam <sup>2</sup>
4	Two dentin pins <sup>1</sup> Bond <sup>4</sup> Resin composite <sup>5</sup>
5	Bond <sup>4</sup> Resin composite <sup>6</sup>
6	Two dentin pins <sup>1</sup> Resin modified glass ionomer cement <sup>6</sup>
7	Resin-modified glass ionomer cement <sup>6</sup> alone
8	Two dentin pins Cermet <sup>7</sup>
9	Cermet <sup>7</sup> alone
<sup>1</sup> Stabilok, Fairfax Dental, London, UK <sup>2</sup> Tytin, Kerr Manufacturing Co, Romulus, USA <sup>3</sup> Panavia 21, Kuraray Co, Osaka, Japan <sup>4</sup> Scotchbond Multipurpose, 3M Health Care Ltd, Minneapolis, USA <sup>5</sup> Prisma TPH, Dentsply, Weybridge, UK <sup>6</sup> Vitremer, 3M Health Care Ltd, Minneapolis, USA <sup>7</sup> Ketac Silver, ESPE GmbH, Seefeld/Oberbay, Germany	

sample teeth (Figure 3). A light bodied, silicone impression material (Provil, Bayer, Leverkusen, Germany) was injected around the floor and walls of the cavity and the tooth was embedded in silicone putty contained within a stock impression tray. Acrylic resin (GC Pattern Resin, Tokyo, Japan) was poured into the impression and following the insertion of an index pin, left to harden for 24 hours. The completed resin replica was mounted on the scanning side of a pantographic

Table 2: Load (KN) to Fracture Values

Group	n	Mean	sd
1	10	0.888	0.20
2	10	0.893	0.10
3	10	0.837	0.16
4	10	0.702	0.17
5	10	0.506	0.12
6	10	0.711	0.24
7	10	0.799	0.23
8	10	0.672	0.15
9	10	0.485	0.15

Key:  
 1. Control  
 2. Pins, varnish, amalgam  
 3. Amalgam bond, amalgam  
 4. Pins, bond, resin composite  
 5. Bond, resin composite  
 6. Pins, resin modified glass ionomer cement  
 7. Resin modified glass ionomer cement  
 8. Pins, cermet  
 9. Cermet

Table 3: Bonferroni Post Hoc Multiple Comparison Test Findings

Group	1	2	3	4	5	6	7	8	9
1	-	-	-	-	*	-	-	-	*
2	-	-	-	-	*	-	-	-	*
3	-	-	-	-	*	-	-	-	*
4	-	-	-	-	-	-	-	-	-
5	-	-	-	-	-	-	-	-	-
6	-	-	-	-	-	-	-	-	-
7	-	-	-	-	*	-	-	-	*
8	-	-	-	-	-	-	-	-	-
9	-	-	-	-	-	-	-	-	-

-, no significant different ( $p>0.05$ ); \*, significantly different ( $p<0.05$ )

Table 4: Mode of Failure by Group

Mode	Gp1	Gp2	Gp3	Gp4	Gp5	Gp6	Gp7	Gp8	Gp9	Gp10
I	10	6	9	9	8	5	1	2	3	-
II	-	1	-	1	-	3	-	-	-	-
III	-	-	-	-	2	-	2	-	2	-
IV	-	-	1	-	-	-	7	-	5	-
V	-	3	-	-	-	2	-	8	-	-

Key:  
 I Fracture of buccal cusp  
 II Fracture of tooth at interface with pin  
 III Adhesive failure of restorative material  
 IV Bulk fracture of restorative material  
 V Fracture in restorative material at interface with pin

The highest mean load-to-fracture values were found for teeth restored with amalgam. The mean load-to-fracture values for the teeth restored with composite placed with dentin pins and bonding, resin-modified glass ionomer cement placed with pins and resin-modified glass ionomer cement only were very similar and significantly higher ( $p<0.05$ ) than those for the teeth restored with reinforced glass ionomer cement and composite placed with bonding only. The teeth restored with reinforced glass ionomer cement placed with pins demonstrated a relatively low mean load-to-fracture value. This value was, however, significantly higher ( $p<0.05$ ) than the value for the teeth restored with reinforced glass ionomer cement alone and composite placed with bonding.

There was a significant difference among the nine groups with respect to the number of mode 1 failures (fracture of the buccal cusp) (Table 4). Groups 1,3,4 and 5 had many failures of mode 1 (8-10), whereas Groups 7,8, and 9 only few (1-3). Group 7 had seven failures in mode IV (bulk fracture of the restorative material) and Group 8 had eight failures of mode V (fracture in the restorative material at the pin interface).

## DISCUSSION

All the teeth in the unrestored control group failed as a result of mesiodistal vertical fractures through the mid-line of the crown. This suggests that under direct axial loading through a two-point contact point on the buccal and palatal cusps, the weakest point in maxillary premolar teeth may be found in a mesiodistal plane through the occlusal fissure. It should, however, be noted that as the majority of the teeth investigated were extracted in young individuals for orthodontic reasons, this finding may be the result of the relatively large pulp chambers found in immature teeth.

In Group 2, the majority (60%) of failures occurred as a result of fracture of the buccal cusp in teeth restored with amalgam, two dentin pins and cavity varnish. As there was no bond between the amalgam restoration and the prepared and weakened buccal cusp, this result could have been expected. Indeed, considering the extent of tooth preparation, a lower mean load-to-fracture value might have been anticipated. Three of the 10 teeth in this group underwent fracture of the amalgam at the interface with the pins. This result concurs with work by Kao (1991), who concluded that pins weaken amalgam, but disagrees with early work by Markley (1958), who believed that pins reinforce amalgam. Haller, Götze & Weiss (1991) also reported an increase in the strength of amalgam cores when pins were placed. It



is likely that the differences in study results are due to differences in the bulk of amalgam surrounding and covering the pins in the experimental protocols. The remaining tooth in this group failed as a result of fracture in the tooth tissue at the interface with the dentin pins. The occurrence of dentinal cracks and crazing as a result of the insertion of threaded dentin pins has been demonstrated in a number of studies (Dilts, Welk & Stovall, 1968; Trabert et al, 1973; Webb, Straka & Phillips, 1989; Standlee, Collard & Caputo, 1970). Examination of the crown morphology of the fractured tooth indicated it had a marked bucco-palatal narrowing towards the amelocemental junction which could permit rapid propagation of any existing defects in the dentin surrounding the pins towards the surface of the tooth, resulting in fracture.

The mean load-to-fracture value for the teeth restored with amalgam and a bonding agent was slightly lower than that for the restorations of amalgam and dentin pins, although this difference was not statistically significant ( $p < 0.05$ ). The modes of failure, however, were distinctly different. Bonding negated the weakening effects of pins on the restoration and tooth tissue. Nine out of 10 failures in this group were due to fracture of the buccal cusp and adhesive failure at the bonding interface. None of the teeth failed as a result of debonding of the amalgam bonding agent and amalgam. Eakle, Staninec & Lacy (1992) demonstrated, using maxillary premolar teeth with mesio-occluso-distal (MOD) preparations, that teeth restored with bonded amalgam restorations were significantly more resistant to fracture than those restored with amalgam alone ( $p < 0.05$ ). The results of this study indicated that the teeth restored with bonded amalgam were equal in fracture resistance to those restored with amalgam using dentin pins.

The mean load-to-fracture values obtained for the teeth restored with composite, dentin pins and a dentin bonding agent were not significantly different from the mean values found for the control teeth and teeth restored with amalgam ( $p > 0.05$ ). This lends support to the findings of other workers who reported that the use of bonded composite restorations does not significantly increase the fracture resistance of teeth (Joynt, Wiecekowsky, 1987; Purk, Eick & Roberts, 1990; Stampalia & others, 1986) but contradicts a number of studies which have demonstrated an increase in fracture resistance (Eakle, Maxwell & Braly, 1986; McCulloch & Smith, 1986;). Kao (1991) demonstrated that the fracture resistance of teeth restored with composite restorations is reduced by the presence of dentin pins, which differs from the findings of Haller, Götze & Weiss (1991), who reported no reduction in strength. As none of the sample teeth failed as a result of fracture through the bulk of the restorative material, it is not possible to verify either of these findings. Only one in 10

sample teeth in this group suffered failure at a site associated with dentin pins. This would indicate that within the parameters of this study, the fracture resistance of composite restored teeth is relatively unaffected by the presence of pins. Nine out of 10 teeth in this group failed as a result of buccal cusp fracture. The lack of pins in the teeth restored with bonded composite may have contributed to the nature of the failures that occurred in this sample group. Two out of 10 teeth failed as a result of debonding of the restorative from the tooth—no damage being observed in the remaining tooth tissue. It is likely that the lack of lateral resistance to displacement, which pins provide, increased the shear stress on the bond leading to failure. The remaining eight teeth in this group failed as a result of buccal cusp fracture. The significantly lower mean load-to-fracture value found in this group is difficult to explain. Had a majority of the failures occurred from adhesive failure of the restorative material, the results could have been more easily interpreted. By contrast, these findings point towards complex fracture mechanics in heavily restored teeth under load. Of particular note is that a bonded composite failed to prevent the fracture of a remaining buccal cusp (as occurred in eight of the 10 specimens in this study).

There has been relatively little research into the fracture resistance of teeth restored with a resin-modified glass ionomer cement. Studies have demonstrated higher compressive and tensile strengths in resin-modified glass ionomer cements than in conventional glass ionomer cement (Kitamura, Aoyama & Miyazaki, 1993; Burgess & others, 1993) and the potential value of the clinical use of a resin-modified glass ionomer cement alone in the transitional restoration of permanent molar teeth (Wilson, & others, 1999). The mean load-to-fracture value for teeth restored with pins and resin-modified glass ionomer cement in this study was not significantly different from the values obtained for Groups 1, 2, 3, 4, 6, 7 and 8 ( $p > 0.05$ ). Three in 10 sample teeth failed as a result of tooth fracture adjacent to the pin sites and an additional two teeth failed as a result of restoration failure at the pin site. In contrast with the previous sample groups, one half of the failures appear to have occurred as a result of the presence of pins. The remaining five teeth failed due to fracture of the buccal cusp. In teeth restored with resin-modified glass ionomer cement alone, a slightly higher mean load-to-fracture value was found compared with teeth restored with resin-modified glass ionomer cement and dentin pins, although there was no statistically significant difference in this value compared with the other groups ( $p > 0.05$ ). The modes of failure were, however, markedly different from those observed in the other groups. Seven out of 10 failures occurred as a result of fracture in the bulk of the restorative material, two more resulted from adhesive failure of the restorative material and

only one tooth failed as a result of fracture of tooth tissue. The absence of pins in this group appears to have reduced the incidence of failure due to tooth fracture without any reduction in the overall fracture resistance.

The mean load-to-fracture value for teeth restored with reinforced glass ionomer cement placed with pins was not significantly different from the other sample groups ( $p>0.05$ ). The principal mode of failure (eight out of 10) was fracture of the restorative material at the interface with the pins. The remaining two teeth failed as a result of buccal cusp fracture. Teeth restored with reinforced glass ionomer alone showed a significantly lower mean load-to-fracture compared with teeth restored with pins and cermet. It appears that pins increase the fracture resistance of teeth restored with a reinforced glass ionomer cement. This concurs with work by Kao (1991), who demonstrated that the presence of pins does not weaken alloy reinforced glass ionomer materials, and in certain situations, it increased fracture resistance. Two out of 10 teeth restored with the reinforced glass ionomer alone failed as a result of debonding. The remaining teeth in this group failed due to fracture through the bulk of the restorative material.

### CONCLUSIONS

The following conclusions may be drawn from this laboratory study:

1. Premolars prepared with extensive compound preparations involving loss of the palatal cusp may be found to have substantially weakened buccal cusps. When completing such preparations, care should be taken to minimize reduction in the buccopalatal width of the buccal cusp.
2. The choice of restorative material and type of transitional restoration may have relatively little effect on the overall fracture resistance of restored premolar teeth, with the possible exception of teeth restored with a reinforced glass ionomer cement alone.
3. Teeth transitionally restored using reinforced glass ionomer cement alone may be found to have significantly lower resistance to fracture than teeth transitionally restored with composite, resin-modified glass ionomer cement, amalgam or reinforced glass ionomer placed with pins.
4. The use of resin-modified glass ionomer alone for transitional restorations in premolar teeth may be found to result in the least damage to tooth tissue when failure from axial loading occurs.

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# Repair of Non-Carious Amalgam Margin Defects

HW Roberts • DG Charlton • DF Murchison

## Clinical Relevance

Flowable resin composite may provide an adequate marginal seal for selected non-carious amalgam margin defects.

## SUMMARY

This study investigated the microleakage associated with the repair of non-carious amalgam defects using flowable resin composite. Occlusal amalgam preparations were accomplished on 36 non-carious mandibular molars. A standardized 40-micron marginal defect was made by condensing amalgam against a mylar matrix strip. Specimens then underwent a corrosion protocol designed to simulate intraoral corrosion seen with amalgam restorations. The resultant specimens were divided into three treatment groups: 1) No treatment (control); 2) Air abrasion of the amalgam defect surface, acid etching of both amalgam and enamel surfaces, then placement of a flowable composite and 3) Air abrasion of the amalgam defect, application of a fifth-generation dentin bonding agent and placement of the flowable composite. Specimens were thermocycled,

sealed with glass ionomer and fingernail polish to within 1 mm of repaired margins, then immersed in basic fuchsin for 24 hours. Specimens were sectioned and microleakage assessed. Results indicated that a flowable resin composite significantly reduced marginal microleakage compared to the control ( $p < 0.05$ ). There was no difference in microleakage between flowable resin composite repairs done with or without the use of a dentin-bonding agent.

## INTRODUCTION

The presence of a narrow or stained non-carious marginal defect has been shown to be an insufficient reason for replacing amalgam restorations (Kidd, Joyston-Bechal & Beighton, 1995; Kidd & O'Hara, 1990; Maryniuk & Brunson, 1989; Merrett & Elderton, 1984; Sölderholm, Antonson & Fischlschweiger, 1989). Even so, clinicians often feel uncomfortable leaving such defects untreated. Rather than replacing the restoration, various ways of sealing the defects have been evaluated. One technique that uses resin materials has been advocated as a viable clinical method (Anderson, 1993). Repairing margin defects may increase the longevity of the restoration (Mjör, 1999), eliminate areas that are difficult for the patient to properly cleanse and conserve remaining tooth structure (Anusavice, 1995). The use of flowable resin composites

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for sealing non-carious amalgam margin defects has not been reported. This investigation evaluated the effectiveness of a flowable composite in sealing non-carious amalgam margin defects.

### METHODS AND MATERIALS

Thirty-six caries-free mandibular molars debrided of soft tissue and previously stored in formalin were used for the microleakage test. The teeth were cleaned with pumice, placed into three groups based on even size distribution and stored in distilled water. Standardized Class I rectangular preparations (three millimeters buccolingually by six millimeters mesiodistally by two millimeters deep) were made by one operator. A 556 bur in a high-speed handpiece with water spray was used, and a new 556 bur was used for every three preparations. A standardized 40-micron margin defect was prepared by placing a 5 mm-wide section of mylar matrix strip lightly coated with a water-soluble lubricant (Surgilube, E Fougera & Co, Melville, NY 11747) against a suitable buccal or lingual wall within each of the preparations. The strip was placed so it extended to the pulpal floor. The preparations were then filled with Dispersalloy amalgam (Dentsply/Caulk, Milford, DE 19963). The amalgam was prepared following manufacturer's recommendations and incrementally placed, condensed and carved. In the area of the embedded mylar matrix strip, the margin was carved back to the approximate marginal contour. After carving, a football-shaped instrument was used to burnish the amalgam surface and margins. Twenty-four hours after the restorations had been placed, the mylar matrix strip was removed. Figure 1 shows the standardized defect that was produced. Specimens were stored in 37°C distilled water for one week to allow the water-soluble lubricant to leach from the marginal defect area of the preparations. The specimens were then subjected to an accelerated aging protocol as described by Matyas, Caputo & Cowie (1978). This protocol consisted of exposing the specimens to ammonium sulfide gas for 24 hours, then immersing them in Ringer's lactate solution for 24

hours. This cycle was repeated three times. The protocol was completed after the specimens were exposed an additional time to Ringer's lactate solution for 24 hours. The accelerated aging regimen approximated corrosion produced in two years of intraoral exposure (Matyas & others, 1978).

The restored teeth were then randomly placed into one of the following treatment groups:

**Group 1:** No treatment (Control).

**Group 2:** The amalgam margin defect was air abraded with 40-micron alumina powder at 40 psi using a hand-held air abrasion unit (Danville Engineering, San Ramon, CA 94526) after which oil-free compressed air was used to remove alumina debris. The enamel and amalgam margins of the defect were then etched for 20 seconds with 37% phosphoric acid, rinsed with distilled water and dried with oil-free compressed air. A flowable resin composite (Permaflow, Ultradent, South Jordan, UT 84095) was carefully injected into the marginal defect with attention to confining it to the defect and adjacent enamel and amalgam margins. The resin was then polymerized with a visible light-curing unit (Optilux 401, Kerr/Demetron, Orange, CA 92867) for 40 seconds. The adequacy of the light unit's intensity (800 mW/cm<sup>2</sup>) was assessed immediately prior to use with a hand-held radiometer (Model 100, Kerr/Demetron). No finishing of the repaired defect was done following light activation. Figure 2 shows a typical repaired defect.

**Group 3:** The amalgam margin defect was air abraded with alumina powder and cleaned with compressed air as described for Group 2. A fifth-generation dentin bonding agent (PQ1, Ultradent) was then applied by first etching the enamel and amalgam margins of the defect for 15 seconds with Ultra-Etch (35% phosphoric acid). The etchant was then removed by rinsing with distilled water and the area was lightly dried with oil-free compressed air. Care was taken to leave the etched area slightly moist. PQ1 Bonding Agent was vigorously applied to the etched area for 15 seconds using the brush tip provided, then air dried using 1- to 2-second

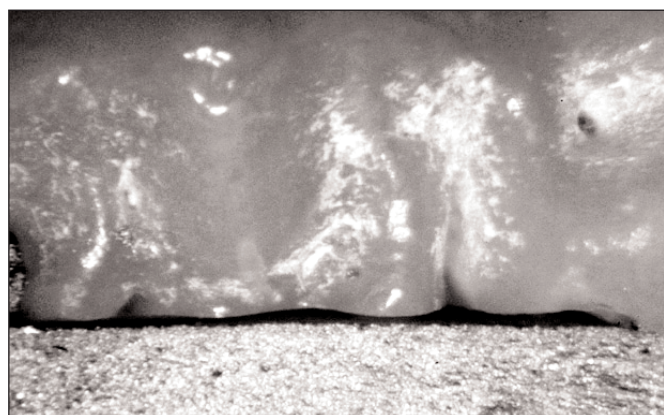


Figure 1. Amalgam margin defect (25X magnification).

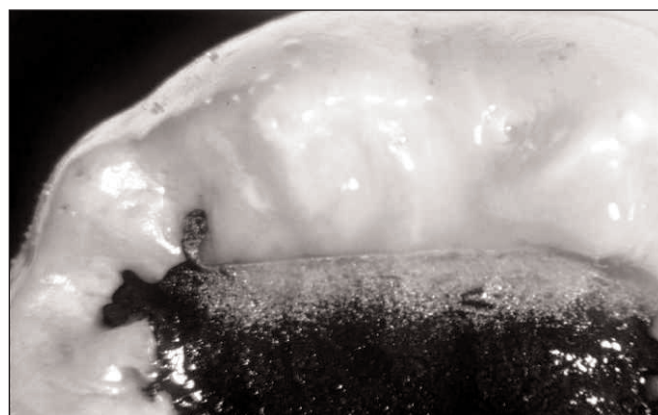


Figure 2. Repaired amalgam margin defect (8X magnification).

intermittent air bursts from an air/water syringe. The treated amalgam and enamel were visually inspected to ensure that they were glossy, and the bonding agent was light activated for 20 seconds. The marginal defect was then repaired by injecting flowable resin composite into it and light activating the resin as described for Group 2. As with the Group 2 specimens, no finishing of the repaired defect was done following light activation.

After repair, all specimens were thermocycled 500 times between a 5°C water bath and a 55°C water bath. A dwell time of 30 seconds was used with a transfer time of five seconds. The root apices of the specimens were occluded with a glass ionomer material (Ketac Bond, ESPE America, Plymouth Meeting, PA 19462), and the entire tooth surface was sealed with two coats of fingernail polish. The fingernail polish was painted to within one millimeter of the treated margin defect. The specimens were then immersed for 24 hours in a 0.5% solution of basic fuchsin dye at 37°C. After removal from the dye, the specimens were mounted in self-cure epoxy (Buehler, Lake Bluff, IL 60044) and sectioned faciolingually with a water-cooled, slow-speed diamond saw (Buehler). One millimeter sections were obtained from both ends of the repair as well as at mid-restoration. Microleakage was then scored by a blinded evaluator using a stereo microscope (Zeiss Stemi SR, Carl Zeiss Inc, Thornwood, NY 10594) at 8X magnification. The following scoring system was used: 0 - no leakage; 1 - leakage up to one-half the length of the wall of the defect; 2 - leakage between one-half and the entire length of the wall of the defect and 3 - leakage along the pulpal floor of the preparation. The highest leakage score for any of the three sections per tooth was recorded as the overall score for that specimen. Statistical analysis was accomplished using the Kruskal-Wallis test and Mann-Whitney U test for non-parametric data ( $\alpha=.05$ ).

RESULTS

Table 1 summarizes the degrees of leakage for the groups. Statistical analysis indicated that the repair groups (Groups 2 and 3) leaked significantly less than the control group (Group 1) (both  $p=0.0059$ ). There was no significant difference in degree of leakage between the repair groups ( $p=0.38$ ).

