

# OPERATIVE DENTISTRY



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

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# The Future of Dentistry From Where I Sit

The annual gatherings of the various academies in Chicago have just ended. The many opportunities for reunions that these meetings provide buoy me up and underscore the pure joy of having chosen to be a dentist. It is a time when we share ideas, experiences and concerns and it highlights the core values to which we jointly aspire. These gatherings are also a time to express the cares, concerns and frustrations that those of us in private practice, the military and academia carry in our hearts.

One such concern and frustration relates to the dental educational process of tomorrow's dentists. A recent article featured in my alumni journal emphasized the dire need for alumni to offer their services in the clinical area of this Eastern school. While the plea was well put, few are accepting the challenge to teach and train future dentists. Many dentists state that choosing private practice over teaching is an issue of economics.

Since private practice certainly provides greater income potential than teaching, it is easy to understand why students who incur more than \$100,000 of indebtedness would have a hard time choosing an academic career over establishing their own practice. However, what about those of us who have cleared the early hurdles of indebtedness?

We cannot expect the recent graduate to be adequately equipped to teach. We cannot reasonably look to private practitioners to be ready to teach if they have little or no experience in ALL aspects of restorative dentistry.

This brings me to a point of deep concern. If dental graduates are not being trained in the varied uses, applications and selection of dental materials, such as cast and direct gold, what will the educators of the future teach? If I were a dental student today, paying the vast amounts of money that current students do for their dental education, yet I was not learning about ALL dental materials in a way that allowed me to make responsible choices with my patients, I would feel cheated.

Enter the Operating Study Club!

It seems that many dental educational institutions have abdicated the role of providing future dentist's with a complete restorative armamentarium and are not even telling them where they can get this information. While I am not positive that this has been a universal decision by all dental schools, when I sit with students during meeting lunches and they ask me, "Where can I learn to do inlays, onlays and gold foils?" I usually reply, "An operating study club." Then, when the students ask, "What is an operating study club?" It gives me cause to wonder.

Several possibilities come to mind following such an encounter.

1. Those who run operating study clubs could make a very strong, concerted effort to connect with recent dental graduates in their area and invite them to a meeting.
2. Dental schools that do not provide opportunities for students to learn direct and indirect gold should realize that they have only partially educated the future dentist and openly seek clinical support from study clubs in their area.
3. The Academy of Operative Dentistry can renew its focus to support operating study clubs by creating a manual of sample constitutions and methods of operation so that existing study clubs can serve as a resource for recent graduates and post it on their website.

Clearly, educators who have made the effort to participate in the mission of the Operative Academy have the high level of appreciation and expertise needed to achieve this goal. Each and every AOD member has a dental school to which they can speak. Please, let these dental schools know your feelings.

From my perspective, during my 43-year career as a dentist, many individuals have contributed to my growth and development as a dentist. Although my efforts to pay back that debt have not yet met with the

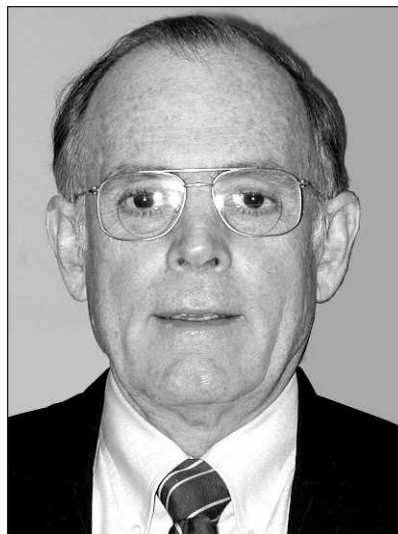


desired results from my dental school, I MUST continue to try. I ask you to do the same. If we do not persist in our efforts to correct the problems we see in our system of dental education, then we have only ourselves to blame if future generations of practitioners are inadequately prepared to meet the restorative needs of their patients.

How about it? Are you willing to accept what you see in dentistry's future?

I am NOT and I hope that you will NOT accept it, either!

Bob Keene, President  
American Academy of Gold Foil Operators



*Robert Keene*

## COMMENTARY

I would like to thank Dr Bob Keene for sharing his thoughts in this guest editorial. Dr Keene currently serves as President of the American Academy of Gold Foil Operators and is in the enviable position of heading the academy as it celebrates its 50th anniversary.

The 50th Annual Session of the AAGFO will be held in Halifax, Nova Scotia on October 9-12, 2002. Please consider joining Dr Keene and the rest of us as we celebrate excellence in clinical dentistry with both lectures and an outstanding clinical operating session.

Michael A Cochran  
Editor

## Buonocore Memorial Lecture

# Dentin Caries: Progression and Clinical Management



Michael Buonocore

L Bjørndal



Lars Bjørndal

### SUMMARY

A fundamental issue of restorative treatment is assessing the different conditions of cases not only from tooth-to-tooth but also from the activity of each caries lesion and the size of the cavity. In addition, restorative treatment is sometimes carried out for prosthetic and cosmetic purposes and involves cutting

sound, unaffected dentin. These aspects are outlined in classic textbooks and are based on the principles described by Black (1908). However, each factor and its relative importance has changed over the years. Significant time has been devoted to improving the important technical aspects of performing restorative treatment. Traditionally, these aspects have primarily been related to cavity design, choice of restorative material and the clinical procedures involved. In this context, Dr Michael Buonocore's contributions must be recognized because the

adhesive technique he introduced has become an integral part of modern operative dentistry.

Operative dentistry today also focuses on cavity design and selecting restorative materials. Less effort has been placed on incorporating what is known about the pattern of caries progression and how it relates to caries removal or excavation. Although the reaction pattern of the pulp-dentin organ is quite different in terms of the nature of active (rapid-progressing) and arrested (slow-progressing) lesions, no widespread major distinction has been made regarding the different restorative treatment approaches in these situations.

This presentation updates the progression and clinical management of dentin caries and how it relates to treating deep caries lesions.

### Definition of Lesion Characteristics

The dentistry of today has developed into many subspecialties. As a result, terms used in operative dentistry may have different meanings. Caries is a good example. Researchers involved with cariology would first associate a lesion with specific bacteria and the progressive and dynamic events of mineral loss starting at the surface enamel, whereas the term "caries" used in operative dentistry indicates a cavity in need of restorative treatment. This is different from the endodontist who would interpret a lesion as being associated with pulpal or apical pathosis.

For the same reasons, the histopathological description of a lesion in operative dentistry is typically illus-

trated as a dentin involvement growing larger, and with dentin caries spreading along the dentino-enamel junction. However, to obtain more information on the biological reactions, when describing caries pathology, more precise terminology needs to be used. For example, what are the first signs of pulp-dentin reactions in relation to caries? At what stage of lesion progression can we expect the spreading and undermining nature of dentin caries?

### Enamel Lesion Without Dentin Exposure

A recent review has focused on the clinical and histopathological pattern of untreated caries in different stages of lesion progression as a way of updating our knowledge of the pulp-dentin organ (Bjørndal & Mjör, 2001). It is important to realize that there is a well-defined structural interrelation between dentinal reactions subjacent to the clinically active progressing enamel lesion without clinical exposure to dentin. Previous interpretation of the first signs of dentin caries focused on the early, rapid spread of decay that undermines sound enamel. Then, an independent description of



Figure 1. Ground section through a proximal lesion viewed in reflected light. When the enamel lesion reaches the dentino-enamel junction the extent of the discolored demineralized dentin is limited to this contact area. (Original magnification X2.5).

dentin's involvement followed with no strict relation to either the enamel or pulp (Silverstone & Hicks, 1985). If a thick section taken from an extracted tooth with caries is examined, it is easy to make such an interpretation. However, as soon as the thin, undemineralized sections are examined, the first alteration in the dentin resulting from caries can be traced all the way to the pulp and the dentin is hypermineralized (Johnson, Taylor & Berman, 1969; Bjørndal & Thylstrup, 1995). The onset of dentin demineralization and dentin discoloration are not observed until the lesion has reached the dentino-enamel junction, and no lateral spread is noted in relation to enamel lesions without dentin exposure (Figure 1). Quantitative analyses have shown that the extent of the discolored, demineralized dentin typically follows the enamel lesion in contact with the dentino-enamel junction (Bjørndal & Thylstrup, 1995; Bjørndal, Darvann & Lussi, 1999). At this stage of lesion progression, dentin reactions are strictly guided by the cariogenic environment on the enamel surface. From a clinical viewpoint, this means that a non-operative treatment of the enamel lesion, including removing cariogenic biomasses at the outer enamel surface, would lead to the arrest of the entire enamel-dentin lesion complex.

It is commonly believed that early dentin caries spreads along the dentino-enamel junction. This concept is important because the clinical consequence of this early spreading has been to remove the affected dentin as soon as possible, with no specific reference to the status of the lesion on the enamel surface. The presence of discolored dentin has, moreover, led to the conclusion that treatment was justified, and if done correctly, will prevent further undermining of sound enamel. However, based on an updated view of the structural characteristics of the enamel-dentin lesion, the most effective way to treat actively progressing, non-cavitated lesions is by using non-operative treatment principles.

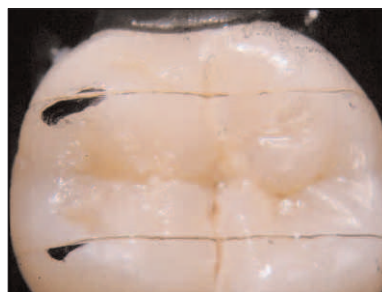


Figure 2. Macroscopical view of a freshly extracted third molar. Note the two cutting lines characterizing the specimen involving rapidly progressing enamel lesions covered with cariogenic plaque. (Original magnification X2.5).

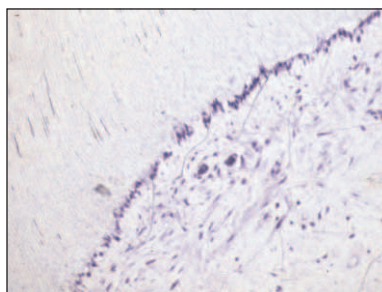


Figure 3. Reduced odontoblastic layer and reduced predentin width subjacent to the active occlusal lesions shown in Figure 2 not reached the dentino-enamel junction. (Toluidine blue-pyronin stain; original magnification X50).



Figure 4. Ground section through an active occlusal lesion with the characteristic feature of a yellowish discoloration of the demineralized dentin. (Reflected light; original magnification X2.5).



Figure 5. Ground section through an arrested occlusal lesion with the characteristic feature of a dark brownish discoloration of the demineralized dentin. (Reflected light; original magnification X2.5).



The spreading pattern of caries following enamel breakdown has led to designs of micro-cavities, for example, tunnel preparation or internal occlusal fossa preparation (Wilson & McLean, 1988), whereby early dentin exposure is treated by a retrograde approach. In addition, introducing dental operating microscopes represents a challenging improvement in relation to the clinical assessment of early caries, but the observations made must be correlated to the knowledge of lesion activity and the spreading pattern of caries. Taken together, the field of micro-procedures and micro-cavities also represents a potential risk of introducing clinical procedures at early stages of lesion progress, where further progression could have been avoided because

the enamel lesion was already passive or because it could be arrested by preventive means.

### Examination of the Pulpal Response to Non-Cavitated Caries

Through the use of thin, undemineralized tooth sections, it is possible to simultaneously examine the events taking place in the hard and soft tissue. As also indicated by Brännström & Lind (1965), cellular changes in the odontoblastic and subodontoblastic regions have recently been shown to present subjacent to subsurface enamel lesions (Figures 2 and 3) confirmed by the use of thin, undemineralized tooth sections and computed histomorphometry (Bjørndal, Darvann & Thylstrup, 1998). It was also noted that cellular reactions along the pulp-dentin interface from rapid- and slow-progressing enamel lesions were different. In clinical terms, this means that removing cariogenic biomasses during non-operative treatment of enamel lesions is not only reflected at the enamel tooth surface, but also along the pulp-dentin interface. In short, the clinical concept of different discolorations of demineralized dentin (Figures 4 and 5) characterizing different lesion activities can be biologically confirmed at the cellular level, thus demonstrating the reversible nature of the early pulpal response (Bjørndal & others, 1998).

### Lesions Involving Dentin

Considerable debate has been devoted to the degree of bacterial infection related to the surface status of the caries lesion (Thylstrup & Qvist, 1987). In non-cavitated enamel lesions, the level of bacterial invasion is very low, if at all present. Even though the enamel is demineralized, the spaces in-between the crystals are too small for micro-organisms to penetrate (Figure 6), and the subjacent discolored and demineralized dentin, in general, is without the presence of micro-organisms in precavitated lesions. As soon as the demineralized enamel layer crumbles, particularly from forces of

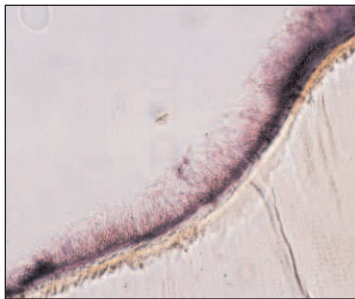


Figure 6. Detail of enamel lesion covered with cariogenic plaque. Note the enamel rods are clearly visible in the 15 mm thin undemineralized tooth section. No bacteria are penetrating the demineralized rod structure. (Transmitted light; original magnification X50).

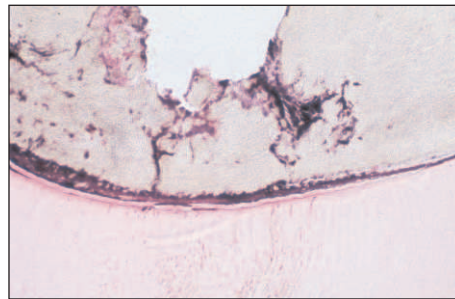


Figure 7. Detail of dentino-enamel junction subjacent to an active enamel cavitated lesion, where the enamel layer has crumbled down to the dentin. Note presence of bacteria in the dentin. (Transmitted light; original magnification X25).

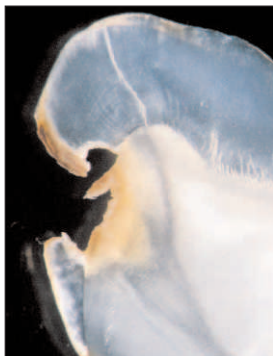


Figure 8. Ground section through a dentin exposed proximal lesion. The focus of spreading is located deep within the cavitation. A lateral spread of dentin demineralization is taking place along the dentino-enamel junction and the enamel has become undermined. (Reflected light; original magnification X2.5).



Figure 9. A maxillary premolar with dentin exposed lesion following loss of restoration. The clinical signs of spread along the dentino-enamel junction can be viewed as a change in the occlusal enamel translucency. The partly removal of overhanging enamel discloses the retrograde pattern of demineralization.



Figure 10. A maxillary molar in a late stage of caries progression with mixed lesion activity. Almost the entire coronal enamel has been broken down. The central occlusal area resembles areas with signs of slowly progressing caries. The demineralized dentin is dark and the surface is not covered with plaque. Enamel is still present in the peripheral lesion parts and a thick plaque accumulation is noted. Caries is rapidly progressing in these parts of the tooth. Eventually the entire crown will be destructed ending up as the neighboring tooth, where only remnants of roots are observed.

mastication and any stress toward the lesion area, a growing accumulation of micro-organisms will occur (Figure 7). Eventually, the dentin becomes infected by micro-organisms, and the bacterial spreading is markedly changed. The bacterial growth conditions change from being a bacterial plaque at the external tooth surface to being protected deep within the lesion environment by the broken down enamel, which can be observed along the dentino-enamel junction (Figure 8). This process has classically been described by Black (1908) as “backway decay of enamel.” Clinically, this spreading pattern can be traced through sound, undermined enamel as a shift in enamel translucency. Looking for these lesion characteristics, one is seldom surprised that the extent of the final preparation will reach deep into the dentin (Figure 9).

### **The Pulpal Response to Dentin Exposed by Caries**

Deep dentinal lesion progress is often presented as the point of no return for the specific pulp involved. The signs and symptoms of irreversible pulpitis followed by apical pathosis enter the discussion. In deep lesions, however, large variations and changes within the lesion environment may be detected. Closed and open ecosystems have been suggested for these phenomena (Edwardsson, 1987) related to the clinical features of the cavity. The open lesion environment represents a lesion where the undermined enamel has been broken down, changing the growth condition for the cariogenic biomasses. As Figure 10 illustrates and with a focus on caries activity, different rates of progression can be present within one tooth. The occlusal and central part of the vital tooth is actually without heavy plaque accumulation because chewing and other functions prevent it. The corresponding dentin also appears to have classical signs of slowly arrested dentin caries shiny with brown discoloration. In contrast, the peripheral parts are still protected by undermined enamel and heavy accumulations of cariogenic biomasses. This marked change within lesion environment can also be reflected within the pulp as different types of tertiary dentin (Bjørndal, 2001). In “closed,” active and rapidly progressing lesions, the initial tertiary dentin is laid down without dentinal tubules, which are also defined as fibrodentin (Baume, 1980) or interface dentin (Mjör, 1985), whereas in slow-progressing lesions, the extra-dentinal matrix laid down is tubular, resembling primary dentin. These observations indicate that deep dentin affected by caries is not unconditionally related to an irreversible pulp pathology as traditionally described in textbooks that advocate pulp invasive treatment procedures (Tronstad, 1991; Ørstavik & Pitt Ford, 1998).

### **Treatment of Deep Caries—The History of Different Concepts**

The philosophy of restorative treatment to deep caries can be traced back to two thoughts “...it will often be a question of whether or not the pulp will be exposed

when all decayed dentin overlaying it is removed.... it is better to expose the pulp of a tooth than to leave it covered only with softened dentine” (Black, 1908). In contrast, Tomes (1859) wrote, “It is better that a layer of discoloured dentine should be allowed to remain for the protection of the pulp rather than run the risk of sacrificing the tooth.” Today, even though maintenance of pulp vitality in deep lesions has been widely discussed, the subject has been dominated by this contradictory and controversial information (Dumsha & Hovland, 1985).

Different methods for preventing exposure and damage to pulp have been advanced. The first is the indirect pulp-capping procedure employed particularly in the primary dentition (Aponte, Hartsook & Crowley 1966) and in mixed dentitions (Eidelman, Finn & Koulourides 1965; King, Crawford & Lindahl, 1965; Kerkhove & others, 1967). The second method is the two-stage excavation procedure (Sowden, 1956; Law & Lewis, 1961; Massler, 1978) or stepwise excavation (Magnusson & Sundell, 1977), which, more recently, has been applied even in the permanent dentition (Leksell & others, 1996; Bjørndal, Larsen & Thylstrup, 1997). The main difference is that the indirect pulp-capping procedure almost completely removes the affected dentin, leaving a thin layer of residual demineralized dentin and re-entry is not made; that is, it is a one-step procedure, while the stepwise excavation procedure involves re-entry at varying intervals.

### **Treatment of Deep Caries Lesions Based on an Understanding of Caries Pathology**

Recently, a less invasive first excavation procedure in the stepwise approach was introduced (Figures 11-14). It aims at further reducing the risk of pulp exposures during the first excavation and promoting physiological reactions in the pulp-dentin organ but with particular focus on the overall change in lesion activity (Bjørndal & others, 1997; Bjørndal & Thylstrup, 1998; Bjørndal & Larsen, 2000). This first excavation procedure is not, as originally attempted, to reach the pulp as closely as possible in order to stimulate the formation of extra dentinal tissue per se. Instead, it is to promote arresting the lesion by changing the cariogenic environment.

Clinical changes before and after such a modified, less invasive procedure (Bjørndal & others, 1997) have shown that caries arrestment can be assessed easily. A harder, more brownish dentin (Figures 12, 13) will be found following temporary sealing of the lesion according to Miller's (1959) description of active and arrested dentinal lesions. This approach also allows reparative dentinogenesis to occur (Bjørndal & Rud, 2000). Dentin permeability will be reduced during treatment and will affect the long-term effectiveness of the permanent restoration.



### Why Perform the Final Excavation?

When using a less invasive first excavation approach, bacterial counts of dentin samples have been shown to decrease in a similar way during a treatment interval (Bjørndal & others, 1997), as seen in studies where the excavation was performed to the residual level, which is much closer to the pulp (King & others, 1965; Aponte, & others 1966). However, since completely sterile conditions are not created, no long-term data supports avoiding the final excavation into deep lesions. Clinical observation of dentin changes during treatment provides the clinician with relevant information about the change in lesion progress. The final excavation is facilitated because it is more convenient to excavate in harder, darker carious tissue close to the pulp (Figure 13) than in soft, yellow demineralized dentin. The dual function of final excavation is therefore to perform clinical control of the tooth reaction and to remove the slow-progressing but still slightly infected discolored, demineralized dentin before carrying out the permanent and final restoration. The 10-year results by Mertz-Fairhurst & others (1998), which make a one-step procedure that arrests dentin lesions by sealants, seems to be a breakthrough in terms of controlling a cariogenic environment, but it is also important to note that these results were based on less advanced dentinal lesions being located in the outer half of the dentin. Therefore, outcome studies that compare the indirect pulp capping procedure with a stepwise excavation approach in deep lesions of similar size are needed.

### Experience from Private Practitioners Performing Stepwise Excavation

Experience from a dental practice environment has shown the effectiveness of the stepwise excavation procedure for the management of deep carious lesions, and long-term recall (3.5–4.5 years) has shown a high success rate (92%) with teeth treated with this approach (Bjørndal, 1999). Although the total group of failed cases was less than 10%, in half of them, insufficient temporary and permanent restorations were noted, underlining the importance of performing high-quality temporary and permanent seals. A two-step excavation procedure will add to the cost of the restorative treatment, including control examinations with regard to pulp sensitivity and vitality because of the possibility of the asymptomatic development of irreversible pulp degeneration over time.

Comparisons with other studies must be done cautiously. However, 5% of the cases treated by general practitioners in the above study had pulpal complications during final excavation. In contrast, the traditional, more invasive step-by-step approach (Magnusson & Sundell, 1977; Leksell & others, 1996) presents a higher proportion of pulp complications during the final treatment (~15%). This could indicate the positive effect of a less invasive first excavation procedure. In addition, it

probably reduces the frequency of iatrogenic pulp exposures.

### Case Selection and Clinical Comments on Stepwise Excavation

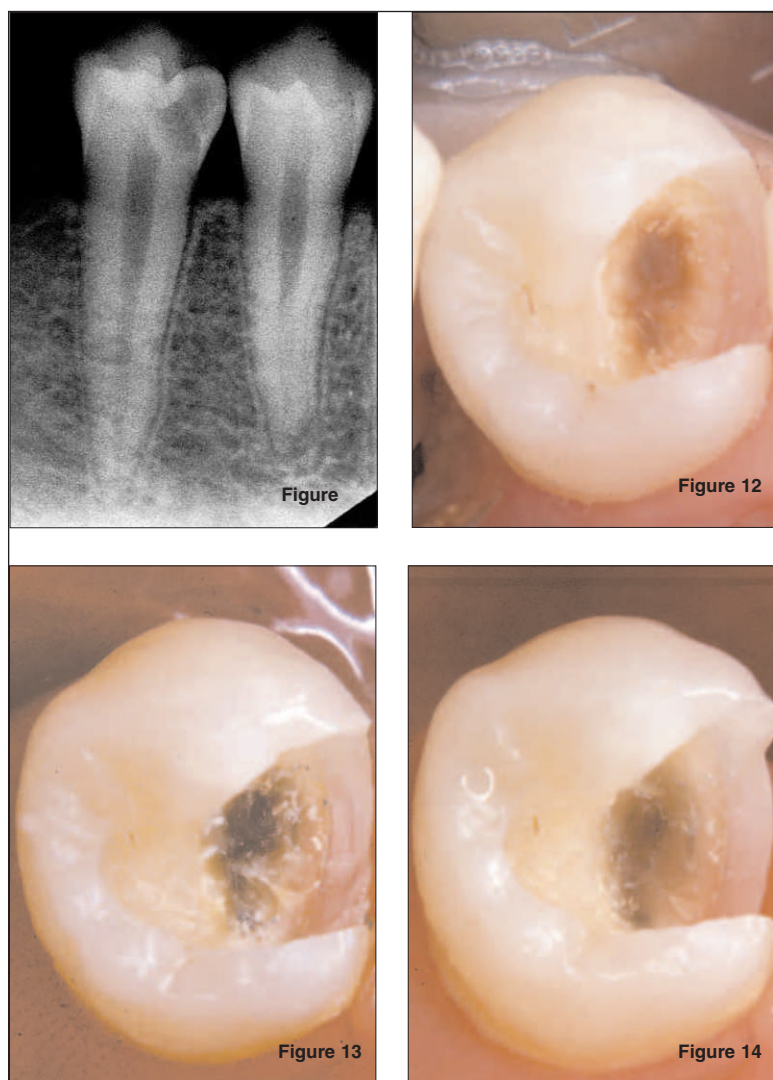
As long as there are no non-invasive tools to assess the histopathological condition of the pulp and for the measurement of the severity of the pulpal inflammation, discussion of reversible or irreversible development of pulpitis will remain controversial in relation to treatment of vital, non-symptomatic, deep dentin lesions. On this basis, the only alternative is to depend on patients' information, the objective evaluation of pulp vitality and information obtained from radiographs (Reit, 1995). Based on data from the Danish dental practice environment (Bjørndal & Thylstrup, 1998), the following guidelines are currently suggested in relation to case selection for the stepwise excavation:

- Deep lesions are likely to result in pulp exposure if treated by a single, terminal excavation. Evaluated by radiographs, the dentinal lesion should involve more than 75% of the entire dentin thickness (Figure 11).
- No history of subjective pre-treatment symptoms, such as spontaneous pain or provoked pulpal pain; however, mild to moderate pain upon thermal stimulation is accepted.
- Positive pulp vitality tested by an electric pulp tester, thermal stimulation or drilling.
- Pre-treatment radiographs to exclude apical pathosis.