Table 1: Frequency Distribution of Degrees of Leakage for Control and Repair Groups					
Groups	Procedure	Leakage Category			
		0	1	2	3
1	Control (no repair)	3	0	1	8
2	Air abrade, acid etch, flowable composite	9	2	0	1
3	Air abrade, bonding agent, flowable composite	7	4	1	0
Category 0 - No leakage					
Category 1 - Leakage up to one-half the length of the wall of the defect					
Category 2 - Leakage between one-half and the entire length of the wall of the defect					
Category 3 - Leakage along the pulpal floor of the preparation					

DISCUSSION

In this *in vitro* study, an attempt was made to simulate common clinical situations involving the repair of a narrow non-carious amalgam marginal defect. Caries-free mandibular molars were used because the occlusal anatomy of these teeth provides a more consistent linear profile for standardized preparations. A 40-micron-wide marginal defect was utilized because it is readily detectable with a dental explorer and is visually discernable. In reality, although great care was used in preparing standardized defects, some were slightly larger than expected. This lack of uniformity was not necessarily undesirable, however, because one can expect to encounter defects of various widths clinically.

The results of this study indicate that both treatment groups had significantly less microleakage than the untreated control. Air abrading amalgam surfaces prior to repair has been recommended because aluminum oxide particles remove corrosion products, increase micromechanical retention and increase the surface energy of the alloy (Anderson, 1993). All of these effects improve the wettability and adaptation of resin to amalgam. It was hypothesized that the application of a fifth-generation bonding agent would increase the wettability of the amalgam and the etched tooth structure and promote more intimate adaptation of the flowable resin to those surfaces. The use of the bonding agent, however, did not measurably improve the degree of protection against leakage provided by the resin composite. Although manufacturers have simplified the application process of each subsequent generation of bonding agent, fifth-generation products remain technique sensitive and relatively time consuming to apply. The fact that a dentin bonding agent is not necessary is a positive finding for clinicians because it means that repairs of these types of marginal defects can be made more quickly and simply.

CONCLUSIONS

Under the conditions of this study, flowable resin composite was found to be an acceptable material for repairing selected non-carious amalgam margin defects. The use of a dentin-bonding agent prior to placement of the flowable resin composite did not appear to improve the protection against leakage afforded by the resin composite. Future research should be directed toward clinical evaluation of this technique.

Disclaimer

The ideas expressed in this article are those of the authors only and do not reflect the official opinion of the United States Air Force or the Department of Defense.

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# Effect of Bonding Variables on the Shear Bond Strength and Interfacial Morphology of a One-Bottle Adhesive

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N Tanaka • Y Katoh

## Clinical Relevance

The use of Prime & Bond and Dyract AP on etched, moist or dry enamel and moist dentin resulted in high bond strengths and intimate adaptation of resin to the substrate.

## SUMMARY

This study's objectives were: 1) to determine the combination of bonding procedures (with or without acid etching, moist or dry substrate, one or two applications of primer/adhesive) that would produce the highest shear bond strength of Prime & Bond and Dyract AP and 2) to characterize the resin-dentin/enamel interface produced by these bonding procedures. Ninety-six bovine incisors were randomly assigned to eight groups for shear bond testing to enamel (n=6) and dentin (n=6). Prime & Bond and Dyract AP were applied and cured following manufacturers' instructions. Shear bond testing was conducted in a Universal Testing Machine. Thirty-

two bovine incisors were sectioned to produce blocks with enamel and dentin, then bonded in pairs for evaluation of interfacial morphology. They were polished and argon ion-etched using a high-speed argon ion-etching machine and examined by SEM. The groups where enamel was etched, kept moist or dry and received a single application of Prime & Bond produced the highest shear bond strength. Dentin bond strengths were high in the groups where dentin was etched and kept moist. The number of Prime & Bond applications had no effect on dentin bond strength. Acid etching results in better adaptation of Prime & Bond to enamel and dentin regardless of whether moisture is present.

## INTRODUCTION

Current dental adhesives have the primer and adhesive combined in a single component, which reduces the number of application steps, making these dental adhesives simpler to use and less technique-sensitive. Although they are commonly marketed as single-component or "one-step" systems, most still require a separate conditioning step and multiple applications of primer/adhesive (Swift & Bayne, 1997). One product not requiring acid conditioning of the tooth surface is Dyract PSA, also known in the US as Prime & Bond. Newer versions have been introduced (Prime & Bond

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2.0, Prime & Bond 2.1 and Prime & Bond NT), but instructions are the same regarding acid conditioning. Their product literature states that these primer/adhesive systems, when used with the polyacid-modified composite Dyract AP (Dentsply/DeTrey, Konstanz 78467, Germany), do not require acid conditioning of enamel and dentin except in stress-bearing Class I, Class II and Class IV cases.

However, it has been found that without acid conditioning, Prime & Bond produces no visible etching pattern on enamel (Fritz, Finger & Uno, 1996). This could explain the findings of independent investigators that acid etching of enamel, not considered necessary by the manufacturer, produces a significant increase in bond strength (Cortes & others, 1998; Abate & others, 1997; Desai & Tyas, 1996; Triolo, Barkmeier & Los, 1995; Cortes, García-Godoy & Boj, 1993). As a result, the manufacturer's other directions, such as bonding to dry enamel, bonding to moist dentin and applying two layers of primer/adhesive, should also be investigated. This has been done in two studies (Barkmeier, Hammesfahr, & Latta, 1999; Nathanson & Ashyeri, 1997) but their experimental groups included no other possible combinations of these three factors (acid conditioning, substrate condition, number of primer/adhesive applications).

In view of the result of a one-year clinical evaluation of these materials, a study to determine the bonding procedure(s) that would produce high bond strengths to both enamel and dentin is relevant. Marginal discoloration around some restorations (8 of 28 restorations) was observed at the enamel margins, which was attributed to the low bond strength to unetched enamel (Tyas, 1998).

This study, on the other hand, aims to: 1) determine the combination of bonding procedures (with or without acid conditioning, moist or dry substrate, one or two applications of primer or adhesive) that would produce the highest shear bond strength of Prime & Bond and Dyract AP to enamel and dentin and 2) characterize the resin-dentin/enamel interface produced by these bonding procedures.

## METHODS AND MATERIALS

This study used 128 bovine mandibular incisors. Studies have shown that bovine teeth are suitable substitutes for human teeth in adhesion tests (Nakamichi, Iwaku & Fusayama, 1983, Schilke & others, 2000). The teeth were cleaned and kept frozen in distilled water for not more than three months prior to the start of the study.

Ninety-six teeth were randomly assigned to eight groups for shear bond testing to

enamel (n=6) and to dentin (n=6). Table 1 shows the experimental groups and the corresponding bonding procedures to enamel and dentin. Prior to the bonding procedures, the crowns were sectioned (Velnus Diamond Saw, Osaka 553-0003, Japan) into rectangular blocks. These tooth blocks were then embedded in self-curing resin (Unifast Trad, GC Corporation, Aichi 486-0844, Japan) to produce rectangular blocks (20 mm X 20 mm X 10 mm). Flat enamel and dentin surfaces were prepared on the labial surfaces (junction of the middle and cervical third) by wet grinding using 180-grit and 600-grit silicon carbide paper (Carbimet, Buehler Ltd, Lake Bluff, IL 60044). For the dentin bonding sites, care was taken to ensure that only superficial dentin was exposed. To limit the area of the bonded surface, a piece of double-sided tape (Nichiban, Tokyo 112-8663, Japan) with a hole (diameter of 4.0 mm) was attached to the specimen surface. A clear plastic tube with an inner diameter of 4.0 mm and height of 5.0 mm was then placed on top of the double-sided tape.

The bonding procedures were done at an ambient laboratory temperature of  $23 \pm 1^\circ\text{C}$  and  $>30\%$  humidity.

In the groups that required acid conditioning, Conditioner 36 (Lot #9809001214, Dentsply/DeTrey, Konstanz 78467, Germany) was directly syringed on the substrate surface. After the required etching times (30 seconds for enamel and 15 seconds for dentin), the etchant was rinsed for 20 seconds with copious amounts of water.

For the moist substrate surfaces, the residual water from the etchant-rinsing step was removed by blot drying (Kimwipes, Kimberly-Clark Corp, Roswell, GA 30076). The surface was visibly moist with no pooling of water. The dry substrate surfaces were obtained by drying the surface with compressed air for at least 20 seconds.

Prime & Bond (Lot #9808000866, Dentsply/DeTrey, Konstanz 78467, Germany) was then applied using a brush which was moved across the surface for three seconds. After 30 seconds, the primer/adhesive was lightly

Table 1: *Experimental Groups and Bonding Variables*

Groups	Bonding Variables		
	Acid-Etching	Condition of Substrate	Number of Prime & Bond Applications
A	Yes	Moist	One
B	Yes	Dry	One
C	Yes	Moist	Two
D	Yes	Dry	Two
E	No	Moist	One
F	No	Dry	One
G	No	Moist	Two
H	No	Dry	Two

air dried for 10 seconds with an air syringe placed approximately 10 centimeters from the surface. It was then light cured (Lightel II, J Morita Corp, Kuraray Co Ltd, Tokyo 103-0027, Japan) for 10 seconds. For the groups requiring two layers of primer/adhesive, a second application was done and immediately air dried and light cured for 10 seconds each. A 1-mm increment of Dyract AP (Lot #60604603, Dentsply/DeTrey, Konstanz 78467, Germany) was syringed, compacted and light cured from three sides for 20 seconds per side. This was also done on the two additional increments (2 mm).

The intensity of the light was checked using a curing radiometer (Demetron Research Corp, Model 100, Danbury, CT 06810) after every six specimens to ensure an output greater than 400 mW/cm<sup>2</sup>.

The specimens were stored in distilled water at 37°C for 24 hours. Shear bond strength was determined using a straight-edge chisel in a universal testing machine (Shimadzu Autograph AG-2000B, Shimadzu Corp, Kyoto 604-8511, Japan) with a crosshead speed of 0.5 mm/min. Shear bond strength was calculated as the ratio of fracture load and bonding area expressed in MPa.

Data were subjected to separate one-way ANOVA for enamel and dentin. Post hoc comparisons were made using the Tukey-Kramer HSD test calculated at 0.05 significance level. No attempt was made to compare the bond strengths to enamel and dentin.

To examine the adhesive resin-dentin/enamel interface, 16 pairs of rectangular tooth blocks with a thickness of approximately 2.0 mm were made from 32 bovine incisors. Wet grinding using 180-grit and 600-grit silicone carbide papers (Carbimet, Buehler Ltd, Lake Bluff, IL 60044) produced the flat dentin and enamel surfaces with enamel being located at both ends of the blocks. Two pairs of blocks were randomly assigned to the eight groups. After light curing the primer/adhesive layer, Dyract AP was placed on both blocks then pressed together to produce an approximately 1 mm-thick layer of restorative material between the blocks. This layer was light cured through the blocks for 80 seconds. The block pairs were then embedded in self-curing resin (Unifast Trad, GC Corporation, Aichi 486-0844, Japan) to prevent possible separation during polishing, especially the groups that produced low shear bond strength values (Figure 1). They were then immersed in 10 percent formaldehyde fixative solution for at least eight hours (Inokoshi & others, 1990). The specimens were successively ground and polished perpendicular to the bonded surfaces with wet silicon carbide papers (Carbimet, Buehler Ltd, Lake Bluff, IL 60044) of 600-, 1000-, 1,200- and 4,000-grit size, followed successively by polishing cloths with diamond suspension (Metadi Diamond Suspension,

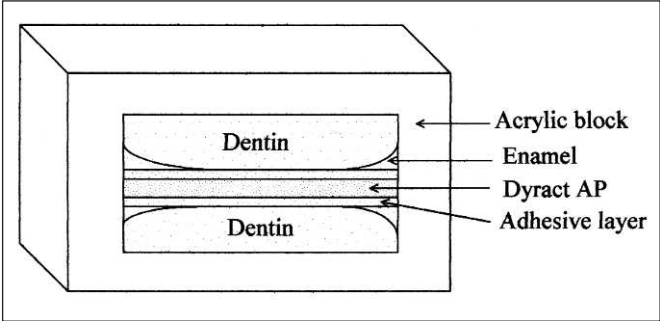


Figure 1. Diagram of the specimen for the evaluation of the resin-dentin and resin-enamel interfaces.

Buehler, Lake Bluff, IL 60044) of 3 µm (Lot #26316) and 1 µm (Lot #25680) grit size. Then the specimens were ultrasonically cleaned for 10 minutes, dehydrated in an ascending ethanol series from 30% to 100% and thoroughly air dried. The polished resin-dentin/enamel interfaces were then etched with an argon ion beam in an argon ion-etching machine (XP-HSI63, JEOL, Tokyo 196-0021, Japan) for 10 seconds. The accelerating voltage was set at 1 kV and gun current to 10 mA/cm<sup>2</sup>. They were then mounted on aluminum stubs, sputter coated with platinum/palladium (Hitachi E101, Hitachi Ltd, Tokyo 105-8430, Japan) and observed with a scanning electron microscope (Hitachi S-800, Hitachi Ltd, Tokyo 105-8430, Japan).

The hybrid layer and primer/adhesive layer thicknesses were then measured in the developed prints using a profile projector (Nikon V-12, Nikon, Tokyo 100-8331, Japan).

RESULTS

Tables 2 and 3 show the results of the enamel and dentin groups.

The enamel bond strengths ranged from 4.39 MPa to 21.01 MPa. Post-hoc testing showed that Groups A and B had significantly higher bond strengths than the other groups.

The dentin bond strengths ranged from 8.14 MPa to 16.43 MPa. Post-hoc testing showed that the bond

Table 2: Shear Bond Strength (MPa) to Enamel	
Group	Mean ± SD
A	21.01 ± 2.53 <sup>a</sup>
B	19.15 ± 2.22 <sup>a</sup>
C	15.58 ± 1.78 <sup>b</sup>
D	13.72 ± 1.83 <sup>b</sup>
E	9.24 ± 1.23 <sup>c</sup>
F	5.15 ± 0.61 <sup>d</sup>
G	6.54 ± 0.55 <sup>c,d</sup>
H	4.39 ± 0.67 <sup>d</sup>
Note: Same superscript letters indicate no significant difference (p<0.05).	



Table 3: Shear Bond Strength (MPa) to Dentin and Hybrid Layer Thickness ( $\mu\text{m}$ )		
Group	Mean $\pm$ SD	Hybrid Layer Thickness ( $\mu\text{m}$ )
A	16.43 $\pm$ 2.17 <sup>a</sup>	1.73 – 4.33
B	9.57 $\pm$ 1.31 <sup>b,d</sup>	1.31 – 5.49
C	14.16 $\pm$ 1.65 <sup>a,c</sup>	1.75 – 3.02
D	11.87 $\pm$ 1.89 <sup>b,c</sup>	1.32 – 2.59
E	11.38 $\pm$ 1.23 <sup>b,c</sup>	0 – 0.66
F	8.14 $\pm$ 0.91 <sup>d</sup>	0 – 0.54
G	11.74 $\pm$ 1.58 <sup>b,c</sup>	0 – 0.87
H	10.16 $\pm$ 1.29 <sup>b,d</sup>	0 – 0.80

Note: Same superscript letters indicate no significant difference ( $p < 0.05$ ).

strengths of Groups A and C were significantly higher than the other groups.

Representative resin-dentin and resin-enamel interfaces are shown in Figures 2-9. All the groups that were acid-etched showed distinct intertubular and peritubular hybrid layer and resin tag formation (Figures 2A, 3A, 4A, 5A). This was not the case for the non-acid-etched groups that had thin, and sometimes indistinct hybrid layers that made taking measurements difficult. Resin tags were rarely seen and most tubules were covered by smear plugs (Figures 6A, 7A, 8A, 9A).

The hybrid layer had a granular, sometimes fibrous appearance. The thickness in the etched groups ranged

from 1.31  $\mu\text{m}$  to 5.49  $\mu\text{m}$ . Those in the non-acid-etched groups were 0–0.87  $\mu\text{m}$ . Ranges are reported here because of the great variability noted among specimens and along different areas of the same specimen.

No difference in the morphology of the hybrid layer could be observed between moist and dry substrate specimens. This was also true between groups that received one and two applications of primer/adhesive. However, the thickness of the adhesive layer in the groups receiving a single application of primer/adhesive was noticeably thinner. In the groups with thick adhesive layers and even those with thin adhesive layers, amorphous hybrid layer-like structures could be seen. These structures were often seen as globules within the hybrid layer (Figures 8 A, B and Figures 9 A, B) and some were continuous with the hybrid layer (not shown).

## DISCUSSION

### Enamel Groups

The highest bond strength to enamel was obtained when the enamel substrate was acid etched and kept moist or dry with only one layer of primer/adhesive applied (Groups A and B). These bonding substrates and the number of primer/adhesive applications differ from the manufacturer's recommendations for bonding to unetched, dry enamel and applying two layers of primer/adhesive. It has already been established that acid etching improves the bond strength of resin to enamel. Even for this particular brand of adhesive resin, studies have reported significantly higher bond strengths to etched enamel (Hse, Leung & Wei, 1999; Cortes, García-Godoy & Boj, 1993; Triolo & others, 1995; Abate & others, 1997). This can be attributed to acid etchants producing microporosities that this primer/adhesive cannot produce on its own as the microporosities show (Figures 6B–7B).

Two other studies had the same SEM observation (Fritz & others, 1996, Ferrari, Goracci & García-Godoy, 1997). Therefore, it is not surprising that the groups where acid etching was done had higher bond strengths to enamel. Furthermore, bonding to etched and moist enamel resulted in bond strengths not significantly different from acid etched and dry enamel. This is consistent with the findings of Kanca (1997) and Finger & Fritz (1997), whose studies also used a hydrophilic adhesive resin with acetone as the solvent. A study using another kind of hydrophilic primer (HEMA) with ethanol/water as the solvent also observed the same result (Jain & Stewart, 2000).

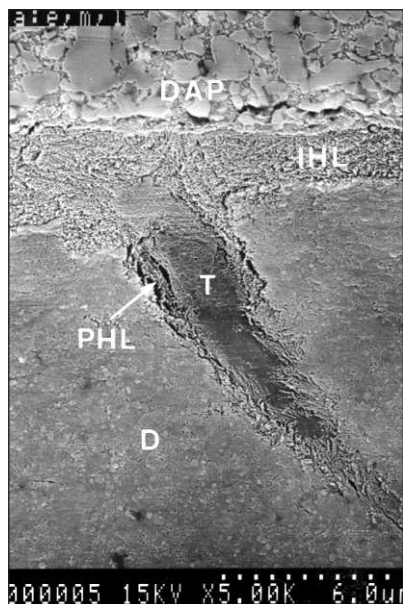


Figure 2A. Resin-dentin interface of group A specimen showing intertubular hybrid layer (IHL) and peritubular hybrid layer (PHL) formation. A resin tag (T) is visible inside a tubule and appear to be bonded to the tubule walls. DAP= Dyract AP. D=Dentin X5000.

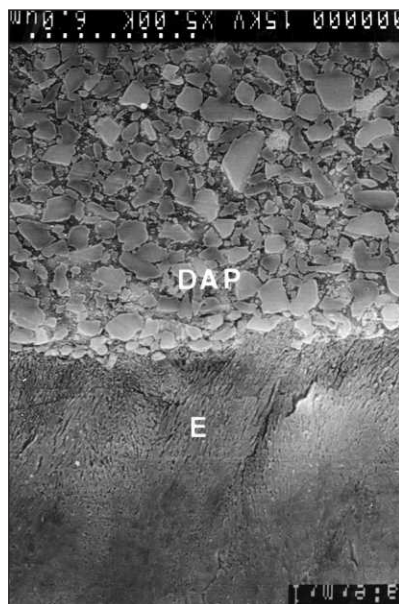


Figure 2B. Resin-enamel interface of group A specimen showing good adaptation of resin to the etched enamel surface. No adhesive layer is visible between Dyract AP (DAP) and enamel (E) X5000.

These results may be explained by the observation that when acetone mixes with residual water, the vapor pressure of water is elevated and surface tension is decreased (Weast, 1970). These twin actions allow the acetone to act as a "water chaser," displace the water, carry the resin into the micro-porosities and evaporate, leaving the resin behind (Gwinnett & Kanca, 1992). It seems that the amount of acetone in a single application of primer/adhesive was enough to "chase away" the water in the moist enamel surface. This would explain the insignificant difference in the bond strengths of Groups A and B.

The lower shear bond strength observed when two layers of primer/adhesive were applied may have resulted from the thicker primer/adhesive layer produced as observed in the SEM pictures. This thick primer/adhesive layer may adversely affect shear bond strength in a number of ways. First, since the adhesive resin is unfilled and has a greater proportion of functional diluent (in most cases, including this brand, triethyl-eneglycol dimethacrylate), there is a substantially greater potential for polymerization shrinkage during curing (Craig, 1989). This could compromise the enamel/ resin bond strength and increase the potential for microleakage (Jensen & Chan, 1985). Second, since the adhesive resin is unfilled, it has low mechanical properties. The mechanical properties have been shown to have an effect on the bond strength (Amory & Yvon, 1994; Finger, Inoue & Asmussen, 1994). Improving the mechanical properties by adding fillers (up to a certain limit) has been shown to improve bond strength (Fanning & others, 1995; Miyazaki & others, 1995). This is also the rationale for the addition of nanofillers in Prime and Bond NT (Dentsply/DeTrey Technical Manual). Lastly, two air-thinning procedures of one-bottle bonding resins may result in more pronounced oxygen inhibition. This may then lead to lower bond strength values (Holderegger & others, 1997).

The values obtained for etched, dry enamel with one and two layers of Prime & Bond were 13.72 MPa and 19.15 MPa, and these are within the range obtained by other studies (Abate & others, 1997; Cortes & others, 1998; Cortes & others, 1993; Triolo & others, 1995; Barkmeier & others, 1999). Although Abate (1997), Triolo (1995) and Barkmeier

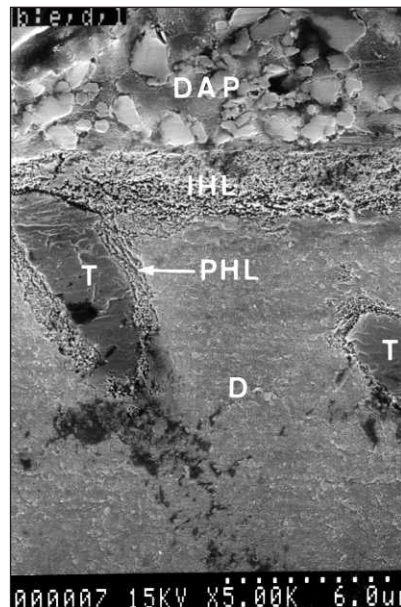


Figure 3A. Resin-dentin interface of group B specimen showing intertubular hybrid layer (IHL) and peritubular hybrid layer (PHL) formation. Similar to group A, a resin tag (T) is visible inside a tubule and appears to be bonded to the tubule walls. No adhesive layer is visible between Dyract AP (DAP) and intertubular hybrid layer (IHL). D= Dentin. X5000.

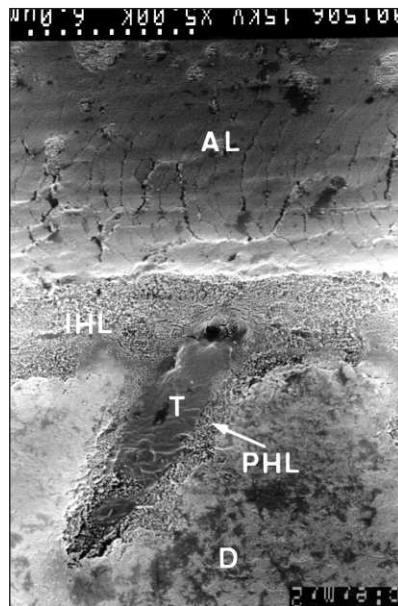


Figure 4A. Resin-dentin interface of group C specimen showing intertubular hybrid layer (IHL) and peritubular hybrid layer (PHL) formation. Resin tag formation can also be seen. Note the thick adhesive layer (AL) above the intertubular hybrid layer (IHL). D= Dentin. X5000.

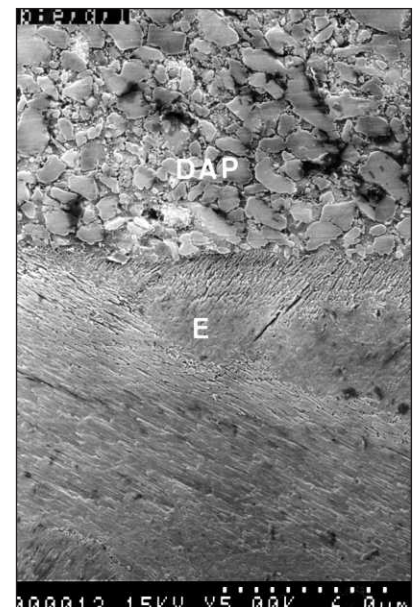


Figure 3B. Resin-enamel interface of group B specimen showing good adaptation of resin to the etched enamel surface. No adhesive layer is visible between Dyract AP (DAP) and enamel (E). X5000.

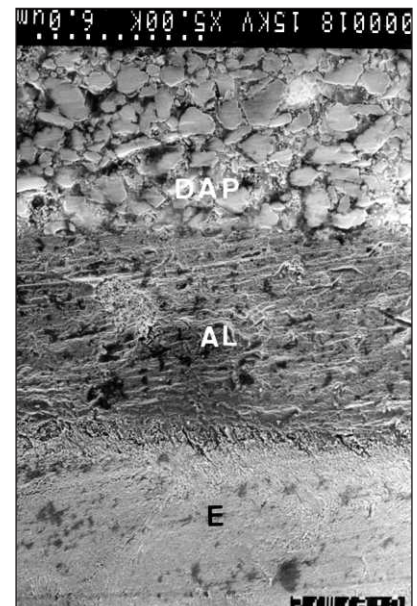


Figure 4B. Resin-enamel interface of group C specimen showing good adaptation of resin to the etched enamel surface. A thick adhesive layer (AL) can be seen between Dyract AP (DAP) and enamel (E). X5000.



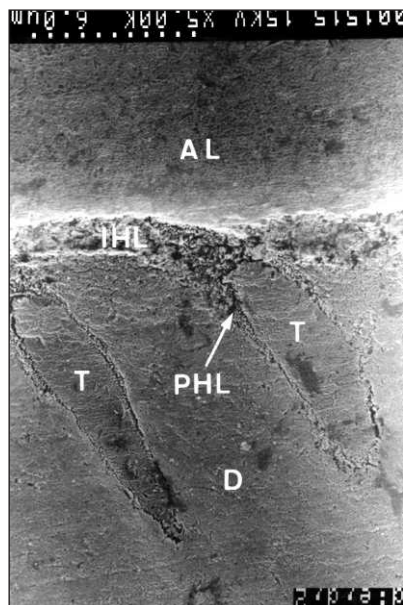


Figure 5A. Resin-dentin interface of group D specimen showing intertubular hybrid layer (IHL) and peritubular hybrid layer (PHL) formation. This picture is similar to Figure 3A, including the resin tag (T) and the thick adhesive layer (AL) above the intertubular hybrid layer (IHL). D= Dentin X5000.

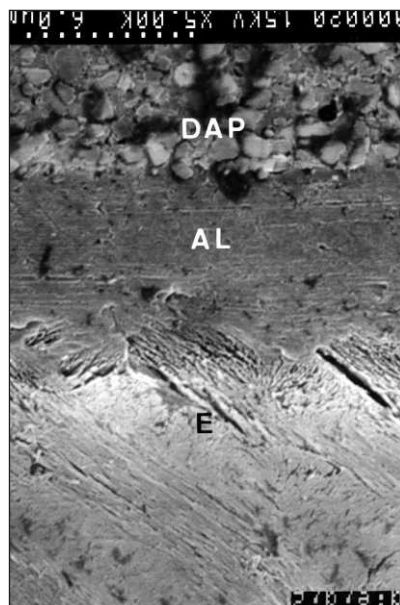


Figure 5B. Resin-enamel interface of group D specimen showing good adaptation of resin to the etched enamel surface. A thick adhesive layer (AL) can be seen between Dyract AP (DAP) and enamel (E) X5000.

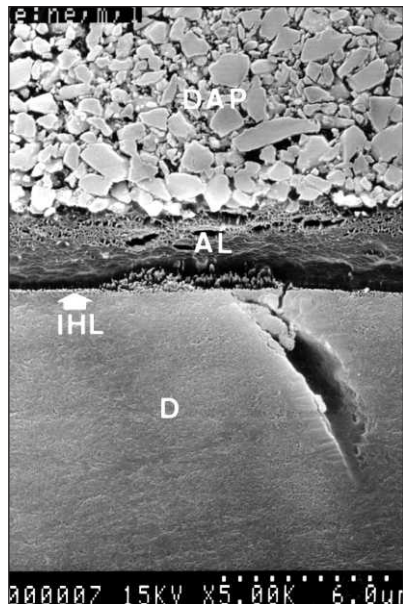


Figure 6A. Resin-dentin interface of Group E specimen showing a very thin intertubular hybrid layer (IHL) but no peritubular hybrid layer formation. A smear plug is seen covering a tubule. An adhesive layer (AL) is visible unlike in Figures 1A and 2A. DAP= Dyract AP. D= Dentin X5000.

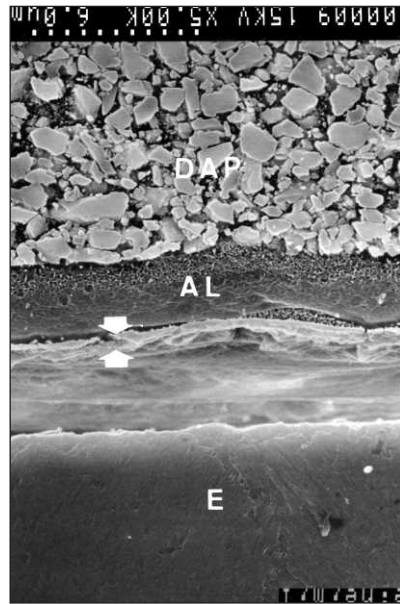


Figure 6B. SEM picture of a debonded resin-enamel interface of Group E specimen showing the adhesive layer (A) that appears to be bonded to the smear layer (between arrows). The smear layer debonded from the enamel (E) during specimen preparation. DAP= Dyract AP X5000.

(1999) observed higher bond strengths compared to those found in this study, the differences could be explained by variations in the methodology. In these three studies, no mention was made regarding how the bonding site was demarcated. This is important because extension of the adhesive beyond the bonding site will result in a significant elevation in bond strength (Van Noort & others, 1991; Retief, 1991; Unterbrink & Liebenberg, 1999). Despite these differences in the absolute values, Abate (1997), Triolo (1995), Cortes (1993, 1998) and this study have observed that bonding to etched enamel results in a higher bond strength than unetched enamel.

### Dentin Groups

The bonding procedure that produced the highest shear bond strength to enamel (Table 1, Group A) also produced the highest shear bond strength to dentin (Table 2, Group A). However, this did not significantly differ from Group C.

Getting a higher bond strength value to etched and moist dentin using Prime and Bond is consistent with an earlier study conducted at Creighton University and cited by Barkmeier (1999). A study that used another brands of "one-bottle" adhesive system also observed this trend (Kanca, 1997). This finding is consistent with the currently accepted mechanism of adhesion to dentin, that of micromechanical interlocking (Nakabayashi, Kojima & Masuhara, 1982; Nakabayashi, 1991). The acid etchant removes the smear layer, widens the orifices of the dentinal tubules and demineralizes the intertubular dentin up to a depth of 7.5  $\mu\text{m}$  (Van Meerbeek & others, 1992). Removal of the hydroxyapatite crystals upon etchant rinsing exposes a mesh of collagen fibers that should be kept moist to prevent collapse (Carvalho & others, 1996; Kanca, 1992; Jacobsen & Söderhold, 1995). When the primer/adhesive is applied, the residual water mixes with the acetone/ hydrophilic resin (PENTA or HEMA) mixture. The acetone/water phase then evaporates, leaving only the PENTA or HEMA molecules inside the collagen mesh (Jacobsen & Söderhold, 1995). The subsequent polymerization of the monomer resulted in a hybrid layer or resin-dentin inter-diffusion zone, which is believed to be the source of bonding to acid-etched dentin (Nakabayashi & others, 1982; Van Meerbeek & others, 1992; Eick & others, 1997). This



mechanism of adhesion (penetration of resin into demineralized hydroxyapatite crystal structure) is really the same as that with etched enamel (Kanca, 1997).

The micrographs of the acid-etched groups (Figures 2A–5A) show the formation of thick, distinct hybrid layers and resin tags. On the other hand, the unetched groups had a thin, sometimes indistinct hybrid layer. Resin tags were also rarely seen. Two studies (Cadroy, Boj & García, 1997; Ferrari & others, 1997) using Prime & Bond 2.1 did not observe any hybrid layer formation in unetched specimens. The difference in the results could be attributed to differences in the methods used to disclose the hybrid layer. The studies mentioned used acid etching, while this study used argon-ion etching. Even with argon-ion etching, the thin hybrid layer was still difficult to disclose. In some specimens, the etching time had to be increased to 20 seconds.

Despite the obvious differences in the thickness of the hybrid layers between the etched and unetched groups, no correlation between hybrid layer thickness/morphology and shear bond strength could be found. Other studies also arrived at the same conclusion (Prati & others, 1999, Prati & others, 1998, Vargas, Cobb & Denehy, 1997, Yoshiyama & others, 1996). They all show that bond strength is probably better correlated with bonded surface area rather than depth (that is, volume) of resin penetration as hypothesized by Pashley (1997). Although the SEM pictures could not explain bond strength results in terms of hybrid layer thickness or morphology, they did show the importance of etching dentin. The acid-etched groups showed intimate adaptation of resin to intertubular hybrid layer and of resin tags to peritubular hybrid layer, resulting in the formation of an excellent dentin seal. The formation of this seal, which includes the obtunding of patent dentin tubules, is crucial in preventing the undesirable effects of microleakage (Cox, 1992). The unetched groups were able to bond to superficial dentin but failed to penetrate the smear plugs and form resin tags.

The SEM micrographs also disclosed the presence of amorphous hybrid layer-like structures within the adhesive layer, which were sometimes continuous with the hybrid layer (Figures 6A,B–9A,B). Their presence made measurement of the thin hybrid layer

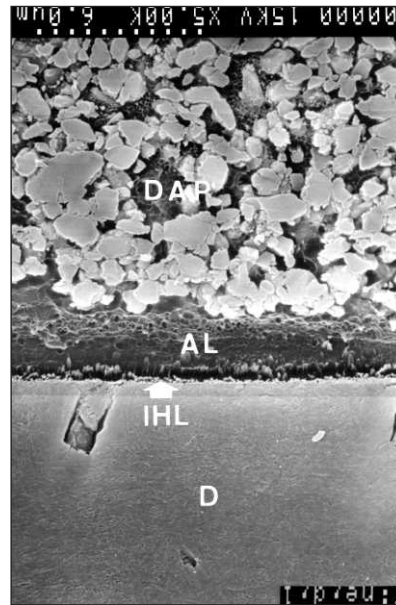


Figure 7A. Resin-dentin interface of group F specimen showing a very thin intertubular hybrid layer (IHL) but no peritubular hybrid layer formation. An adhesive layer (AL) is visible unlike Figures 1A and 2A. DAP= Dyract AP. D= Dentin X5000.

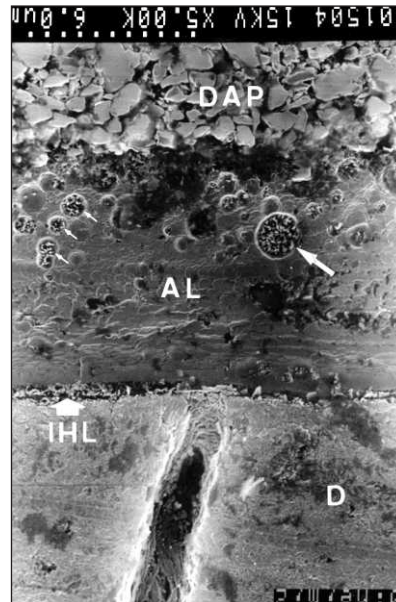


Figure 8A. Resin-dentin interface of group G specimen showing a very thin intertubular hybrid layer (IHL) but no peritubular hybrid layer formation. A smear plug is seen covering an empty tubule. An adhesive layer (AL), thicker than that found in Figure 5A, is observed. Note the presence of circular amorphous hybrid layer-like structures (arrow) inside the adhesive layer. DAP= Dyract AP D= Dentin X5000.

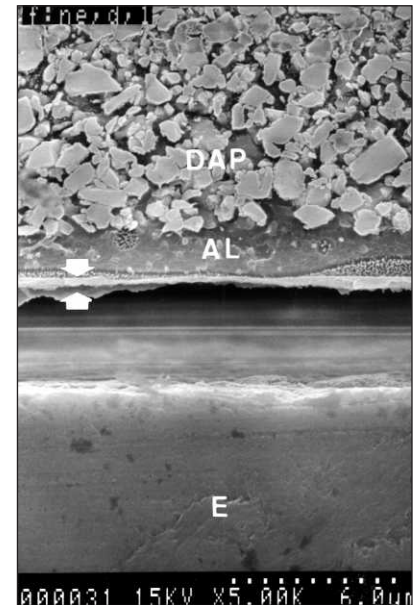


Figure 7B. SEM picture of a debonded resin-enamel interface of group F specimen showing the adhesive layer (AL) that appears to be bonded to the smear layer (between arrows). The smear layer debonded from the enamel (E) during specimen preparation. DAP= Dyract AP X5000.

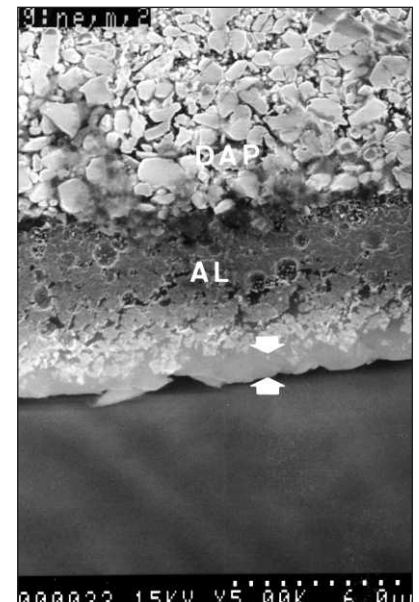


Figure 8B. SEM picture of a debonded resin-enamel interface of group G specimen showing the thick adhesive layer (AL) that appears to be bonded to the smear layer (between arrows). The smear layer debonded from the enamel during specimen preparation. The width of the gap was big enough to exclude the enamel from this picture. DAP= Dyract AP X5000.

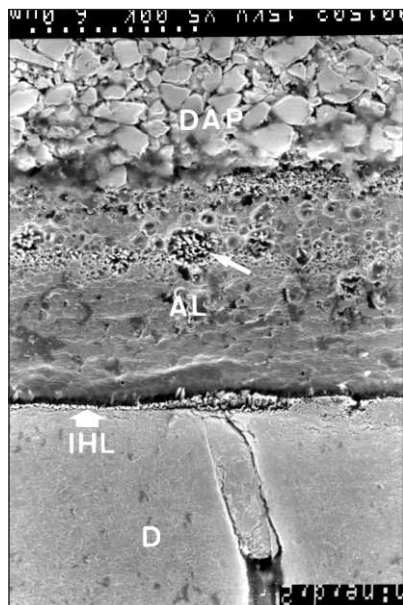


Figure 9A. Resin-dentin interface of group H specimen showing a very thin intertubular hybrid layer (IHL) but no peritubular hybrid layer formation. A smear plug is seen covering an empty tubule. The two applications of primer/adhesive resulted in an adhesive layer (AL) that is thicker than that found in Figure 6A. Note the presence of circular amorphous hybrid layer-like structures (arrow) inside the adhesive layer. DAP= Dyract AP. D= Dentin X5000.

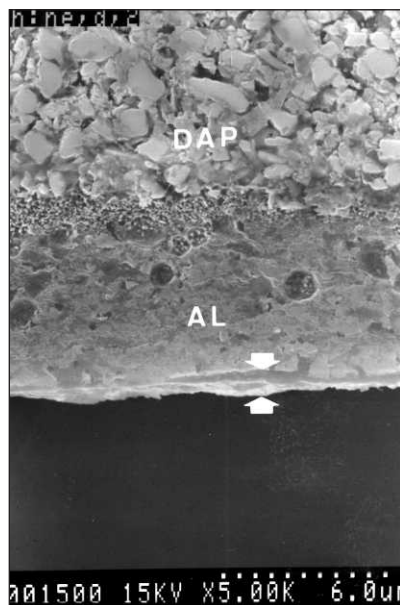


Figure 9B. SEM picture of a debonded resin-enamel interface of group H specimen showing the thick adhesive layer (AL) that appears to be bonded to the smear layer (between arrows). The smear layer debonded from the enamel during specimen preparation. The width of the gap was big enough to exclude the enamel from this picture. DAP= Dyract AP X5000.

of the unetched groups even more difficult. Similar structures have also been previously observed in other brands of one-bottle primer/adhesives, Single Bond and Scotchbond Multi-Purpose Plus (Vargas & others, 1997). However, this is the first time that these structures have been reported in dry substrates. They are believed to be primer globules, as suggested by Vargas (1997) and Tay, Gwinnett & Wei (1996).

Unlike the results obtained for the enamel groups, the number of primer/adhesive applications had no effect on bond strength. This finding is similar to that of Barkmeier (1999) and Nathanson & Ashyeri (1997). The two applications of primer/adhesive also resulted in a thicker primer/adhesive layer but did not adversely affect the shear bond strength, unlike what occurred in the enamel groups. The reason for this is unclear.

This study's finding, which is similar to those of others (Barkmeier, 1999; Nathanson, 1997)—that one application of Prime & Bond can produce bond strength not significantly different from two applications—does not imply that only one application is needed. It should be remembered that the experimental set-up might be different from what is clinically obtained. The plastic tube used to confine Dyract AP also confined Prime & Bond and made it possible for a single application to ade-

quately penetrate the enamel and dentin substrate. This may not be true *in vivo*, especially in Class V cavities, because there is nothing to prevent the spreading out of the primer/adhesive. The most important point is that enough adhesives must penetrate the etched surface to provide adequate mechanical interlocking (Swift & Bayne, 1997). The production of a uniform, glossy appearance with a single application may mean that a second application might not be necessary.

## CONCLUSIONS

- 1) High shear bond strengths can be achieved with Prime & Bond and Dyract AP when bonding is done to acid etched, moist enamel and dentin using a single application of Prime & Bond. Clinicians would welcome this single bonding procedure for enamel and dentin. Separate treatments of enamel and dentin are a practical impossibility for the clinicians in all but the most accessible and simple preparations (Kanca, 1997).
- 2) Acid etching of dentin and the application of Prime & Bond are required for the formation of a distinct hybrid layer and resin tags. However, there was no significant correlation between shear bond strength and the thickness of the hybrid layer.
- 3) Acid etching of enamel results in better adaptation of Prime & Bond to the enamel surface.

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# Re-Attachment of Anterior Fractured Teeth: Fracture Strength Using Different Techniques

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MRO Carrilho • LE Rodrigues Filho

## Clinical Relevance

The re-attachment of coronal fragments to the remaining tooth, using an overcontour or an internal groove technique as well as a composite build-up, can provide high fracture strength to restored teeth.