These guidelines for case selection do not differ significantly from those for direct and indirect pulp capping.

Clinical comments concerning a less invasive stepwise excavation approach include:

- Decide at an early treatment stage whether it will be a stepwise excavation case.
- Finish the peripheral excavation of the lesion followed by a central excavation that removes the outermost necrotic, infected demineralized dentin (Figure 12) so that a provisional restoration can be properly placed.
- Do not attempt to excavate as close to the pulp as possible during the first step, thus reducing the risk of pulp exposure.
- Decide which material (usually calcium hydroxide or zinc oxide-eugenol cement) will be used to cover the remaining carious dentin.
- Decide which provisional restorative material will be used in relation to the length of the treatment interval, ranging between six and eight months (amalgam, glass ionomers and composites may be excellent temporary materials).



Figures 11-14. Radiograph of a mandibular premolar with a deep dentin exposed lesion. No evidence of apical pathosis (Figure 11). A stepwise excavation is decided and a detail of the cavity following first excavation is observed (Figure 12). After a six months treatment interval and removal of base material (calcium hydroxide) and temporary filling (amalgam) the retained carious dentin shows signs of a slowly progressing lesion (Figure 13). The premolar with final excavation completed and final restoration can be performed (Figure 14).

- Performing the final excavation often ends up being less invasive than expected due to the altered dentinal changes gained during the treatment interval (Figure 14).

### FINAL COMMENTS

Change in dentin permeability, including tertiary dentin formation following a stepwise excavation procedure, represents secondary biological reaction that is not only produced for the protection of the pulp, but also as a consequence of a change in the cariogenic environment. Therefore, tissue changes per se cannot permanently serve as a barrier against new, potential

cariogenic biomasses to create the reestablishment of low pH-gradients or prevent a re-invasion of bacterial antigens.

A full understanding of the pulp-dentin organ in restorative dentistry also includes knowledge of its limitations. Control and prevention of further secondary damage to the restored tooth will, besides providing an optimal restoration, include the care of proper oral hygiene procedures for removing cariogenic biomasses that tend to accumulate where it all began—in the area of the restored tooth surface.

*Presented at the 31st Annual Meeting of the Academy of Operative Dentistry, February 21, 2002.*

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## Clinical Research

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# One-Year Clinical Performance of a Self-Etching Adhesive in Class V Resin Composites Cured by Two Methods

WW Brackett • DA Covey • HA St Germain, Jr

### Clinical Relevance

No difference was observed between “soft-start” and high-intensity light curing. The self-etching adhesive that was evaluated was not effective in resin composite restorations of unprepared cervical erosion/abfraction lesions.

### SUMMARY

**This study evaluated the clinical performance of a self-etching adhesive for resin composites over one year. Thirty pairs of restorations of Pertac II, using the adhesive Prompt L-Pop, were placed in caries-free cervical erosion/abfraction lesions without tooth preparation. One of each pair was cured using “soft-start” polymerization, while the other was polymerized with high-intensity halogen light. Restorations were clinically evaluated at baseline, six and 12 months using modified Ryge/USPHS criteria. Although no significant difference ( $p>0.05$ ) was observed between**

**the curing methods, adhesive performance was poor, with a 35% loss of restorations overall.**

### INTRODUCTION

Dentin adhesive resins were originally formulated with separate etchants, primers and adhesives, but have evolved in such a way that all three are combined into a single component in some products. Combined or “self-etching” adhesives are likely to be popular because of the reduced number of steps necessary prior to the placement of resin composite. Laboratory tests indicate that such products are equivalent to multiple-component systems in bond strength to enamel (Hannig, Reinhardt & Bott, 1999) and dentin, (Hannig, Reinhardt & Bott, 2001; Oberlander, Friedl & Schmalz, 2001) and in resistance to microleakage (Gordan & others, 1998). Clinical data are limited to trials of one year or less, but indicate the performance of single-component adhesives to be equivalent to earlier formulations in Class I and II resin composite restorations, (Deneghy & others, 2001) in Class III restorations and in Class V restorations placed in prepared cavities (Muñoz & others, 2001). One study, conducted at the same time as this investigation and with a similar protocol, has shown clinical performance in

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unprepared Class V resin composite restorations over six months to be comparable to an earlier formulation of adhesive with a separate etchant (Wilder & others, 2001).

Visible light polymerization of resin composites has also changed markedly in recent years with the introduction of high-intensity laser, plasma arc and halogen light sources. Although clinical data are lacking, laboratory data suggest that these produce polymerization stresses along resin/tooth interfaces greater in magnitude than those observed with conventional halogen curing lights (Bouschlicher, Vargas & Boyer, 1997) and greater microleakage in Class V resin composites than conventional lights (Brackett, Haisch & Covey, 2000). Concern over polymerization stresses has prompted the development of curing lights that slowly ramp upward in intensity at the beginning of the curing cycle (“soft-start”) and appear in laboratory data to reduce stresses on resin/tooth interfaces due to polymerization relative to ordinary curing lights (Sakaguchi & Berge, 1998; Watts & al Hindi, 1999). Leakage data (Friedl & others, 2000) and one clinical study of Class V polyacid-modified resin composites (Oberlander & others, 1999) have not shown significant advantages relative to ordinary light curing, however. No study to date has made any comparison of ramped versus high-intensity light curing of resin composites.

This study evaluated the clinical efficacy of a single-component dentin adhesive in resin composite restorations of unprepared Class V abrasion/abfraction lesions while also evaluating any differences in the restorations produced by ramped curing versus high-intensity curing of the resin composite.

METHODS AND MATERIALS

The self-etching adhesive chosen for this study was Prompt L-Pop (ESPE Dental AG, Seefeld, Germany), which etches tooth structure because of a methacrylated phosphoric acid ester content. This adhesive was used

with the same manufacturer’s resin composite, Pertac II (ESPE Dental AG), and Elipar TriLight (ESPE Dental AG) curing unit. The two curing methods were attained through the use of different operating modes of the same curing light. The ramped or “soft-start” curing was conducted using the “exponential” setting in which light intensity increases from 0 to 800 mW/cm<sup>2</sup> over the first 15 seconds of the curing cycle, then remains level for 25 seconds. High-intensity curing was 30 seconds at 800 mW/cm<sup>2</sup> using the unit’s “standard” setting. The variable times were selected so that restorations in both groups received approximately equal amounts of radiant energy.

This study was conducted according to the protocol for clinical studies set forth in the 1994 American Dental Association acceptance program for dentin and enamel adhesive materials. Thirty pairs of equivalent-sized cervical erosion/abfraction lesions, primarily in premolar and anterior teeth, were identified in 30 healthy patients under recall in the student clinics of the University of Nebraska Medical Center (UNMC) College of Dentistry. The study was conducted according to the guidelines of the UNMC Institutional Review Board, with informed consent obtained from each patient. The patient median age was 60 years, while age ranged from 42 to 83 years. Each pair of cervical erosion/abfraction lesions received one restoration cured by each method, assigned randomly. Each patient received one pair of restorations. Included in the study were pairs of lesions of varied size and axial depth. The approximate size of each lesion and any sensitivity of the lesion to air from the dental unit were recorded (Table 1) prior to restoration.

One investigator (WWB) placed all restorations. Isolation was accomplished using cotton rolls, with gingival retraction cord placed if necessary. The self-etching primer was applied continuously to tooth surfaces for 15 seconds, then air-thinned according to manufacturers’ instructions. Other than cleaning with

Table 1: Distribution of Curing Method to Teeth Restored; Axial Depth and Preoperative Sensitivity of Erosion/Abfraction Lesions																			
Pertac II/Prompt L-Pop/Standard																			
	Incisors					Canines					Premolars					Molars			
	n	s	o	a	b	c	n	s	o	a	b	c	n	s	o	a	b	c	
Maxillary	1		1		1		5	2	3		2	3	11	8	3		2	9	
Mandibular	1		1		1		0						8	4	4		3	5	
Pertac II/Prompt L-Pop/Exponential																			
	Incisors					Canines					Premolars					Molars			
	n	s	o	a	b	c	n	s	o	a	b	c	n	s	o	a	b	c	
Maxillary	0						8	2	6			8	7	2	5		3	4	
Mandibular	1		1		1		0						13	7	6		4	9	
n = number of teeth involved s = sensitive to air o = insensitive to air a = axial depth < 1 mm b = axial depth 1-2 mm c = axial depth > 2 mm																			

plain pumice and water in a rubber prophylaxis cup, no mechanical preparation or abrasion of tooth surfaces was done. Each restoration was placed in one increment and light-cured by one of the two methods specified above. The curing light was calibrated at the beginning of each half-day clinic session, and light output verified to be 800 mW/cm<sup>2</sup> between patients, using the unit's built-in radiometer and calibration mode. For each restoration, the shade considered the closest match using a Vita shade guide (Vita-Zahnfabrik, Bad Säckingen, Germany) was selected. Restorations were shaped with a plastic instrument prior to light curing, contoured at high speed with ET (Brasseler USA, Savannah, GA 31419, USA) finishing diamonds using

air/water coolant and polished with wet EP abrasive disks (Brasseler USA).

At baseline, six and 12 months, the restorations were clinically evaluated by two other calibrated investigators using modified Ryge/USPHS criteria, (Cvar & Ryge, 1971) which are listed in Table 2. Baseline evaluations were conducted two weeks after placement of the restorations so that dehydration of the teeth during placement did not influence the evaluation of color match. The examiners were unaware of which method of polymerization had been used for any restoration, and any discrepancy between examiners was resolved before the patient was dismissed.

For purposes of statistical analysis, restorations receiving a score of "charlie" in any category were classified as failed restorations. The incidence of failures was analyzed as a pairwise comparison using an exact binomial test.

## RESULTS

The only scores of "charlie" assigned in this study were for retention. Two restorations were lost between the placement appointment and the baseline evaluation that was conducted two weeks later. At the end of one year, 26 pairs of restorations were available for evaluation, a recall rate

Table 2: Modified USPHS Rating System

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

Table 2: Results of Clinical Evaluation for Class V Resin Composites Placed with a Self-Etching Adhesive and Polymerized by Two Methods (%).

Pertac II/Prompt L-Pop/Standard																
Retention**				Sec. Caries			Color Match		Marg. Disc.		Anat. Form		Marg. Adapt.		Surf. Smooth	
n*	alfa	charlie		n*	alfa	charlie	alfa	bravo	alfa	bravo	alfa	bravo	alfa	bravo	alfa	bravo
baseline	30	93	7	28	100	0	18	82	96	4	100	0	21	79	96	4
6 months	29	79	21	23	100	0	43	57	96	4	100	0	22	78	96	4
12 months	26	69	31	17	100	0	41	59	76	24	100	0	24	76	100	0
Pertac II/Prompt L-Pop/Exponential																
Retention**				Sec. Caries			Color Match		Marg. Disc.		Anat. Form		Marg. Adapt.		Surf. Smooth	
n*	alfa	charlie		n*	alfa	charlie	alfa	bravo	alfa	bravo	alfa	bravo	alfa	bravo	alfa	bravo
baseline	30	100	0	30	100	0	27	73	96	4	100	0	23	77	93	7
6 months	29	72	28	23	100	0	39	61	96	4	100	0	22	78	89	11
12 months	26	62	38	17	100	0	53	47	76	24	100	0	18	82	100	0

\* sample size larger for retention than for other criteria because lost restorations unavailable for evaluation.

\*\* cumulative throughout the study.



of 87%. Relative to the standards set forth in the ADA acceptance program, this material would not qualify to receive provisional acceptance with either method of polymerization used. There was no significant difference (exact binomial test;  $p=0.72$ ) between the two curing methods in the number of failed restorations. All retained restorations were clinically sound, with no incidence of secondary caries. Approximately 80% of gingival margins were detectable with an explorer, and a 24% incidence of discoloration without axial penetration was observed. None of the teeth with either retained or lost restorations that exhibited sensitivity to air at the beginning of the study were sensitive at any recall. The anatomic form and surface smoothness of all retained restorations were considered continuous and enamel-like surface, respectively, after one year. The color match of approximately 50% of the restorations was scored as an acceptable mismatch rather than as matching the tooth at six- and 12-month recalls. Complete results are presented in Table 3.

## DISCUSSION

Although this study showed no significant difference between the two curing methods in the number of failed restorations, the relatively poor performance of the adhesive somewhat overshadows the comparison of curing methods. Although concerns related to repeated measures on the same data preclude running a statistical test for each criterion, little difference was observed in marginal adaptation or marginal discoloration of the retained restorations—the two measures most likely to show adverse effects from polymerization shrinkage. “Soft-start” polymerization might also be more advantageous in larger restorations than in the Class V restorations evaluated in this study. If the results of this study are representative, this product would not qualify for acceptance as a dentin and enamel adhesive under the ADA acceptance program.

It is difficult to explain why the results of this study differ so markedly from a previously cited similar study by Wilder & others (2001) which evaluated the same adhesive, albeit with conventional light curing. Personal communication with that group of authors has revealed that their protocol differed from this study in that the tooth surfaces to be restored were lightly abraded with a diamond prior to applying pumice and the adhesive, and that patients in this study were from an older age group. Perhaps this product has been reformulated after the start of production, so that the two studies may have evaluated different formulations.

It may be that older patients' dentin is relatively unresponsive to etching by this self-etching adhesive. This is somewhat supported by the observation that while approximately half the teeth included in the study were sensitive to air prior to restoration, two-

thirds of the lost restorations were from erosion/abfraction lesions that were initially insensitive. The sensitive areas that lost restorations remained insensitive, implying that failure in these teeth occurred at the adhesive/resin interface rather than at the dentin/adhesive interface.

The author who placed the restorations observed that after air-thinning, this product left tooth surfaces less “tacky” than other dentin adhesives, which made adaptation and contouring of the resin composite prior to curing difficult because the resin was too easily displaced from the tooth surface. This resulted in most gingival margins of restorations being detectable with an explorer and may imply that an insufficient amount of resin remained on the tooth surface to form a strong interface with the restorative resin. The authors believe that the relative translucence of this resin composite renders it more suitable for coronal rather than root-surface restorations. This, and the tendency of this resin to be somewhat lighter than the Vita shade guide, accounted for the relatively high percentage of “bravo” scores for shade matching. Better shade matching would likely occur by using either the manufacturer's shade guide or a direct shade matching.

## CONCLUSIONS

No significant difference in the number of lost restorations was observed between the two curing methods employed. The self-etching dentin adhesive proved relatively ineffective in retention of resin composite restorations of unprepared cervical erosion/abfraction lesions when used according to the protocol of this study.

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# Clinical Evaluation of a Polyacid-Modified Resin Composite (Dyract) in Class III Cavities: Three-Year Results

M Demirci • H Ersev • M Üçok

## Clinical Relevance

Dyract exhibited significant marginal discoloration after three-year clinical performance in Class III cavities.

## SUMMARY

This study evaluated the three-year clinical performance of a polyacid-modified resin composite material, Dyract (DeTrey/Dentsply, Konstanz, Germany), in Class III cavities.

Sixty-two Class III cavities in 30 patients were restored with Dyract. Restorations were clinically evaluated at baseline, one-, two- and three-year recalls according to the modified Ryge criteria by two experienced, calibrated examiners.

After three years, the retention rate was 96.7%. At the one-year interval, one restoration had to be replaced due to sensitivity. At the two-year recall, one restoration, with a caries lesion adjacent to its margin, was clinically unacceptable and had to be replaced. Except for these two

restorations, all other restorations were clinically acceptable in regard to color match, marginal discoloration, wear or loss of anatomical form, caries, marginal adaptation and surface texture after three years. At the end of three years, marginal discoloration was statistically significant ( $p=0.017$ ) but did not require replacement of any of the restorations.

Dyract exhibited significant marginal discoloration after three-year clinical performance in Class III cavities.

## INTRODUCTION

Glass ionomer cements became popular due to their fluoride ion releasing capacity over a prolonged period (Swartz, Phillips & Clark, 1984) and their adhesive bonding to enamel and dentin (Powis & others, 1982). Their limitations, low mechanical properties (van Dijken, 1996) and inconvenient setting characteristics were first addressed through resin-modified glass ionomer cements in which the set is accelerated by the presence of light-cured resins that are cross-linked to polyacids in the cement liquid (Brackett & others, 2001).

More recently, to overcome the technique-sensitive mixing and handling properties of resin-modified glass ionomer cements (Welbury & others, 2000) that were

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basically formed by adding methacrylate derivatives to the glass ionomer formula, polyacid-modified resin composites, also known as compomers, were developed by adding acidic polymers to the original methacrylate resin matrix (Gladys & others, 1999). Compomers contain acid-decomposable aluminosilicate glass and acidic, polymerizable monomers substituting the polyalkenoic acid polymers (Welbury & others, 2000), and they are one-component materials that do not require mixing, in contrast to glass ionomer and resin-modified glass ionomer cements. Since they also contain no water in their formulation, an acid-base reaction does not occur during their setting process, unlike the typical setting process of glass ionomer cements (García-Godoy, 2000). The dominant setting reaction of compomers is resinous photopolymerization (Welbury & others, 2000). The resin matrix in Dyract, an example of polyacid-modified resin composites is TCB, which has two methacrylate groups and two carboxylate groups. Thus, not only can the monomer cross-link when initiated through radical polymerization, it can also undergo an acid-base reaction with the filler and reactive silicate glass particles (72%) containing fluoride if water is absorbed from the tooth and the oral environment (Gladys & others, 1997). As a result of this reaction, fluoride is released (Berg, 1998; Hammesfahr, 1994; Tyas, 1998), but at a lower degree than that released by glass ionomer or resin-modified glass ionomer cements (Rothwell, Anstice & Pearson, 1998; Shaw, Carrick & McCabe, 1998; Yip & Smales, 2000).

Practitioners have seen the multi-step application of dentin bonding agents for composites as a clinical inconvenience. Like composite resins, compomers cannot bond to dentin and enamel, therefore, they also require the use of a bonding system (Çehreli & Altay, 2000). Bonding Dyract to tooth structure is achieved by means of PSA Primer/Adhesive that is applied to clean, unetched, enamel and dentin (Tyas, 2000). This self-etching system (Berg, 1998) uses an acetone solvent with PENTA (dipentaerythritolpentacrylate phosphoric acid ester) as the primary adhesion promoter (Barkmeier, Hammesfahr & Latta, 1999). It replaces the conditioner, primer and adhesive resin, thus providing great ease of application (Roeters & others, 1998).

Dyract seems to offer an advantage over composite resins since its suggested application technique eliminates the acid-etching procedure. Due to this ease of application and its handling characteristics, Dyract has been recommended as a restorative material in primary teeth (Mass, Gordon & Fuks, 1999). In primary molar restorations, Dyract was reported to provide satisfactory results and low failure rates after three years (Marks & others, 1999; Roeters & others, 1998). In another study, however, it showed significant color change and was far less satisfactory in esthetic performance than conventional resin composites after 18 months (Gladys & oth-

ers, 1999). Dyract is also indicated for definitive Class III and cervical restorations (Di Lenarda, Cadenaro & De Stefano Dorigo, 2000). It is reported that Dyract had high clinical performance in cervical restorations (Folwaczny & others, 2000; Abdalla & Alhadainy, 1997; Abdalla, Alhadainy & García-Godoy, 1997; Tyas, 1998). However, very little data is available on the long-term clinical performance of Dyract as an anterior restorative material. van Dijken has reported low levels of failure in Class III cavities after three and five years (van Dijken, 1996; van Dijken, 1999).

The results of a one-year study of the clinical performance of Dyract restorations in Class III cavities have previously been reported (Demirci & Üçok, 2001). This paper presents the results of the three-year follow-up.

## METHODS AND MATERIALS

This clinical study was performed at the Department of Conservative Dentistry, Istanbul University. Sixty-two Class III carious lesions in 30 patients were selected. The average patient age was 29.9 years (range 13-78 years). All cavities were prepared and restorations placed by the same operator. Cavity preparation was limited to the removal of caries. The cervical margins were at/or incisal to the cemento-enamel junction and no incisal edges were involved in the preparations. After the cavities were prepared, the manufacturer's instructions were closely adhered to regarding cavity treatment and placement of the restorative material. Isolation was achieved with cotton rolls and saliva ejectors. After air drying the cavities, PSA Primer/Adhesive was applied in two coats. The first coat was left on for 30 seconds, gently air dried and cured for 10 seconds. The second coat was immediately placed, air dried and cured for 10 seconds. Color matching was done with a Vita shade guide (Vita Zahnfabrik, Bad Sackingen, Germany) and the material, delivered in compules, was injected into the cavities. With restoration depths of more than 2 mm, the material was applied by using the incremental technique. The first material layer was applied on the pulpal walls and light cured for 40 seconds. Then, a second layer was applied and light cured for an additional 40 seconds. In shallow cavities, the material was placed in a single increment and light cured for 40 seconds from both the buccal and palatal sides. The intensity of the curing light (XL3000, 3M Dental Products, St Paul, MN 55144, USA) was measured before and after application and the light output was never below 450 Mw/cm<sup>2</sup>. Following the removal of excess material with fine diamond burs and strips, the restorations were finished and polished with Sof-Lex abrasive disks (3M Dental Products).

The restorations were evaluated by two experienced, calibrated examiners according to the modified Ryge

Table 1: Direct Clinical Evaluation Criteria (Modified Ryge Criteria)

Rating	Aspect	Method
<b>Retention</b>		
Alpha (A)	No loss of the restoration.	Visual inspection
Charlie (C)	Loss of the restoration.	Visual inspection
<b>Color Match</b>		
Alpha (A)	There is no a mismatch in color, shade and/or translucency between the restoration and the adjacent tooth structure.	Visual inspection
Bravo (B)	There is a mismatch in color, shade and/or translucency between the restoration and the adjacent tooth structure, but the mismatch is within the normal range of tooth color, shade and/or translucency.	Visual inspection
Charlie (C)	The mismatch is between restoration and adjacent tooth structure outside the normal range of tooth color, shade and/or translucency.	Visual inspection
<b>Cavosurface Marginal Discoloration</b>		
Alpha (A)	There is no discoloration anywhere on the margin between the restoration and the tooth structure.	Visual inspection
Bravo (B)	There is discoloration anywhere on the margin between the restoration and the tooth structure, but the discoloration has not penetrated along the margin of the restorative material in a pulpal direction and can be polished away.	Visual inspection
Charlie (C)	The discoloration has penetrated along the margin of the restorative material in a pulpal direction.	Visual inspection
<b>Wear/Anatomic Form</b>		
Alpha (A)	The restoration is not under-contoured, that is, the restorative material is not discontinuous with existing anatomic form.	Visual inspection and explorer
Bravo (B)	The restoration is under-contoured, that is, the restorative material is discontinuous with existing anatomic form, but sufficient restorative material is not missing so as to expose the dentin or base.	Visual inspection and explorer
Charlie (C)	Sufficient restorative material is missing so as to expose the dentin or base.	Visual inspection
<b>Caries</b>		
Alpha (A)	There is no evidence of caries contiguous with the margin of the restoration.	Visual inspection
Bravo (B)	There is evidence of caries contiguous with the margin of the restoration.	Visual inspection
<b>Marginal Adaptation</b>		
Alpha (A)	There is no visible evidence of a crevice along the margin into which the explorer will penetrate.	Visual inspection and explorer
Bravo (B)	There is visible evidence of a crevice along the margin into which the explorer will penetrate. The dentin or base is not exposed.	Visual inspection and explorer
Charlie (C)	There is visible evidence of a crevice along the margin into which the explorer will penetrate. The dentin or base is exposed.	Visual inspection and explorer
Delta (D)	The restoration is fractured or missing in part or <i>in toto</i> .	Visual inspection and explorer
<b>Surface Texture</b>		
Alpha (A)	Surface of restoration is smooth.	Explorer
Bravo (B)	Surface of restoration is slightly rough or pitted, can be refinished.	Explorer
Charlie (C)	Surface deeply pitted, irregular grooves (not related to anatomy), cannot be refinished.	Explorer
Delta (D)	Surface is fractured or flaking.	Explorer

criteria (Ryge, 1980) (Table 1). Inter- and intra-examiner agreement for the evaluated criteria was 91%. At baseline, one-, two- and three-year recalls, color match, wear or loss of anatomical form, marginal discoloration, caries, marginal adaptation and surface texture were evaluated.

Data gathered by interviewing the patient provided information about the hypersensitivity status of the restored teeth.