## SUMMARY

Fracture of anterior teeth by trauma is a common problem in children and teenagers. Complex metal-ceramic crowns with considerable loss of remaining sound structure are no longer necessary due to adhesive techniques, such as composite restorations and re-attachment techniques. This study compared the fracture strength of sound

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and restored anterior teeth using a resin composite and four re-attachment techniques. A “one bottle” adhesive system (One-Step, BISCO) and a dual cure resin cement (Duo-Link, BISCO) were applied. Thirty-five sound permanent lower central incisors were fractured by an axial load applied to the buccal area and randomly divided into five groups. The teeth were restored as follows: 1) bonded only = just bonding the fragment; 2) chamfer-group = after bonding, a chamfer was prepared on the enamel at the bonding line and filled with composite; 3) overcontour group = after bonding, a thin composite overcontour was applied on the buccal surface around the fracture line; 4) internal dentinal groove = before bonding, an internal groove was made and filled with a resin composite; 5) resin composite group = after a bevel preparation on the enamel edge, the adhesive system was applied and the fractured part of the teeth rebuilt by resin composite. Restored teeth were subjected to the same loading in the same buccal area. Fracture strength after restorative procedure was expressed as a percentage of the original fracture strength and the results analyzed by Kruskal-Wallis statistical analysis. The mean percentages of fracture strength were: Group 1: 37.09%, Group 2: 60.62%, Group 3: 97.2%,

**Group 4: 90.54% and Group 5: 95.8%. It was concluded that the re-attachment techniques used in Groups 3 and 4, as well as the composite restored group (Group 5), were statistically similar and reached the highest fracture resistance, similar to the fracture resistance of sound teeth.**

## INTRODUCTION

Coronal fracture by trauma is the most frequent type of dental injury in the permanent dentition (Andreasen & Andreasen, 1993). Recent studies on the incidence of dental trauma, mainly among children and teenagers, have shown that this injury affects up to 25% of patients at this age (Hamilton, Hill & Holloway, 1997; Murchison, Burke & Worthington, 1999). The most affected teeth are upper incisors due to their anterior position and protrusion caused by the eruptive process (Andreasen & Andreasen, 1993).

In the past, fractured teeth were restored using acrylic resin or complex ceramic restorations associated with metals. These restorations did not promote adequate long-term esthetics and also required a significant tooth reduction during preparation. Because of difficulty in obtaining good retention, there were few attempts to re-attach fragments. Chosack & Eildeman, 1964, were the first to report it. The amputated anterior crown was cemented to a cast post. Spasser, in 1977, used interlocking mini-pins associated with light-cured composites. With the acceptance of the acid-etch technique, re-attachment of the tooth fragment became a more usual procedure because of the excellent retention obtained with the fluid resin on etched enamel.

Re-attachment of a tooth fragment is the first choice for restoring fractured teeth, whether or not the technique is combined with resin composites (Tennerly, 1978; Simonsen, 1979; Simonsen, 1982; Silva Filho & Esberhard, 1982; Franco & others, 1985; Osborne & Lambert, 1985; Fontana & others, 1986; Martens & others, 1988; Burke, 1991; Diangelis & Jungbluth, 1992; Dickerson, 1994). This treatment may offer several advantages over conventional acid-etch composite restoration. Improved esthetics is obtained since enamel's original shape, color, brightness and surface texture are maintained. In addition, the incisal edge will wear at a similar rate to adjacent teeth, whereas a composite restoration will likely wear more rapidly (Leinfelder, 1993; Lutz, Krejci & Oddera, 1996). Furthermore, this technique can be less time-consuming and provide more predictable long-term appearance. Despite the fact that this technique is less than ideal, the advantages associated with the shortcomings of composite buildup have also led some clinicians to use tooth fragments from extracted teeth when the original is not available (Gabrielli & others, 1981; Santos & Bianchi, 1991; Busato & others, 1998).

Despite the fact that re-attachment of tooth fragments is commonly suggested in the literature, there is no agreement about which technique may provide higher mechanical strength and longevity. In several clinical reports, the enamel margins of tooth and fragment were beveled circumferentially before re-attaching the fragment in order to obtain a better retention and enhancement of the finishing line with a resin composite (Simonsen, 1979; Amir, Bar-Gil & Sarnat, 1986; Burke, 1991; van der Vyner & Morais, 1996; Walker, 1996). However, this technique requires additional enamel preparation, and in certain cases, the precise fit between the segments is lost, which makes the correct positioning of the fragment more difficult. This may explain why some authors prefer creating a chamfer in the fracture line after performing the bonding procedure (Davis, Roth & Levi, 1983; Franco & others, 1985; Andreasen & others, 1995).

The techniques described above require placement of a resin composite on the buccal surface of the tooth, which may compromise long-term esthetics. The abrasion and discoloration process that occurs in composites when exposed over time to the oral environment is well documented. In order to improve esthetics, Silva Filho & Esberhard (1982) attempted to place a bevel only on the lingual surface. Another technique widely reported is placement of an internal V-shaped notch in enamel (Simonsen, 1982; Diangelis & Jungbluth, 1992). However, due to limited enamel thickness in anterior teeth, this procedure is difficult to perform.

Some clinical reports suggest placement of an internal groove in the tooth fragment by removing a sufficient amount of dentin to accommodate a cement liner applied to protect the pulpal tissue (Simonsen, 1982; Franco & others, 1985) or a glass ionomer cement to obtain some adhesion (Diangelis & Jungbluth, 1992; Burke, 1991). Currently, the space provided by this procedure is most often filled by a resin composite to reinforce bonding (Walker, 1996). In pulpless teeth, part of the pulp chamber has been used for additional mechanical retention (Amir, Bar-Gil & Sarnat, 1986; Diangelis & Jungbluth, 1992).

The improvement of hydrophilic adhesives combined with the acid-etch technique has also made the re-attachment technique possible with no additional preparation of the fracture site (Osborne & Lambert, 1985; Martens & others, 1988; Dickerson, 1994).

Even considering the proposed advantages of these techniques and their widespread use, the majority of these design features are selected empirically, and little is known about their influence in the long-term success of restorations. Research has reported that the primary cause of fragment loss is a new dental trauma or a non-physiological use of restored teeth, with a survival rate of 25% after seven years (Andreasen & others, 1995).



It seems reasonable to study fracture resistance of the different designs used to re-attach tooth fragments (bonding without additional preparation; placement of a chamfer, an overcontour or an internal groove comparing to a resin composite buildup), since the restored teeth may be exposed to new dental trauma. The null hypothesis to be tested was that there was no difference in fracture resistance of the different techniques used to re-attach tooth fragments.

## METHODS AND MATERIALS

Sixty sound human lower incisors extracted due to periodontal disease were selected under optical magnification (x2). Teeth free from cracks or other structural defects were chosen. They were disinfected in 0.5% chloramina for 15 days and stored for less than six months in 0.9% saline solution (DeWald, 1997). The test basically consisted of three procedures: (1) fracture of sound teeth; (2) restoration of fractured teeth using different techniques (3) and fracture of restored teeth, as in procedure 1.

### 1. Fracture of the Sound Teeth

The buccal surface of each tooth was divided into transversal and longitudinal thirds. Figure 1 shows the area (point) for application of the perpendicular loading.

The roots of the teeth were confined in a special device (holder) and adapted in a universal testing machine Riehle (Riehle Testing Machine, MODEL FS-5, ILLINOIS, USA) (Figure 2).

The load was applied to each tooth in a buccal-to-lingual direction by means of a small stainless steel ball (2 mm<sup>2</sup>) inserted at the end of a pin held in the crosshead of the universal testing machine at a speed of 0.6 mm/min. The force required to fracture the tooth was recorded.

### 2. Restoration of the Fractured Teeth

Out of 60 teeth, only 35 were chosen for this *in vitro* study because they showed a Class II Ellis fracture type (Ellis & Davey, 1970), the pattern chosen for this *in vitro* study. Both the fragment and the remaining fracture surface were randomly divided into five groups and kept in 0.9% saline solution until the restoration procedure was performed (Farik & others, 1998b; 1999). All materials and techniques used are shown in Tables 1 and 2 and Figure 3.

Teeth from Groups 1, 2 and 3 had the fragments re-attached using a "one-bottle" adhesive system (One-Step, BISCO, Inc, Itasca, IL) and a dual cure resin luting cement (Duo-Link, BISCO, Inc). First, the adhesive system was applied following the manufacturer's instructions (Table 1) to both the fragment and the remaining fracture surface. The adhesive was not initially light cured to allow better positioning of the fragment to the tooth. After that, the resin luting cement was applied, the

fragment was re-attached on the tooth and the buccal and lingual surfaces were light cured for 40 seconds each.

In Group 1 no additional preparation was made (Figure 3A). In Group 2 following re-attachment, a 1.0 mm-depth chamfer was placed in the fracture line in the buccal surface using a diamond round bur (ref #1016, KG Sorensen, São Paulo, Brazil) (Table 2 and Figure 3B). In teeth from Group 3 following re-attachment, a preparation was placed on the buccal surface by means of a cylindrical diamond-finishing bur (ref #2135F, KG Sorensen) extending 2.5 mm coronally and apically from the fracture line with a depth

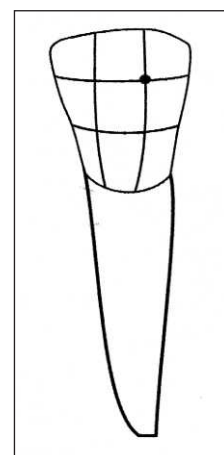


Figure 1. Tooth divided into transversal and longitudinal thirds to standardize the area (point) for the perpendicular load application.

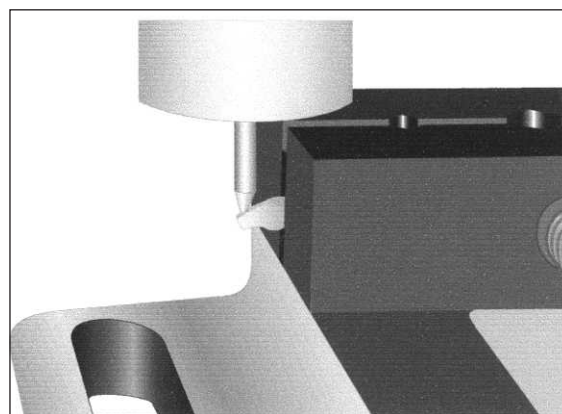


Figure 2. The root of the teeth confined in a special device and adapted in a universal-testing machine RIEHLE.

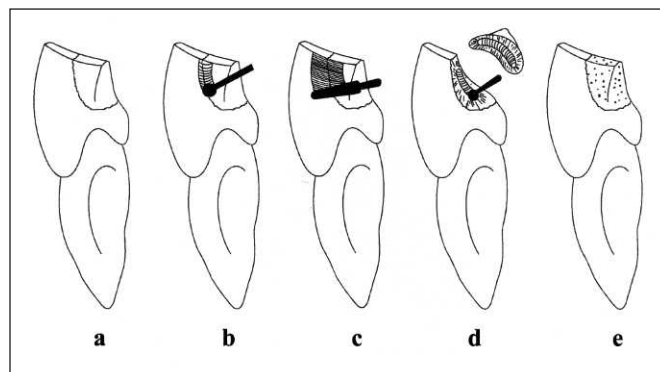


Figure 3. Techniques used to restore the fractured teeth. (3A) Bonded only; (3B) Chamfer; (3C) Overcontour; (3D) Internal groove; (3E) Composite buildup.

Table 1: *Materials and Treatment Sequences*

Code	Material (*)	Composition	Application Sequence	Batch
A	One-Step Adhesive system "One-bottle"	- Uni-etch 32% PA semigel - Adhesive BPDM, Bis-GMA, HEMA, Acetone, Photoinitiator	1. Acid etching 15 seconds 2. Rinse 15 seconds 3. Blot dry 4. Apply two coats of adhesive 5. Light-cure 10 seconds	- Uni-etch 01323 - Adhesive 00444
B	Duo-Link Dual resin luting cement	- Catalyst and base Bis-GMA, TEG-DMA (catalyst), UDMA (base), alumina (catalyst), Glass frit and particles not otherwise classified (PNOC)	1. Similar increments of past base and past catalyst 2. Mix for 15 seconds	9900006255
C	Aelitefil Resin Composite	- High filler content (60%wt) - Microfine radiopaque glass (0.7-0.8 $\mu$ m) hybridized with SiO <sub>2</sub> (0.04 $\mu$ m) - Bis-GMA, Urethane dimethacrylate, Polyalkylenoglycol Dimethacrylate and Aminoakyl Methacrylate	1. Incremental placement ( $<2$ mm) 2. Light-cure for 40 seconds	029024

(\*) BISCO, Inc, Itasca, IL

Table 2: *Technique Description and Sequence Material*

Groups	Technique	Description	Sequence of the Materials
1	Bonded only	Re-attachment of the fragment with no additional preparation	A + B
2	Chamfer	Re-attachment of the fragment + chamfer in the buccal surface	A + B + C
3	Overcontour	Re-attachment of the fragment + superficial preparation on the enamel	A + B + C
4	Internal groove	Internal dentinal groove + re-attachment of the fragment	A + B + C
5	Composite buildup	No fragment was used	A + C (enamel bevelled)

of 0.3 mm (Table 2 and Figure 3C). The preparation areas in Groups 2 and 3 were then treated in the following manner: One Step adhesive was applied as in Table 1, followed by placement of one increment of resin composite (shade A2 dentin—Aelitefil, BISCO). The resin was then light cured for 40 seconds. This created a slightly overcontoured tooth surface.

In Group 4 prior to performing the re-attachment technique, an internal groove (1 mm deep and 1 mm wide) was placed within the fragment and the remaining tooth by means of a carbide bur (ref #329, KG Sorensen) with a high-speed handpiece (Table 2 and Figure 3D). The One Step adhesive system (BISCO) was applied to each surface. Prior to light curing, a resin composite (Aelitefil, BISCO) was placed within the groove. The fragment was re-attached and the excess composite removed. Each surface was then light cured for 40 seconds.

No re-attachment technique was used in the fractured teeth from Group 5 (Table 2 and Figure 3E). A 45° bevel extending 1 mm on the buccal surface was prepared using a cylindrical diamond finishing bur (ref #2135F, KG Sorensen), and a resin composite buildup (Aelitefil, BISCO) was performed after adhesive application (One-Step, BISCO) (Table 1). Restorations were made following the incremental technique (+/- 3 increments were used). Each increment was light cured for 40 seconds.

For polymerization, a light-curing unit, OPTILUX 400 (Demetron Research Corp, Danbury, CT) at 450 mW/cm<sup>2</sup>, was used. The teeth were finished and polished with flexible discs (Sof-Lex Pop On polishing disks, 3M Dental Products, St Paul, MN).

### 3. Fracture of the Restored Teeth

The specimens were loaded in the same pre-determined area used in procedure 1 (Figures 1 and 2) until failure.

Groups	Sound Teeth		Restored Teeth		Recovery (%)	
	Mean	SD	Mean	SD	Mean	SD
1	29.02	6.5	10.50	2.82	37.09	10.90
2	24.78	4.18	14.39	4.77	60.62	25.64
3	22.39	9.25	21.06	8.13	97.2	33.2
4	24.23	1.26	21.94	3.92	90.54	16.53
5	25.91	3.06	25.02	9.68	95.8	33.3

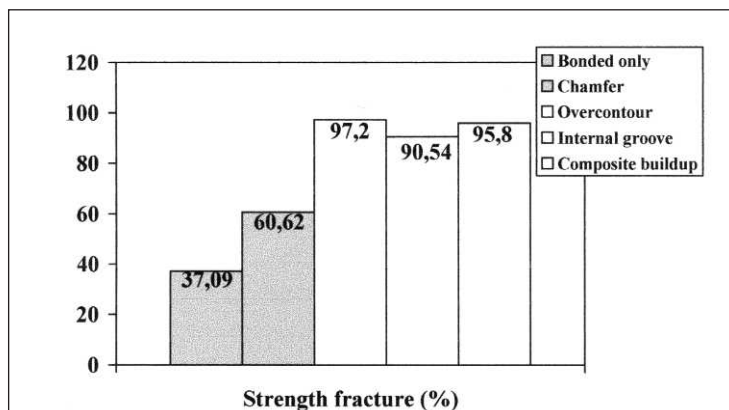


Figure 4. Mean percentage values of the strength recovery of the techniques used and the statistical significance (different shades = significance statistical).

The force required to detach each fragment was recorded. For each tooth, fracture strength was expressed as a percentage of the load required to fracture the sound tooth (strength recovery) so that it established a relationship between fracture strength of an intact tooth and the fracture strength obtained by the restorative procedures previously described. A non-parametric Kruskal-Wallis analysis using a 5% level of significance was performed on the recorded data.

## RESULTS

The mean force and standard deviation required to fracture the sound teeth was  $25.26 \pm 5.7$  Kgf, and after restoration, this force decreased to  $18.58 \pm 8.1$  Kgf. Table 3 presents the mean fracture resistance (Kgf) and standard deviation of sound and restored teeth as well as the strength recovery (%) of each group. Only the mean percentage values of strength recovery was statistically analyzed (Figure 4).

The statistical analysis showed no difference between results from Group 1 (bonded only) and Group 2 (chamfer). However, these results were significantly lower than the mean fracture strength found in Group 3 (overcontour), Group 4 (internal groove) and Group 5 (composite buildup), which did not significantly differ ( $p < 0.05$ ). The fracture path of the restored teeth followed the bonded interface in all specimens.

## DISCUSSION

The experimental design used in this study simulated real trauma. A fracture pattern without loss of enamel structure was obtained, which was not found in previously reported *in vitro* studies (Munksgaard & others, 1991; Andreasen & others, 1993; Badami, Dunne & Scheers, 1995; Farik & others, 1998a,b; Farik &

Munksgaard, 1999; Farik & others, 1999). Despite the fact that maxillary incisors are most affected by traumatic injuries (Andreasen & Andreasen, 1993), the experimental design was performed with lower incisors because they were easier to obtain.

The lower fracture strength found in Group 1 (bonded only) may be partly due to the smaller bonded area. This bonding technique may have a shorter longevity since only 37% of the strength of sound teeth was achieved. These findings oppose the results published by Farik & others (1998a,b), Farik & Munksgaard (1999) and Farik & others (1999), which may be due to differences in methodology used as well as the inclination of the load.

Teeth fragments from the 35 teeth used in this study fit the remaining teeth perfectly with no discernible disruption of the cavosurface margin at the fracture site. Therefore, the thickness of the adhesive and resin cement in the interface was low, which may have contributed to the lower fracture strength found in Group 1. These results are in agreement with the observations of Silva Filho & Esbehard (1982) in which a lower survival rate was obtained when the fragments were bonded only using a sealant without additional mechanical preparation of the fracture site.

Andreasen & others (1993) concluded that materials with relatively high mechanical properties (flexural strength and flexural modulus), such as resins, should be used in conjunction with adhesive systems instead of single application in order to withstand functional stresses.

Several authors advocate placement of a bevel around the fracture site to increase fragment retention and longevity. Its placement alters the enamel prisms orientation, allowing a more effective enamel acid etch (Bagheri & Denehy, 1983). Nevertheless, this recommendation is based on studies involving Class IV resin restorations and not studies concerned with the re-attachment of tooth fragments. Although this technique is widely reported, this study did not evaluate its performance due to the difficulty in positioning the fragment (Franco & others, 1985).

A technique similar to the previous one does not have that shortcoming. Placement of a chamfer on the buccal



surface can be done after the bonding procedure. According to the results of this study, this technique increased the fracture strength of restored teeth from 37% (found in Group 1) to 60.6%. However, this difference was not considered statistically significant. Similar results were obtained by *in vitro* studies (Dean, Avery & Swartz, 1986; Munksgaard & others, 1991) and in a multi-centre clinical study by Andreasen & others (1995).

Groups 3, 4 and 5 had excellent results, with fracture resistance close to sound teeth. In Group 3, good performance cannot be exclusively attributed to enlargement of the adhesion area provided by tooth preparation around the fracture site. According to Andreasen, Daugaard-Jensen & Munksgaard (1991), the greater the extension of material on that surface, the better the force distribution over a large enamel area, contrary to what occurred in Group 2, where the stress was concentrated in the fracture line. Nevertheless, this technique has some inconveniences. Greater exposure of a resin composite to the oral environment, as in Group 2, will diminish the long-term esthetics due to the process of abrasion and discoloration that occurs to composites with time. Polishing at recall appointments may solve this problem. This drawback does not occur when bonding is performed without additional preparation (Group 1), however, this technique should be avoided because of its low fracture strength and consequently the highest vulnerability to future fractures.

Placement of an internal groove may provide higher esthetic durability as well as excellent fracture strength of the restoration (similar to Groups 4 and 5). It is likely that the greater adhesion area and placement of an internal resin bar, which acts as an opponent to the compression load applied on the buccal surface, were responsible for the good results obtained in this group. Also, this technique did not alter the precise fit between the fragment and the remaining tooth. Esthetically, the most favorable situation exists when there is minimal disruption of enamel at the labial fracture site, and the segments fit together with no discernible defects. This facilitates an accurate apposition of the fragment and minimizes an enamel/composite interface. As placement of an internal groove can also provide good fracture strength to restored teeth, it should be preferable in such cases.

Resin composite buildup (Group 5) in regard to fracture strength was equally satisfactory. The high toughness of resin composite is likely to be responsible for absorbing the load used to fracture the tooth before its failure, which may explain the good results obtained in this group. This is the most popular way to restore a fractured tooth when the fragment is not available. Although survival time is similar in both treatments (re-attachment technique and composite build-up) according to Andreasen & others (1995), the authors

questioned whether the greater exposition of composite in the latter case can lead to esthetics problems in the short-term (seven years). In addition to this and low wear resistance, achievement of correct contours and establishment of interproximal contacts are more complex, requiring longer chair time.

According to Busato (1986), the re-attachment technique must be chosen based on the quality of fit between segments. When the segments fit together with no discernible disruptions or defects, techniques that avoid resin composite exposition would be preferable. On the other hand, when enamel structure is lost in the trauma event, it may be more convenient to use an overcontour technique so that the esthetics can be obtained simultaneously with increase in adhesion area.

Sometimes, after bonding the fragment by placing an internal dentinal groove, the fracture line continues to be evident even when the segments approximate well. If this happens, it necessitates a superficial buccal preparation around the fracture line to improve the esthetics of the restoration (Davis, Roth & Levi, 1983; Franco & others, 1985). This represents a drawback to the technique, since only after performing the re-attachment can the evidence of a fracture line be observed.

Another relevant variable concerning re-attachment techniques is the material used. Badami & others (1995) showed that the strength of restored teeth was dependent on the adhesive system applied. They found fracture strength as low as 43% when Scotchbond 2 was used. Farik & others (1998a,b), Farik & Munksgaard (1999) and Farik & others (1999) testing different adhesive systems found values of fracture strength statistically similar to sound teeth. This may be explained by the differences in methodology used in these studies and the current one. However, these studies indicate that there are more factors involved in the retention of the fragment and re-attachment longevity.

Based on the results of this study, the null hypothesis tested was rejected since there were differences among the techniques used to restore fractured teeth.

## CONCLUSIONS

According to the methodology used, it was concluded that the overcontour technique, the placement of an internal dentinal groove and the composite buildup technique provided fracture strength similar to those in sound teeth. Bonding with no additional preparation and placement of a chamfer are not indicated due to the low fracture strength obtained.

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# Mechanical Properties of an Improved Visible Light-Cured Resin-Modified Glass Ionomer Cement

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## Clinical Relevance

The maximum mechanical properties of Fuji II LC Improved were achieved at one week. Therefore, finishing/polishing should be delayed and not conducted immediately after light polymerization. A decrease in mechanical properties was observed at one month and may be attributed to water sorption. Mechanical properties were significantly affected by increased powder:liquid ratio.

## SUMMARY

This study investigates the mechanical properties (hardness, flexural strength and compressive strength) of a new light-cured resin-modified glass ionomer cement (Fuji II LC Improved). Effects of the increased powder:liquid ratio on mechanical properties and the correlation between different mechanical properties were also studied. Mechanical properties of the

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cement at manufacturer's recommended powder:liquid ratio (F), 2% (F2) and 4% (F4) increased powder weight were measured after one day, one week and one month storage in distilled water at 37°C. Hardness testing (KHN; n=5) was done with a digital microhardness tester (load=500g, dwell time=15 seconds). Flexural and compressive strength testing (MPa; n=5) were conducted based on ISO 4049 and BS6039, respectively. Results were analyzed using ANOVA/Scheffe's test ( $p<0.05$ ) and Pearson's correlation ( $p<0.01$ ). The maximum mechanical properties of Fuji II LC Improved were achieved at one week. The hardness, flexural and compressive strength at one week was significantly higher than at one day. A decrease in all mechanical properties was observed at one month. Mechanical properties were significantly affected by increased powder:liquid ratio. After one month storage, significance was as follows: Hardness-F,  $F2>F4$ ; Flexural strength- $F4>F$ ,  $F2$  and  $F>F2$ ; Compressive strength-F,  $F2>F4$  and  $F2>F$ . A significant very strong and negative correlation was observed between flexural and compressive strengths ( $r=-0.97$ ).

## INTRODUCTION

Glass ionomer cements consist of a basic glass and acidic polymer, and set by an acid-base reaction between these components. Their favorable fluoride-releasing and adhesive properties have led to their extensive use as luting, lining/base and restorative materials. As a water-based cement, maintenance of water balance is important for the development of proper translucency and full physical properties (Mount, 1999). Resin-modified glass ionomer cements were introduced to help overcome the problems of moisture sensitivity and low early mechanical strength associated with conventional glass ionomer cements, while maintaining their clinical advantages (Sidhu & Watson, 1995). Setting characteristics were also improved, and finishing/polishing of restorations can be carried out almost immediately after photo-curing with resin-modified cements (Mount, 1993). Resin-modified glass ionomer cements contain the components of conventional glass ionomer cements, that is, basic glass and an acidic polymer as well as additional monomeric ingredients, usually 2-hydroxyethyl methacrylate (HEMA). Set cements consist of an interpenetrating network of poly(HEMA) and polyacrylate salts (Wilson, 1990). They have been shown to absorb water, and the degree of water sorption appears to be influenced by the resinous phases (Small & others, 1998; Kanchanavasita, Anstice & Pearson, 1997; Yap, 1996). The degradation effect caused by the uptake of water competes with the maturation of the cement. Water results in hydrolysis and plasticization of the cement resin-polyacrylate matrix, which may cause deterioration of mechanical properties (Kanchanavasita, Anstice & Pearson, 1998a; Kanchanavasita, Anstice & Pearson, 1998b).

Studies on the mechanical properties of resin-modified glass ionomers as a function of storage time are limited (Kanchanavasita & others, 1998b). Most published literature involved the use of Fuji II LC (GC Corp, Tokyo, Japan), and little work had been done on Fuji II LC Improved (GC Corp, Tokyo, Japan), which is an enhanced version of Fuji II LC. It has been reformulated with finer powder particles to provide better mechanical properties, higher abrasion resistance and smoother restoration surfaces. The average particle size of Fuji II LC Improved is 1.8  $\mu\text{m}$ , while that of Fuji II LC is 4.8  $\mu\text{m}$ . The maximum particle size has been reduced from 25  $\mu\text{m}$  to 5  $\mu\text{m}$ . Despite using a smaller glass particle size, the powder:liquid ratio has been increased from 3.0g/1.0g to 3.2g/1.0g (GC Corporation, 1998).

This study investigated the mechanical properties (hardness, flexural strength and compressive strength) of Fuji II LC Improved as a function of storage time. The effects of increased powder:liquid ratio on mechanical properties of Fuji II LC Improved and the correlation between the different mechanical properties were also studied. The hypotheses to be tested were that storage

time in water and the powder:liquid ratio can affect the mechanical properties of Fuji II LC Improved.

## METHODS AND MATERIALS

Fuji II LC Improved is currently only available in hand-mixed form. Fuji II LC Improved (Powder lot #020781; Liquid lot #030781) of B2 shade was selected for this study. The cement was dispensed at manufacturer's powder:liquid ratio (F; 3.2g/1g), at 2% (F2; 3.26g/1g) and at 4% (F4; 3.32g/1g) increased powder weight. All weight measurements were made with an electronic weighing machine (A&D Co Ltd, Tokyo, Japan) to an accuracy of  $\pm 0.01\text{g}$ . The powder was divided into equal halves; the first half was incorporated into the liquid and mixed for 10 seconds. The second half was then added and mixed for an additional 15 seconds. The mixed cements were injected into different molds using a Centrix syringe (Centrix Inc, Shelton, CT 06484). All light-polymerization was done with a Max light-cure unit (Dentsply Inc, Milford, DE 19963), and the light intensity was checked with a radiometer (Cure Rite; EFOS Inc, Ontario, Canada) before each experimental session to ensure a consistent output of  $420 \pm 2 \text{ mW/cm}^2$ .

### Hardness Testing

The cements were injected into circular Teflon molds with internal dimensions of 5 mm diameter and 1 mm height and confined between two opposing acetate strips (Hawe-Neo Dental, Bioggio, Switzerland). A glass slide (1 mm thick) was then placed on the molds and excess cement extruded by pressure application. The cements were then irradiated from the top through the glass slide and an acetate strip for 20 seconds and the whole assembly was placed in a water bath at  $37 \pm 1^\circ\text{C}$  for 10 minutes. The hardness specimens were subsequently removed from the molds and stored in distilled water at  $37 \pm 1^\circ\text{C}$ . Five specimens were made for each cement (F, F2 and F4), and hardness testing was conducted at one day, one week (seven days) and one month (30 days). At each time interval the specimens were blotted dry and positioned centrally beneath the indenter of a digital microhardness tester (FM7, Future-Tech Corp, Tokyo, Japan) to assess Knoop hardness of the top surface. A 50g load was applied through the indenter with a dwell time of 15 seconds. The Knoop hardness number (KHN) corresponding to each indentation was computed as follows:

$$\text{KHN} = 1.451 \times P/D^2$$

Where

P is the test load (Newtons)

D is the longer diagonal length of an indentation (mm)

### Flexural Strength Testing

Flexural strength testing specimens were fabricated according to ISO 4049 (International Standards Organization, 1992) specifications (25 mm length x 2

mm width x 2 mm height) in customized stainless steel molds. The cements were injected into molds, which were positioned on top of glass slides overlaid with acetate strips. A second acetate strip and glass slide was placed on top of the molds and gentle pressure applied to extrude excess cement from them. The top and bottom surfaces of the specimens were then light polymerized in three overlapping irradiation of 20 seconds starting from the center. The exit window of the light-cure unit was 13 mm. The assemblies were maintained in a water bath at  $37 \pm 1^\circ\text{C}$  for 10 minutes before the mold was removed and the specimens stored in distilled water at  $37 \pm 1^\circ\text{C}$ . For each cement, 15 specimens were made and five tested at each time interval (that is, one day, one week and one month). At each time interval, the specimens were blotted dry and smoothed with wet 400-grit silicon carbide paper. The dimensions of the specimens were then measured using a digital veneer caliper (Mitutoyo Corp, Tokyo, Japan) to an accuracy of  $\pm 0.01$  mm. Measurements for length, width and height were taken in two locations, and the average of the two values was used to calculate the flexural strength. Flexural strength testing was done with an Instron universal testing machine (Model 4502; Instron Corp, Canton, MA 02021) at a crosshead speed of 0.5 mm/minute until the specimen fractured. The maximum load exerted on the specimens was recorded and flexural strength  $\sigma$ , in megapascals (MPa), was calculated using the following equation:

$$\sigma = 3 P L / (2 B H^2)$$

Where

P is the maximum load (Newtons)

L is the distance between the supports (mm)

B is the width of specimen measured prior to testing (mm)

H is the height of the specimen measured prior to testing (mm)

### Compressive Strength Testing

Compressive strength testing was conducted based upon the British Standard specification for dental glass ionomer cements BS 6039: 1981 (British Standards Institution, 1981) with the exception of specimen dimensions and time before mold removal. The molds were made of Teflon and the internal dimensions were 3 mm diameter and 4 mm height. The specimen dimensions (6 mm diameter and 12 mm height) advocated by British Standards were not feasible for this experiment due to the need for light polymerization (cure depth of 2 mm). The cements were injected into the cylindrical molds that were positioned on top of glass slides overlaid with acetate strips. A second acetate strip and glass slide was placed on top of the molds and gentle pressure was applied to extrude excess cement from them. The cements were light polymerized from both ends through

the glass slides and acetate strips. The assemblies were maintained in a water bath at  $37 \pm 1^\circ\text{C}$  for 10 minutes before the mold was removed and the specimens stored in distilled water at  $37 \pm 1^\circ\text{C}$ . Fifteen specimens were made for each cement and five specimens were tested at different time intervals. At each time interval, the specimens were blotted dry and smoothed with wet 400-grit silicon carbide paper. The diameter of specimens was calculated by taking the mean of four readings, two at each end of the specimens at right angles to each other. Compressive strength testing was done using the Instron Universal Testing Machine, with a crosshead speed of 1 mm/minute. The specimens were placed with the flat ends between the platens of the apparatus so that load was applied in the long axis. The maximum load applied to fracture the specimens was recorded and the compressive strength C (MPa) calculated using the following formula:

$$C = 4P/\pi D^2$$

P is the maximum applied load (Newtons)

D is the measured diameter of the specimen (mm)

A significance level of 0.05 was used for all statistical analysis with the exception of correlation. One-way analysis of variance and Scheffe's multiple-range tests were performed to compare the mechanical properties of the cements over time. Inter-material comparison at various time intervals was also done using the above-mentioned statistical tests. Correlation between the different mechanical properties was done using Pearson Correlation at a significance level of 0.01.

## RESULTS

Table 1 and Figures 1-3 reflect the mean hardness, flexural and compressive strengths of the cements as a function of storage time. Tables 2 and 3 show results of the statistical analysis. When mixed according to manufacturer's powder:liquid ratio of 3.2g/1g (F), the cement achieved its maximum hardness, flexural and compressive strength at one week. The cement was the softest and strength (flexural and compressive) the lowest at one day. With the exception of hardness, a similar trend was observed for F2. The lowest hardness of F2 was observed after one-month storage in water. Although the trend of flexural strength development was similar to F and F2, a progressive decrease in compressive strength and hardness from one day to one month was observed with F4.

The results of inter-material comparison were similar at all time intervals for flexural and compressive strength. The flexural strength of F4 was significantly greater than F and F2. In turn, the flexural strength of F was significantly greater than F2. The compressive strengths of F and F2 were significantly greater than F4. In addition, the compressive strength of F2 was greater than F. At one day, F2 and F4 were significantly



Table 1: Mean Hardness, Flexural and Compressive Strength at Various Time Intervals

Material	Time	Flexural Strength (MPa)	Compressive Strength (MPa)	Hardness (KHN)
<b>F</b> 3.2g P : 1g L	1 day	39.09 [0.79]	147.11 [0.74]	25.36 [0.11]
	1 week	42.91 [0.30]	149.37 [0.75]	27.76 [0.11]
	1 month	41.36 [0.90]	148.06 [0.99]	27.26 [0.61]
<b>F2</b> 3.26g P : 1g L	1 day	31.78 [0.48]	160.64 [0.70]	27.82 [0.08]
	1 week	34.77 [0.44]	163.31 [0.40]	28.22 [0.31]
	1 month	34.69 [0.71]	162.69 [1.07]	27.70 [0.43]
<b>F4</b> 3.32g P : 1g L	1 day	57.38 [0.46]	130.56 [0.77]	27.92 [0.13]
	1 week	60.92 [0.73]	127.62 [0.73]	25.74 [0.20]
	1 month	60.14 [1.04]	125.99 [0.92]	25.08 [0.23]

Standard deviations in [ ]; P = powder and L = liquid

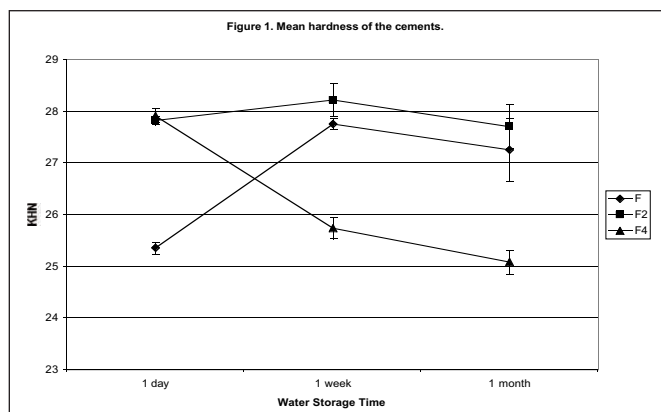


Figure 1. Mean hardness of cements.

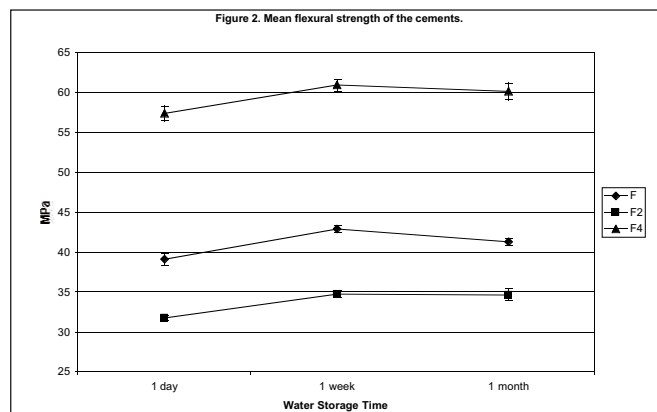


Figure 2. Mean flexural strength of cements.

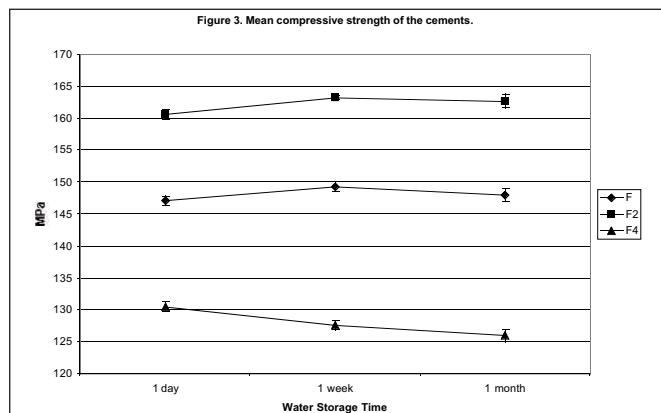


Figure 3. Mean compressive strength of cements.

harder than F. At one week, F and F2 were significantly harder than F4 and F2 was harder than F. At one month, significant differences in hardness were only observed between F/F2 and F4. Changes in powder: liquid ratio resulted in significant changes in strength properties. A 4% increase in powder weight resulted in significantly better flexural strength but lowered compressive strength. A 2% increase in powder weight improved compressive strength significantly but lowered flexural strength. With regard to hardness, no obvious advantage of increased powder:liquid was

observed over time. After one month of storage, a 4% increase in powder weight resulted in significantly lower hardness.

Correlation between flexural strength and compressive strength was significant, very strong and negative, with a correlation coefficient of  $r=-0.97$ . Therefore, a higher flexural strength was coupled with lower compressive strength. Significant correlation was also observed between hardness and flexural/compressive strength. The correlation was, however, weak and correlation coefficients were  $r=-0.53$  and  $0.62$ , respectively.

## DISCUSSION

Little published data are available on the long-term mechanical properties of resin-modified glass ionomer cements (Kanchanasita & others 1998a, Kanchanasita & others, 1998b; Azillah, Anstice & Pearson, 1998; Yap, 1997; Momoi & others, 1995). The majority of studies determined mechanical properties at one day (Li & others, 1996; Attin, Vataschki & Hellwig, 1996), whereas fewer studies determined mechanical properties at longer periods of seven or 30 days (Irie & Nakai, 1995). A one-month period was selected for this study, as long-term studies have indicated that resin-modified cements reached their maximum strengths within one to seven days. In addition, when resin-modified glass ionomers are immersed in

Table 2: Results of Statistical Analysis

Independent Variable Material	Mechanical Properties	Differences
<b>F</b>	<b>Flexural strength</b> <b>Compressive strength</b> <b>Hardness</b>	1 day<1 week & 1 month; and 1 month<1 week 1 day<1 week 1 day<1 week & 1 month
<b>F2</b>	<b>Flexural strength</b> <b>Compressive strength</b> <b>Hardness</b>	1 day<1 week & 1 month 1 day<1 week & 1 month NS
<b>F4</b>	<b>Flexural strength</b> <b>Compressive strength</b> <b>Hardness</b>	1 day<1 week & 1 month 1 week & 1 month<1 day; and 1 month<1 week 1 week & 1 month<1 day; and 1 month<1 week
<b>Time interval</b>		
<b>1 Day</b>	<b>Flexural strength</b> <b>Compressive strength</b> <b>Hardness</b>	F4>F & F2; and F>F2 F & F2>F4; and F2>F F2 & F4>F
<b>1 Week</b>	<b>Flexural strength</b> <b>Compressive strength</b> <b>Hardness</b>	F4>F & F2; and F>F2 F & F2>F4; and F2 > F F & F2>F4; and F2>
<b>1 Month</b>	<b>Flexural strength</b> <b>Compressive strength</b> <b>Hardness</b>	F4>F & F2; and F>F2 F & F2>F4; and F2>F F & F2>F4

> denotes statistical significance and NS denotes no statistical significance (Results of one-way ANOVA and Scheffe's test ( $p<0.05$ )).

Table 3: Pearson's Correlation of the Different Mechanical Properties

Property	Flexural Strength	Compressive Strength	Hardness
<b>Flexural Strength</b>	-	-0.97*	-0.53*
<b>Compressive Strength</b>	-0.97*	-	0.62*
<b>Hardness</b>	-0.53*	0.62*	-

\* indicates that correlation is significant at the 0.01 level.

distilled water, 90% of the equilibrium water uptake occurs within seven days and the cements are equilibrated within two to three weeks (Kanchanasvita & others, 1997).

An increased powder:liquid ratio of 3.26g/1g and 3.32g/1g was chosen, as the manufacturer had already investigated the mechanical properties of Fuji II LC Improved at 3.0g/1g, 3.2g/1g and 3.4g/1g (GC Corporation, 1996). Mechanical properties of Fuji II LC Improved mixed at ratios of 3.2g/1g and 3.4g/1g were better than Fuji II LC Improved and Fuji II LC mixed at 3.0g/1g. Fuji II LC Improved mixed at 3.4g/1g exhibited only a slight improvement in compressive, diametral tensile and flexural strengths over 3.2g/1g cement. The working time and bond strengths to tooth of the 3.4g/1g cement were, however, reduced compared to the 3.2g/1g mixture (GC Corporation, 1996). A powder:liquid ratio of 3.2g/1g was therefore recommended by the manufacturer. Whether the optimal powder:liquid ratio was reached at 3.2g/1g is not known and the powder:liquid ratio range investigated provided adjunct information to the manufacturer's data.

Of primary concern are the effects of water sorption on the different mechanical properties of resin-modified glass ionomers. Kanchanasvita & others (1997) have showed that resin-modified cements can absorb water up to 7% by mass for restoratives and 15% by mass for liner/base cements. Water sorption had been attributed to the hydrophilic nature of poly (HEMA), which

can be likened to the structure of a synthetic hydrogel (Anstice & Nicholson, 1992). It is well known that on exposure to moisture, hydrogels will take up water and swell (Chirila, Constable & Crawford, 1993). The amount of water uptake by resin-modified glass ionomers is dependent on the poly(HEMA) content of the cements (Kanchanasvita & others, 1997; Yap, 1996).