According to the modified Ryge criteria, Alpha (A) indicates the clinically ideal situation, Bravo (B) indicates a clinically acceptable situation except for caries, which requires the replacement of the restoration,

Charlie (C) is a clinically unacceptable situation where the replacement of the restoration is required and Delta (D) indicates a situation where the restoration is unacceptable due to fracture, mobility or loss and has to be replaced. Data obtained by evaluating each assessment criteria were statistically analyzed using the Friedman test for comparison of years followed by the Wilcoxon matched pairs test (Bonferroni corrected) for multiple comparisons.

## RESULTS

All patients returned for one- and two-year recalls and the recall rate for patients was 100%. At the end of three years one patient dropped-out (two restorations), bringing the recall rate down to 96.7%.

During the first year one restoration was lost to endodontic treatment and the retention rate was 98.39%. After two years one restoration had to be replaced due to a caries lesion adjacent to its margin and the retention rate was therefore 98.36%. During the third-year, no restorations were lost. During the three-year evaluation period, only two restorations were lost and the retention rate was 96.7%.

Direct clinical evaluation rates at baseline, one-, two- and three-year recalls are given in Table 2. During the first year one restoration was clinically unacceptable due to postoperative sensitivity and had to be replaced following endodontic treatment. After two years, one restoration was clinically unacceptable and had to be replaced due to a caries lesion adjacent to its margin. At the end of the three-year period, except for these two restorations, there were no other clinically unacceptable restorations in regard to color match, marginal discoloration, wear or loss of anatomical form, caries, marginal adaptation and surface texture. However, after three years, marginal discoloration in the restorations was statistically significant ( $p=0.017$ ) but the discoloration was clinically acceptable (Bravo) and did not require replacement of any of the restorations (Table 2).

In spite of the statistically significant increase in marginal discoloration at the end of three years when compared with the baseline, differences between the one- and two-year evaluation rates as well as the one-year and baseline evaluation rates were not statistically significant.

Statistical analysis revealed no significant difference among each evaluation period in regard to color match, wear or loss of anatomical form, caries, marginal adaptation and surface texture after three years (Table 2).

## DISCUSSION

During three-year evaluation period only two restorations were lost and retention was 96.7%. As stated in the previous paper's one-year results (Demirci & Üçok, 2001), one restoration was lost due to acute apical abscess formation two months after baseline. After two years, another restoration had to be replaced due to a caries lesion adjacent to its margin. There have been few published clinical studies of Dyract in Class III cavities (van Dijken, 1996; van Dijken, 1999). In the present study, the retention rate after three years agreed with the findings of van Dijken's (1996) study, in which the replacement of only two restorations, one lost due to heavy bruxism and the other from secondary caries within three years, was reported. The retention of restorations depends on the effectiveness of the bond at Dyract and both the enamel and dentin interface. Dyract's adhesive agent (PSA Primer/Adhesive) is an acid primer and HEMA-like resin monomer (Ferrari & others, 1998) that contains acetone. Acetone wets the enamel surface, penetrates the dentin, and by diffusing into the dentinal tubules, forms a layer of interdiffusion between the surface-treated dentin and the adhesive system (Abate & others, 1997; Dentsply DeTrey-DeDent, 1994; Ferrari & others, 1998). Furthermore, the hydrophylic phosphate groups in the PENTA molecule, which is the active ingredient of PSA Primer/Adhesive, reacts with the tooth surface and

	Retention		Color Match			Marginal Discoloration			Wear/Anatomic Form			Caries		Marginal Adaptation				Surface Texture			
	A	C	A	B	C	A	B	C	A	B	C	A	B	A	B	C	D	A	B	C	D
Baseline <i>n</i> =62	100	0	95.2	4.8	0	100	0	0	100	100	0	100	0	96.7	3.3	0	0	100	0	0	0
1 year <i>n</i> =61	98.39	1.61	85.2	14.8	0	90.2	9.8	0	91.8	8.2	0	100	0	91.8	8.2	0	0	95.1	4.9	0	0
2 year <i>n</i> =60	98.36	1.64	80	20	0	81.7	18.3	0	86.7	13.3	0	98.4	1.6	90	10	0	0	93.3	6.7	0	0
3 year <i>n</i> =58	100	0	77.6	22.4	0	63.8	36.2	0	82.8	17.2	0	100	0	86.2	13.8	0	0	89.7	10.3	0	0
<i>p</i>	<i>p</i> =1.00 (ns)		<i>p</i> =0.509 (NS)			<i>p</i> =0.017 (S)			<i>p</i> =0.486 (NS)			<i>p</i> =1.00 (NS)		<i>p</i> =0.855 (NS)				<i>p</i> =0.855 (NS)			
S= significant (p<0.5) NS= not significant																					



forms an ionic bond with the calcium ions of the hydroxypapatite (Abate & others, 1997; Çehreli & Altay, 2000; Dentsply DeTrey-DeDent, 1994; Toledano & others, 1999; Tyas, 1998; Yap, Lim & Neo, 1995). Moreover, carboxylic (COOH) groups of TCB resin in Dyract enable the material to be self-adhesive (Hickel & others, 1998; Hse, Leung & Wei, 1999). In addition to the ionic bonding of the material to tooth structure, macromechanical retention obtained from the cavity preparation might contribute to this high retention rate. However, high percentages of marginal discoloration observed in this study are most likely due to deterioration of the bond between the material and enamel. Per the manufacturer's instructions (Dentsply DeTrey-DeDent, 1994), no acid etching of the cavity margins was carried out prior to restoration with Dyract. While enamel is harder to etch due to its substantially more inorganic composition than dentin (Ferrari & others, 1998), the self-etching primer system could only produce more shallow etch patterns than conventional acid etchant (Perdigão & others, 1997). Phosphoric acid conditioning before applying the self-etching primer system had produced improved compomer bond strength to enamel (Abate & others, 1997) and therefore has been recommended by several authors (Abate & others, 1997; Ferrari & others, 1998).

There was no statistically significant difference in color match after three years. However, statistically significant color change at the end of the first year was previously reported (Demirci & Uçok, 2001). At two-year recall 80% of the restorations had ideal color match (Alpha) and 20% had clinically acceptable color match (Bravo), a 5.2% increase with respect to one-year recall. After three years, the rate of ideal color match (Alpha) was 77.6%. On the other hand, clinically acceptable color match increased by 2.4% at two years to 22.4%. This shows that color change was more pronounced within the first year (Demirci & Uçok, 2001), but the change in color match began to decrease between one and two years. This decrease was more pronounced at the third year. These results are consistent with those of van Dijken (1996), who reported that a slight mismatch in color was detected in 19.6% of the restorations and an obvious mismatch in 2% after three years. van Dijken (1996) stated that the hybrid materials' high content of hydrophylic monomer causes a high rate of water sorption, which results in a color change. It is also reported that hydrophylic methacrylate components of Dyract are susceptible to water uptake and water-based staining (Mount, 1999). The manufacturer claimed that after the light curing process, Dyract begins to absorb water in a moist environment and, depending on the volume of the restoration, this may continue for a couple of months and ends when the entire filling material reaches its maximum water content (Dentsply DeTrey-DeDent, 1994). Cattani-Lorente

& others (1999) reported that the water absorption of Dyract continued for at least three months. It is also claimed that Dyract can absorb water up to its 3% w/w (Dentsply DeTrey-DeDent, 1994). In addition, the absorption of water during the days and weeks after photopolymerization starts an acid-base reaction with the carboxyl groups in the TCB molecule of the material and the setting continues (Berg, 1998; Dentsply DeTrey-DeDent, 1994; Tyas, 2000). These factors might explain the color change observed in this study, especially within the first year. Gladys and others (1999) found that the color changes of Dyract occurred in the first six months and were less pronounced in the following 12 months of their study. They observed color changes ranging from "excellent color match" to "slight color mismatch" by the end of the first 12 months. Compared with their observations, the color changes observed in this study ranged from "ideal color match (Alpha)" to "clinically acceptable color match" and were more pronounced after one year. This difference might be due to the different cavity types selected in the two studies. In the present study, there was a substantial drop in color change during the second year, which was more pronounced within the third year. The color change detected in the study by Gladys and others (1999) showed a tendency to decrease with time, which is similar to our observations. However, it is difficult to make an exact comparison between these two studies because of the differences in evaluation periods. Roeters and others (1998), in contrast to these results, observed an improvement in the color match of Dyract in primary Class I and II restorations after one year. However, they also reported a pronounced color change from "ideal color match" (Alpha) towards "clinically acceptable color match" (Bravo) at the end of three years. At the end of the three-year evaluation period although none of the restorations was clinically unacceptable in regard to color match, it should be noted that esthetic quality is an important feature for an anterior restorative system (van Dijken, 1999). It has been shown that composite resins exhibited significantly better color match than resin-modified glass ionomers and polyacid-modified resin composites (van Dijken, 1996; Vargas & others, 2001; Yap, Tan & Bhole 1997) and resin composite should be the material of choice when an excellent color match is preferred (van Dijken, 1999).

At one-year recall, marginal discoloration was observed only in 9.8% of the restorations and was only located on an unspecific point on the enamel surrounding the restoration. The discoloration could be polished away, indicating that it did not progress towards the pulp. This was a clinically acceptable situation. However, by the end of the three years, the same type of marginal discoloration (Bravo) showed a statistically significant increase up to 36.2% ( $p=0.017$ ), causing con-

cern regarding deterioration of the bond between the material and the tooth structure (Tyas, 1998). In accordance with the results of the present study, several authors reported finding marginal discoloration with Dyract (Çehreli & Altay, 2000; van Dijken, 1996; Tyas, 2000). Çehreli and Altay (2000) reported that marginal discoloration affected 9.5% and 16.4% of the restorations in minimally-invasive occlusal cavities at 12 and 24 months, respectively, and further discoloration could have been observed if occlusal wear at the margins had not occurred. After three years van Dijken (1996) found significantly higher marginal discoloration scores for Dyract and a resin-modified glass ionomer cement than a resin composite in Class III restorations. At the end of the five-year observation period, the same author (van Dijken, 1999) reported that the marginal discoloration rate had increased to 40.8% from 33.3%, which was observed at the three-year recall. Tyas (2000) observed some degree of severe marginal discoloration in 16 of the 36 non-carious cervical restorations after three years and also reported that restorations showing the most marginal discoloration did so at the enamel margin, reflecting the comparative enamel and dentin bond strengths (Tyas, 1998) that is given by the manufacturer as less to enamel (9.6 MPa) than to dentin (14.5 MPa) (Dentsply DeTrey-DeDent, 1994). Although the manufacturer recommends applying the material without acid-etching the enamel, the bond strength should be adequate with the use of PSA Prime/Adhesive alone (Dentsply DeTrey-DeDent, 1994), Tyas (1998) emphasized that it would be reasonable to expect a high bond strength to etched enamel since Dyract is essentially a resin composite. Dyract's resin content is approximately 28% (Toledano & others, 1999) and its initial volumetric shrinkage was reported to be 2.7% (Miyazaki, Fukuishi & Onose, 1999). Attention should also focus on all resin-containing materials undergoing some degree of polymerization shrinkage (Campanella & Meiers, 1999). If the shrinkage is great enough and the resulting contraction stresses exceed the strength of the compomer-tooth bond, then bond failure occurs (Asmussen & Jorgensen, 1972), resulting in marginal discrepancies that lead to microleakage, marginal discoloration, secondary caries and sensitivity (Davidson, 1986; Feilzer, de Gee & Davidson, 1990; Kaplan & others, 1992). The results of the histopathological study conducted on monkey teeth by Tarim & others (1997) have shown pulpal inflammatory response due to bacterial penetration, and they concluded that this pulpal reaction is possibly a result of poor marginal adaptation of Dyract to tooth structure in Class V cavities. Several studies have shown a higher bond strength and more intimate marginal adaptation of compomers when the enamel was acid etched (Abate & others, 1997; Yap & others, 1995; El Kalla & García-Godoy, 2000). Di Lenarda & others (2000) observed marginal discoloration in 40% of the non-etched and 16.7% of the orthophosphoric acid-etched cervical restora-

tions after 48 months with a statistically significant difference between these two groups, and they concluded that Dyract's marginal adaptation is enhanced by etching. Ferrari and others (1998) also concluded that treatment with phosphoric acid improves the sealing ability of Dyract based on the results of their *in vivo* study conducted on Class V restorations. Tyas (2000) stated that although Dyract has now been superseded by Dyract AP with a finer filler particle, a cross-linking resin and an optimized initiator system, the manufacturers should still consider specifying mandatory enamel etching. His contention is supported by the findings of Luo & others (2000), who noted a gradual discoloration of the restoration margin during the one-year period, in which they evaluated the clinical performance of Dyract AP in Class I and II cavities and reported that marginal adaptation and sealing ability of Dyract had improved, but the marginal discoloration remained problematic and needs to be improved. One of the unique features of the compomer's adhesion to enamel is the omission of acid etching (Yap & others, 1995) to simplify the bonding procedure. However, without acid etching, the finding of marginal discoloration has been reported in a number of studies (Çehreli & Altay, 2000; van Dijken, 1996; Tyas, 2000). Several authors noted that etching should be performed (Abate & others, 1997; Di Lenarda & others, 2000; El-Kalla & García-Godoy, 2000), so that one of the advantages of compomers, their placement without the need for etching, would be negated. Marginal discoloration seriously deteriorates the appearance of restorations and may lead to accelerated replacement due to esthetic concerns. To meet the esthetic demands, composite resin, which has been shown to be esthetically superior to resin-modified glass ionomer and polyacid-modified resin composite due to its better adaptation to enamel (Yap & others, 1995), should be considered in anterior restorations.

There was no statistically significant difference in regard to wear/anatomic form after three years for 82.8% of the restorations. They were clinically ideal (Alpha) in regard to wear and loss of anatomical form, while only 17.2% had a Bravo rating. That is, they were still clinically acceptable. Wear was limited to the restorative material and did not extend to sound tooth structure.

After two years, secondary caries was detected in only one restoration that had to be replaced. Except for this restoration with caries lesions adjacent to its margin, no other cases of caries were observed after three years.

Statistical analysis showed no significant difference in marginal adaptation and surface texture. After three years, 86.2% of the restorations had ideal marginal adaptation (Alpha). Only 13.8% of the restorations had a crevice along the margins (Bravo). But in the restorations with a crevice, dentin was not exposed, and this

was clinically acceptable. At the three-year recall, in regard to surface texture, 10.3% of the restorations were slightly pitted and had rough surfaces (Bravo) that could be restored by repolishing.

Except for the only case of postoperative sensitivity that was observed during the first year, no other cases of sensitivity were reported after three years.

### CONCLUSIONS

1. Except for one case of sensitivity reported during the first year, there were no cases of sensitivity after three years.
2. There was no statistically significant difference in color match after three years. Color change showed a decrease between one and two years and this decrease was more pronounced within the third year, compared to one-year results.
3. After three years, marginal discoloration in restorations was statistically significant ( $p=0.017$ ). There were 36.2% of the restorations with marginal discolorations but the discolorations were clinically acceptable (Bravo).
4. Statistical analysis showed no significant difference in regard to color match, wear or loss of anatomical form, caries, marginal adaptation and surface texture at the end of three years.
5. Dyract exhibited significant marginal discoloration after three-year clinical performance in Class III cavities.

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# Relating Visual and Radiographic Ranked Scoring Systems for Occlusal Caries Detection to Histological and Microbiological Evidence

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## Clinical Relevance

A ranked visual and a radiographic scoring system for occlusal caries diagnosis can be used to predict the level of infection of dentin.

## SUMMARY

This study compared a visual ranked scoring system and a radiographic ranked scoring system for occlusal caries detection with the level of infection of dentin. Seventy-five third-molars, designated for extraction, were professionally cleaned. Caries was scored according to a visual ranked scoring system at a selected site in the groove-fossa system. Radiographs of the teeth were available and caries was recorded along a five-point ranked scoring system. Each

tooth was extracted and hemi-sectioned through the investigation site under aseptic conditions. A burful of dentin was removed from the EDJ of one of the section faces and these samples were processed to establish the level of dentin infection. The depth of the lesion was assessed on the other section face using a five-point ranked histological scoring system. A strong relationship was observed between the histological lesion depth and visual score ( $r_s=0.93$ ) while a moderate relationship was seen between lesion depth and radiographic scores ( $r_s=0.77$ ). The dentin from teeth with cavities exposing dentin was heavily infected. The dentin from teeth with microcavities or grey discoloration of the dentin was less infected than the lesions with frank cavitation (score 4) ( $p<0.05$ , t-test), but more infected than the initial lesions ( $p<0.05$ , t-test). The latter lesions showed minimal infection. A similar tendency was seen with respect to increasing radiographic scores and the level of infection of the dentin.

## INTRODUCTION

Occlusal caries accounts for the majority of carious tooth surfaces in children and young adults. However,

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there is a perceived difficulty with its accurate diagnosis and logical management (Ricketts & others 1997). As such, there have been many attempts at improving the accuracy and reproducibility of diagnosis by various new techniques such as fibre optic transillumination, probing, radiography, electronic caries detection, ultrasound (Kidd, Ricketts & Pitts 1993) and more recently, a laser fluorescence system (Lussi & others 1999, Attrill & Ashley 2001).

Each method incurs an additional purchase cost for the dentist, however, the authors have shown that it is possible to detect occlusal carious lesions and assess their depth and activity based on a simple clinical and radiographic examination (Ekstrand, Ricketts & Kidd, 1997; Ekstrand & others, 1998). Logical management of carious lesions depends on these assessments. For instance, an arrested lesion requires no treatment, whereas active caries requires preventive and therapeutic treatment and some lesions also need operative intervention. It is generally considered that once an occlusal lesion is cavitated, the area is no longer cleansable, and the dentin is infected. At this stage operative treatment is a necessary part of managing the carious process. While information is available on the relationship between the ranked visual criteria, appearance on radiograph and lesion depth, the authors do not know how the visual and radiographic criteria relate to the level of infection of the dentin. The latter may be important in deciding the threshold between applying sealants or operative intervention.

Thus, the aim of this study is to relate the ranked visual and radiographic scoring systems to the level of infection of dentin.

METHODS AND MATERIALS

The Sample

The study was carried out on 75 third molars scheduled for extraction by dentists other than the authors.

Visual and Radiographic Examination of the Teeth

Just prior to extraction, the occlusal surface of the tooth to be extracted was thoroughly cleaned with a rotating bristle brush and copious water. The most demineralized site in the groove-fossa system was selected (primarily the central fossa) by one examiner, and the stage of caries was characterized using the visual scoring system presented in Table 1 and Figure 1.

An intra-oral periapical radiograph was taken, using an Eggen film holder (Kwik-Bite, Hawe-Neos, Switzerland), as part of the normal assessment prior to extraction. The radiographs were examined by the

Table1: The Visual Classification System	
0	No or slight change in enamel translucency after prolonged air drying (>5s) (Figure 1a).
1	Opacity (Figure 1b) or discoloration (Figure 1c) hardly visible on a wet surface, but distinctly visible after air drying.
2	Opacity (Figure 1d) or discoloration (Figure 1e) distinctly visible without air drying.
3	Localized enamel breakdown in opaque or discolored enamel (Figure 1f) and/or greyish discoloration from the underlying dentin (Figure 1g).
4	Cavitation in opaque or discolored enamel exposing the dentin (Figure 1h).

Table 2: Criteria Used in the Radiographic Examination (Figure 2)	
0	No radiolucency visible.
1	Radiolucency visible in the enamel.
2	Radiolucency visible in the dentin but restricted to the outer third of the dentin.
3	Radiolucency extending to the middle third of dentin.
4	Radiolucency in the pulpal third of dentin.

Table 3: Criteria Used in the Histological Examination	
0	No enamel demineralization or a narrow surface zone of opacity (edge phenomenon).
1	Enamel demineralization (opacity) limited to the outer 50% of the enamel layer.
2	Demineralization (brown discoloration) involving between 50% of the enamel and 1/3 of the dentin.
3	Demineralization (brown discoloration) involving the middle third of the dentin.
4	Demineralization (brown discoloration) involving the inner third of dentin.

same examiner and the caries recorded using the radiographic scoring system presented in Table 2 and Figure 2.

Histological Examination

After extraction each tooth was immediately hemi-sectioned through the investigation site using an aseptic technique (Mejàre, Mejàre & Edwardsson, 1979). Initially, the root component was removed horizontally at the enamel cementum junction (ECJ), exposing the dentin and the pulp chamber. A superficial groove was made with a sterile disc through the investigation site occlusally on the buccal and lingual enamel and cervically on the exposed dentin. Care was taken so that the occlusal aspect of this groove did not reach the enamel dentin junction (EDJ). The tooth was then hemi-sectioned using a sterile chisel in the groove.

A sample for microbiological examination was taken from one section face by dipping a sterile round steel bur into reduced transport fluid (RTF) (Syed & Loesche, 1972) and using it to gather a “burful of dentin” (Kidd, Joyston-Bechal & Beighton, 1993) just



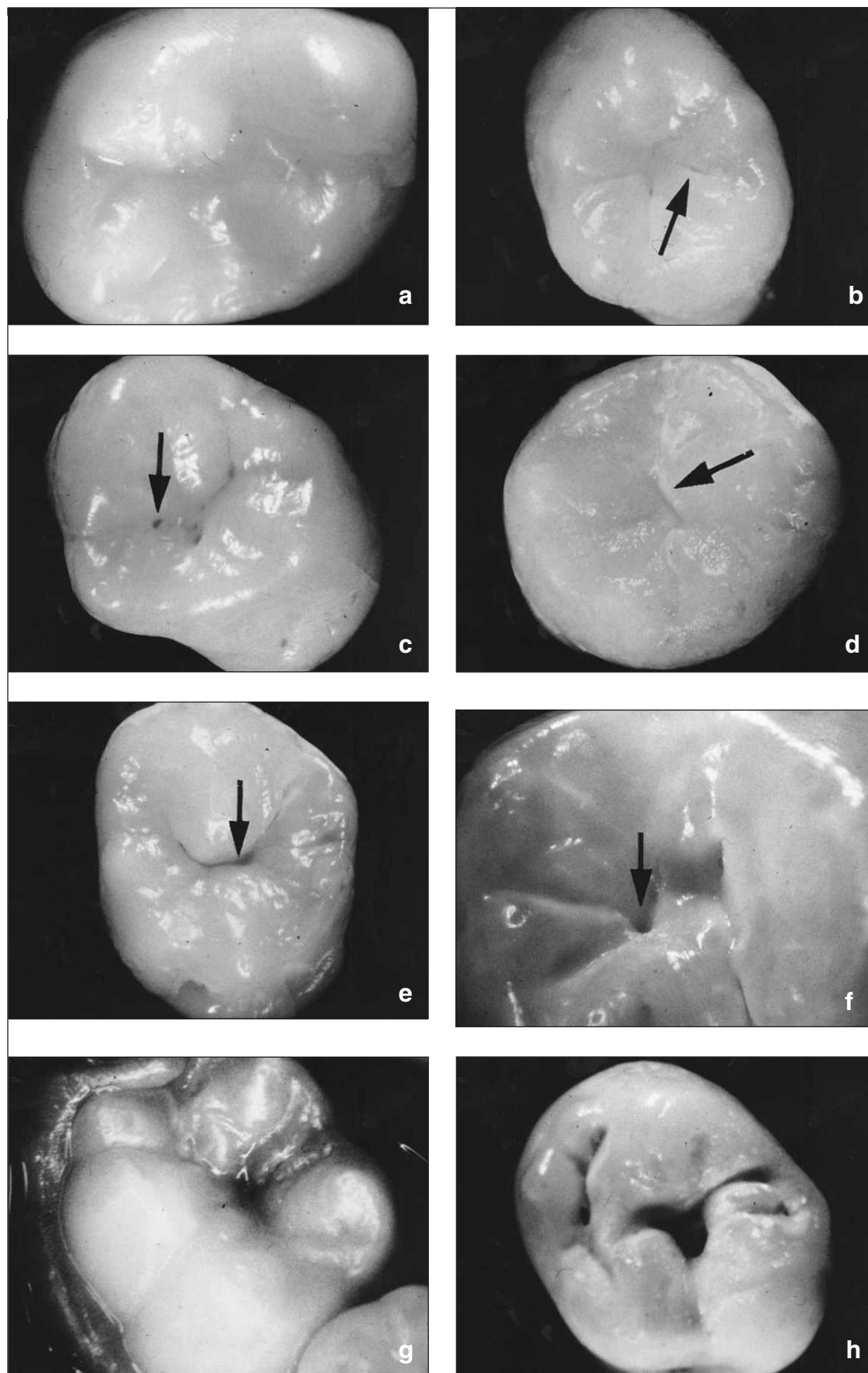


Figure 1 a-h: Clinical photographs illustrate the visual classification system described in Table 1. In Figure 1b, the tooth is wet. In all other figures the teeth are dry. Reproduced from *Dental Update* with permission of George Warman Publications (UK) Ltd.

below the EDJ. The bur was placed in a vial of RTF that was immediately taken to the laboratory. The other section face was now photographed in the stereomicroscope at  $\times 10$  magnification. The depth of the lesion on the section face was recorded using the criteria in Table 3.

#### *Microbiological Technique*

The samples were vortex mixed with sterile glass beads for 60 seconds to evenly disperse the sample. They were then ten-fold serially diluted in RTF before 0.1 ml was plated onto tryptic soy agar (TSA, Difco Laboratories, Detroit, MI 48232, USA) supplemented with horse blood (5%) haemin (5 mg/ml) and menadine (0.5 mg/ml) for growth of total colony-forming units (CFU). Each colony-forming unit is produced from an organism in the



Figure 2. A bitewing radiograph of extracted molar teeth showing the various radiographic appearances described in Table 2. The upper right second molar shows a sound occlusal surface, an enamel lesion would not be visible radiographically. The lower right first molar has a radiolucency confined to the outer third of dentin, the upper right first molar shows a radiolucency extending into the middle third of dentin and the lower right second molar shows a radiolucency extending to the pulpal third of dentin.

original sample and thus the CFU count represents the number of bacteria or level of infection of the dentin.

#### *Reproducibility of the Radiographic and Histological Scores*

Due to the design of the study, the examiner could not re-examine the teeth visually *in vivo*. In contrast, repeated examination of the radiographs was carried out after a week had elapsed. The histological depth of the lesion was reassessed using the photographs at least one week after the initial examination.

#### *Statistical Analysis*

The reproducibility of the radiographic and histological scores were assessed by repeating these readings for the whole sample, then noting the percentage of scores achieving perfect agreement.

The Spearman ranked correlation coefficient was used to investigate whether there was any relationship between the visual, radiographic and histological scores. For radiographic and histological scores, the first set of readings was used.

Differences in the level of infection of the dentin associated with each visual and radiographic classification were assessed using Student's *t* test.

## RESULTS

#### *Material Available*

Seventy-five third molars from 75 patients were used, and of these, five were completely unerupted. Material for microbiological investigation was gathered from 74 teeth; one tooth was excluded because the aseptic conditions failed. Four radiographs were unreadable.

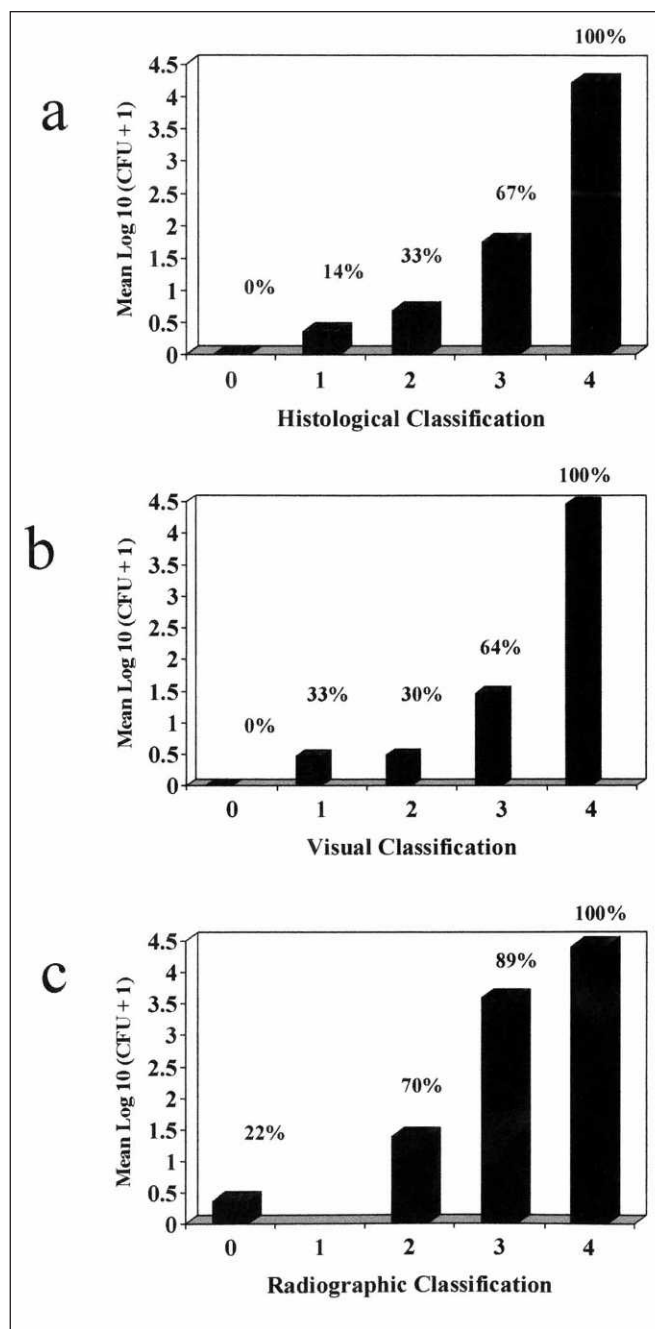


Figure 3. Relationship between the microbiological data (how heavily infected the dentin samples were) and the histological (a), visual (b) and radiographic (c) scores. The proportion of dentin samples that were infected in each group is also shown.

Organization of the data by the ranked visual scores showed there were approximately equal numbers of sound/early lesions (scores 0-2) and deeper lesions (3 and 4).

#### *Clinical, Radiographic and Histological Assessment*

A 5% disagreement between the two sets of radiographic and histological scores was noted. Strong rela-

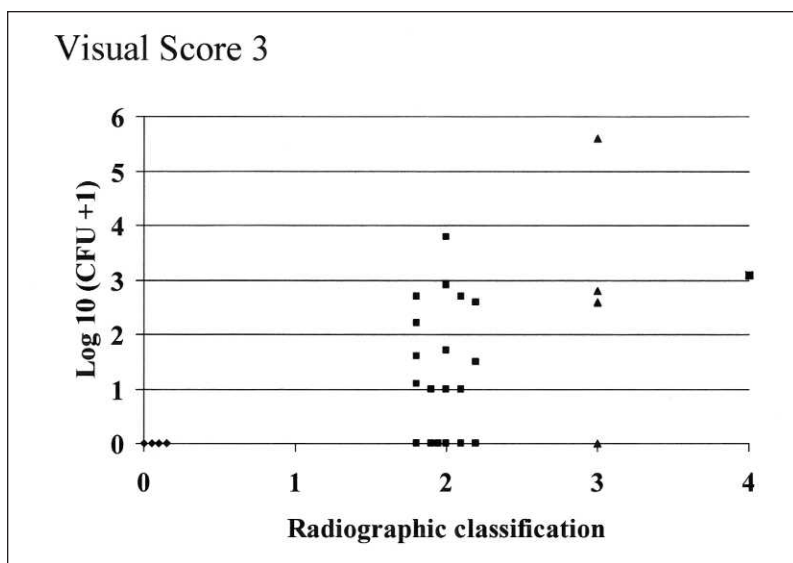


Figure 4. A scattergram shows the relationships of the radiographic scores to the level of infection of the dentin for visual score 3.

tionships were observed between the histological lesion depth and the visual criteria ( $r_s=0.93$ ). There was a moderate relationship between the histological and radiographic criteria ( $r_s=0.77$ ).

### *Relationships Between Histological, Visual and Radiographic Scoring and Level of Infection of the Dentin*

Figure 3a-c shows the relationships between the ranked histological, visual and radiographic scoring systems and the level of infection of the dentin, that is, how heavily infected the dentin sample was (total CFU). It is obvious from Figure 3a that as the depth of the lesion increased, as did the level of infection of the dentin. Student's *t* tests showed a significant difference between histological scores 2 and 3 ( $p<0.05$ ), and between 3 and 4 ( $p<0.05$ ).

Figure 3b pertains to the visual scores. There was no statistically significant difference in the level of infection of the dentin in teeth with visual scores 0, 1 and 2. However, there was a statistically significant rise in the number of recoverable organisms in teeth with a visual classification of 3 and 4 ( $p<0.05$ ). There was also a significant increase in the total colony forming units between visual classification 3 and 4 ( $p<0.001$ ).

Figure 3c concerns the radiographic scores. When caries was seen as a radiolucency in the outer third of dentin (classification 2), there was a statistically significant increase in the number of recoverable microorganisms (total CFU) compared to those where no radiolucency was detected ( $p<0.005$ ). There was a further increase in the number of recoverable microorganisms (total CFU) when the lesions extended radi-

ographically to the middle-third of dentin (classification 3) ( $p<0.05$ ).

It can be seen from Figure 3b that for visual classification 3, while there was a significant rise in the number of micro-organisms recovered, in 36% of the specimens no organisms were recovered. Figure 4 pertains only to visual score 3. It plots the radiographic classification and the corresponding infection of the dentin for each specimen as a scattergram. It is noticeable that the majority of the teeth that visually scored as 3 were radiographic score 2, demineralized in the outer third of dentin only. However, within this category, only six sites showed a level of infection greater than  $\log_{10}(\text{CFU} + 1) = 2$ .

## DISCUSSION

The ranked visual scoring system was devised to link the appearance of carious lesions on the occlusal surfaces with their histological depth (Ekstrand & others, 1997). This system has the potential to allow the trained dentist to interpret the surface appearance in terms of a histological hemisection provided the teeth are perfectly clean and drying facilities are available. To date, no one has validated the system microbiologically. It seems reasonable to suggest that the ranked system should correspond to varying levels of infection of the dentin, and this study was designed to evaluate this.

This evaluation necessitated a clinical examination followed by tooth extraction and subsequent histological and microbiological study. Five unerupted teeth were included in the analysis, as these were bound to be sound and served as negative controls. No microorganisms were cultured from the teeth, which shows that the sectioning technique did not introduce contamination. Third-molar teeth, designated for extraction, were ideal specimens. Since it is good practice to have a radiograph prior to extraction, the study also provided the opportunity to compare the microbiological and radiographic findings. It may be possible that the rather simple tools available to all dentists, namely eyes and radiographs, would allow the practitioner to assess lesion depth and gauge the level of infection of the dentin.

The sample size is extensive—75 teeth, with no less than eight specimens in any visual score. The study design was dictated by ethical considerations. From a purely scientific viewpoint, it would have been preferable to collect the microbiological sample *in vivo*. However, this would have subjected the patient to cavity preparation on a tooth that was about to be extracted, and this would have been ethically unacceptable. Sectioning and sample collection were carried out immediately after extraction, and the sectioning tech-



nique was designed to prevent cooling water washing away organisms or a blade dragging organisms from the tooth surface to the sample site. Thus, only the enamel was scored with a cutting disc before a chisel was used to split the tooth.

The results showed the expected high correlation ( $r_s=0.93$ ) between the visual scores and lesion depth and a moderate correlation between radiographic scores and lesion depth ( $r_s=0.77$ ) (Ekstrand & others, 1997 & 1998). The latter is explained by the fact that early demineralization is not apparent radiographically.

The results also showed the expected correlations between the visual scores and the level of infection of the dentin. Visual appearances 0, 1 and 2 represent either sound teeth or uncavitated lesions. It is not surprising that micro-organisms were cultured from only a few of these specimens. These results may represent contamination and may therefore be artifacts, but it is also possible that micro-organisms were indeed present in these specimens, *in vivo*. Brännström & others (1980) showed invasion of micro-organisms in white spot lesions. However, it is important to note that there were no statistically significant differences in the levels of infection of the dentin in these groups. At this point, it should be noted that visual score 2 may correspond to demineralization in the outer third of dentin. The enamel-dentin junction (EDJ) is not seen as important in the histological scoring system. What is important is the integrity of the tooth surface. In these scores no cavity is present, plaque is still on the surface of the tooth and most importantly, the disease process is arrestable by plaque control alone (Carvalho, Thylstrup & Ekstrand, 1992).

Visual appearances 3 and 4 represent cavitation, 3 being a microcavity or greyish discoloration and 4 a hole with dentin at its base. As expected, these visual scores showed increasing levels of infection of the dentin. With respect to teeth scored visually as 4, some form of operative intervention seems warranted so that plaque control can be re-established and the disease process arrested.

The results from visual score 3 are interesting. The arithmetic means expressed in Figure 3b potentially hide important information. Sixty-four percent of teeth with visual score 3 had infected dentin, but this obviously implies that organisms were not recovered from 36% of the specimens. It also seems important to explore the level of infection of the specimens and investigate any relationship with the other diagnostic tool available to the dentist, namely the radiograph; hence, the production of the scattergram, Figure 4. This shows that in most of these teeth—19 out of 28—the radiolucency was in the outer-third of the dentin but even these sites seem lightly infected, 13 of them having less than 100 organisms per sample. As far as the clinician

is concerned, this probably implies that teeth scored visually 3 are not infected or lightly infected and require only fissure sealing rather than operative intervention. This approach was suggested by Handelsman in 1991. He fissure sealed lesions visible in dentin on radiograph and two weeks after sealing showed a major reduction in cultivable bacteria with a gradual reduction in total count thereafter. The radiographically monitored lesions did not progress over two years (Handelman & others, 1986). More recently, a remarkable randomized clinical trial was reported by Mertz-Fairhurst & others (1998). In this work, large occlusal lesions were treated with acid etched composite restorations, leaving soft, demineralized dentin both at the enamel dentin junction and in the base of the cavity. The teeth were followed over 10 years and there were no reports of failed restorations, pulpitis or pulp death. Taken together, these studies are disturbing to the operative dentist because they question the importance of removing infected dentin during operative treatment.

## CONCLUSIONS

In conclusion, this study shows a relationship between the ranked visual and radiographic scoring systems, and the level of infection of the dentin. The operative dentist can use this information to help decide the appropriate management of occlusal lesions of varying severity. Lesions with visual classification 1 and 2 can be managed with preventive treatment such as oral hygiene, fluoride applications and fissure sealants. Lesions with visual classification 3 and no radiographic evidence of caries can also be treated with fissure sealants, as the dentin will be minimally infected. Those lesions with a visual classification of 3 and a radiolucency on radiographic examination, and those with a visual score of 4 will be heavily infected and should be treated operatively. However, the literature has even questioned the conventional treatment of such lesions by caries removal and placement of a restoration. Simply sealing the carious biomass from the intra-oral sugar substrate may be sufficient to arrest lesion progression and this requires further research.

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## Laboratory Research

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# Effect of Caries Disclosing Agents on Bond Strengths of Total-Etch and Self-Etching Primer Dentin Bonding Systems to Resin Composite

RB Kazemi • JC Meiers • K Peppers

### Clinical Relevance

The use of two tested caries disclosing agents did not alter the shear bond strengths of various (a total-etch and two self-etch, non-rinsing primers) dentin bonding systems tested in this study.

### SUMMARY

This study examined the effect of caries disclosing dyes on composite to dentin shear bond strengths of a total etch, one-bottle and two self-etching, non-rinsing primer dental adhesives. Two caries disclosing dyes were evaluated, Seek and Snoop, with three dentin adhesives, Prime & Bond NT, Prompt L-Pop and Clearfil SE Bond. Extracted human molars stored in 0.2% sodium azide were sectioned longitudinally to expose dentin and embedded in acrylic, leaving the dentin exposed. Each dentin adhesive had three

test groups (n=12); a control and one with each of the caries disclosing dyes. The control group had the dentin conditioned and the adhesive applied following the manufacturer's instructions. The caries disclosing dye groups had the dentin first treated for 10 seconds with the disclosing dye, rinsed, then the dentin adhesives were applied as in the controls. A column of Tetric Ceram was bonded after dentin adhesive placement to each specimen and light cured. Specimens were stored in room temperature water for 24 hours, thermocycled for 1,000 cycles between 5°C and 55°C and tested in shear until failure. Mean  $\pm$  SD shear bond values (SBV) were determined in MPa. A one-way ANOVA and Student Neuman Keuls multiple comparison test within each DBS were performed at a significance level of  $p < 0.05$  to analyze the caries disclosing dyes input on SBV versus the controls. Surface analysis to determine the nature of the type of dentin/composite fracture/separation was also performed. For the fracture analysis data, a Chi-Square test was performed at

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**a significance level of  $p<0.05$ . The results of this study indicate that using the two tested caries disclosing dyes, with a total etch, one bottle and two self-etching, non-rinsing primer dental adhesives did not negatively affect the dentin-to-composite shear bond strengths of the three tested dentin bonding systems ( $p>0.05$ ).**

INTRODUCTION

An important aim of adhesive dentistry is to restrict cavity preparation to the removing of carious dentin and preventing sound and healthy dentin removal (Demarco & others, 1998; Palma & others, 1998). Carious dentin, often defined as containing two layers of decalcified dentin (Fusayama, 1979), should be excavated by removing only the bacterial infected layer. The infected outer layer of carious dentin is soft, insensitive, non-remineralizable with irreversibly deteriorated collagen fibers and odontoblast processes (Fusayama, 1988; Yoshiyama & others, 2000). On the other hand, the underlying bacteria-free dentin/affected layer is harder, non-infected, sensitive, remineralizable, with reversibly denatured collagen fibers and vital odontoblast processes that should be preserved (Fusayama, 1988; Yoshiyama & others, 2000). Various caries disclosing dyes that stain only the bacterial infected dentin have been widely used as a guide to removing the infected layer of dental caries (Anderson, Loesche & Charbeneau, 1985; List & others, 1987; Kidd & others, 1989). The dye solution is applied for a few seconds over the entire cavity preparation. Using dye solution allows practitioners to perform an ideal and minimally invasive cavity preparation for adhesive restorations (Fusayama, 1993).

There is very little data in the literature (and what is there is conflicting) on whether using various caries disclosing dye solutions affects the ability of dentin adhesives to bond-to-dentin when the cavity preparation is restored with restorative resin-based composite materials (Demarco & others, 1998; Palma & others, 1998; Miller, 2000; Miller, 2001). More importantly, no data has been published in scientific journals that relates to the impact of dye application on dentin on shear bond strengths of the newly intro-

duced self-etching primer class of Dentin Bond Systems (DBS).

This study investigated the effect of two caries disclosing dye solutions on the resin composite-to-dentin shear bond strengths employing two categories/generations of currently used dentin adhesive systems; total etch/single bottle and non-rinsing self-etching primer dental adhesives.

METHODS AND MATERIALS

Fifty-four non-carious human molars stored in 0.2% sodium azide solution were sectioned longitudinally parallel to the long axis of the teeth by means of a rotating diamond coated saw/Isomat Slow Speed Saw (Buehler Inc, Lake Bluff, IL 60044, USA) under distilled water irrigation. The sections were individually embedded in Teflon molds using auto-cure acrylic resin (Repair Material, Dentsply International Inc, York, PA 17405, USA) leaving the cut surface protruding above the resin. Each dentin section was ground with 320-grit sandpaper under copious amounts of water to create a flat, uniform smooth dentin surface. Care was taken to assure that the top and bottom surfaces of the specimens were parallel. All specimens were randomly divided into nine experimental groups of 12 specimens each and stored in room temperature water until bonding tests were conducted.

For each specimen the materials used and the sequence that was followed are specified in Tables 1 and 2. In this study, two caries disclosing dye solutions, D & C red dyes in a glycol base-Seek (Ultradent Products, Inc, South Jordan, UT 84095, USA) and a dark blue dye in a propylene glycol base-Snoop (Pulpdent Corp, Watertown, MA 02471, USA), and

Table 1: Groups Design and Specimen Distribution										
Caries Disclosing Agent	Prime & Bond NT			Prompt L-Pop			Clearfil SE Bond			Particulate Composite Tetric Ceram
	1	2	3	4	5	6	7	8	9	
Control	n			n			n			X
Seek		n			n			n		X
Snoop			n			n			n	X
n=12 X: used for all groups										

Table 2: The Experimental Sequence							
I*	II*	III**	IV**	V	VI***	VII	VIII
Disclosing Dye	Rinsing	Dentin Conditioning (37% H <sub>3</sub> PO <sub>4</sub> )	Rinsing	DBS Light Curing	Composite Column Light Curing	Thermo-Cycling 1000 Cycles 5°C & 55°C	Fracturing
* Skipped steps I and II in groups 1, 4, 7 (Control groups). See Table 1 for details. ** Skipped steps III and IV in groups 4 though 9 (Self-etching/non-rinsing primer DBS). *** Plus 20 second more light curing after removing the specimens from the assembly.							



their effect on three different dentin bonding systems—a total etch, acetone based and filled one bottle dentin bonding system, Prime & Bond *NT* (Caulk, Milford, DE 19963, USA), and two self-etching primer adhesive/ non-rinsing dentin bonding systems, Prompt L-Pop (ESPE Dental AG, Seefeld, Germany) and Clearfil SE (Kuraray Co, Ltd, Osaka, Japan)—were evaluated. Tetric *Ceram* (Ivoclar Vivadent, North America, Amherst, NY 14228, USA) particulate resin composite was used as the rod (column) material. A bonding assembly (Ultradent Products) was also used to place the particulate resin composite rods.

Before placing the specimens into the bonding assembly, the caries dye solutions were applied on the dentin surface of the specimens for 10 seconds, except for the control groups. The solutions were rinsed with water for 20 seconds and dried. The coronal dentin surface of each specimen was conditioned with 37% H<sub>3</sub>PO<sub>4</sub> for 20 seconds, rinsed for another 20 seconds and the excess of water removed with five-second light air drying. Care was taken not to desiccate the dentin. The dentin surface was left visibly moist, followed by the application of either dentin bonding system according to the manufacturers' instructions. Each DBS was applied with a sponge applicator saturating the dentin surface for 20 seconds. A uniform glossy surface should appear. If not, the last step was repeated. At this point the DBS was not light cured. The specimens were individually placed into the bonding assembly, then light cured for 10 seconds from two different directions (vertically and horizontally). Tetric *Ceram* resin composite was then packed incrementally into the insert of the assembly and light cured according to the manufacturer's instructions to build-up the composite column.

The specimens were removed from the assembly, light cured again and stored in room temperature water for 24 hours before the thermocycling phase was conducted. Specimens were thermocycled 1,000 times between 5°C and 55°C with a dwell time of 30 seconds. The specimens were then placed in a positioning jig (Ultradent Products) and tested in shear failure in an Instron (Instron, Canton, MA 02021, USA) using a crosshead speed of 0.5 mm/minute. The values were calculated in MPa.

Surface analysis to determine the nature of the type of dentin/composite fracture/ separation was also performed using a binocular light microscope (Olympus Co, Lake Success, NY 12422, USA) at x20 with the fractures classified as either adhesive or cohesive.

Adhesive: Debonded surface had more than 60% of dentin showing.

Cohesive: Debonded surface had more than 60% resin covering dentin surface.

A one-way ANOVA was performed on the shear dentin bond data using the variable at a significance level of  $p<0.05$ . If significance was found, then the Student Neuman Keuls multiple comparison test was performed to determine where the significant difference between the groups occurred at a significance level of  $p<0.05$ . For the surface analysis data of the fracture site, a Chi-Square test was performed at a significance level of  $p<0.05$ .

### RESULTS

Figure 1 and Table 3 show the results of this study. In respect to the shear bond strength of all three dentin bonding systems, there were no significant differences between the groups treated with either tested dental caries disclosing solution and the control groups ( $p>0.05$ ). The results of analysis of the dentin surface fracture (adhesion and cohesion) also indicated that there were no significant differences between the control group and the two applied dye groups for each dentin bonding system ( $p>0.05$ ).

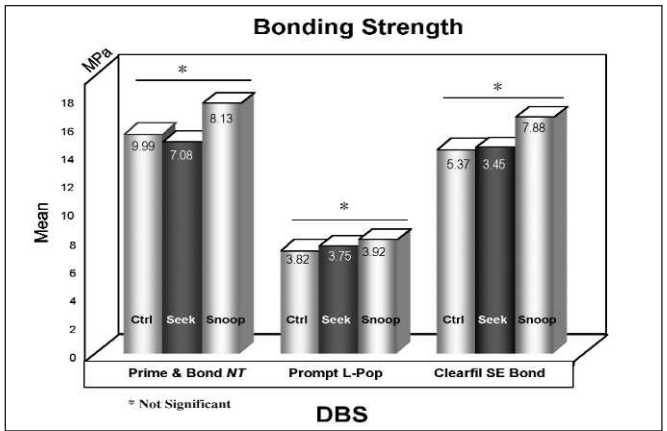


Figure 1. Mean ± SD effect of two caries disclosing dyes on shear bond strengths of three different dentin bonding systems to resin composite. Results of one-way ANOVA test indicate no significant statistical differences within each DBS, between control group and two disclosing dye solutions ( $p>0.05$ ).

Table 3: Dentin Surface Fracture Analysis						
Group	Prime & Bond NT		Prompt L-Pop		Clearfil SE Bond	
	Adhesive	Cohesive	Adhesive	Cohesive	Adhesive	Cohesive
Control	1	11	6	6	1	11
Seek	2	10	8	4	0	12
Snoop	4	8	4	8	0	12
p<value	NS*	NS*	NS	NS*	NS*	NS*
* Not Significant						

## DISCUSSION

Caries disclosing agents have been widely used for removing infected layers of dental caries (Fusayama, 1979; Anderson & others, 1985; List & others, 1987; Kidd & others, 1989). The advantage of using the caries dye solutions is to preserve more non-infected dentin over the pulp. However, an important question that needs to be answered is how the dyes could affect the ability of dentin adhesives to bond-to-dentin when the teeth are restored with bonded restorations. The results of this study indicate that there was no negative affect on shear bond strength of composite-to-dentin with the use of dyes with the tested dentin bonding systems.