Surface hardness is the resistance to the localized indentations of the surface by an indenter. The load applied results in both plastic and elastic deformation of the surface. As with composite resins, the deformation occurred mostly in the resin-polyacrylate matrix (Watts, McNaughton & Grant, 1986). Fuji II LC Improved mixed at manufacturer's recommended ratio of 3.2g/1g, achieved maximum hardness at one week. This significant increase in hardness from one day to one week can be accounted for by the delayed acid-base reaction resulting from resin modification (Wan, Yap & Hastings, 1999). The acid-base reaction of a conventional glass ionomer was essentially complete after 24 hours, while that of its resin-modified counterpart took 168 hours or one week for completion (Wan & others, 1999). Finishing/polishing of Fuji II LC Improved ideally should be conducted one week after placement, as immediate finishing/polishing could cause irreparable damage to and excessive loss of the soft, maturing polyacrylate salt phase. As no significant difference in hardness was observed between one week and one month's

storage, it can be implied that post-hardening reaction overcame the plasticizing effect of absorbed water. This corresponded well with other studies on the surface hardness of other resin-modified glass ionomers, including Fuji II LC (Kanchanasita & others, 1998a; Uno, Finger & Fritz, 1996).

Although no significant difference in hardness was observed between the different storage times for the cement mixed at 3.26g/1g (that is, F2), a significant difference was observed for the cement mixed at 3.32g/1g (that is, F4). For F4, a significant decrease in hardness was observed from one day to one month. This significant softening cannot be attributed to water sorption alone, as the HEMA content was the lowest among the three ratios investigated. One possible explanation is that the critical powder:liquid ratio was reached and there was insufficient polyacid for reaction with the glass powder. Post-hardening may not be sufficient to compensate for both water sorption and dissolution of unreacted glass powder. After one month of storage in water, both F and F2 were significantly harder than F4. No significant difference in hardness was observed between F and F2. Therefore, there was no obvious advantage of increased powder:liquid ratio over time.

The flexural strength of Fuji II LC Improved mixed at all powder:liquid ratios was highest at one week. The increased flexural strength can be attributed to the maturation and cross-linking of aluminum polyacrylate phase. Flexural strength at one week and one month was significantly higher than at one day. At increased powder:liquid ratios of 3.26g/1g and 3.32g/1g, no significant difference in flexural strength was observed between one week and one month storage. The pattern of changes in flexural strength were similar to that observed by Miyazaki, Moore & Onose (1996). They found that after seven days the flexural strength of resin-modified glass ionomers was maintained or slightly increased for a period up to six months. Flexural strength at one month was, however, significantly lower than flexural strength at one week when Fuji II LC Improved was mixed at the manufacturer's powder:liquid ratio of 3.2g/1g. This can be attributed to the greater water sorption by the bulk of the material over time, which is due to the higher resin content present in F. Water acts as a plasticizer, weakening and decreasing the physical properties of resin-based materials (Watts, 1986).

At all time intervals the flexural strength of F4 was the highest. The higher powder:liquid ratio results in a higher amount of unreacted glass per unit volume of cement. The latter acts as fillers that can retard crack growth and propagation during flexural strength testing, resulting in higher flexural strength. Theoretically, the flexural strength of F2 should be higher than F, as the powder:liquid ratio is higher. The greater softening

of the surface and the plasticization of the cement matrix following water sorption in F could, however, reduce the stress concentration effect of any surface flaws present and improve flexural strength.

Compressive strength of the cement mixed at 3.2g/1g (that is, F) and 3.26g/1g (that is, F2) ratios was greatest at one week. The compressive strength at one week was significantly greater than at one day. Both F and F2 showed a decrease in compressive strength with extended storage in water. For F2, the compressive strength at one month was still significantly higher than at one day, while the compressive strength of F at one day and one month was not significantly different. Results can once again be attributed to the effects of water sorption associated with resin content. The high powder:liquid ratio of F4 was detrimental to compressive strength. The results of statistical analysis were identical to that for hardness. There was a progressive decrease of compressive strength from one day to one month. Compressive strength at one day was significantly greater than that of one week and one month. Compressive strength at one week was significantly greater than one month. Although the unreacted glass particles can act as fillers and retard crack growth/propagation during flexural strength testing, they can also act as foci of stress concentration during compressive strength testing. The hard unreacted glass particles can transmit compressive forces to the surrounding polyacrylate/resin matrix, leading to microcracking and propagation of cracks, which results in lower compressive strength. At all time intervals the compressive strength of F4 was significantly lower than F and F2. The compressive strength of F2 was significantly greater than F. A 3.26g/1g ratio appears to be optimal in terms of compressive strength.

Correlation between flexural and compressive strength was significant and negative. The correlation was very strong, with a correlation coefficient of  $r = -0.97$ . A high flexural strength was associated with a low compressive strength. The possible explanation has been given in the previous paragraph. This correlation does not apply to all resin-modified glass ionomer cements. Further investigations on a wide spectrum of resin-modified glass ionomers and other tooth-colored restoratives are warranted before a definitive conclusion can be made on material behavior. Significant correlations were also observed between hardness and flexural/compressive strength. The correlation was, however, weak and may not have any important implications.

## CONCLUSIONS

Under the conditions of this *in vitro* study:

1. Fuji II LC Improved, at manufacturer's recommended powder:liquid ratio of 3.2g/1g, achieved its maxi-



mum hardness, flexural and compressive strength at one week. A decrease in all mechanical properties was observed with continued storage in water at 37°C.

2. A 4% increase in powder weight (powder:liquid ratio of 3.32g/1g) significantly improved flexural strength but lowered compressive strength.
3. A 2% increase in powder weight (powder:liquid ratio of 3.26g/1g) significantly improved compressive strength but lowered flexural strength.
4. With regards to hardness, no obvious advantage of increased powder:liquid was observed over time.

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# Microleakage of Posterior Packable Resin Composites With and Without Flowable Liners

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## Clinical Relevance

When used as a liners in Class II cavities, flowable composites reduced, but did not eliminate, microleakage of the tested packable and microhybrid resin composites at gingival margins apical to the CEJ.

## SUMMARY

**The use of flowable composites as liners in Class II packable composites has been suggested by some manufacturers. However, the contributions**

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of this technique are unproven. This study evaluated marginal microleakage in Class II packable composite restorations with and without the use of a flowable composite liner. A conventional microhybrid composite was used as a control. Microleakage at occlusal and gingival margins of Class II cavities was evaluated using  $^{45}\text{Ca}$  and autoradiographs.

Fifty non-carious, restoration-free human molar teeth were used. Separate mesio-occlusal and disto-occlusal Class II cavity preparations were made in each tooth. Gingival margins of all cavities were placed 1 mm apical to the cemento-enamel junction (CEJ). Four Packable composites (Alert, Surefil, Pyramid and Solitaire) and one conventional microhybrid composite (Renew) with their respective manufacturer's bonding agents were used to restore the cavities. One side of each tooth was restored with composite alone, while the other side was restored with the composite lined with that manufacturer's flowable liner. The restored teeth were thermally stressed and  $^{45}\text{Ca}$  was used to evaluate microleakage. Two independent evaluators scored leakage based on the autoradiographs.

The results showed flowable composites helped reduce microleakage at gingival margins of Class II restorations ( $p < 0.05$ ). Gingival margins had higher microleakage than occlusal margins ( $p < 0.05$ ). Without flowable liners, three packable

**composites (Alert, Pyramid and Surefil) showed higher leakage ( $p < 0.05$ ) than the microhybrid control. Only Solitaire packable composite without liner showed no significant difference in microleakage to the control ( $p > 0.05$ ). Although the flowable liners help reduce microleakage, Alert and Pyramid packable composites with liners still showed higher leakage than the control ( $p < 0.05$ ). Surefil and Solitaire packable composites with flowable liners showed no significant difference in microleakage ( $p > 0.05$ ) to the control.**

## INTRODUCTION

Resin composites have been used as restorative materials by the dental profession for nearly four decades. With early resins, posterior clinical use on occlusal surfaces revealed serious problems in terms of restoration deterioration including severe leakage, secondary caries, loss of anatomic form and high rate of wear (Phillips & others, 1973). In the past decade, however, resin composites have been used increasingly as posterior restoratives. Wear resistant formulations of resin composites have been developed and adhesive techniques have gone through a tremendous amount of research, providing the clinician with improved materials. Moreover, the increasing demands for esthetic restorations and public concerns related to mercury in dental amalgam have produced increased interest in composite resin as an alternative posterior restorative. Traditional composite resins have never been the ideal amalgam substitute. Resin composites require more time to place compared to amalgam and are more difficult to manipulate. Some of the problems confronting clinicians when placing resin composites in Class II cavities include difficulty in obtaining proximal contact, adhesion to placement instruments and poor adaptation (Belvedere, 1999; Leinfelder, Radz & Nash, 1998). Traditional composites do not offer any resistance to placement forces in their unpolymerized state and tend to be "sticky," resulting in a tendency to pull away from the cavity wall when the placement instrument is withdrawn. Resin composites cannot serve as a potential replacement for amalgam until handling characteristics are improved.

Recently several manufacturers have introduced "condensable" or "packable" composites to the marketplace as alternatives to amalgam (Leinfelder, 1997; Leinfelder & others 1998; Adams, 1999; Leinfelder & Prasad, 1998). Several methods used to achieve packability of new materials include the use of high filler loading with glass fibers (Alert, Jeneric/Pentron, Wallingford, CT 06492), porous filler particles (Solitaire, Heraeus Kulzer, South Bend, IN 46614), irregular filler particles (Surefil, Dentsply/Caulk, Milford, DE 19963), "glass frit" filler particles (Pyramid,

BISCO, Schaumburg, IL 60193) and viscosity modifiers (Prodigy Condensable, Kerr, Orange, CA 92867). Packable composites use amalgam techniques for placement and produce acceptable interproximal contacts (Leinfelder & others, 1998). Because of the high depth of cure and low polymerization shrinkage of packable composites, a bulk-fill technique may be possible (Affleck & others, 1999; Aw & Nicholls, 1999; Kerby & others, 1999; So, Roeder & Powers, 1999). However, concerns related to the ability of these stiffer materials to adequately adapt to internal areas and cavosurface margins have been raised, particularly at the cervical. To offset this problem, using "flowable" composites as liners has been suggested. Flowable composites have low viscosity and may adapt to the cavity better than packable composites (Bayne & others, 1998). However, the efficacy of this technique is unproven.

This *in vitro* study compared marginal microleakage in Class II cavities restored with packable composites alone to packable composites used in conjunction with a flowable liner. The study also compared marginal microleakage in Class II cavities of packable resin composites to one conventional microhybrid composite with and without a flowable liner. Microleakage of occlusal and gingival margins of Class II cavities was evaluated using  $^{45}\text{Ca}$  and autoradiographs.

## METHODS AND MATERIALS

Fifty recently extracted, non-carious, restoration-free human molar teeth were stored in saline until use. Mesio-occlusal and disto-occlusal Class II cavity preparations were made in each tooth using a #269 tungsten carbide bur (Brassler USA, Savannah, GA 31419) in a high-speed handpiece with water spray. These slot preparations were separate with no occlusal connection. The buccolingual width was 3 mm and the gingival margins of all cavities were placed 1 mm apical to the cemento-enamel junction (CEJ). Buccal and lingual walls of the preparations were approximately parallel and connected to the gingival wall with rounded line angles. All margins were smoothed with an enamel hatchet. The materials used in the study, including bonding agents, packable resin composites, microhybrid resin composite and flowable liners, are listed in Table 1.

Each tooth was restored with the same brand of packable or microhybrid resin composite. One side was restored with packable or microhybrid composite alone, while the other side was restored with packable or microhybrid resin composites lined with flowable liner. The teeth were divided into five groups of 10 teeth as follows:

**Group 1:** One side was restored with Alert. The other side was lined with Flow It then restored with Alert. The bonding agent was Bond 1.



Table 1: *Products Used in This Study*

Group	Product		Batch Number	Manufacturer
1	Packable Composite	Alert	21538	Jeneric/Pentron
	Flowable Liners	Flow-it	18939	
	Bonding Agent	Bond-1	19060	
2	Packable Composite	Surefil	990420	Dentsply/Caulk
	Flowable Liner	Dyract Flow	990805	
	Bonding Agent	Prime&Bond NT	990807	
3	Packable Composites	Pyramid	9900004978	BISCO
	Flowable Liners	AEIite Flo LV	9900004983	
	Bonding Agent	One-step	9900004782	
4	Packable Composite	Solitaire	VP150499/2	Heraeus Kulzer
	Flowable Liner	Flow-line	010021	
	Bonding Agent	Gluma comfort bond Plus	VP010499Mo	
5	Microhybrid Composite	Renew	9900000172	BISCO
	Flowable Liners	AEIite Flo LV	9900004983	
	Bonding Agent	One-step	9900004782	

**Group 2:** One side was restored with Surefil. The other side was lined with Dyract Flow then restored with Surefil. The bonding agent was Prime&Bond NT.

**Group 3:** One side was restored with Pyramid. The other side was lined with Aelite Flo LV then restored with Pyramid. The bonding agent was One-Step.

**Group 4:** One side was restored with Solitaire. The other side was lined with Flow-line then restored with Solitaire. The bonding agent was Gluma Comfort Bond Plus.

**Group 5:** One side was restored with Renew. The other side was lined with Aelite Flo LV then restored with Renew. The bonding agent was One-Step.

Teeth were treated and bonding agents applied following manufacturers' instructions. A Tofflemire matrix retainer and soft metal band were then placed on the tooth. The matrix was tightened and held by finger pressure against the gingival margin of the cavity so the preparations could not be overfilled at the gingival margin. On one side of the tooth, composite was placed using an incremental technique. Each 1.5 mm layer (approximate) was polymerized for 60 seconds using a VIP light-curing unit (BISCO, USA) with a light intensity of 500 mW/cm<sup>2</sup>. The other side was lined with a thin layer of flowable liner on the pulpal and axial walls with approximately a 1 mm thickness at the gingival floor of the cavity preparation. Composite was then placed using the same horizontal incremental technique. The matrix was removed after both restorations were completed. A #15 surgical blade was used to remove any excess material, especially at the gingival margin. A series of Sof-Lex disks (3M, USA) were used to finish margins that would be accessible clinically. Gingival margins were not disked.

All restored teeth were stored in distilled water at 37°C for one week. They were then thermally stressed between an 8°C and a 48°C water bath for 2,500 cycles with a 30-second dwell time and a 10-second transfer time. After that, the teeth were again stored in distilled water at 37°C for an additional five days.

<sup>45</sup>Ca, as the tracer with autoradiographs, was used to test microleakage. Prior to the

microleakage test, the apices of the specimens were sealed with resin. The unprepared coronal and root surfaces were painted with one coat of dark fingernail polish to within 1 mm of the restoration margin to prevent <sup>45</sup>Ca penetration anywhere other than through the restorations' margins. Tin foil was wrapped over the teeth with the restoration exposed. The specimens were immersed in an aqueous solution (pH 5.5) of <sup>45</sup>CaCl<sub>2</sub> for two hours at room temperature. After removal from <sup>45</sup>CaCl<sub>2</sub> and rinsing under water for one hour, each tooth was sectioned mesiodistally across the center of the restoration in a sectioning machine using copious amounts of water. Pulpal tissue was removed, and the specimens were rinsed, scrubbed and allowed to air dry.

Each specimen was placed on an ultra-speed intra-oral radiographic film supported by a plastic plate with the cut side down. The specimens were coded with numbers to help with identification, then secured by a rubber band around the plastic plate. Specimens were individually wrapped with tin foil and placed in a light-proof container for 24 hours. Exposed films were developed in an automatic film processor. Radiographs from both halves of each sectioned tooth were examined under a magnifier at X2 by two independent evaluators. Each evaluator scored the microleakage of the two halves of each specimen three times, with a time lapse of at least 24 hours between evaluations. The maximum score from each examiner for each half was selected, then the highest of the four scores (two halves x two examiners) was selected to record the microleakage score. Occlusal and gingival margins were scored separately. The following scoring criteria was used to rate the degree of marginal microleakage:

gingival margin

1 = No evidence of isotope penetration.

2 = Superficial penetration of isotope at the margin but less than 1/3 of the gingival width.

3 = Penetration along the margin beyond 1/3 of the gingival width, up to the axial wall.

4 = Penetration along the axial wall.

#### occlusal margin

1 = No evidence of isotope penetration.

2 = Superficial penetration of isotope at the margin but less than 1/3 of the width of mesial/distal wall.

3 = Penetration along the margin beyond 1/3 of the width of mesial/distal wall, up to the pulpal wall.

4 = Penetration along the pulpal wall.

The groups were compared for differences in microleakage ratings. Agreement between the examiners was assessed using two-way contingency tables and Kappa and Weighted Kappa Statistics. Kappa measures exact agreement and Weighted Kappa accounts for the ordered nature of the ratings. Kappa interpretations were made using the description by Landis & Koch (1977). The effects of presence of a flowable composite, location (gingival or occlusal) and materials on micro-leakage rating were analyzed using analysis of variance (ANOVA). The ANOVA model contained a random effect for specimens to correlate multiple measurements from the same specimen. Fixed effects for the three main effects and all interactions were also included in the model. Pairwise comparisons between treatment combinations were made using Tukey's method to control the overall confidence level at 95%. Although ANOVA is typically used for continuous data, ANOVA was used for this data because the ratings are ordered and the complicated nature of the study design required a more complex analysis than traditional categorical analysis methods can handle.

## RESULTS

Agreement between the examiners was moderate overall (Kappa=0.50, Weighted Kappa=0.65) and for

cervical (Kappa=0.54, Weighted Kappa=0.65) but only fair for occlusal (Kappa=0.33, Weighted Kappa=0.42). Disagreements reflected higher ratings from one examiner to the other.

The results in Table 2 show the number of teeth in each microleakage-rating category. They show that cervical margins had significantly higher leakage than occlusal margins ( $p=0.0001$ ) for all materials. For the occlusal, the leakage ratings for flowable and without flowable sides showed no significant difference ( $p=0.83$ ) for all materials. The cervical margins of flowable sides showed significantly lower leakage than without flowable ( $p=0.0001$ ) for all materials.

Renew had significantly lower leakage ratings than Alert ( $p=0.0001$ ) and Pyramid ( $p=0.0006$ ) both with and without flowable liner. Renew also had significantly less leakage than Surefil when used without flowable liners ( $p=0.0214$ ). However, when flowable liner was used, microleakage ratings of Renew and Surefil were not significantly different ( $p=0.32$ ). Renew and Solitaire leakage ratings were not significantly different with or without flowable liners ( $p=0.16$ ).

Alert with and without flowable liner showed significantly higher leakage than Solitaire ( $p=0.0420$ ) and Surefil ( $p=0.0003$ ) but was not different from Pyramid ( $p=0.88$ ). Pyramid with and without flowable liner was not significantly different from Solitaire ( $p=0.33$ ).

Leakage of Surefil without flowable liner was not significantly different from Pyramid ( $p=1.00$ ) and Solitaire ( $p=0.99$ ). However, when used with flowable liners, Surefil had significantly lower microleakage ratings than Pyramid ( $p=0.0004$ ) and Solitaire ( $p=0.0478$ ).

## DISCUSSION

This *in vitro* study examined microleakage of four packable composites and a microhybrid composite control with and without the use of flowable resin liners. The dentin bonding systems and flowable liners used

Table 2: Distribution of the Microleakage Ratings of Class II Resin Composite Restoration With or Without Flowable Liners

Materials	Flowable	Occlusal				Gingival			
		1	2	3	4	1	2	3	4
Alert	No	0	5	4	1	0	0	0	10
	Yes	1	5	4	0	0	0	7	3
Surefil	No	0	9	1	0	0	0	1	9
	Yes	3	6	1	0	0	9	1	0
Pyramid	No	0	5	5	0	0	0	4	6
	Yes	1	3	6	0	0	1	7	2
Solitaire	No	0	7	3	0	0	1	5	4
	Yes	1	7	2	0	0	4	2	4
Renew	No	6	2	2	0	0	1	7	2
	Yes	2	7	1	0	0	2	7	1

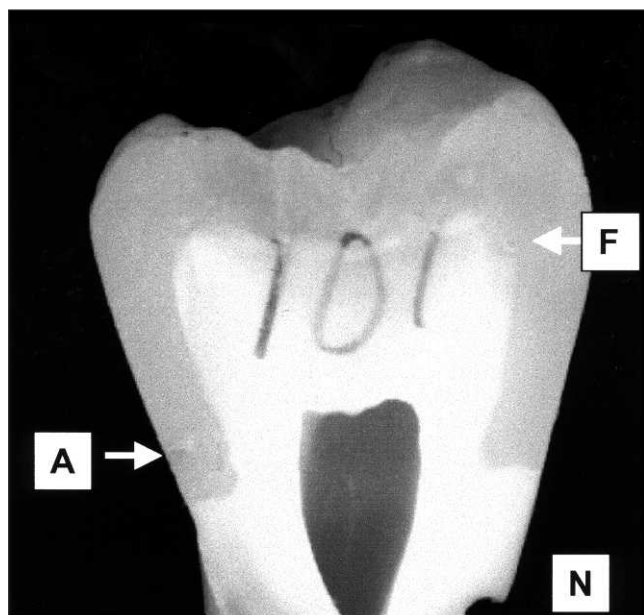


Figure 1.

with each composite were the materials supplied by the respective manufacturers. Class II cavities were prepared with enamel occlusal margins and cementum/dentin gingival margins. Cervical margins showed significantly higher leakage than occlusal margins in all materials in this study. This was expected as bond strength to enamel is usually higher than bond strength to dentin because dentin is a less favorable bonding substrate (Nakabayashi, Ashizawa & Nakamura, 1992) and enamel margins of composite restorations are reported as having less leakage than the cementum/dentin margins. (Nakabayashi & Pashley, 1998).