The results of this study differ from those obtained by Demarco & others (1998), who reported that the bond strength of resin composite could adversely be affected by the use of two different caries-disclosing dyes. These investigators observed some dentin samples with light staining after the dentin was rinsed, acid etched and rinsed again. Their interpretation was that the remaining dye solution trapped in dentin might have adversely affected the wetting of dentin by the dentin bonding agent and contributed to a decrease of micromechanical retention and bond strengths of the resin composite to dentin. In their study, they used a total etch technique with an acetone based DBS (Prime & Bond 2.0) and all their debonded samples failed adhesively.

In this study, a light staining of dentin after rinsing, acid etching and rinsing in some specimens was observed, but the results do not support the findings of Demarco & others (1998). In this study, a total etch with a later version of their DBS (Prime & Bond NT) was used along with two self-etching/non-rinsing primer DBSs (Prompt L-Pop and Clearfil SE Bond). For the total etch samples, the assumption is that the acid etch process that includes rinsing the dentin surface is capable of removing the dissolved mineral components of dentin and the remaining or interaction byproducts of caries dye solution. In the case of the non-rinsing primer DBS, the smear layer and any possible free or interacted caries dye solution would remain in the bonding site with the formed hybrid layer.

Introducing a self-etching primer class of dentin bonding systems which does not require rinsing and serve simultaneously as conditioner and primer (NRC) is the most recent advancement in adhesive dentistry (Rosa & Perdigão, 2000). There is, however, no data published in scientific journals related to the impact of caries disclosing dye application to dentin on shear bond strengths of this new generation of DBS. The results of this study provide the first documentation of the innocuous native of the formulation of the two caries disclosing solutions with this class of dentin adhesives.

This study confirms the results of other studies that show the use of caries dye solutions did not significantly

lower the tensile bond strength of any tested resin based DBS (Palma & others, 1998; Miller, 2000; Miller, 2001). In addition, it was reported that although the bond strengths of the DBS to the affected/relatively soft layer of dentin was lower than normal/hard dentin, the caries dye solutions did not alter the bond strengths of all tested resin composite and compomer materials (Palma & others, 1998).

This study assessed the shear bond strengths of RBC to the treated hard/normal dentin surface with caries dye solutions. This could be considered different from the bonding of resin composites on the relatively soft affected layer of dentin. Although it has been reported that the caries disclosing solution did not significantly alter the bonding development onto the artificially partially demineralized dentin (Palma & others, 1998), Nakajima & others (1995) also reported that the affected layer of carious dentin cannot be considered normal dentin because it has a lower Knoop hardness value. Also, the SEM studies of Yoshiyama and his co-investigators (2000) revealed that the lower bond strengths produced by either a single bottle dentin bonding system or a new self-etching dentin bonding system to caries affected dentin versus normal dentin are due to the presence of acid-resistance mineral precipitated in both dentinal tubules and in adjacent inter-tubular dentin. This barrier could also interfere with resin tag formation. This data would indicate that it is more important to have hard, unaltered dentin tissue available on the bonding site rather than any possible affect from the short application of caries dye solution.

This study did not intend to compare the shear bond strengths of the three different dentin bonding systems. The plan was to evaluate the influence of caries dye solution on the shear bond strengths within each of the tested dentin bonding system. It is interesting to note that one of the new generation of the DBS (Prompt L-Pop) did not perform as well as the other two dentin bonding systems of different generations in terms of shear bond strength. However, applying the caries disclosing dyes did not affect this adhesive, already showing low bond strengths.

## CONCLUSIONS

The use of two glycol based caries disclosing solutions (Seek and Snoop) did not effect the shear bond strengths of a total etch (Prime & Bond NT) and two self-etching, non-rinsing primer (Prompt L-Pop and Clearfil SE Bond) dentin bonding systems. The clinical correlation of these results may be interpreted to mean that the use of these specific types of caries disclosing dye formulations to the dentin in a cavity prep will not negatively impact the dentin-to-composite shear bond strength of the three tested adhesives, though this needs further investigation in clinical studies.

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# Surface Roughness of Various Packable Composites

TM Ryba • WJ Dunn • DF Murchison

## Clinical Relevance

Surface roughness of packable composites differs among various manufacturers and this difference cannot be overcome with different polishing methods.

## SUMMARY

Packable composite restorations have become a popular alternative to dental amalgam restorations in posterior teeth. A drawback inherent to composites is their difficulty in polishing, which often results in a dull or rough surface. This study compared the surface roughness of a resin-based hybrid composite material and five packable resin-based composites polished with either aluminum oxide disks or a rubber polishing system. Sixteen specimens of each of the six composite materials were polished with either Sof-Lex XT disks or Enhance rubber polishers followed by fine and superfine polishing pastes. The specimens were evaluated for surface roughness using surface profilometry. Mean values were calculated for each material type and method of polishing. Data were subjected to a two-way ANOVA. Post hoc comparison was accomplished

using Tukey's HSD. No significant difference in surface roughness was detected among polishing techniques ( $p=0.067$ ); however, a strong trend—that aluminum oxide disks provided a smoother surface than rubber polishers in five out of six materials—was noted.

## INTRODUCTION

Packable resin-based composites have recently been introduced as a dental restorative material option. These composites have packable characteristics by various proprietary manufacturing methods, such as increasing viscosity, increasing level of filler content beyond 80%-85% by weight, unique particle morphology and/or particle size distribution (Bernighaus & Tyler, 1999). Packable resin-based composites differ mainly in their inorganic filler, the size of the filler, size distribution and the extent of filler loading. Such factors may influence their ability to be polished.

A unique drawback to most composites is their inherent resistance to polishing, which often results in a rough or dull surface. This problem has been attributed to the differences in hardness between the inorganic filler and the polymeric matrix (Pratten & Johnson, 1988). Increased plaque retention, gingival inflammation and staining of the restoration may result in sub-optimally finished restorations (Quirynen & Bollen, 1995; Weitman & Eames, 1975). All composites should be polished following polymerization as rough, unpolished

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restorations increase the coefficient of friction and may increase the rate of wear (Krejci, Lutz & Boretetti, 1999). In addition, composites polymerized with a clear matrix on the surface will leave a resin rich surface layer that is easily abraded in the oral environment, exposing unpolished, rough, inorganic filler material (Krejci & others, 1999). Polishing with different methods has been shown to produce various results (Chung, 1994; Kaplan & others, 1996; Hondrum & Fernandez, 1997). Aluminum oxide disks and rubber polishing systems have been suggested as standard protocol (Jefferies, 1998).

Studies investigating the effects of different polishing methods on newer packable composite restorative materials is limited. This study compared the surface roughness of a hybrid resin-based composite and five packable resin-based composites polished with aluminum oxide disks or a rubber polishing system.

METHODS AND MATERIALS

Six commercially available resin-based composite restorative materials were evaluated. All were visible light-activated composites intended for clinical use. Table 1 lists the composites used in this study. Z-100 (3M Dental Products, St Paul, MN 55144, USA), a hybrid resin-based composite, was used as the control. The same investigator performed all specimen preparation, finishing, polishing and evaluation in order to reduce variability. Experimental groups consisted of 16 specimens from each of the six different composite materials. The specimens were formed by placing the composite material within a 2 mm deep by 8 mm diameter Delrin ring covered on both sides with mylar strips and a 1.2 mm thick glass slide. Specimens were polymerized with an Optilux 400 visible light-curing unit (Demetron/Kerr, Danbury, CT 06810, USA) with a 13 mm tip. Each

specimen was polymerized for 30 seconds, then the matrix assembly was removed and the specimens were further polymerized for an additional 30 seconds on each side. A curing radiometer (Demetron/Kerr) confirmed a light intensity of at least 400mW/cm<sup>2</sup> immediately after each sample was polymerized. To simulate the clinical finishing procedure, all specimens were ground flat with 320 grit silicon carbide paper (Buehler Ltd, Lake Bluff, IL 60044, USA) under running water in a polishing lathe (Buehler Ltd). The ground surfaces were used as the baseline finish prior to polishing. The specimens were labeled with cross-referencing identification. Each material group was further divided randomly into two groups of eight specimens each for the two different polishing techniques.

The first half of each material group was polished using an aluminum oxide polishing system, Sof-Lex XT (3M Dental Products). The polishing sequence of medium (360), fine (600) and superfine (1200) was used. The disks were mounted on a slow speed handpiece (KaVo USA, Lake Zurich, IL 60047, USA) rotating at approximately 60,000 rpm. Uniform light pressure and a circular pattern for 10 seconds for each abrasive step was used to polish the specimens (Chung, 1994). The disks were discarded following each use. A total of 48 specimens were polished with this method.

The second half of each material group was polished using a rubber polishing system, Enhance (Dentsply/Caulk, Milford, DE 19963, USA). The polishing sequence included the rubber-like polishing disks followed by the fine and super-fine polishing pastes. A slow-speed handpiece (KaVo) was used rotating at approximately 60,000 rpm, with light pressure and circular pattern applied for 15 seconds for each polishing sequence as recommended by the manufacturer. Polishing disks and cups were discarded after each use.

A total of 48 specimens were polished with this method. The specimens were stored in water at 37°C at all times.

Following polishing treatment, all specimens were steam cleaned and evaluated under a stereomicroscope (Nikon model SMZ-1B, Tokyo, Japan) for grinding debris or surface defects. Any defects in specimen surfaces were noted in order to correlate any unusual data. Specimens were then evaluated for surface roughness using mechanical surface profilometry. Profilometry was performed using the Surfanalyzer 4000 surface analyzer (Mahr Federal, Providence RI 02940, USA) to evaluate surface roughness for each specimen. A 5 µm stylus was used with a traverse speed of 0.25 mm per second, 15 mN and a cutoff control of 0.8 mm/0.03 inches. Length of traverse was 1 mm. Three runs at three different angles (0°,

Table 1: <i>Materials Tested</i>		
Product	Lot #	Manufacturer
ALERT	210217	Jeneric/Pentron Wallingford, CT 06492
Filtek P-60	9AH	3M Dental Products St Paul, MN 55144
Pyramid	9900003818	BISCO Dental Products Schaumburg, IL 60193
Solitaire II	020224	Heraeus Kulzer South Bend, IN 46614
SureFil	990224	LD Caulk/Dentsply Milford, DE 19963
Z-100	19970401	3M Dental Products St Paul, MN 55144
Sof-Lex XT Contouring and Polishing Discs	NA	3M Dental Products St Paul, MN 55144
Enhance Finishing and Polishing System	9806122	Dentsply/Caulk Milford, DE 19963

120°, 240°) were averaged to determine the mean surface roughness ( $R_a$ ).

Mean  $R_a$  values were calculated for each material type and each method of polishing. A two-way Analysis of Variance (ANOVA) was used to compare these scores for differences with respect to material type, method of polishing and interaction of the two factors. Post-hoc comparison was accomplished using Tukey's HSD (honestly significant difference). All testing was performed at  $\alpha=0.05$ . The sample of eight disks/material/polishing technique provided a 96% chance of detecting a relatively significant difference between polishing techniques ( $ES=4$ ). The sample provided an 82% chance of detecting a vast difference among materials.

### RESULTS

Two-way ANOVA revealed that no significant interaction between material and polishing method existed ( $p=0.895$ ). The analysis for main effects detected a significant difference among the six composite materials ( $p=0.002$ ). The type of polishing technique did not achieve statistical significance ( $p=0.067$ ); however, a trend was noted that aluminum oxide disks provided a smoother surface than rubber polishers in five of the six materials (Figure 1). Tukey's HSD was used to identify where differences occurred (Table 2). The only significant difference detected was that Filtek P-60 polished smoother than ALERT when using rubber as the polishing medium. All six composite materials achieved similar smoothness when polished with aluminum oxide disks.

### DISCUSSION

The dental profession welcomed packable composite restorative materials because their handling characteristics were supposed to resemble amalgam. To change the viscosity of composites, manufacturers used higher volume fraction fillers and monomers with higher viscosity

and larger molecular weight. Packable composite restorative materials, although marketed in similar ways because of their packability, are manufactured with very different filler particles. The filler phase of ALERT consists of colloidal silica and silanated chopped glass fibers. ALERT contains the largest filler particles of the composites examined, ranging from 1.0 to 3.0  $\mu\text{m}$  in diameter, with a volume filler fraction of 70%. The chopped glass fibers average 40  $\mu\text{m}$  in diam-

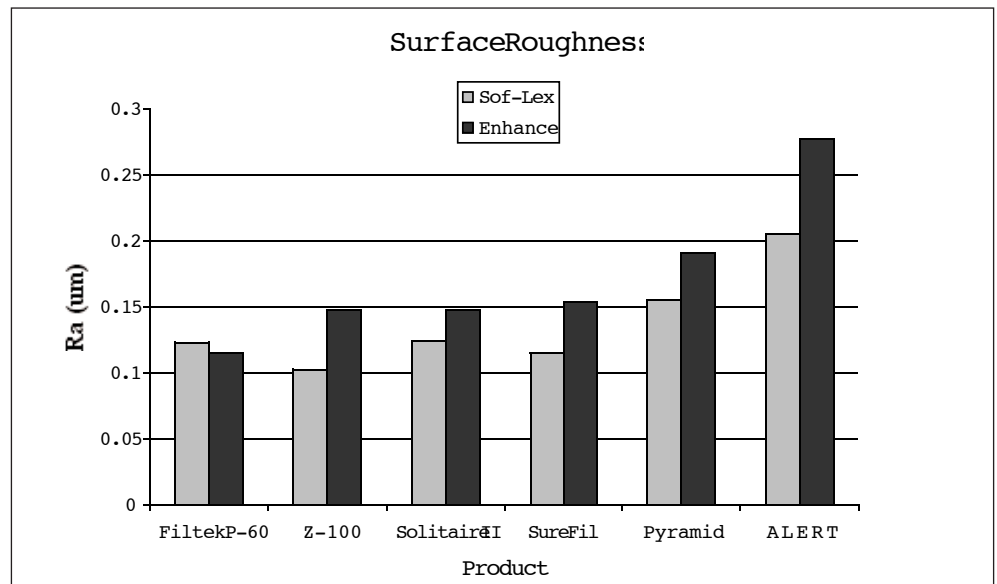


Figure 1. Graphical representation of mean surface roughness ( $R_a$ ) of packable composites and control.

Table 2: Comparison of Mean Surface Roughness ( $R_a$ ) in Microns and Tukey's HSD

Product	Enhance		Sof-Lex	
	$R_a$	SD	$R_a$	SD
Filtek P-60	0.116	0.119	0.123	0.040
Z-100	0.147	0.060	0.102	0.041
SureFil	0.154	0.055	0.115	0.069
Solitaire II	0.147	0.075	0.124	0.077
Pyramid	0.191	0.101	0.155	0.095
ALERT	0.277	0.162	0.205	0.119

Groups connected by the same line are not statistically different from each other.

Table 3: Two-Way ANOVA Table

Combined	Type III Sum of Squares	df	Mean Square	F	Sig
Product	0.170	5	0.034	4.08	0.002
Polishing Technique	0.029	1	0.029	3.43	0.067
Product/Technique Interaction	0.014	5	0.027	0.328	0.895
Error	0.912	84	0.010		

eter. The manufacturer reports that the resultant clinical feel is typical of amalgam due to the microfibers, which are added to the filler base. The manufacturers also claim that a 5 mm depth of cure is possible, a claim which is disputable (Kerbey & others, 1999). As these composite materials wear, the particle size will give an indication of how rough the composite surface will become. Because ALERT has the largest particle size (1-110  $\mu\text{m}$ ), ALERT, over time, will have the roughest surface. The high stiffness of ALERT may make cavity adaptation difficult and may also lead to the incorporation of voids in critical areas of a proximal box in Class II preparations. On the other hand, the high stiffness would allow easier shaping of occlusal anatomy prior to polymerization. Regardless of the finishing and polishing procedure, the  $R_a$  values for ALERT were higher than for all of the other composites tested in this study. Naturally, these observations confirm that larger filler particle sizes result in larger  $R_a$  values.

SureFil has an average particle size of 0.8  $\mu\text{m}$ , with some particles as large as 6  $\mu\text{m}$ . The filler content is 66% by volume and is composed of barium fluoroaluminoborosilicate glasses and fumed silica. According to the manufacturer, packability is achieved by using four distinct particle size distributions and morphologies. This mixture of different sized glass fibers has been termed "Interlocking Particle Technology." Under these conditions, the resin matrix is minimized. Decreased matrix volume would reduce the preferential loss of the resin phase during polishing, thereby, reducing areas of filler showing positive relief. A decreased matrix volume may contribute to dislodgement of the larger filler particle during finishing and polishing procedures due to an inability to adequately stabilize these particles, increasing the  $R_a$  value. The manufacturer also claims that a 5 mm depth of cure is possible, but this is again disputable (Kerbey & others, 1999).

Solitaire II is characterized by the manufacturer as having a variable filler particle size of 2-20  $\mu\text{m}$  and is 75% filled by volume. Filler particles consist of silicon dioxide, barium aluminoborofluorosilicate glass and aluminum fluoride glass. Solitaire II does not contain BISGMA or TEG-DMA. Instead, it consists of an indirect high heat and pressure-cured polycarbonate vitroid glass ceramic material. Each filler particle is covered or coated with nodules to prevent the free flow of one particle past another when subjected to condensing forces. This morphology imparts its characteristic packability. The surface topography may also prevent the filler particle from dislodging during finishing and polishing resulting in lower  $R_a$  values.

Pyramid enamel consists of stratified aggregate filler with ethoxylated resin to reduce water sorption. Pyramid Dentin contains coarser fillers and is more heavily filled, giving it a higher viscosity. Pyramid

Dentin is the packable component. Pyramid has a particle size range of 1-15  $\mu\text{m}$ .

Z-100, a hybrid resin-based composite, contains zirconia/silica filler particles that range in size from 0.01-3.5  $\mu\text{m}$ , averaging 0.6  $\mu\text{m}$ , in a resin system of BisGMA and TEGDMA. It is 66% filled by volume. The smaller filler particle sizes account for increased polishability. Made by the same manufacturer as Z-100 with similar composition, Filtek P-60 is marketed as a posterior resin-based condensable hybrid composite. It has an average filler particle size of 0.6  $\mu\text{m}$  with a 0.1-3.5  $\mu\text{m}$  range and is 61% filled by volume. The resin components differ from Z-100, with the incorporation of BisEMA and UDMA. BisEMA has a high molecular weight and forms fewer double bonds resulting in a higher viscosity and slightly softer matrix. The lower fill ratio and smaller particle size are responsible for P-60 having a low  $R_a$  value.

As expected, overall surface roughness of the different materials correlated well with the average particle size. The selection of a polishing system proved to be less important as far as the final surface polish is concerned. An acquired final polish is not as important as the inherent surface finish of a material, which is dependent on particle size.

The trend of Sof-Lex polishing disks providing a slightly smoother surface may be explained by the aluminum oxide abrasive on a rigid matrix. This has the ability to flatten the filler particles and abrade the softer resin matrix at an equal rate. The Enhance polishing system is made from a flexible, rubber-like material that is actually a proprietary light-cured resin impregnated with an abrasive. The abrasive may abrade the softer matrix and only round the protruding filler particles. This may result in a higher surface roughness. When comparing the two polishing techniques used on posterior composites, finishing and polishing access must be considered. Circular aluminum oxide disks are not practical for polishing occlusal anatomy.

Results of this study correlate well with that of Chung's (1994), where no difference was detected between rubber polishing instruments and aluminum oxide disks in assessing surface roughness of hybrid composites and a micro-filled composite. In any case, finishing procedures should be kept to a minimum as they are inherently destructive to the restoration surface (ADA Council on Scientific Affairs, 1998) and may lead to subsurface micro-crack formation (Leinfelder, Wilder & Teixeira, 1986). In addition, the composite surface closest to the light tip during polymerization, which has the best physical properties, is removed during finishing procedures (Hilton, 2000). The decision to use a packable composite is a personal one and should be based on the operator's preference of handling characteristics and not on any expectations of improved clinical performance.



## CONCLUSIONS

Under the conditions of this study, the method of finishing and polishing condensable composite restorative materials does not significantly affect the final polished smoothness of the material. There are, however, significant differences in surface roughness among various condensable composite materials. Filtek P-60 achieved a significantly smoother surface than ALERT when using a rubber polishing 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, United States Navy or the Department of Defense.

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# Flow Characteristics and Film Thickness of Flowable Resin Composites

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## Clinical Relevance

In the selection of flowable composites, higher flow characteristics for adaptation of liners, sealants, margin repairs and a low film thickness for veneer cementation are important. A lower flow can be helpful for control of the placed material for example in a Class IV, core build-up, composite veneer and porcelain repair.

## SUMMARY

Flowable resin composites have been recommended for many clinical uses and have been formulated in a variety of compositions and viscosities to meet various uses. This study compared the variation in viscosity of flowable resin composites using the ADA Flow Test and measured film thickness with a test to simulate flow during cementation. The film thickness test for the flowable resin composites was performed at three different seating pressures to simulate pressure variation during seating of porcelain veneers, one of the potential uses of flowable resin composites that may favor a lower viscosity. The following flowable resin composites were eval-

uated: Revolution, StarFlow, Aeliteflo LV, Aelite, Flow-It, FloRestore, Versaflo, Durafill Flow and Tetric Flow, with Nexus2, a composite luting resin used as a control.

Flow characteristic measurements suggest that resin composites may be divided into high flow (StarFlow, Revolution, Aeliteflo LV), medium flow (FloRestore, Durafill Flow, Flow-It) and low flow (Tetric Flow, Versaflo, Nexus2, Aelite) groupings. The film thickness measurements agreed with the ADA flow test, except for two exceptions. Durafill Flow resin composite had a higher film thickness than expected based on the ADA flow test. Also, Aeliteflo unexpectedly had a lower film thickness.

At the lowest seating pressure (.016MPa) tested, eight out of the nine resin composites tested as well as the control luting resin had a film thickness greater than the 25 microns used as the clinical standard for cement film thickness. However, at the highest seating pressure (.038MPa) tested, only two and the control, Versaflo, Durafill Flow and Nexus2, had film thicknesses significantly greater than 25 microns. The highly filled Nexus2 luting resin had the highest film thickness at all seating pressures.

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INTRODUCTION

Flowable composites have been available for a relatively short time, yet many possible uses have been recommended by the manufacturers with limited testing for efficacy in the laboratory or clinic. Flowable composites are used to solve clinical problems, often for which no specific material had previously served. For example, as the repair material for non-carious amalgam margin defects (Roberts, Charlton & Murchison 2001) or to bond back together part of a chipped tooth for use as a long-term emergency temporary (Small 1996). The materials are a modification of the restorative resin composites, thus, they tend to contain a lower filler content and more of a resin matrix (Tabassian & Moon, 1999). The glass filler content and sizes, as well as the viscosity of the resins used, can influence the flow characteristics of commercial products.

The development and acceptance of flowable composites are driven by the manufacturers' perceived benefit of having a resin composite that is easier to place or nearer to self-adapting compared to more conventional restorative resin composites (Bayne & others, 1998). An example of the benefit of self-adapting flowable resin composites was demonstrated by Leevailoj & others (2001), in which they showed reduced microleakage when a flowable resin composite was used as a

liner under all packable resin composites, but only half of the flowables reduced the microleakage to the level of a hybrid composite. Some functions these materials could perform, as suggested by manufacturers, are listed in Table 1 and include aiding in margin repair, simplifying restoration placement, improving lining by adaptation, marginal sealing, speeding build-up, cementation, sealants and block-out placement. In selection, emphasis is put on the flow characteristics of these materials by manufacturers, and the manufacturer justifies them to the dentist as meeting an immediate goal for improved ease of placement. Research is needed to clinically test these materials, their procedures and develop techniques that optimize their usefulness or define areas where their use is contra-indicated. A balance must be struck between ease of placement and physical properties of the material to minimize technique sensitivity and ensure long-term clinical performance. Therefore, other properties should be considered for determining the long-term clinical potential for these materials, especially in applications where occlusal forces are exerted. Properties, as reported by Bayne & others (1998), showed that they had sufficient wear resistance for use in low stress-bearing areas and Carter, Moon & Barnes (1999) showed marginal chipping can be related to the transverse strength of resin composites, which should be considered for evaluating longer-term use of these materials.

When selecting commercial materials, a variation in the flow exists among the materials from flowing, to slumping, to slump resistant. Table 2 lists the flowable composites tested, with their weight filler and manufacturer and address as reported by Tabassian & Moon (1999). Nexus2 VLC base component was evaluated as a control for comparison for use as a luting resin for porcelain veneers. It is more highly filled and expected to have a high viscosity and more wear resistance than flowable composites in general. At least three companies manufacture flowable composites with two different viscosity ranges (a higher and lower viscosity product) BISCO, Inc, Aeliteflo and Aeliteflo LV, Jeneric/Pentron, Flow-It LF and Flow-It, and Danville Engineering, Aria and StarFlow. The first product listed for each company has higher viscosity than the second. Companies with only one product are divided between those with only a high, medium or a low viscosity product. When selecting the most useful viscosity for a particular application, it is suggested that some applications may lend themselves more to a low or a high viscosity. For example, a low modulus liner for bond stress relief or an abfraction-related root restoration is likely to benefit from a self-adapting low viscosity flowable composite, as it implies more resin that decreases the modulus to reduce stress from bond shrinkage and occlusion. A Class IV application may work better with a high viscosity flowable resin composite because its

Table 1: Some Applications Suggested by Manufacturers for Flowable Composites Which Need To Be Verified for Efficacy by Clinical Research

1.	Margin repair (amalgam, composite, crown porcelain, temporary)
2.	Class I (small)
3.	Class II (gingival box) Class III (small)
4.	Class V (shallow)
5.	Class IV (small)
6.	Pit and Fissure Sealants
7.	Air-Abrasion Restorations
8.	Preventive Resin Restorations
9.	Incisal Edge Restoration
10.	Liner
11.	Porcelain Veneer Cementation
12.	Composite Re-surfacing
13.	Build-up Worn Composite
14.	Forming Contacts on Bis-acrylic Temporary
15.	Tunnel Restoration
16.	Worn Provisional or Denture Teeth
17.	Block Out
18.	Porcelain Repair
19.	Core Build-up
20.	Composite Veneer
21.	Splinting



Table 2: *Products Tested, Weight % Filler and Size Range in Microns(u)\**

Product	Filler Wt. %	Size Range	Manufacturer	Address
AeliteFlo	58.8	<.5 – 14u	BISCO, Inc	1100 W Irving Park Blvd Schaumburg, IL 60193
AeliteFlo LV	50.5	<.5 – 11u	BISCO, Inc.	1100 W Irving Park Blvd Schaumburg, IL 60193
Durafill Flow	48.3	<.1 – .2u	Heraeus Kulzer	4315 S Lafayette Blvd South Bend, IN 46614
FloRestore	44.3	<.5 – 2u	Den-Mat, Inc	PO Box 1729 Santa Maria, CA 93456
Flow-It	68.9	<.5 – 4.5u	Jeneric/Pentron	153 N Plains Industrial Rd Wallingford, CT 06492
Revolution	53.3	<1 – 4u	Kerr, Inc	1717 W Collins Avenue Orange, CA 92867
Nexus2	78	N A	Kerr, Inc	1717 W Collins Avenue Orange, CA 92867
StarFlow	60.0	<.5 – 1.2	Danville Engineering	2021 Omega Road Ramon, CA 94583
Tetric Flow	65.2	<.5 – 10u	Ivoclar-Vivadent	175 Pineview Drive Amherst, NY 14228
Versaflo	61.0	<.5 – 2.5u	Centrix, Inc	770 River Road Shelton, CT 06484

\*Weight % and size range reported by Tabassian and Moon (1999) for flowable composites. Weight % for Nexus2 is from Kerr Inc.

greater filler content should help resist chipping and wear better and hold its shape during placement. Also, a less flowing composite may make clean up easier during cementation, as claimed by Nexus2. The ease of use depends on the dentist's skill and familiarity with the placement procedure and material used (Chuang & others, 2001). The viscosity of composites has been noted to decrease with increasing temperatures (Van Meerbeek & others, 1994) so that material chilled in the refrigerator will be more viscous than the same material at room temperature, and when the material warms in the mouth, the viscosity decreases.

This study measured the relative flow of some commercial flowable resin composites for selection for use based on their relative flow. In addition, the film thickness was measured so that the seating ability of these materials for porcelain veneer cementation could be evaluated (Peumans & others, 2000 and Shaini, Shortall & Marquis, 1997).

## METHODS AND MATERIALS

To measure the relative flow of nine flowable resin composites listed in Table 2, a procedure similar to the ADA specification #8 for the consistency of dental cement, was followed as described (Standford, 1972). A half-milliliter of resin composite to be tested was measured out and centered on a glass plate. Immediately, a second glass plate of equal size was placed over the first, with an added weight centered on top so that the total of the added weight and glass plate was 120

grams. This weight compelled the resin composite to flow out in a circular shape between the glass plates for one minute. The added weight was then removed, and the thin, circular layer of composite formed from the flow of the resin composite between the glass plates was light cured through the top glass plate to prevent further changes in the diameter of the flow sample. Sample diameters at 90° to one another were measured using a micrometer caliper that allowed measurement to 25 microns to characterize the flow. The mean and standard deviations for each resin composite diameter (n=3) were calculated. The average flow diameters and standard deviations were linearly normalized to

the same pressure. The normalization is justified because the composite is caused to flow by a fixed load acting on the composite so, as the composite flows out into a larger diameter, it has less pressure continuing to cause it to flow. This means that at the end of one minute, the larger diameter samples have less pressure acting to cause them to flow. A linear normalization calculation was developed to account for the variation in pressure between composites at the end of one minute of flow because of their flow area differences. The flow resistance is related to the pressure needed to drive the flow, as can be shown from analysis by Braden, 1975. The average diameter of a composite being normalized was multiplied by the ratio of the average flow area for this composite, divided by the average flow area of the composite with the highest pressure (that is, the smallest flow area). This procedure calculates a corrected diameter ( $D_N$ ) for the measured diameter ( $D_M$ ) compensating for the pressure difference between the composite with the most pressure (PS) acting (that is, the smallest diameter— $A_S$ ) and the other composites ( $A_M$ ) as given by the following equation:

$$D_N = D_M P_S / P_M = D_M A_M / A_S$$

The significant differences between the normalized flow diameter characteristic were compared using ANOVA and for a priori  $\alpha=0.5$ , the Newman-Keuls multiple comparison *t*-test was used to rank order the flowable composites into significantly different groups based on their normalized flow diameters.

Table 3: The Average Diameter of Flow and Standard Deviation Normalized for the Pressure Applied to the Flowable Resin composites Listed in the Order of Increasing Flow with the Newman-Keuls Multiple Comparison Groups of Significant Difference					
Material	Flow Diameter DN (DM) cm, St Deviation & Significant Groups				
AeliteFlo	2.77	(2.77)	±	0.39	a Low Flow Group
Nexus2	3.15	(2.89)	±	0.14	a Low Flow Group
VersaFlo	3.36	(2.95)	±	0.39	a Low Flow Group
Tetric Flow	3.52	(3.02)	±	0.34	a Low Flow Group
Flow-It	4.91	(3.35)	±	0.58	b Medium Flow Group
DuraFill Flow	5.46	(3.47)	±	0.44	b Medium Flow Group
FloRestore	5.61	(3.50)	±	0.14	b Medium Flow Group
AeliteFlo LV	7.21	(3.81)	±	0.60	c High Flow Groups
Revolution	8.56	(4.03)	±	1.08	c' High Flow Groups
StarFlow	10.62	(4.33)	±	0.42	c" High Flow Groups

The procedure for measuring the film thickness as it relates to the use of flowable composite for bonding porcelain veneers is also modeled after ADA Specification #8 for the film thickness of dental cement (Standford, 1972). However, the pressure for seating was varied and reduced to evaluate the affect of seating pressure and to model the lower forces for seating veneers as opposed to full crowns. The amount of resin composite that was weighed out for each sample was 0.1 grams, as this was sufficient to cause excess material to be squeezed out from between the pressure plates. The glass plate had an area of approximately 13.3 square centimeters. Three different load values of 21.3 newtons, 36.0 newtons and 50.7 newtons were placed on three samples for each material for three minutes, then the load was removed and the samples light cured. Fifteen kilograms (147 newtons) are normally used to test the film thickness of dental cements as part of ADA specification #8 and on a much smaller area, 2 square centimeters. The film thickness was measured with a dial gage micrometer as the difference at the center between the glass plates with the cured film thickness sample present minus the thickness of

only the glass plates. The micrometer allowed measurements of 1 micron as its smallest division. The seating pressures used in this test were 0.016 MPa, 0.027 MPa and 0.038 MPa as calculated from the area and the seating force. The highest seating pressure (0.038 MPa) is equivalent to 0.42 lbs of force being used to seat a veneer on a central incisor with an area of 0.5 cm<sup>2</sup>. The criteria normally used as clinically acceptable—25 microns or less for cement film thickness—was used to judge the acceptability of the different flowable composites resins for use in porcelain veneer cementation for the different seating pressures and compared to the values for Nexus2.

RESULTS

Table 3 lists the normalized average diameters and standard deviations of the flow test measurements. The larger the diameter, the greater the flow and the lower the viscosity of resin composite. ANOVA calculations indicated the normalized diameters where significantly different to a level of confidence of *p*=.001. The Newman-Keuls multiple comparison *t*-test analysis indicated that the flow characteristic diameters could be divided into five groups in the order of increasing flow. Group “a,” with low flow (*D<sub>N</sub>* =2.77-3.52 cm), was composed of AeliteFlo, Nexus2, Versa Flow, Tetric Flow and represents the most viscous of the flowable resin composites. Group “b,” with medium flow (*D<sub>N</sub>* = 4.91-5.61 cm), was composed of Flow-It, DuraFill Flow, and FloRestore. The high flow composites (*D<sub>N</sub>* = 7.21-10.62 cm) are composed of Groups “c, c’ and c,” three statistically significantly different composites, Aeliteflo LV, Revolution and StarFlow, which may be defined as a high flow grouping.

In Table 4, the average film thicknesses and standard deviations calculated are reported for flowable resin composites at different pressures. At the lowest pressure (.016 MPa) only Revolution had a film thickness less than 25 microns, a usual ADA recommendation for cementation. This accepted clinical criteria of less than 25 microns in film thickness for cements is used to judge the adequacy of these materials for clinical cementation rather than a statistical evaluation comparing their mathematical differences. At the middle pressure (0.027 MPa), Revolution, Starflow and FloRestore met the cement film thickness of less than 25 microns. At the higher pressure (0.038 MPa), Revolution, StarFlow, AeliteFlo LV, FloRestore, Flow-It and Aelite Flo met cement film thickness

Table 4: The Average Film Thickness and Standard Deviation of Flowable Resin Composite Measured in Microns at Three Applied Seating Pressures Simulating Porcelain Veneer Cementation			
Material/Pressure	.016 MPa	.028 MPa	.039 MPa
Revolution	20 ± 5	10 ± 4	9 ± 2
StarFlow	35 ± 5	16 ± 6	10 ± 5
Aeliteflo	37 ± 3	31 ± 5	20 ± 8
Aeliteflo LV	41 ± 7	29 ± 7	10 ± 1
Flow-it	43 ± 6	27 ± 7	17 ± 1
FloRestore	51 ±10	23 ± 7	17 ± 1
VersaFlo	63 ± 8	48 ± 7	41 ± 9
DuraFill Flow	75 ± 3	54 ± 9	45 ± 1
Tetric Flow	77 ± 2	44 ± 8	26 ± 1
Nexus2	79 ± 4	63 ± 1	54 ± 1

requirements of less than 25 microns. Tetric Flow was not considered different within the error of measurement. Versaflo and Duraflow Flow did not meet the film thickness criteria for cementation of porcelain veneers at the pressures tested. Nexus2 had the highest film thickness at all pressures tested.

## DISCUSSION

The flow characteristics of these flowable composites are such that they might be divided into three groups. A high flow group composed of StarFlow; Revolution and Aeliteflo LV; a medium flow group composed of FloRestore, Duraflow Flow and Flow-It; and a low flow group composed of Tetric Flow, Versa Flow and Aeliteflo. When considering selection of a product for applications as indicate in Table 1, selection consideration should be given to the advantages of the flow characteristic to the intended use.

Comparison of the film thicknesses tested at various pressures to the flow diameter tested suggested that there are some anomalies in that the highest flow diameter did not guarantee the lowest film thickness. For example, Revolution had a lower film thickness than StarFlow and met the 25 micron criteria at a lower pressure, yet, StarFlow was judged to flow more by the flow diameter tests. Duraflow Flow had a high film thickness but a medium diameter flow test. Duraflow Flow never met the film thickness criteria even though AeliteFlo and Flow-It, two resin composites with less flow as gauged by the flow diameter test, met the 25-micron criteria at the higher pressure. These variations in characteristics may be related to the filler content, particle size and distribution, and the resin monomers used.

It should be noted that the two materials with the highest film thickness, Duraflow Flow and Versaflo, have different filler particles and degrees of fill as found by Tabassian & Moon (1999). Duraflow is a lower filled microfill composite and Versaflo is highly filled hybrid composite. Another difference between these two materials as found by Carter & others (1999) was that Versaflo had the highest transverse strength of the nine flowable composites tested and Duraflow Flow had the lowest transverse strength, about 50% lower.

An encouraging result of this study was that seven of the flowable composites could be used to cement porcelain veneers based on the ADA film thickness criteria at the higher seating pressure. This pressure (0.038 MPa) represents a seating force of 1.9 newtons (0.43 lbs.) on a 0.5 centimeter square area, an area typical of a central maxillary incisor veneer. The other seating pressures (0.016 MPa and 0.027 MPa) represent seating forces on a 0.5 cm<sup>2</sup> area veneer of 0.17 lbs and 0.31 lbs, respectively. All the flowable composites had a film thickness less than Nexus2, the control luting resin composite.

According to Kerr Inc, Nexus2 has an average particle size of 0.6 microns. The particle size range for flowable composites as found by Tabassian & Moon (1999) are given in Table II. The greater filler content of Nexus2 is an explanation of its greater film thickness.

A high film thickness can play a role in several clinical concerns regarding porcelain veneers, including marginal discrepancy, wear of the luting resin and fracturing from resin shrinkage stresses, along with thermal cycling stresses and fracturing during placement. Nexus2 would be expected to have a larger marginal discrepancy than flowable composites when used to seat porcelain veneers at low pressure. An increased marginal gap is a concern for resin wear or ditching at the margins. However, the increased filler in Nexus2, compared to the flowable composites, may compensate for the increased discrepancy.

## CONCLUSIONS

1. The range of flow characteristics measured among flowable resin composites indicated that a selection of flowable resin composite can be made of a high, medium or low flowable composite if it is advantageous for a specific application. For most applications, which flow characteristic is preferred is yet to be proved.
2. Film thickness measurements suggest that many flowable composites, even those in the high flow range, may increase the marginal discrepancy at the margins of close fitting porcelain veneers if used for veneer cementation and seated at the lowest seating pressure tested (.016 MPa). However, they had a lower film thickness than the control Nexus2 luting resin at all pressures tested.
3. Many flowable composites in the high and medium flow ranges can produce film thicknesses of less than 25 microns and may be suited to lute porcelain veneers if seated at the highest seating pressure tested (.038Mpa). However, other factors such as wear resistance, bond strength, ease of clean up and esthetic concerns need to be evaluated to confirm their use by laboratory and clinical testing.

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# Fracture Strength of Amalgam Crowns with Repaired Endodontic Access

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## Clinical Relevance

Endodontic access through an amalgam crown and subsequent amalgam occlusal restoration significantly compromises the fracture resistance of the original restoration.

## SUMMARY

Endodontic therapy is accessed occlusally in posterior teeth, many of which have large, pre-existing amalgam restorations. These teeth are also commonly restored with an occlusal amalgam to repair the access opening. This study determined the fracture resistance of complex amalgam restorations that have repaired endodontic access compared with original, unrepaired, complex amalgams on endodontically-treated teeth. Two groups of 30 molars were used in the study. The first group was decoronated and received an endodontic access preparation. These teeth were restored using chamber retention and four TMS pins. The second group was decoronated and restored using pin retention. Later, they received an endodontic access through the restoration. The access was then repaired with amalgam. The

samples were loaded in an Instron Universal Testing Machine until failure. The Group 1 samples failed at a mean force of 2297.5 N. The mean failure load for the samples in Group 2 was 1586.1 N. Student's t-test found this difference to be statistically significant. Endodontic access through an amalgam crown significantly compromises the fracture strength of the original restoration.

## INTRODUCTION

Dental amalgam remains the material of choice in many clinical situations due to its ease of manipulation, adequate physical properties, low cost and long-proven clinical track record (Anderson & McCoy, 1993). A complex amalgam is defined as a restoration that replaces one or more cusps of a tooth with amalgam when inadequate tooth structure remains, providing for the retention and resistance of the restoration (Plasmans, Welle & Vrijhoef, 1986). The literature reveals that complex amalgams have good long-term survivability. Martin & Bader (1997) found that four and five surface amalgams had a favorable five-year outcome. Smales & Hawthorne (1997) reported that the median survival time for extensive amalgam restorations was 14.6 years. Smales (1991) demonstrated that amalgam crowns might last more than 15 years. Robbins & Summitt (1988) reported that complex amalgam restorations had a 50% survival rate at 11.5 years, compared with only six years for full cast crowns.

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Complex amalgam restorations require adequate resistance and retention features to be successful. Resistance is defined as the ability of a restoration to resist dislodgement by oblique forces (Plasmans & others, 1987). Retentive features designed to increase the longevity of complex amalgam restorations have been studied (Plasmans & others, 1986; Buikema & others, 1985; Burgess, 1985). Summitt & others (1991) found that threaded stainless steel pins and amalgapins provide superior resistance over a peripheral shelf. Burgess, Alvarez & Summitt (1997) demonstrated that pins combined with adhesive bonding provide greater resistance to fracture than pins alone.

The ability to repair a complex amalgam restoration can increase its longevity. Cowan (1983) demonstrated that bonding new amalgam to set amalgam may be a clinically acceptable technique. However, the bond strength of new amalgam condensed against set amalgam is less than half the strength of unrepaired amalgam (Walker & Reese, 1983). Jessup & others (1998) found that repaired amalgam is only 7-18% as strong as the original amalgam. Conversely, Brown & others (1986) and Hadavi & others (1992) reported repair strengths of 84% and 79%, respectively. Amalgam repair appears to be an unreliable technique from a strength standpoint, therefore, the operator must be sure to include adequate retention and resistance features (Terkla, Mahler & Mitchem, 1961). MacInnis & others (1992) investigated the repair of endodontic access in amalgam cylinders and found that wetting the amalgam with mercury-rich amalgam significantly increased the compressive strength of the repair as compared to the cores that were restored without wetting. Unfortunately, wetting the amalgam did not improve the diametral tensile strength of the repaired specimens. The original, unrepaired specimens were significantly stronger in both categories than either the wet or unwet specimens. Still, MacInnis & others (1992) concluded that amalgam refilling of access preparations through amalgam cores had potential as a clinical technique and should be investigated further.

Teeth restored with complex amalgam restorations may have had significant caries, necessitating cusp reduction and/or intracoronal pin placement. The pulpal health of these teeth may have been affected by deep caries or placement of multiple restorations (Abou-Rass, 1982). It has also been shown that pin placement can lead to pulpal pathology (Suzuki, Goto & Jordan, 1973).

One area where complex amalgam restorations are frequently placed is in the restoration of endodontically-treated teeth. Kane, Burgess & Summitt (1990) demonstrated that when endodontically-treated teeth are restored with amalgam, the amalgam should be limited

to the pulp chamber. Extension into the canal space did not increase resistance. However, another study advocated the use of an amalgam radicular dowel in the restoration of endodontically-treated teeth (Nayyar, Walton & Leonard, 1980). Plasmans & others (1986) concluded that in multi-rooted teeth, the operator should not remove too much tooth structure for a cast dowel and core and that chamber retention was a better method to preserve sound dentin.

The clinical question is whether repair of the endodontic access preparation in complete cuspal coverage amalgam restorations is adequate to resist fracture. Repair of the access preparation would certainly be preferable to removing the entire existing amalgam and pins and placing a new restoration. This study compared the fracture resistance of complex amalgam restorations that had an endodontic occlusal access repaired with amalgam versus original, unrepaired complex amalgam restorations on endodontically-treated teeth.

## METHODS AND MATERIALS

A total of 60 human mandibular third molars extracted within six months were collected and sorted according to size, then divided into two groups of 30 so that equal size distribution was represented in both groups. The teeth were stored in 0.5% chloramine-T solution from the time of extraction until their use in the study.

The teeth were embedded in orthodontic resin (Dentsply/Caulk, Milford, DE 19963, USA) with occlusal surfaces parallel to the floor and the cemento-enamel junction (CEJ) 2 mm above the level of the resin. The first group of 30 teeth was decoronated with an Isomet saw (Beuhler, Ltd, Lake Bluff, IL 60044, USA) to a level 2 mm coronal to the CEJ. They then received an endodontic access preparation. Canal orifices were filled with gutta percha to prevent amalgam from being condensed into the canals. Four TMS regular pins (Coltene/Whaledent Inc, Mahwah, NJ 07430, USA) were placed in each tooth at the line angles 1 mm inside the dentoenamel junction (DEJ) and 2 mm deep into dentin. The pins were cut so that they extended 2 mm occlusally from the preparation. A copper band (Union Broach, Long Island, NY 11735, USA) was placed on the tooth as a matrix. Dispersalloy dental amalgam (Johnson & Johnson, East Windsor, NJ, USA) was triturated according to manufacturer's instructions in a Vari-Mix II M triturator (Dentsply/Caulk) and hand condensed by the same operator into each preparation and pulp chamber. The copper band was removed after 10 minutes and any flash or over-extension of amalgam was removed. The occlusal surface was adjusted to provide a 2 mm height of amalgam above the occlusal extent of the pins. The specimens were then stored in water for 30 days at 37°C.

The second group of 30 teeth was decoronated, then four TMS regular pins were placed as previously described. Copper bands were placed and the teeth were restored with Dispersalloy amalgam as previously described. These specimens were stored in water for 30 days at 37°C. This group then received an endodontic occlusal access through the amalgam restoration with a #557 crosscut fissure bur (SS White, Piscataway, NJ 08812, USA). The canal orifices were filled with gutta percha to prevent amalgam from being condensed into the canals. The access preparations were repaired with Dispersalloy amalgam. Specimens in this group were then stored in water for seven days.

Each specimen had a groove machined into the occlusal surface with a Degusa milling machine (GB Dental, Frankfurt, Germany). Using a positioning device, each specimen was loaded on the occlusal surface at 45° with a flat-ended rod in an Instron Universal Testing Machine (Instron Corp, Canton, MA 02021, USA) at a crosshead speed of 5 mm per minute until failure. The specimens in Group 1 were tested 30 days after they were prepared. The samples in Group 2 were tested seven days after the restoration of the endodontic access. The force required to fracture each specimen was recorded. Data were analyzed using a two-tailed Student's *t*-test at  $\alpha = 0.05$ .

## RESULTS

The mean fracture force for Group 1 was 2297.5 N  $\pm$  903.1. The mean fracture force for Group 2 was 1586.1 N  $\pm$  586.2. Student's two-sample *t*-test was found to be statistically significant ( $p < 0.05$ ). Twenty-two of the 30 teeth of Group 1 fractured through the pulp chamber, rendering them non-restorable. Only seven of the teeth of Group 2 were non-restorable.

## DISCUSSION

A common cause of failure in endodontically-treated teeth is inadequate final restoration (Sorensen &

Martinoff, 1984). It may be tempting to repair the endodontic access in a tooth that has a pre-existing full coverage amalgam restoration with an occlusal amalgam. The fact that the tooth already has cuspal coverage may lull the practitioner into a false sense of security. The results from this study suggest that complex amalgam restorations are significantly weakened when an occlusal endodontic access opening is made and subsequently refilled with amalgam. This finding is consistent with other reports that studied the poor repair potential of set amalgam (Jessup & others, 1998; Leelawat & others, 1992; Lacy, Rupprecht & Watanabe, 1992).

Under the conditions of this study, repair or refilling of occlusal access openings will lower the fracture resistance of the restoration. In contrast, an earlier study (MacInnis & others, 1992) concluded that amalgam refilling of access preparations through amalgam cores had promise as a clinical technique. The basis for this conclusion was that the compressive strength of specimens from the repair group using mercury-rich amalgam was as strong as the original controls. The results from MacInnis' tensile strength test, however, revealed that repaired samples were weaker than the original controls. Amalgam, being a brittle material, will fail in tension before compression; therefore, MacInnis' (1992) research actually supports the current study.

The repaired (Group 2) specimens failed at the margin of the repaired portion of the restoration. Since repaired amalgam is less than half as strong as the original amalgam, this is not unexpected: these restorations fractured at their weakest point. An interesting observation is that in the repaired (Group 2) specimens, the crack usually propagated to a location that could still be restored clinically. Only 23% of Group 2 failures were deemed non-restorable. In contrast, 73% of original unrepaired (Group 1) complex amalgams failed catastrophically (Figure 1). The significance of the undesirable location of Group 1 complex amalgam failures may not be clinically important, however, because all samples were tested until they fractured. The important mechanical property to test would be the yield stress, or stress that first starts to permanently deform a material, but this is difficult to measure in amalgam (Ashby & Jones, 1996). Forces that caused failure in Group 1 specimens are not likely to occur in the normal human dentition. The force required to cause catastrophic failure of both experimental groups was well above the mean maximal clenching force of humans with natural teeth, reported as 721 N (Gibbs & others, 1981). However, a biting force of 4337 N was recorded for a bruxer (Gibbs & others, 1986). While numerical data from *in vitro* tests should not be extrapolated to other studies, this information still suggests that the forces required to cause clinical failure in repaired complex amalgams might be within reach, particularly



Figure 1. Catastrophic failure of the complex amalgam restoration.



those forces that caused failure in the repaired (Group 2) specimens. This is especially true if we consider the case of a premature contact on a small spot of the restoration or biting into a small hard object. Since stress is a function of force per unit area, small areas can generate tremendous stresses capable of tooth or restoration fracture.