Some manufacturers suggest flowable composites for use as liners in areas of difficult access or flow, such as irregular internal surface and proximal boxes of Class II preparations. The assumptions are that these less viscous materials flow easily into, adapt to and fill the tooth surface, resulting in less leakage and post-operative sensitivity (Bayne & others, 1998; Leinfelder & Prasad, 1998; *The Dental Advisor*, 1999). In this study, flowable liners helped reduce microleakage in all composite restorations at cervical margins. While flowable liners may provide better adaptation, they may also act as a flexible intermediate layer, which helps relieve stresses during polymerization shrinkage of the restorative resin (Kemp-Scholte & Davidson, 1990; Rooklidge, Boyer & Bouschlicher, 1999). However, flowable composites are reported to shrink more than traditional composites because they have less filler loading (Tolidis & Setcos, 1999). Perhaps the relatively thin layer minimizes this effect.

Packable composites have mechanical and physical properties comparable to hybrid composite (Kelsey,

Latta & Barkmeier, 1999; MacGregor, Cobb & Vargas, 1999; Ruddell & others, 1999; Susuki, 1999). The wear rate of flowable composites was higher than that of packable composites; therefore, flowable composites should be used only at contact-free areas (Bayne & others, 1998; Dang & Sarrett, 1999). In this study, the application of approximately 1-mm thickness of flowable liner at the gingival wall was considered acceptable clinically since this is a contact-free area. The liners also did not extend to the occlusal cavosurface and results showed no significant difference in leakage at occlusal margins for any material or technique.

Compared to conventional hybrid composites, packable composites need greater forces to “condense” the material into the cavity preparation. Although the packable composites used in this study did not stick to dental instruments, they were difficult to adapt to the preparations. Alert, Surefil and Pyramid were stiffer than Solitaire and Renew. The stiffest material, Alert, showed the greatest microleakage at gingival margins. Alert, Surefil and Pyramid without flowable liners showed higher microleakage than Solitaire and the control, Renew, at gingival margins. Solitaire showed comparable microleakage to Renew with and without flowable liners. For the materials used in this study, stiffness appeared to relate to microleakage of the restorations.

Flowable liners helped reduce microleakage at gingival margins for all materials. However, the microleakage rates of Alert and Pyramid with flowable liner were significantly higher than the control hybrid composites (Renew) with flowable liner. Surefil with flowable liner showed lower microleakage compared to Alert, Solitaire and Pyramid. With flowable used, Solitaire, Surefil and Renew had no significant difference in microleakage.

Current flowable materials can easily be syringed into the cavity but are sometimes difficult to manipulate because of their stickiness. Air is sometimes trapped in the restorations while removing the syringe tip from the cavity. Porosities were also observed in packable composite restorations (Figure 1). Operators need to use these materials with caution.

Microleakage might relate to compatibility of flowable composites, bonding systems and packable composites. Future research needs to be directed towards this issue. Clinical research needed to support the use of these relatively new materials.

## CONCLUSIONS

In the current experiment, evidence of microleakage in Class II restorations was detected in both occlusal and gingival margins. Gingival margins demonstrated higher microleakage than occlusal margins. Flowable composite or compomer liners reduced microleakage in



Class II packable and microhybrid resin composite restorations at gingival margins. Without flowable liners, packable composites (Alert, Pyramid and Surefil) showed higher leakage than a microhybrid resin composite (Renew). Only Solitaire packable composite did not show a significant difference in microleakage compared to Renew. Although the flowable liners reduced microleakage, Alert and Pyramid packable composites with liners had higher leakage than the microhybrid resin, Renew. Surefil and Solitaire packable composites with flowable liners showed no significant difference in microleakage compared to the microhybrid composite (Renew). The results of this research are limited to the composite/flowable combinations studied. Since each resin was tested with only a single flowable liner, caution should be used in generalizing these conclusions to other products.

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# Evaluation of Cutting Patterns Produced with Air-Abrasion Systems Using Different Tip Designs

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## Clinical Relevance

The variety of cutting patterns produced with the different air abrasion tip designs indicated that clinicians must select specific tip parameters to achieve expected outcomes.

## SUMMARY

This study assessed cavity preparations produced with different air abrasion tip parameters. Twelve test groups of extracted teeth were prepared to evaluate the parameters of 80° or 45° nozzle angles and 0.38 or 0.48 mm inner tip diameters. All other factors were held constant. A device was made to hold the specimen and air abrasion handpiece that standardized the distance and position relative to the tooth and time of application. The cavities were evaluated by assessing the rounding of the cavosurface mar-

gins and cavity floor. Measurements of cavosurface angles and the angle of concavity were made at the deepest portion of the abraded surface using scanning electron micrographs. The cavosurface angles were compared using paired *t*-test, and the effects of the tip design parameters were analyzed by ANOVA and Duncan's Multiple Range test. From the cavity patterns found in this study, the authors suggest that 80° angle tips are more appropriate than 45° angle tips for making narrow, deep cuts for preventive resin restorations. Conversely, when shallow preparations are needed, as in the case of Class V cavity preparations, cutting patterns of 45° angle tips are more suitable.

## INTRODUCTION

In the 1950s, air abrasion systems met with limited success, in part because it was difficult to create GV Black preparations with this technology (Berry & Ward, 1995; Laurell & Hess, 1995). Today, adhesive dentistry has altered concepts about cavity preparations (Feilzer, DeGee & Davidson, 1987; Mount & Hume, 1998; Yoshikawa & others, 1999). Rounded, minimal-sized cavity preparations have replaced conventional cavity preparations of converging walls with precision line

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angles and flat cavity floors. Thus, there is renewed interest in air-abrasion technology because of the potential to produce cavity contours compatible with the needs of adhesive dentistry (Laurell & Hess, 1995; Roeder & others, 1995). Rounding of cavo-surface margins produced by air abrasion techniques has been shown to improve the longevity of resin composite restorations (Nordbø, Leirskar & von der Fehr, 1998). Christensen (1998) and Ferdianakis & White (1999) reported that adaptation of resin composites to the cavity wall was better for samples prepared with air abrasion techniques than to cavities prepared with #330 burs. Since these findings may lead to increased use of air abrasion systems, it is important to understand the cutting patterns produced by the many accessories available with air abrasion units.

There has been speculation that cutting with air abrasion systems results in minimal loss of tooth structure when preparing rounded cavities. However, Boston, Alperstein & Boberick, (1997) found no significant differences in the amount of tooth structure removed at margins with air abrasion systems versus conventional burs. Thus, the advantage for the air abrasion system would simply be the ease of making rounded preparations.

As with any new technology, a complete understanding of the parameters that affect the quality of the methodology is needed so that clinicians using these systems can reproduce the desired results. This study assessed the differences in cavity preparations produced with air abrasion systems using four combinations of available tip parameters.

METHODS AND MATERIALS

Erupted molars were collected from patients 18 to 25 years of age. These extracted teeth were stored in a solution of saline and 1% thymol for one month prior to testing. They were sectioned longitudinally and embedded in resin, exposing a flat surface of the mesial enamel, distal cementum or internal dentin. The 72 specimens were randomly divided among the test groups, representing six specimens from each of three tooth substrates for each of the four experimental conditions.

The test parameters used with the air abrasion system (PrepStar, Danville Eng, San Ramon, CA, USA 94583) consisted of the angle of the handpiece tip, either 80° or 45°, and the inner diameter of the tip, either 0.38 or 0.48 mm. Table 1 gives the experimental conditions. A number of parameters were held constant, including the air pressure (80 psi) used to operate the air abrasion system and the size and type of abrasive power (27 µm Al<sub>2</sub>O<sub>3</sub>). Operator variables were eliminated because the

Table 1: <i>Description of Test Conditions for Group</i>												
Tooth Surface	Mesial Enamel				Internal Dentin				Distal Cementum			
Tip angle	80°		45°		80°		45°		80°		45°	
Tip diameter (mm)	0.38	0.48	0.38	0.48	0.38	0.48	0.38	0.48	0.38	0.48	0.38	0.48
Group	1	2	3	4	5	6	7	8	9	10	11	12

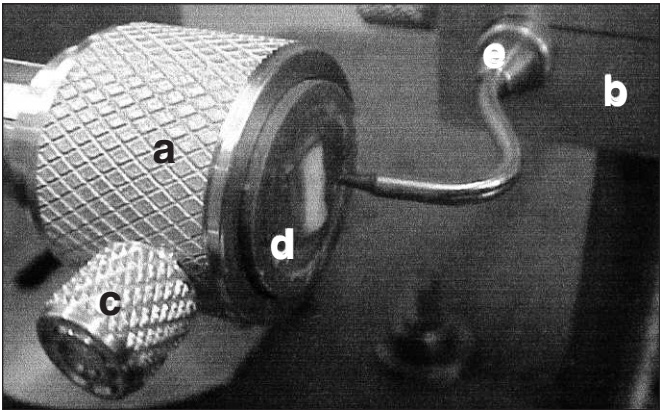


Figure 1. Apparatus used to standardize the application of the air abrasion system: a: specimen holder, b: handpiece holder, c: locking screw, d: specimen e: air abrasion nozzle.

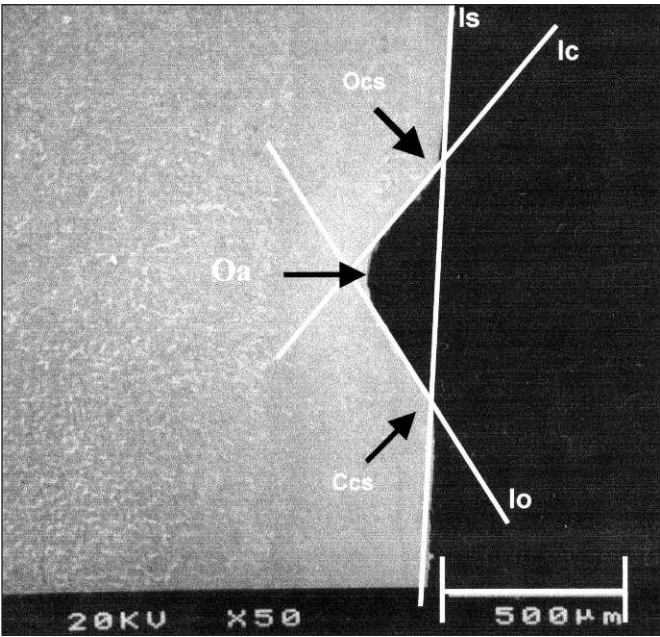


Figure 2. Micrograph of the sectioned enamel surface showing cross section of cut made with the air abrasion system and diagram of the traced lines and the measurements made using the digitizing program. ls - approximately the original surface of the uncut enamel lo - internal wall of the cut enamel from the occlusal side lc - internal wall of the cut enamel from the cervical side Ocs - occlusal surface angle Ccs - cervical surface angle Oa - concavity angle The magnification marker on the micrograph was used in calculating the angles.



Table 2: Means and Standard Deviations of Measured Angles Made with Different Air Abrasion Handpiece Tip Parameters

Tooth Surface	Tip Angle (degree)	Tip Diameter	Occlusal Cavo-surface Angle (Ocs)		Cervical Cavo-surface Angle (Ccs)		Concavity Angle (Oa)	
			Mean (µm)	SD	Mean (µm)	SD	Mean (µm)	SD
Enamel	80	0.38	144.9	6.6	148.6	6.7	113.5	10.1
		0.48	150.1	8.1	144.7	7.9	114.8	12.8
	45	0.38	175.9	2.0	175.7	2.0	171.6	4.0
		0.48	173.9	3.5	174.9	1.7	168.8	4.7
Dentin	80	0.38	145.2	8.2	146.3	4.4	111.6	7.9
		0.48	150.2	6.6	149.2	5.9	119.3	11.1
	45	0.38	170.3	2.2	168.8	2.7	159.1	3.1
		0.48	175.5	1.2	175.2	1.3	170.7	2.4
Cementum	80	0.38	149.1	4.5	151.6	6.0	120.6	6.8
		0.48	151.4	7.5	152.0	4.9	123.4	10.6
	45	0.38	154.6	17.1	161.0	7.5	135.6	17.4
		0.48	166.7	2.9	169.4	1.5	156.1	3.6

Table 3: Summary of the Statistical Analysis: Comparison of Cavo-surface Angles on Tooth Surfaces

Tooth Surface	Occlusal Cavo-surface Angles (Ocs)				Cervical Cavo-surface Angle (Ccs)			
	80°		45°		80°		45°	
	0.38 mm	0.48 mm	0.38 mm	0.48 mm	0.38 mm	0.48 mm	0.38 mm	0.48 mm
Enamel vs Dentin	ns	ns	ns	ns	ns	ns	ns	s
Enamel vs Cementum	ns	ns	ns	s	ns	ns	s	s
Dentin vs Cementum	ns	ns	ns	s	ns	ns	ns	s

s – statistically significant  
ns – not statistically significant

Table 4: Summary of Statistical Analysis: Effect of Tip Angle and Orifice Size on Cavo-surface Angle

Tooth Surface	Occlusal Cavo-surface Angle (Ocs)		Cervical Cavo-surface Angles (Ccs)	
	80°	45°	80°	45°
	0.38 vs 0.48 mm	0.38 vs 0.48 mm	0.38 vs 0.48 mm	0.38 vs 0.48 mm
Enamel	ns	ns	ns	ns
Dentin	ns	ns	ns	ns
Cementum	ns	s	ns	s

s – statistically significant  
ns – not statistically significant

Table 5: Summary of the Statistical Analysis: Comparison of Concavity Angle on Tooth Surfaces

Tooth Surface	Concavity Angle (Oa)			
	80°		45°	
	0.38 mm	0.48 mm	0.38 mm	0.48 mm
Enamel vs Dentin	ns	ns	s	ns
Enamel vs Cementum	ns	ns	s	s
Dentin vs Cementum	ns	ns	s	s

s – statistically significant  
ns – not statistically significant

apparatus used to hold the specimens also held the handpiece and established a constant rate of travel and a constant distance (2 mm) between the air abrasion tip and the tooth surface (Figure 1). Cutting time was standardized at 15 seconds for all specimens. Rather than holding the handpiece stationary, it was moved across the surface during the 15 second application. Since the size of the teeth was approximately the same, the apparatus moved the handpiece tip across the tooth a total of 20 times. Each specimen was exposed to only one 15-second application of the air abrasion system.

To assess cavity preparation patterns produced by the different air abrasion tips, cross-sectional views of the cut surfaces were

Table 6: Summary of Statistical Analysis: Effect of Tip Angle and Orifice Size on Cavo-surface Angle

Tooth Surface	Concavity Angle (Oa)	
	80°	45°
	0.38 vs 0.48 mm	0.38 vs 0.48 mm
Enamel	ns	ns
Dentin	ns	ns
Cementum	ns	s

s – statistically significant  
ns – not statistically significant

examined using SEM micrographs (50x). The degree of rounding between the plane of the original surface (ls) and the internal walls (lo and lc) of the cut were measured (Figure 2). These angles were labeled occlusal cavosurface angles (Ocs) and cervical cavosurface angles (Ccs) to distinguish between the upper and lower portion of the cuts. In addition, the angles of concavity at the base of the cuts (Oa) were measured (Figure 2). All measurements were made using a modified cephalometric analysis. The angles were calculated using DF Plus 6.5 1995 (Dentofacial Software Inc, Toronto, Canada, M5W 1G5).

The occlusal (Ocs) and cervical (Ccs) cavosurface angles of each cut were compared by a paired *t*-test. An ANOVA and Duncan's multiple range test were used to compare the effects of the different tip parameters on the shapes and rounding of cavities produced in the different tooth surfaces.

## RESULTS

Table 2 reports the means and standard deviations of measured cavosurface angles and the angles of concavity at the floor of the cut for each experimental condition. The paired *t*-test analysis indicated no difference in the occlusal (Ocs) versus cervical (Ccs) cavosurface angles for any test conditions, except when the 45° angle tip with large inner

diameter was used to cut cementum.

Tables 3 and 4 show results of statistical analysis comparing the effect of test parameters on cavo-surface angles for the different tooth substrates. The 80° angle tip produced significantly smaller cavo-surface angles than were produced with the 45° tip, regardless of the inner diameter size used. The cavo-surface angles (175° for enamel versus 155° for cementum) produced with the 45° tip varied depending upon the hardness of the substrate surface. These differences were significant when the small inner diameter tip was used.

Results of the statistical analysis of the concavity angle (Oa) measurements are shown in Tables 5 and 6. The concavity angles produced with the 80° tip were significantly narrower than the angles produced with the 45° tip (Figure 3). No differences in enamel were detected in the concavity angles produced with the 80° tip, dentin or cementum regardless of the inner diameter used. With the 45° tip, the effect of surface substrate was apparent in significant differences of concavity angles produced with both inner diameter tips. Overall, the 80° degree tip produced cuts with consistent shapes in enamel, dentin and cementum with either inner diameter size (Figure 4).

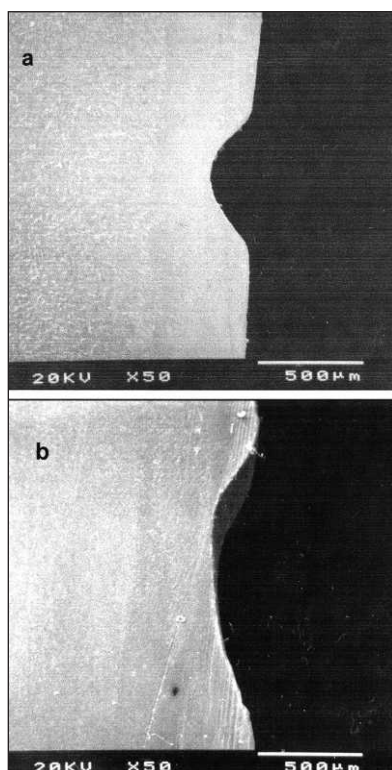


Figure 3. Micrographs showing cross-sections of cuts in cementum.

- a—using the 80° nozzle with 0.48 inner diameter.
- b—using the 45° nozzle with 0.48 inner diameter.

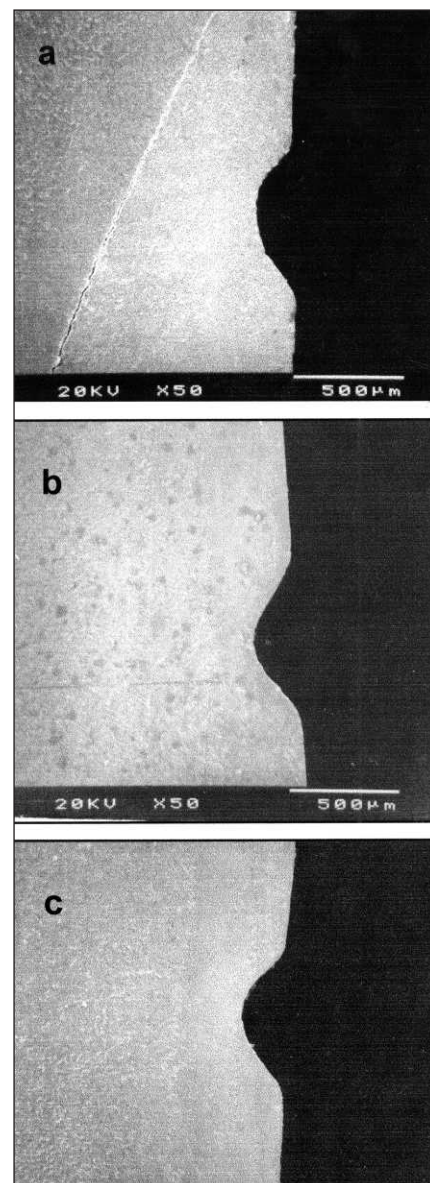


Figure 4. Micrographs showing cross-sections of cuts in a) enamel, b) dentin, and c) cementum. Note the consistent shape and dimensions produced with the 80° nozzle and the 0.38 inner diameter tip irrespective of the tooth substrate.

## DISCUSSION

The patterns of cavity preparations produced with different tip designs were significantly different. The degree of bend in the tip used in the air abrasion handpiece had the greatest effect on cavosurface angles. Both tips used in this study created cuts that were significantly more rounded than cavities cut in a conventional manner using a bur. These results are similar to those found by Laurell & Hess (1995), who reported the cavosurface angles, lateral walls and floor of the cavity preparation were rounded because the cutting efficiency of the spray diminished as it was dispersed from the tip.

The 80° tip consistently produced smaller angles, resulting in narrower cuts than the 45° tips (Figure 3). Except in cementum, this difference in cavosurface angles produced with the 80° tip was at least 10% smaller than those produced with the 45° tip. Thus, the authors could make the assumption that less tooth structure is removed at cavosurface margins when cavosurface angles are smaller. This interpretation is supported by findings reported by Boston & others (1997) that air abrasion systems were not responsible for more reduction in tooth structure at the margins than conventional burs.

Myers (1954) found that when the tip was held 2 mm from the tooth, a U-shaped cut is produced in tooth surface. In this study, all of cuts produced with the 80° nozzle were U-shaped (Figure 4). Cuts made with these parameters were consistent in shape and dimensions in all the different tooth surfaces. To consistently cut rounded surfaces regardless of tooth substrate is a significant benefit of using the air abrasion system since conventional burs do not consistently cut all dental substrates equally.

It is interesting to note that the shape of cuts at the cavosurface margin produced with the 45° tip were less symmetrical than with the 80° tip. However, operator variables were eliminated in this study, so that the effects of the tip parameters were assessed. Thus, in clinical practice, the difficulty of maintaining a constant 2 mm distance may reduce the consistency obtained in cut patterns. Patterns may be less reproducible when an operator holds the handpiece.