Another problem with making an endodontic access through amalgam crowns is the possibility of disturbing the pin retention. The operator may cut into the pins when making the access preparation or may simply leave a thin shell of amalgam around the pins. In either case, retention and resistance are compromised and the restoration is significantly weakened. This risk is especially high when the operator did not place the original restoration and does not know the location of the pins.

*In vitro* data and retrospective studies suggest that endodontically-treated posterior teeth require coronal coverage (Sorensen & Martinoff, 1984). The results of the current study suggest that occlusal amalgam repair of endodontic access openings significantly weakens the tooth-restoration complex. However, it must be emphasized that the results of *in vitro* tests should be viewed cautiously as they may not always correlate well to the clinical situation. Although a statistically significant difference was detected between the two experimental groups, there still is no compelling clinical evidence to prove that repaired occlusal access openings may not suffice as a final restoration. Clearly, clinical research is necessary to confirm the preliminary findings from this study.

### CONCLUSIONS

Under the conditions of this study, complex amalgam restorations that were later accessed endodontically and subsequently repaired with amalgam in the occlusal access were significantly weaker than unrestored original amalcore restorations. Observation of failure modes revealed that 23% of the repaired complex amalgams failed catastrophically.

### 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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# Short-Term Fluoride Release from Various Aesthetic Restorative Materials

AUJ Yap • SY Tham  
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## Clinical Relevance

Giomers may not be as beneficial as glass ionomer cements in patients who are at risk for recurrent caries, as their long-term fluoride release is questionable.

## SUMMARY

The short-term fluoride release of a giomer (Reactmer), a compomer (Dyract AP), a conventional glass ionomer cement (Fuji II Cap) and a resin-modified glass ionomer cement (Fuji II LC) was evaluated and compared. Specimen discs ( $6 \pm 0.2$  mm diameter and  $1 \pm 0.2$  mm thick) were prepared for each material using custom molds. Each disc was placed in 1 ml of deionized for 24 hours at 37°C. After one day, the water was extracted and analyzed. The specimen discs were then re-immersed into another 1 ml of fresh deionized water. The procedure of removing and refilling the water was repeated for 28 days. Sample solutions taken during the first seven

days and at days 14, 21 and 28 were introduced into a capillary electrophoresis system using field amplified sample injection (FASI) to determine fluoride release. Data was analyzed using factorial ANOVA/Scheffe's post-hoc test at significance level 0.05. An initial fluoride "burst" effect was observed with glass ionomers. Both compomer and giomer did not show an initial fluoride "burst" effect. With the exception of the compomer, fluoride release at day one was generally significantly greater than at the other time intervals. The glass ionomers released significantly more fluoride than the compomer and giomer at day one. Although fluoride release of the giomer was significantly greater than the other materials at day seven, it became significantly lower at day 28.

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## INTRODUCTION

Secondary or recurrent caries is by far the most frequent reason for replacement of restorations (Mjör, 1985). It is by definition found at the tooth-restoration interface and is, in general, the result of microleakage (Arends, Dijkman & Dijkman, 1995). Microleakage is defined as the clinically undetectable passage of bacteria and fluids between cavity walls and restorative materials. The loss of marginal integrity between the aforementioned provides potential pathways for reinfection,

as cariogenic bacteria can easily penetrate into the underlying dentin through these defects (Brännström & Nordenvall, 1978). These micro-organisms are responsible for the demineralization of adjacent dentin and/or enamel via a chemical process presumed to be similar to those in primary caries (Arends & others, 1995). As the marginal seal of tooth-colored restoratives to tooth tissues are still not perfect (Yap, Lim & Neo, 1995; Sjodin, Uusitalo & Van Dijken, 1996), anti-bacterial properties are desirable.

Glass ionomer cements have demonstrated *in vitro* anti-bacterial properties (DeSchepper, White & Von der Lehr, 1989; Scherer & others, 1989). Secondary caries initiation and propagation were found to be significantly reduced when glass ionomer restorations were placed (Retief & others, 1984; Donly & others, 1999; Hicks & Flaitz, 2000; Torii & others, 2001). This favorable result has been attributed in part to the release of high concentrations of fluoride ions (DeSchepper & others, 1989; Scherer & others, 1989; McComb & Ericson, 1987). Fluoride acts in several ways to prevent caries. It inhibits demineralization and promotes remineralization, thus encouraging repair or arrest of carious lesions. Depending on its concentration and pH, fluoride can also exert a bactericidal or anti-enzymetic effect (Joyston-Bechal & Kidd, 1994). Because of the beneficial effects of fluoride, extensive work on the rate of fluoride release from restorative materials has been conducted. Fluoride ion-selective electrodes (with ion analyzer) and ion chromatography (IC) are the most frequently employed techniques for measurement of fluoride release (Yap, Khor & Foo, 1999; Verbeeck & others, 1998). Capillary electrophoresis (CE) is a relatively new method of analysis that has been recently introduced for dental research (Yap, Lee & Sabapathy, 2000). It is a separation technique that uses a simple capillary and high voltage to separate components of a sample. The sample is introduced into a capillary field filled with an electrolyte. A high voltage is applied, causing the components to migrate toward the end of the capillary at different rates depending upon charge and size (Sabapathy & others, 2000).

Resin-modified glass ionomers and compomers were developed to overcome the problems of conventional glass ionomer cements, which include early, low mechanical strengths and poor esthetics resulting from moisture sensitivity. Although the fluoride release of

these tooth-colored restoratives had been widely reported (Yap & others, 1999; Verbeeck & others, 1998; Attin & others, 1999; Eliades Kakaboura & Palaghias, 1998; Forsten, 1998), no literature is available regarding the release of fluoride by giomers. The latter, also known as PRG composites, is a new class of hybrid composite restoratives that employs the use of pre-reacted glass ionomer (PRG) technology. Unlike compomers, the fluoroaluminosilicate glass is reacted with polyacrylic acid prior to inclusion into the urethane resin. The manufacturer's claims include fluoride releases, fluoride recharge, biocompatibility, smooth surface finish, excellent aesthetics and clinical stability. Like compomers, giomers are light polymerized and require the use of bonding systems for adhesion to tooth structure.

The study evaluated the amount and pattern of fluoride release by a giomer over 28 days. The results were compared to those of conventional and resin-modified glass ionomer cements and a compomer.

## METHODS AND MATERIALS

The materials used in the study are summarized in Table 1. They included a conventional glass ionomer cement (Fuji II Cap [FC]), a resin-modified glass ionomer cement (Fuji II LC [FL]), a compomer (Dyract AP [DY]) and a giomer (Reactmer [RA]). Four specimen discs, six  $\pm$  0.2 mm diameter and one  $\pm$  0.2 mm thick, were fabricated using custom-made molds for each material. A polyester film (Hawe-Neos Dental, Bioggio, Switzerland) was first secured on a glass slide. This first glass slide formed the base of the mold. The restorative material was placed into the mold and covered with a second polyester film. A second glass slide was then placed over the mold and pressure was applied to extrude excess material. The light-cured restorative materials were then light-polymerized according to manufacturer's cure times through the glass slide with a polyLUX II light-cure unit (KaVo Dental, Warthausen, Germany). The intensity of the light source was checked with a radiometer (CureRite, EFOS Inc, Ontario, Canada) before the start of the experiment. The mean output was  $540 \pm 15$  mW/cm<sup>2</sup> and output was not affected by illumination through the glass slide and polyester

Table 1: *Materials Used in the Study*

Material Type	Product	Manufacturer	Lot #/Color	Light-Curing Time
Conventional Glass Ionomer Cement	Fuji II Cap	GC Corporation, Tokyo, Japan	9905255 Shade 22	NA
Resin-modified Glass Ionomer Cement	Fuji II LC	GC Corporation, Tokyo, Japan	990817 Shade A2	20 seconds
Compomer resin	Dyract AP	Dentsply-DeTrey, Konstanz, Germany	0003001083 Shade A2	40 seconds
Giomers	Reactmer	Shofu Inc, Kyoto, Japan	0400 Shade A3	30 seconds

films. The chemically-cured conventional glass ionomer cement FC was allowed to set for 15 minutes. Immediately after light-polymerization/setting, the polyester films were discarded and the discs immersed in 1 ml of deionized water in small, airtight containers for 24 hours at 37°C. After 24 hours, the containers were thoroughly shaken and the water removed and analyzed. The specimen disks were then re-immersed into another 1 ml of fresh deionized water. The removing and refilling of the water procedure was repeated for 28 days. Sample solutions during the first seven days and at days 14, 21 and 28 were analyzed by capillary electrophoresis (CE) (Prince Technologies, Emmen, Netherlands) immediately after collection. The anions were identified via indirect UV detection at 245 nm using an ultra-violet/visible detector system (Bischoff, Leonberg, Germany).

Pyromellitic acid buffer solution (pH 7.7) was used as the background electrolyte (BGE), as it gave optimal sensitivity. The capillary (50 µm internal diameter and 64 cm long) was conditioned at each experimental session by hydro-dynamically pumping 1 M sodium hydroxide through it at 1200 mBar for two minutes. This was followed by pumping 0.2 sodium hydroxide at 1200 mBar for six minutes. Water was then pumped through at 1200 mBar for 20 minutes. For field amplified sample injection, the BGE was pumped through at 1200 mBar for eight minutes. A water plug was injected at 50mBar for 0.1 minutes to pre-concentrate the sample/standard solutions. The sample/standard solutions were injected electrokinetically for two minutes by applying a negative voltage of -10 kV. This reverse polarity ensured that the mobility of the anions is directed towards the outlet. The inlet vial was replaced with fresh run buffer and a negative voltage of -25 kV was applied at both ends of the capillary for 20 minutes to initialize the stacking and separation. In-between runs, the capillary was flushed with 1M sodium hydroxide, 0.2 M sodium and water for two minutes at 1200 mBar.

Standard concentrations of 5, 1, 0.5, 0.1 and 0.05 ppm fluoride solutions were first analyzed by CE. The corresponding areas of their peaks were plotted against their concentrations to obtain a calibration plot. One ppm fluoride standard solution was injected five times to ensure reproducibility. The fluoride released by the different materials was

then calculated from the calibration plot. A significance level of 0.05 was used for all statistical analysis. One-way ANOVA and Scheffe's post-hoc test were performed on the data with materials as independent variable and fluoride release as dependent variable. Significant differences between mean fluoride release of the materials at 1, 7, 14, 21 and 28 days was also determined.

## RESULTS

The mean fluoride release at days 1-7, 14, 21 and 28 are reflected in Table 2. Cumulative mean fluoride released by the various materials during the first seven days is shown in Figure 1. Figure 2 shows the mean fluoride release at days 1, 7, 14, 21 and 28. Results of statistical analysis are shown in Tables 3 and 4.

An initial fluoride "burst" effect was observed with the conventional and resin-modified glass ionomer cements during the first five days. It was, however, not observed with the compomer and giomer. Fluoride release by the latter materials was more gradual during the first week. Cumulative fluoride release over seven days was 38.67, 39.03, 5.40 and 17.74 ppm for FC, FL, DY and RA, respectively (Figure 1). Ranking of fluoride release differed at the various time intervals. Ranking by materials was as follows: day 1 - FC > FL > RA > DY; day 7 - RA > FC > FL > DY; day 14 - FL > FC > DY > RA; day 21 - FL > FC > DY > RA; and day 28 - FL > DY > FC > RA. Fluoride release by the giomer was consistently the lowest at days 14, 21 and 28. Excluding the compomer, fluoride release at day one was generally significantly greater than at days 7, 14, 21 and 28. Fluoride release ranged from 8.78 to 4.54 ppm at day one but dropped to 3.18 to 0.06 ppm at day 28. For FC, fluoride release at day 14 was also significantly greater than at days 7, 21 and 28. Fluoride release at day 14 was significantly greater than at day seven for both FL and DY. RA released significantly more fluoride at day seven compared to days 21 and 28.

The glass ionomers released significantly more fluoride than the giomer and compomer at day one.

Table 2: Mean Fluoride Release (ppm) at Different Time Intervals

Material	Fuji II Cap	Fuji II LC	Dyract AP	Reactmer
Day 1	8.78 (0.03)	7.19 (0.34)	1.44 (0.03)	4.54 (1.00)
Day 2	7.89 (0.76)	5.77 (0.81)	0.94 (0.28)	1.35 (0.16)
Day 3	6.83 (0.92)	6.51 (0.97)	0.88 (0.11)	2.75 (0.18)
Day 4	6.32 (0.56)	7.52 (1.24)	0.70 (0.04)	2.58 (0.44)
Day 5	4.67 (0.21)	7.29 (0.80)	0.56 (0.37)	1.72 (0.54)
Day 6	3.03 (0.27)	3.81 (0.44)	0.65 (0.14)	2.63 (0.39)
Day 7	1.15 (0.11)	0.95 (0.21)	0.22 (0.13)	2.00 (0.46)
Day 14	5.44 (0.67)	6.29 (1.94)	3.01 (0.67)	1.22 (0.16)
Day 21	1.27 (0.58)	3.24 (0.57)	1.03 (0.66)	0.11 (0.11)
Day 28	1.51 (0.36)	3.18 (0.30)	1.69 (0.27)	0.06 (0.13)

Standard deviations are reflected in parenthesis.



Table 3: Significant Difference in Mean Fluoride Release Between the Different Time Intervals	
Materials	Differences
Fuji II Cap	Day 1 > Days 7, 14, 21, 28 Day 14 > Days 7, 21, 28
Fuji II LC	Day 1 > Days 7, 21, 28 Day 14 > Day 7
Dyract AP	Day 14 > Day 7
Reactmer	Day 1 > Day 7, 14, 21, 28 Day 7 > Days 21, 28
Results of one-way ANOVA and Scheffe's post-hoc test ( $p < 0.05$ ) > indicates statistical significance.	

Table 4: Significant Difference in Mean Fluoride Release Between Material	
Time Interval	Differences
Day 1	FC > FL, DY, RA FL > DY, RA
Day 7	RA > FC, FL, DY FC, FL > DY
Day 14	FC, FL > RA
Day 21	FL > FC, DY, RA
Day 28	FL > FC, DY, RA FC, DY > RA
Results of one-way ANOVA and Scheffe's post-hoc test ( $p < 0.05$ ) > indicates statistical significance. FC = Fuji II Cap; FL = Fuji II LC; DY = Dyract AP; RA = Reactmer.	

Although fluoride release of the giomer was significantly greater than the other materials at day seven, it became significantly lower at day 28. Significant differences in fluoride release were also observed between the glass ionomers and DY at day seven and RA at day 14. The resin-modified glass ionomer cement (FL) released the most fluoride at days 14, 21 and 28, with significantly greater amounts than the other materials evaluated at days 21 and 28.

## DISCUSSION

CE is a relatively new analytical tool and its wide applications have not been fully exploited. The amount of sample solutions required with CE is much lower than with IC and ion-selective electrodes. Separation efficiency of CE is also higher than that of IC due to the number of theoretical plates and lesser band broadening. Since CE uses electro-osmotic flow rather than hydrodynamic flow, mixing is minimized. All the aforementioned contributes to the shorter retention times in CE compared to IC. Although the sensitivity of CE is lower than IC, this disadvantage can be overcome by on-line concentration of the sample using field amplified sample injection (Yap & others, 2000). A 28-day period was chosen because fluoride-releasing composites cause a substantial caries reduction related to fluoride release after 28 days (Dijkman & Arends, 1992). The current experimental set-up does not simulate the clinical situation but gives an indication of the maximum amount of fluoride release possible. Clinically, fluoride is probably not washed away as

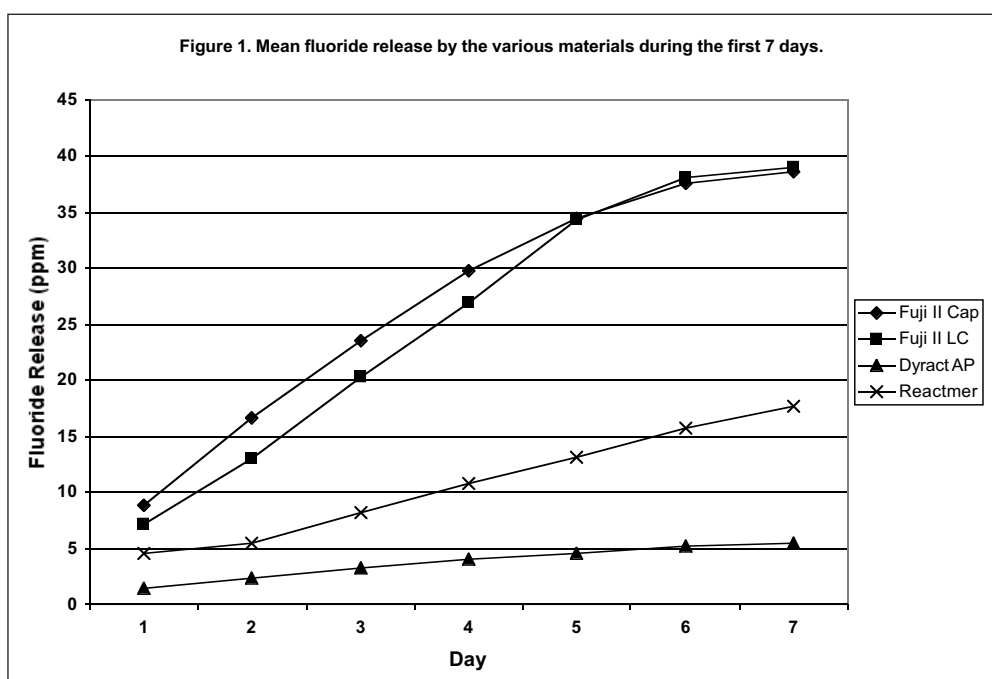


Figure 1. Mean fluoride release by the various materials during the first seven days.

completely as in this experiment. Biofilms and layers on the surface of restorations may reduce fluoride release, as part of the released fluoride will be accumulated in these layers. Uptake of part of the released fluoride back into the restoration is therefore possible (Tam, Chan & Yim, 1997). Deionized water was selected as more fluoride is released in deionized water than in artificial saliva (El-Mallakh & Sarkar, 1990). Determination of fluoride release is very much dependent on methodology. The frequency with which the storage water is changed is perhaps the most critical factor. It has been reported that fluoride levels in test solutions can equilibrate within one hour for some lining materials while little change occurs for luting materials after 24 hours (Dunne & others, 1996). Hence, changing the storage water at 24-hour intervals is a minimum requirement (McCabe, 1998).

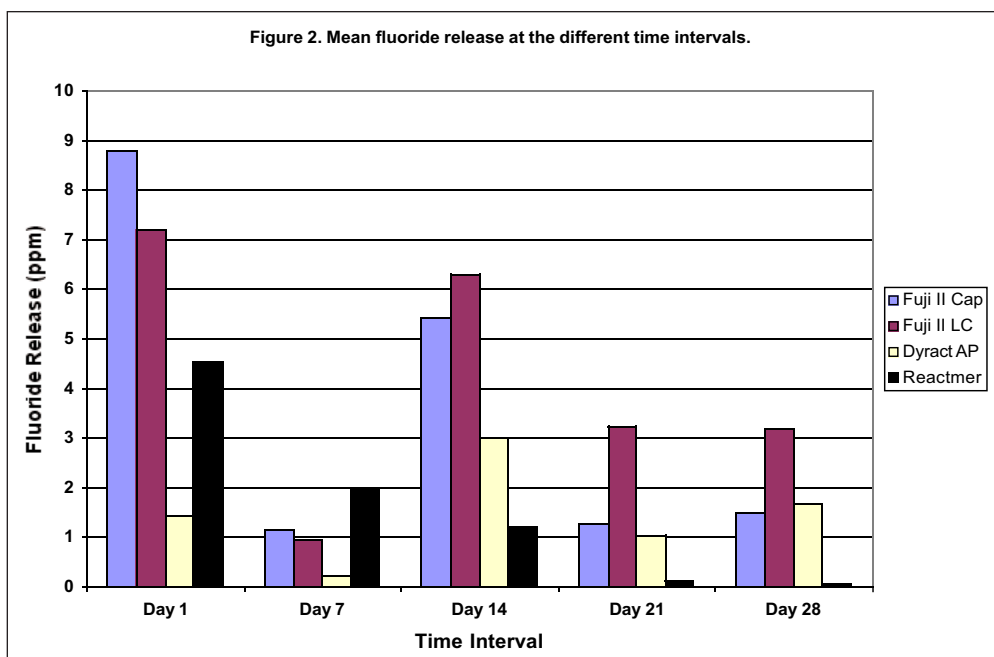


Figure 2. Mean fluoride release at different intervals.

The amount of fluoride release necessary for “curing” carious lesions and for anticariogenic effects has not been documented. Such a therapeutic dose of fluoride may not exist and warrants further investigation. The content of fluoride in restorative materials should, however, be as high as possible without adverse effects on physico-mechanical properties and the release should be as great as possible without causing undue degradation of the filling. An initial fluoride “burst” effect is desirable, as it will reduce the viability of bacteria that may have been left in the inner carious dentin and induce enamel/dentin remineralization (Forsten, 1998). This initial “burst” effect was only observed with the glass ionomer cements. This finding is consistent with other studies conducted on fluoride release of materials using IC and ion-selective electrodes (Yap & others, 1999; Verbeeck & others, 1998; Forsten, 1998; Shaw, Carrick & McCabe, 1998). Both compomer and giomer did not display an initial fluoride “burst” effect. In compomers, the functional groups of polyacrylic acid and methacrylates are combined into one molecule. Light-curing results in a setting process analogous to that of composite resins. Subsequent water sorption leads to ionization of the acid groups and an acid-base reaction resulting in fluoride release in a similar manner to that of glass ionomers. As the aforementioned reaction takes place at a slow rate and reaches a saturation point after approximately four weeks (Eliades & others, 1998), cumulative fluoride release during the first seven days is expected to be low. Although the giomer did not have an initial “burst” effect, its cumulative fluoride release during the first week was higher than the compomer.

Unlike the latter, the fluoroaluminosilicate (FAS) glass particles in Reactmer had already been pre-reacted with polyacrylic acid. Water sorption is therefore not critical in the acid-base reaction process. Fluoride release is probably via an exchange mechanism in the direction of the lowest fluoride concentration. As the existence of a concentration gradient is the driving force for fluoride release, the amount of fluoride release is expected to decrease with time due to diminishing gradient, as fluoride is leached out from the material. The aforementioned explains the steady decline of fluoride release of Reactmer over the 28 day period. The fluoride release by the giomer was lower than the

glass ionomer cements, as “fresh” glass ionomers release more than twice the amount of fluoride that is released by matured specimens (Forsten, 1991). Large standard deviations were observed for both compomer and giomer specimens when fluoride release was approximately below 1 ppm. This may be attributed to a combination of the lower sensitivity of CE, the small but clinically relevant specimens used and the small volume of sample solutions utilized. Standard deviations at low concentrations of fluoride release could possibly be improved with larger specimens and greater volumes of sample solutions (Yap & others, 2000).

Excluding the compomer, fluoride release at day one was generally significantly greater than at days seven, 14, 21 and 28. For the glass ionomers, this finding may be attributed to the high instability and erosion of glass ionomers during the early setting period (DeSchepper & others, 1991; Crisp, Lewis & Wilson, 1980). The high fluoride release at day one by Reactmer is probably due to the significant difference in fluoride concentration gradient between the material and the deionized water. For both conventional and resin-modified glass ionomer cements, fluoride release at day 14 was significantly greater than at day seven. This was also observed by Verbeeck & others (1998) with other conventional and resin-modified glass ionomers. Fluoride release by glass ionomers is therefore intermittent and the exact kinetics and mechanism warrants further investigation. Eliades & others (1998), using micro-multiple internal reflectance Fourier transform infrared spectroscopy, found that

the net peak absorbance area ratio of the carboxylate salts formed to the unionized carboxyls (which reflects the extent of the acid-base reaction) and fluoride release of compomers reached a plateau after 14 days. They suggested that salt formation stabilized the structure from where fluoride ions were initially released due to dissolution. This may explain the significantly higher fluoride release at day 14 compared to day seven for Dyract. For giomer, the fluoride concentration gradient was still high at day seven. Fluoride release at day seven was significantly greater than at days 21 and 28.

Reasons for the significantly higher fluoride release by the glass ionomers compared to the compomer and giomer at day one have been mentioned earlier. Although fluoride release of the giomer was significantly greater than the other materials at day seven, it became significantly lower at day 28 due to the diminishing fluoride gradient. Fluoride release was consistently the lowest among the various materials at days 14, 21 and 28. At day 28 only 0.06 ppm of fluoride was released. This was 25-to-53 times lower than the other materials. The anticaries efficiency of such low fluoride release values has not been confirmed both *in vivo* and *in vitro*. The resin-modified glass ionomer cement released the most fluoride at days 14, 21 and 28. Fluoride release was significantly greater than the other materials evaluated at days 21 and 28. This is primarily due to ion exchange, but a degree of "wash-out" or dissolution may also contribute to the higher fluoride release. The latter is characterized by the leaching of other ions, in particular, calcium, along with the fluoride and a gradual disintegration of the material (Tam, McComb & Pulver, 1991). A reduction in hardness, flexural and compressive strength has been observed with Fuji II LC after continued storage in water at 37°C (Yap & others, 2001). However, the relationship between water sorption/solubility, which appears to be influenced by the resin (2-hydroxyethyl methacrylate) content (Yap, 1996) and fluoride release, has not been defined.