These results provided further evidence that some tip designs can minimize tooth removal. When the 80° tip was used, resulting concavity angles were 70% of those formed with 45° tips for enamel and dentin. Thus, these results indicate that the 80° tip will produce a sufficiently narrow cut that is appropriate for cleaning fissures.

From the patterns of angles produced with the 45° tip, the authors recommend this component of the system for areas that require disperse cuts without depth, such as in the case of cervical erosion. The 45° tips lend themselves to achieving access in these areas. However,

the operator must be aware that these tips are not recommended for cleaning fissures where a narrow cut is more appropriate. Thus, selection of the tip for each clinical application must be made on specific evidence rather than assumptions made by the clinical operator.

## CONCLUSIONS

Cavity patterns produced in this study indicated that the air abrasion method of preparing tooth structure is compatible with the requirements of adhesive dentistry. With properly selected tip design, a saucer-shaped cavity can be produced in all three types of tooth substrate: cementum, dentin and enamel. When using air abrasion systems, it is important for the clinician to be knowledgeable about variations in cavity preparations that result from different tip parameters. For precision cutting, as might be required for a preventive resin restoration, the 80° tip is more appropriate than the 45° tip. When shallow preparations are needed, as in the case of cervical erosion, the cutting patterns of the 45° tip are more appropriate.

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# The Effect of Double Adhesive Application on the Shear Bond Strength to Dentin of Compomers Using Three One-Bottle Adhesive Systems

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B Rhodes • BK Moore

## Clinical Relevance

One of three one-bottle adhesives tested had more reliable performance when the number of manufacturer recommended applications was doubled. Doubling the number of applications had no effect on the other one-bottle adhesives tested.

## SUMMARY

The development of one-bottle dentin adhesive systems resulted in much optimism about providing simplified predictable esthetic dentistry. However, laboratory testing of these systems continues to provide significant variations between facilities. A potential effect of the number of applications was noted in this author's laboratory. This study evaluated the effect of doubling the manufacturer's recommended number of applications on shear bond strength to dentin.

Ninety human molars were divided into groups of 15. The occlusal surfaces were finished to 600

grit SiC to provide a flat dentin bonding surface. Prime & Bond NT—Dyract, Optibond Solo—Elan and One Step—Dyract were evaluated. Each material was tested using: (1) the recommended number of adhesive applications and (2) twice the number of applications recommended. All adhesive applications were accomplished before light curing the adhesive. The specimens were thermocycled after one week of storage and tested in shear after two weeks.

Specimens were also fabricated after adding Rhodamine D to the adhesive to allow for visualization using confocal microscopy. These teeth were sectioned and viewed 24 hours after fabrication.

A *t*-test was used to compare differences within product groups. The results showed a significant effect ( $p < 0.001$ ) when a double application of Prime & Bond NT was used. No difference was seen with Optibond Solo or One Step. All specimens appeared to have a uniform, glossy appearance of adhesive during fabrication. Therefore, the appearance of the adhesive after application may not be a reliable predictor of acceptable bonding. Confocal microscopy showed that single application Prime & Bond NT specimens did not

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**exhibit a uniform thickness of adhesive across the entire interface. Tubule penetration and hybridization was apparent for all specimens.**

INTRODUCTION

The use of shear bond strength testing is widely used in evaluating dental adhesives, in spite of there being little or no correlation to clinical performance (Platt & others, 1996). The test may have greater impact in investigating handling characteristics of these materials (Jacobsen & Soderholm, 1998; Frankenberger & others, 2000).

One bottle adhesive systems have become popular materials, which may be related to a perceived ease of use because of fewer components. However, a wide range of dentin shear bond strengths has been reported for these materials (Perdigão, Baratieri & Lopes, 1999; Swift & Bayne, 1997; Price & Hall, 1999). This may indicate technical concerns which are not well identified.

Swift & others (1997) evaluated the effect of multiple applications of three one-bottle adhesives. They used multiple applications of Prime & Bond (Dentsply Caulk, Milford, DE 19963), One-Step (BISCO, Inc, Schaumburg, IL 60193) and Tenure Quik (Den-Mat Corp, Santa Maria, CA 93456). They reported no significant benefit from multiple applications of the adhesive, with a trend toward decreased bond strengths.

The manufacturers of Prime & Bond NT (Dentsply Caulk, Milford, DE 19963) and One-Step state that if the first application of the adhesive does not leave a uniform glossy appearance, a second application is indicated. The manufacturer of Optibond Solo (Kerr USA, Orange, CA 92867) recommends the use of two coats of adhesive when the restoration retention area lies mainly in dentin. Experience has shown that it is difficult to determine the presence of a uniform or smooth glossy appearance.

This study determined whether routinely applying a double layer of one-bottle adhesives would improve dentin shear bond strength.

METHODS AND MATERIALS

Shear Bond Strength

The materials tested were Prime & Bond NT—Dyract (NT), Optibond Solo—Élan (OP) and One-Step—Dyract (ON). The components of these systems are provided in Table 1. Human molars were treated in formalin for less than two weeks and stored in deionized water until use. Six groups of 15 teeth were prepared. The occlusal surfaces were flattened using SiC paper to provide a flat dentin surface for bonding. The teeth were mounted in acrylic tubing using autopolymerizing poly(methyl methacrylate) resin. Just prior to bonding, each surface was finished with wet 240, 320, 400 and 600 grit SiC paper, rinsed and verified to be free of enamel with a stereomicroscope (20x). Placement of the compomer was accomplished with a compression jig which held a vinyl tube approximately 4 mm in internal diameter and 2 mm tall. All curing was accomplished with a visible light-curing unit (ESPE Elipar Highlight) operating in standard mode with an intensity >400 mw/cm<sup>2</sup>, as verified with a Demetron radiometer—Model 100 (Demetron Research Corporation, Danbury, CT 06810).

**Group NT1:** 1) 34% Tooth Conditioner Gel was applied to the dentin for 15 seconds; 2) the surface was rinsed for 10 seconds and blotted dry with a Kimwipe (Kimberly-Clark Corp, Roswell, GA 30076); 3) Prime & Bond NT adhesive was lightly rubbed onto the surface with a microbrush and the surface kept wet for 20 seconds; 4) the surface was air dried for five seconds to evaporate the solvent; 5) the adhesive was light cured for 10 seconds; 6) the compomer Dyract (Dentsply Caulk, Milford, DE 19963) was placed in a 2 mm increment and light cured for 40 seconds.

**Group NT2:** The steps for Group NT1 were followed plus steps 3 and 4 were repeated before proceeding to step 5.

**Group OP1:** 1) 37.5% H3PO4 (KERR Gel Etchant) was applied to the dentin for 15 seconds; 2) the surface was rinsed for 15 seconds; 3) the surface was blotted dry; 4) Optibond Solo was applied with a light brushing motion with a microbrush for 15 seconds; 5) the adhesive was light cured for 20 seconds 6) the compomer Élan (Kerr USA, Orange, CA 92867) was placed in a 2 mm increment and light cured for 40 seconds.

**Group OP2:** The steps for Group OP1 were followed plus step 4 was repeated before proceeding to step 5.

**Group ON1:** 1) 32% H<sub>3</sub>PO<sub>4</sub> UNI-ETCH was applied to the dentin for 15 seconds; 2) the surface

Table 1: Adhesive Systems Tested		
Adhesive System	Etchant(Code/Lot #)	Adhesive (Code/Lot #)
Prime & Bond NT	34% Tooth Conditioner Gel (990503)	Di & Trimethacrylate resins, PENTA, acetone (107336/0)
Optibond Solo	Kerr Gel Etchant 37.5% Phosphoric Acid (803288)	BISGMA, HEMA, ethyl alcohol (806051)
One-Step	Uni-Etch 32% Phosphoric Acid, Benzalkonium Chloride (9900005387)	BPDM, BISGMA, HEMA, acetone (9900005417)
PENTA = dipentaerythritol penta acrylate monophosphate; BISGMA = bisphenyl-glycidyl-methacrylate; HEMA = 2-hydroxyethylmethacrylate; BPDM = bisphenyl-dimethacrylate		

was thoroughly rinsed; 3) the surface was blot dried with a "moist" Kimwipe; 4) with a fully-saturated brush tip, two consecutive coats of One-Step were applied with slight agitation; 5) a gentle air stream was used for 10 seconds to evaporate the solvent; 6) the adhesive was light cured for 10 seconds; 7) Dyract was placed in a 2 mm increment and light cured for 40 seconds.

**Group ON2:** The steps for Group ON1 were followed plus steps 4 and 5 were repeated before proceeding to step 6.

After fabrication, the specimens were stored in distilled water at 36°C. At one week, the teeth were thermocycled for 2500 cycles between 5°C and 48°C water baths with a 30-second dwell time and 10-second transit time. Specimens were then returned to 36°C deionized water until testing.

At two weeks the shear bond strength was determined using a circular knife-edge applied to the interface in tension. A MTS 1123 Renew testing machine with MTS software was used to apply the load with a 0.5 mm/min crosshead speed. Differences between the recommended application and the double application groups for each product were evaluated with *t*-tests using the SigmaStat 2.0 (Jandel Scientific) statistical software package.

Fracture mode was determined using a stereomicroscope (20x). Failure was classified as cohesive in dentin if any dentin failure was evident, adhesive if the dentin bonding area was intact with no material evident or mixed if there was evidence of retained material on the dentin surface. Chi-square analysis was used to test for differences in failure mode between the groups for each material.

### Confocal Microscopy

Twelve caries-free human molars were prepared as described above without mounting in acrylic tubing. Two teeth were assigned to each treatment group. Bonding was accomplished as described in the shear bond strength methodology. One drop of aqueous 0.5% Rhodamine B was placed in the well of a disposable dappen dish for each tooth to be bonded. The water was evaporated in a dry heat oven. The adhesive was placed into the well and mixed with the Rhodamine B prior to placement on the tooth.

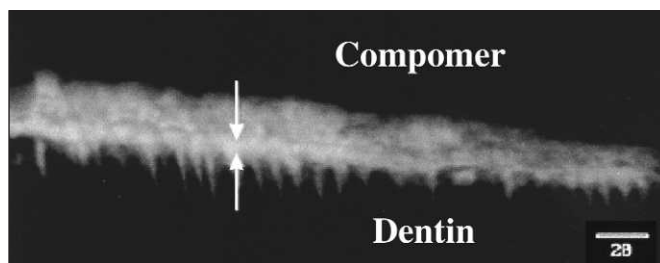


Figure 1. Confocal micrograph of specimen prepared with one application of Prime & Bond NT. The adhesive layer is indicated by the arrows.

Table 2: Shear Bond Strength

	Mean (MPa)	Standard Deviation	Coef of Variation
NT1	12.34*	6.34	51.38
NT2	21.39*	6.16	28.79
OP1	20.02	2.46	12.27
OP2	19.42	2.44	12.54
ON1	20.13	3.11	15.43
ON2	19.46	3.06	15.74

\* = means significantly different at  $p < 0.001$ .

Table 3: Failure Mode

	Dentin	Material	Interface
NT1	3*	8	4
NT2	7*	7	1
OP1	8	7	0
OP2	8	7	0
ON1	12	3	0
ON2	10	5	0

Dentin = Cohesive Failure in Dentin Evident  
 Material = Cohesive Failure in Compomer Evident with no Dentin Failure  
 Interface = No Evidence of Cohesive Failure Within Dentin or Compomer  
 \* = means significantly different at  $p < 0.05$

After bonding, the teeth were sectioned with a diamond saw (Hamco Machines, NY), rinsed, dried and mounted on glass slides for viewing under the confocal microscope (Noran Odyssey, Noran Instruments, Middleton, WI 53562). An argon laser (488 nm wavelength) was used in a reflective mode. Images were captured using computer software (Image I Image Analysis ver 4.14c, Universal Imaging, West Chester, PA 19380).

### RESULTS

The results of the shear bond strength test are given in Table 2. Upon running the *t*-tests, the NT groups failed the normality test and a Mann-Whitney Rank Sum Test performed. The group NT1 was significantly different ( $p < 0.001$ ) than NT2. The means for the OP and ON groups were not significantly different.

Fracture modes are given in Table 3. There was an increase in the number of dentin cohesive failures in NT2 when compared to NT1. There was no difference between the OP and ON groups.

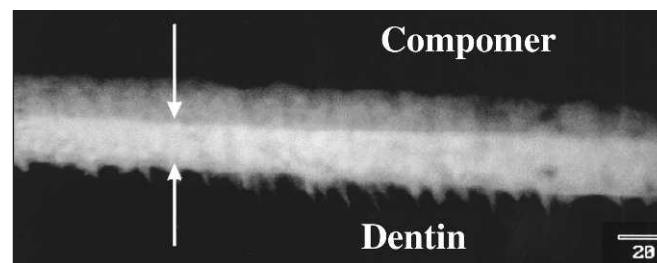


Figure 2. Confocal micrograph of specimen prepared with two applications of Prime & Bond NT. The adhesive layer is indicated by the arrows.



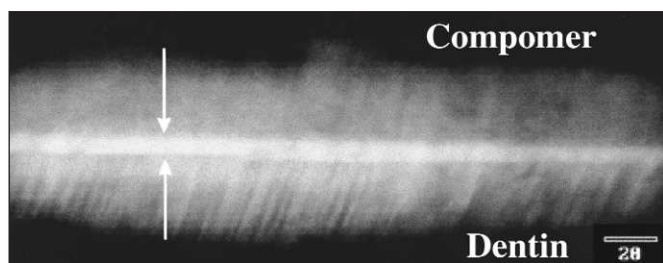


Figure 3. Confocal micrograph of specimen prepared with one application of Optibond Solo. The adhesive layer is indicated by the arrows.

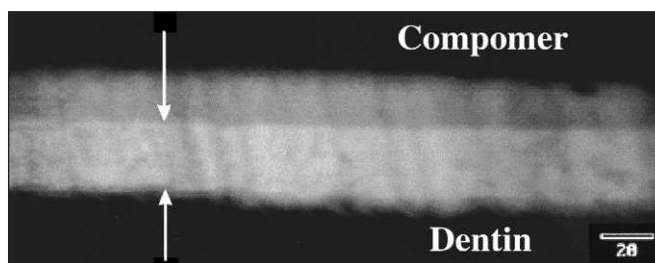


Figure 4. Confocal micrograph of specimen prepared with two applications of Optibond Solo. The adhesive layer is indicated by the arrows.

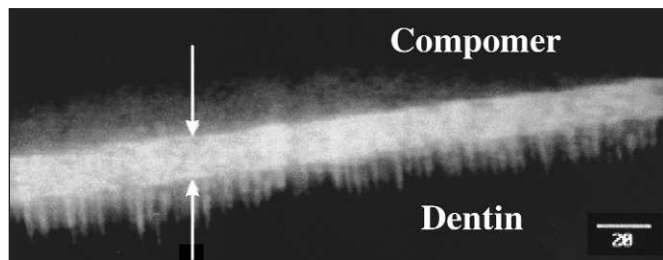


Figure 5. Confocal micrograph of specimen prepared with one application of One-Step. The adhesive layer is indicated by the arrows.

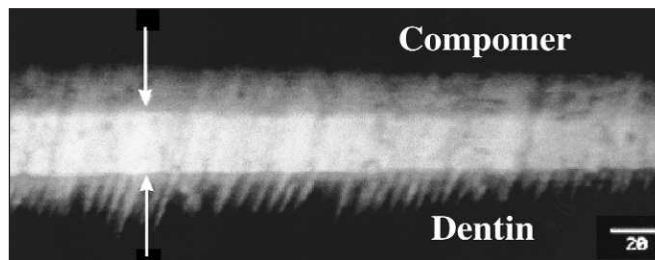


Figure 6. Confocal micrograph of specimen prepared with two applications of One-Step. The adhesive layer is indicated by the arrows.

The confocal micrograph presented in Figure 1 shows a lack of continuity within the adhesive layer that is evident when using the NT1 methodology. Tubule penetration and hybrid layer formation appear to be present. Incorporation of the adhesive into the restorative material can be seen. Figure 2 demonstrates that a second application of adhesive (group NT2) provides a more uniform adhesive interface.

All other groups presented with adhesive layers, which were continuous across the interface (Figures 3-6).

## DISCUSSION

Under the stated conditions, this study demonstrated that doubling the recommended applications of Prime & Bond NT improves performance when evaluating dentin shear bond strength. It was initially thought that this could be a function of the acetone solvent. It has been shown that shear bond strength results can vary between solvent types depending on the conditions during placement (Jacobsen & Soderholm, 1998; Iwami & others, 1998). However, a second material with an acetone solvent was employed, One Step. Its bond strengths did not differ between one and two applications. The results of this study did not appear to be solely dependent on the type of solvent used in the adhesive.

Hilton & Swartz (1995) evaluated the effect of air thinning of the adhesive on the shear bond strength of two-bottle systems. They demonstrated that air thinning of the adhesive reduced dentin shear bond strengths. They also presented scanning electron

micrographs to demonstrate a thinner adhesive layer of variable thickness after air thinning. They reported “waves” or “ripples” within the adhesive layer after air thinning.

In this study, the NT and One-Step solvents were evaporated by the use of a gentle air stream, while the Optibond Solo specimens did not receive this treatment. Every specimen appeared to have a smooth, glossy surface after one application of adhesive and drying of the solvent. However, the micrographs suggest that the NT1 adhesive layer may have had very thin areas at the time of polymerization. Finger & Dreyer Jorgenson (1976) demonstrated that the minimum thickness of adhesive layer whose polymerization is not inhibited by oxygen varies with materials and that layers less than 25 microns may not polymerize. It is possible that one application of Prime & Bond NT may not polymerize due to oxygen inhibition. The adhesive might be incorporated into the overlying restorative material. This would have an obvious effect on the composition of the adhesive interface. A One Step application actually contained two coats, as delineated in the manufacturer's instructions. One might expect that the second coat, during the first application, would be adequate to compensate for this oxygen inhibitory effect.

Others have demonstrated that the thickness of the adhesive layer can positively or negatively effect bond strength data, (Zheng & others, 2000). The average stress in the bonding area has been shown to be dependent on the elasticity of that area (Wakasa, Yamaki & Matsui, 1995). The transition from hybrid layer to restorative material without a definite adhe-

sive layer may be too abrupt of a transition. The expected end result would be decreased shear bond strength. The results for NT are consistent with this scenario.

Swift et al (1997) showed a statistically insignificant trend toward decreased shear bond strength with multiple applications of the single bottle adhesives. In this study, the decreased shear bond strengths seen with a double application of Optibond Solo and One Step were also statistically insignificant.

### CONCLUSIONS

1. The dentin shear bond strength of Prime & Bond NT is improved by the use of a second application, when following manufacturer's instructions.
2. The dentin shear bond strength of One Step and Optibond Solo are not improved by the use of a second application, when following manufacturer's instructions.

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## Departments

### Announcements



#### University Dental Hospital of Manchester PRESS ANNOUNCEMENT 25<sup>th</sup> Anniversary of Visible Light Curing

The 25<sup>th</sup> Anniversary of the placement of the first ever-visible light cured restoration has been celebrated. The celebrations took the form of a commemorative lecture, the unveiling of a plaque in the clinical area in which the restoration was placed at the University Dental Hospital of Manchester and an Anniversary Dinner at the University. The commemorative lecture entitled "25 Light Years" was given jointly by:

Dr Mohamed Bassiouny of Temple University School of Dentistry, Philadelphia—the individual who placed the first ever visible light-cured restoration during his time as a postgraduate PhD student in Manchester.

Dr John Yearn, Manager, Technical Operations, GC Europe, Leuven, Belgium—the individual who headed up the ICI research team that developed the first visible light cured composite restoratives and light source. Dr Yearn is also the individual who received the first visible light cured restoration.

and

Dr Nairn Wilson and Professor David Watts of the University Dental Hospital of Manchester, both of whom have been involved in laboratory and clinical research on visible light cured systems throughout the first 25 years of visible light curing in dentistry.

It is of special note that the visible light cured restoration which Professor Bassiouny placed in Dr Yearn's upper right second premolar on 24<sup>th</sup> February 1976 remains in clinical service (Figure 1). This large Class V restoration was placed without the benefit of etching and bonding. The light source used by Professor Bassiouny was a prototype Fotofil "activator light unit."

As concluded at the 25<sup>th</sup> Anniversary commemorative lecture, it is estimated that globally several million light curing procedures are presently completed on a daily basis. Visible light curing is widely recognized to have made a major contribution to recent developments in the clinical practice of dentistry.

Further details are available from Professor Nairn Wilson, Restorative Dentistry, University Dental Hospital of Manchester, Higher Cambridge Street, Manchester M15 6FH, Tel: 0161 275 6660; Fax: 0161 275 6710; e-mail: Nairn.H.F.Wilson@man.ac.uk.

#### 2001 Meeting of the Academy of Operative Dentistry European Section

The 2001 meeting of the Academy of Operative Dentistry, European Section will be held in conjunction with the 107<sup>th</sup> meeting of the American Dental Society of Europe (ADSE) from June 26<sup>th</sup>-29<sup>th</sup>, 2001 in Barcelona. Details of arrangements to attend this meeting as guests of the ADSE may be obtained from:

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University  
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#### 2002 Meeting of the Academy of Operative Dentistry European Section

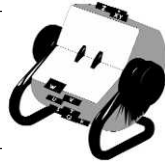
The 2002 meeting of the Academy of Operative Dentistry European Section will be hosted by the University of Nijmegen College of Dental Science in Nijmegen, The Netherlands. The meeting, to be entitled "Adhesive dentistry today. Transfer of research into practice" will be held December 5<sup>th</sup>-7<sup>th</sup>, 2002. Details may be obtained from:

Dr. E H Verdonshot,  
University of Nijmegen College of Dental Science  
PO Box 9101, NL-6500 HB  
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