The results of this study show that giomers and compomers do not have the initial fluoride "burst" effect associated with glass ionomer cements. Although fluoride release of the giomer was significantly greater than the other materials at day seven, it became significantly lower at day 28. This shortcoming may, however, be overcome by fluoride "recharging" using topical fluorides. Unlike compomers, which cannot be replenished with fluoride (Attin & others, 1999; Forsten, 1998), giomers are claimed to be fluoride "rechargeable." This has yet to be validated by independent studies. As the long-term fluoride release of giomers is questionable, they may not be as beneficial as glass ionomer cements in patients who are at risk to recurrent caries.

## CONCLUSIONS

Under the conditions of this *in vitro* study:

1. The initial fluoride "burst" effect observed with glass ionomer cements was not demonstrated by the giomer and compomer.
2. With the exception of the compomer, fluoride release by all materials at day one was generally significantly greater than at other time intervals.
3. Although the fluoride release of the giomer was significantly greater than the other materials at day 7, it became significantly lower at day 28.
4. The resin-modified glass ionomer cement released the most fluoride at days 14, 21 and 28.

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# The Effect of Prolonged Packing on the Surface Hardness of Posterior Composites

LW Stockton • PT Williams • C Attallah

## Clinical Relevance

This study suggests that resin-based composites that require an increased packing time may have a lower KHN once polymerized. This may have a negative effect on the wear rate, and therefore, the lifespan of the composite

## SUMMARY

This study evaluated the effect of four different packing times on the Knoop Hardness Number of three composites (Surefil, Z100 and Spectrum TPH). Ten samples of each composite were prepared for each packing time and 10 readings were made on each sample to produce the KHN.

Photomicrographs were made of 147 indentations to determine whether the indentations had routinely been made on porosity-free sites.

Statistical analysis was made using two-way ANOVA and least square means. Generally, as the packing time increased, the KHN decreased, and although the indentations appeared to have been made in porosity-free composite, the potential effect of porosities was not discounted. Increasing the packing time for clinical compos-

ites may result in a lower KHN and increased clinical wear, which would further increase if also associated with porosities within the composite.

## INTRODUCTION

Recent advances in resin-based composite systems have led dentists to expect a high degree of success when restoring posterior teeth. However, to be clinically successful, resin-based composite systems must possess not only excellent adhesive, esthetic and mechanical properties, but also a high degree of long-term wear resistance (Roulet, 1987a; Roulet, 1987b). Properties that characterize the outer surface of resin-based composites are important parameters in determining their wear rate (Gladys & others, 1997).

Wear, which has been cited as a limiting factor for the use of resin-based composites as posterior restorations (Roulet, 1987b), is an exceedingly complex mechanism (Roulet, 1987b) involving numerous factors related to the material's mechanical properties and the resistance of the material's surface-to-chemical degradation. Since most mechanical and chemical properties are adversely affected by a decreased degree of polymerization, the clinician must strive for the highest degree of cure possible. And because the degree of cure may influence

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wear, factors that negatively affect cure may therefore also affect wear (Powers & others, 1993; Eldiwany, Powers & George, 1993).

Oxygen inhibits the polymerization of resins (Powers & others, 1993; Eldiwany & others, 1993; Ferracane & Condon, 1992) by reacting with the free radicals so that they are not available to induce the polymerization reaction. The inhibition can be significant. For example, thickness of the uncured layer resulting from exposure of the resin surface to air has been reported to vary from 50  $\mu\text{m}$  to 500  $\mu\text{m}$  depending on the sensitivity of the photo initiators used (Ferracane, 1985; Li & others, 1985). Therefore, it is hypothesized that air mixed into the composite during packing may not only increase the porosity, but also inhibit polymerization of composite resins, both of which can lead to a reduction in the composite's hardness.

Hardness, which is defined as the resistance of a material to indentation, cutting, scratching or abrasion (*Materials Handbook*, 1961), can be used as one indicator for the completeness of polymerization since the hardness of a polymer is directly related to its degree of cure (Powers & others, 1993).

This study determined what affect an increase in packing time would have on the Knoop hardness of three posterior resin-based composites.

## METHODS AND MATERIALS

A total of 120 samples were prepared within four groups with packing times of 20, 30, 45 and 60 seconds for each group. For the 20-second group, 10 samples of a condensable resin-based composite (Surefil, Dentsply Caulk, Milford, DE 19963, USA), and 10 samples each of two additional resin-based composites (Spectrum TPH, Dentsply Caulk, and Z100, 3M Dental Products, St Paul, MN 55144, USA) were prepared by packing the resin, using a Hu Friedy Goldstein #2 composite instrument (Hu Friedy, Chicago, IL, 60618, USA), into a plastic mold 9 mm in diameter by 4 mm deep in two approximately 2 mm-thick increments for each sample. Each increment was condensed for 20 seconds and light cured (Optilux 500, Demetron, Kerr Dental Products, Danbury, CT 06810, USA @500 milliwatts per  $\text{cm}^2$ ) for 20 seconds using a 14 mm diameter light-curing tip. The final increment was packed to give a slight overfill of the mold. The remaining three groups of samples were prepared in an identical manner

with the same three composites except the packing time for each group was 30, 45 and 60 seconds, respectively. The shade used for all composites was A<sub>2</sub>. The room in which the samples were prepared had a 12-foot ceiling with minimal ambient lighting from overhead fluorescent light fixtures.

After storage in a thermador (Precision Scientific Co, Chicago, IL 60618, USA) with no interior light at 37°C for 24 hours, all samples were trimmed to remove the resin-rich layer using 38  $\mu\text{m}$  grit paper followed by polishing through to .05  $\mu\text{m}$  aluminum oxide powder.

Ten random readings were made on each of the 10 samples for the three composites in each group using a Kentron Microtester (Torsion Balance Co, Clifton, NJ 08809, USA) and a 200 gram load. For the 120 samples, 1200 hardness measurements were made. The indentations were made in an area that appeared to be free of porosity when viewed at a magnification of x125 through the hardness tester's eyepiece. To determine whether the indentations had been routinely made on porosity-free sites, 147 indentations made on the 45-second samples were evaluated at magnifications up to x625. These sites were randomly selected from seven indentation sites on seven randomly selected samples chosen from each of the three composite brands.

The Knoop Hardness Number (KHN) for each composite was calculated as an average of the 100 readings for the 10 samples.

A two-way ANOVA was completed for both packing times and materials. Since there was a significant interaction between the two factors, pairwise comparisons by least square means tests were done.

## RESULTS

Although bands and aggregates of porosity of varying sizes were visible in all samples evaluated (Figure 1), only nine of 147 indentations evaluated appeared to involve a visible porosity.

Figure 1 a-d shows the surface characteristics of composite resin samples packed for 45 seconds. Horizontal polishing scratches are visible on all samples.

In Figure 1a, bands of almost continuous porosity ( $\Rightarrow$ ) were occasionally seen in some samples. Diffuse

Table 1: Comparison of Knoop Hardness Number (KHN) at Each Packing Time of Three Composites Following Packing of 20, 30, 45 and 60 Seconds. Same Letter Indicates No Statistical Difference. Significance Level Set at  $\alpha=0.05$

Composite:	20 seconds	30 seconds	45 seconds	60 seconds
Surefil	95.25 (23.19) a	96.98 (10.88) a	89.22 (6.79) ab	86.98 (6.06) b
Spectrum TPH	80.38(10.55) c	76.56(10.52) c	78.68 (13.59) c	69.55 (4.44) d
Z 100	141.92 (22.72) e	127.47 (19.39) f	126.44(15.51) f	120.80(15.13) g

KHN for packing times of 20, 30, 45 and 60 seconds; SD in parentheses

regions believed to be porosity ( $\Rightarrow$ ) that lies just beneath the surface are visible through the translucent composite. These were visible in most samples.

In Figure 1b, the diffuse regions of porosity, when seen at higher magnifications, appear to be composed of a large number of porosities lying at various depths beneath the resin surface.

In Figure 1c, an exposed porosity approximately 80  $\mu\text{m}$  in diameter can be seen in the upper left corner. Diffuse objects believed to be subsurface porosity are also visible. A vertical hardness indentation in a porosity-free region is faintly visible between the two arrows ( $\Rightarrow$ ).

In Figure 1d, a vertical hardness indentation is faintly visible between the two arrows ( $\Rightarrow$ ) in a porosity-free region of a sample riddled with porosity. Most of the porosity appears to lie just beneath the surface. Some porosity has been exposed and the largest of these is approximately 90  $\mu\text{m}$  in diameter.

The KHN of all three composites are very different from one another (Table 1), with Z100 having the highest hardness values and Spectrum TPH the lowest.

For each packing time, all types of resin-based composites showed a highly significant decrease in KHN (Table 1) after 60 seconds of condensing ( $p < 0.01$ ). Although the 30-second Surefil samples and the 45-second Spectrum TPH samples showed a slight but statis-

Figure 1. Surface characteristics of several different composite resins that have been packed for 45 seconds.

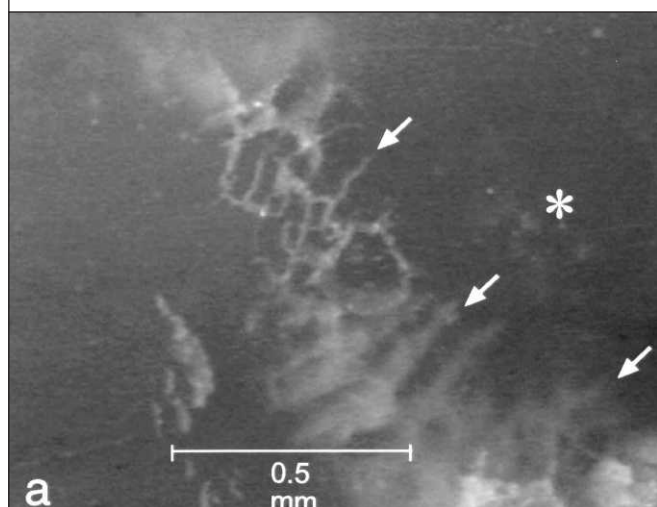


Figure 1a. Continuous bands of densely packed porosity ( $\Rightarrow$ ) and regions of subsurface porosity are visible immediately below the asterisk (\*) (magnification  $\times 80$ ).

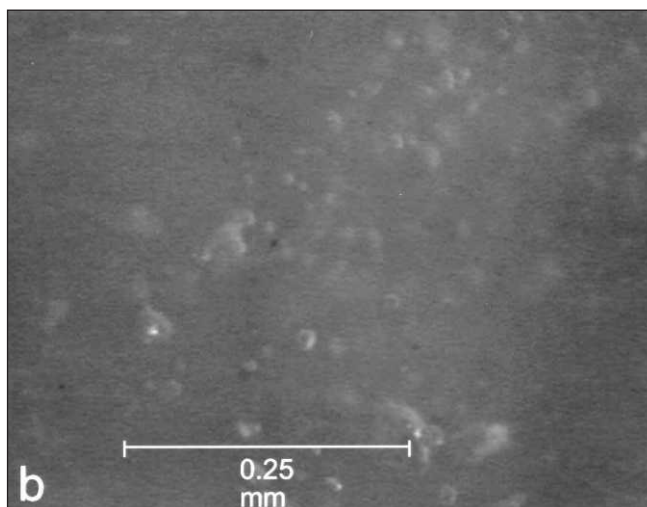


Figure 1b. At a higher magnification, the individual porosities can be seen (magnification  $\times 200$ ).

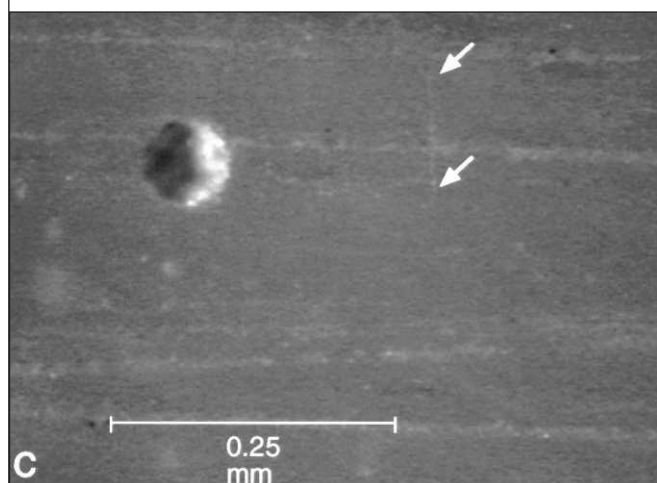


Figure 1c. A single large exposed porosity is present. A hardness indentation can be seen between the two arrows ( $\Rightarrow$ ) (magnification  $\times 200$ ).

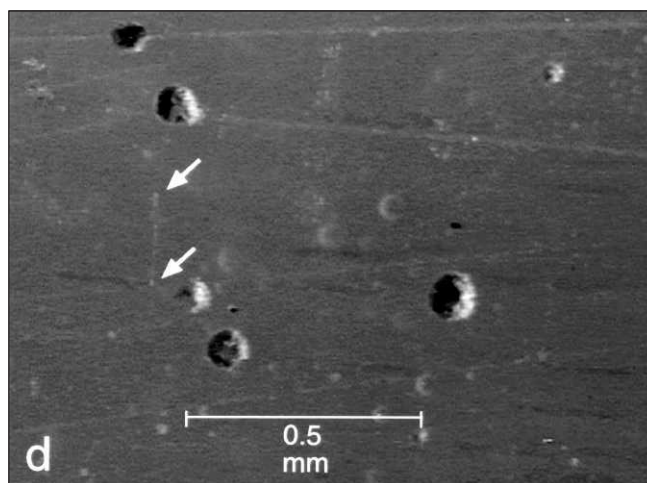


Figure 1d. Sample contains both subsurface and exposed porosities. A hardness indentation which lies in a region free of porosity can be seen between the two arrows ( $\Rightarrow$ ) (magnification  $\times 80$ ).



tically insignificant increase in KHN ( $p=0.20$  and  $0.70$ , respectively) when compared to the previous packing time, all other samples showed a decrease in KHN with increased packing time. For Surefil and Spectrum TPH, the decrease in KHN between the 20- and 45-second samples was not statistically significant ( $p=0.13$  and  $p=0.18$ , respectively). The Z100 samples showed a highly significant decrease in KHN between the 20- and 30-second samples ( $p=0.01$ ), but not between the 30- and 60-second samples ( $p=0.87$ ).

## DISCUSSION

Although the indentations were made in composite that appeared to be free of porosity, the presence of microscopic porosity not visible at the magnifications used cannot be discounted. Although this porosity would reduce the resin's hardness directly, its volume in these apparently porosity-free regions may be so small that the oxygen inhibition effect on hardness would be predominant. In contrast, hardness measurements made in regions containing visible porosity would be lowered greatly because of the direct effect of the porosity.

The authors are unable to explain the slight increase in KHN of the 30-second Surefil and the 45-second Spectrum TPH samples, or the large decrease in KHN in the 60-second Z100 samples. They expected a more uniform decrease with increased packing time.

In clinical practice, prolonged packing could result in decreased wear resistance even in those areas that appeared free of porosity. Inadequate polymerization of a resin-based composite can result in a decrease in hardness (Powers & others, 1993) as well as retention failures; diffusion of the monomers into the pulp (Hamid & Hume, 1997); decreased strength of the restoration (Ruyter & Oysaed, 1982; Swartz, Phillips & Rhodes, 1983; de Gee, ten Harkel-Hagenaar & Davidson, 1984; Asmussen, 1982) resulting in breakage and/or excessive softness and wear (Koran & Kurschner, 1998; Mante, Saleh & Mante, 1993).

Although tiny porosities not visible at a magnification of  $\times 625$  would contribute to a decrease in composite hardness, it is believed that most of the decline in hardness in these apparently porosity-free regions was caused by oxygen inhibition of the polymerization of the resin component. The continued manipulation would mix more air into the resin, thus increasing the oxygen content. A decrease in the degree of polymerization, regardless of the cause, results not only in a decrease in hardness, but also in other mechanical and physical properties of the resin. Wu (1982) reported that decreasing the degree of polymerization increased the diffusion rate of chemicals into the resin. Because the solubility parameters of many organic substances allow them to easily soak into the resin surface (McKinney & Wu, 1985), many foods will contain compounds that can

soften a resin restoration (Wu, Pestaner & Bowen, 1982; Yap, Low & Ong, 2000).

Mante & others (1993), using solutions of ethanol in water to simulate certain beverages, fruits and heptane to simulate fatty foods, found that immersion in these solutions significantly reduced the hardness of composites that had a lower degree of cure when compared to those with a higher degree of cure. In a study of three composites by Santerre, Shajii & Tsang (1999), Z100 composite, which had the lowest degree of cure, also showed the highest amount of degradation following immersion in the enzyme cholesterol-esterase, an enzyme usually present in the oral cavity.

Wu & others, 1982 described a wear mechanism based on the continuous solvent softening of the resin surface. The resin surface, once sufficiently softened, is sheared away under exposure to masticatory forces to expose a new layer of composite to the solvents in the oral environment. The process repeats, resulting in a substantial amount of composite surface wear.

All current resin-based composites wear more than silver amalgam (Jaarda, Wang & Lang, 1997). Although manufacturers bear most of the responsibility for correction of this problem, dentists can minimize wear by using proper curing and proper placement techniques (Kanca, 1985; Pagniano & Johnston, 1993).

In addition to the packing time effect described in this paper, other factors that dentists must consider in using a light-curing system are time, intensity, temperature, light distance and angle, resin thickness and shade, surface air inhibition, filler type, accelerator quantity and ambient room light (Mante & others, 1993; Bouschlicher, Vargas & Boyer, 1997).

## CONCLUSIONS

Resin-based composites that require an increased packing time may have a lower KHN once polymerized, even in areas that appear to be clinically free of porosity. Clinically, this may have a negative effect on the wear rate, and therefore, the life span of the composite.

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# Pulpal Response to a Fluoride-Releasing All-in-One Resin Bonding System

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## Clinical Relevance

Giomers may have better surface finish than conventional/resin-modified glass ionomer. The fluoride-releasing all-in-one resin bonding system, Reactmer, has only slight pulpal irritation and provides a good seal, eliminating bacterial microleakage.

## SUMMARY

**Pulp tissue reactions to a fluoride-releasing all-in-one resin bonding system (Reactmer Bond and Reactmer Paste) in non-exposed monkey teeth were histopathologically evaluated at three, 30, and 90 days after restoration. No serious inflammatory reactions of the pulp, such as necrosis or abscess formation, were observed. At 90 days in the Reactmer group, odontoblastic change and**

**inflammatory cell infiltration were not observed, and slight irritation dentin formation was formed. The pulpal response of the Reactmer group was minimally different from that of the control group. Consequently, the Reactmer system was determined as being biologically compatible with vital pulps.**

## INTRODUCTION

A wide range of restorative materials appropriate to various clinical uses in dentistry have been provided by continuous development of new materials. Usually, conventional and light-cured resin-modified glass polyalkenoate cements are the first choice restorative materials for high caries-risk sites since these materials promote an anti-cariogenic effect and an ion-exchange reaction with tooth tissues (Swartz, Phillips & Clark, 1984; Takahashi, Emilson & Birkhed, 1993; Diaz-Arnold & others, 1995; Dunne & others, 1996). Nevertheless, up to now the poor physical properties of these materials account for their limited usage (Six, Lasfargues & Goldberg, 2000). Since fluoride was shown to have a significant effect in inhibiting caries (Norman & others, 1972; Corpron & others, 1986; Okuda & others, 2001), fluoride-containing restorative materials have been developed with the attempt to pre-

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vent recurrent caries. Modifications of conventional and resin-modified glass polyalkenoate cements have led to the development of fluoride-releasing light-cured composite materials. New formulations have been successfully developed to overcome some clinical demerits of the previous glass polyalkenoate materials.

The total-etch technique of both enamel and dentin with phosphoric acid has revolutionized restorative dentistry by providing a strong interfacial bond between restorations and dental tissues (Fusayama & others, 1979). Recently, many adhesive systems have employed total etching for the bonding of resin composite systems. However, total etching removes not only the smear layer and dentinal plugs, but increases dentin permeability as well as promoting collapse and alteration of the remaining collagen network (Pashley & Carvalho, 1997). Once marginal microleakage occurs at the cavosurface margin after restoration with total etching, bacteria can enter the gap between the restoration and tooth structure and penetrate the dentinal tubules easily. The consequence of this is inflammation of the pulp tissue (Sasafuchi & others, 1999). Sealing of dentin with adhesive bonding systems protects the underlying pulpo-dentinal complex from bacterial irritation (Kitasako & others, 1999). The importance of the marginal seal should be emphasized when considering biological properties of restorative materials (Hilton, 1996; Tarim & others, 1997; Sonoda & others, 2001a).

The multi-step adhesive bonding systems usually consist of separate bottles of etchant, primer and adhesive. The drawback of these multi-step adhesive-bonding systems include technique sensitivity (Mjör & Tronstad, 1972), which may result in lower bond strength, as well as being more time-consuming. On the

other hand, some resin bonding systems have included all the steps in one procedure only. These self-etching-priming systems combine the etching and priming steps to simultaneously treat enamel and dentin. Although recent bonding systems are reliable in conservative treatment, improvement of adhesive resins is still necessary to reduce technique sensitivity and greater durability (Kitasako & others, 2000).

Recently, the fluoride-releasing all-in-one resin bonding system "Reactmer Bond" and a fluoride-releasing composite material "Reactmer Paste" have been developed (Shofu Inc, Kyoto, Japan). This new bonding system requires a reduced number of steps and chairside time, and the new composite material combines the major benefits of glass polyalkenoate cements (fluoride release and biocompatibility) with the easy handling of a light-curing resin composite. This *in vivo* study investigated the monkey pulpal response of this all-in-one resin bonding system in non-exposed monkey teeth. In this study, the authors used the resin bonding system "Imperva Fluorobond" (Shofu Inc) and the light-cured resin composite "Beautifil" (Shofu Inc) for histological comparison.

## METHODS AND MATERIALS

The materials employed in this study are listed in Table 1. Reactmer Bond is an adhesive system, consisting of Reactmer Bond A and B as a self-etching/priming/bonding resin system and requires only one step for the bonding procedure. Reactmer Paste is a light-cured composite filling material. Imperva Fluorobond is an adhesive system consisting of FB Primer A and B as a self-etching priming material and FB Bond as the adhesive. Beautifil is a light-cured resin composite.

Table 1: *Restorative Materials Employed*

Material	Brand Name	Content	Batch	# Manufacturer
Fluoride-releasing all-in-one bonding	Reactmer Bond	Bond A: Fluoro aluminosilicate glass, Full reaction type pre-reacted GI resin system Water, Acetone, Catalyst	099900	Shofu Inc, Kyoto, Japan
		Bond B: UDMA, HEMA, 4-AET 4-AETA, Photoinitiator	099900	
Fluoride-releasing composite material	Reactmer Paste	Full reaction type pre-reacted GI, Glass filler, UDMA, HEMA, Photoinitiator	099900	Shofu Inc, Kyoto, Japan
Resin bonding system	Imperva Fluorobond	FB Primer A: Water, Acetone, Catalyst	089953	Shofu Inc, Kyoto, Japan
		FB Primer B: UDMA, HEMA, 4-AET, 4-AETA, Catalyst	89968	
		FB Bond: Full reaction type UDMA, HEMA, 4-AET, Photoinitiator	089961	
Light-cured resin composite	Beautifil	Surface reaction type pre-reacted GI Multi-functional glass filler, Bis-GMA Photoinitiator	010002	Shofu Inc, Kyoto, Japan

A total of 48 intact teeth of two Japanese monkeys (*Macaca Fuscata*) were randomly distributed throughout the dentitions. Two adult monkeys used for this study were housed in the Tokyo Medical and Dental University Animal Research Center. This study was performed according to the guidelines of the Center. During the restorative operation, total anesthesia was completed with an intramuscular injection of 20 mg/kg ketamine hydrochloride (Ketalar, Sankyo Co, Tokyo, Japan) and an intravenous injection of 10 mg/kg sodium pentobarbital (Nembutal Sodium Solution, Abbott Laboratories, North Chicago, IL USA). Before each cavity preparation, the operation field was cleaned with dilute iodine tincture and 3% hydrogen peroxide. Deep Class

V cavities were prepared on the buccal surfaces of 48 intact teeth with a high-speed tapered diamond bur (#170, GC Co, Tokyo, Japan) under water spray coolant.

The experimental procedure is shown in Figure 1. The 48 experimental teeth were divided into two groups (n=8 for each time period). In the first group (Reactmer group), Reactmer Bond A and B were mixed and applied to the cavity for 20 seconds using a micro-brush to reduce the formation of blisters, then light-cured for 20 seconds. During this treatment the authors did not air-dry the bonding agent in order to keep the required thickness of the adhesive layer. Then, the cavity was restored with Reactmer Paste and light-cured for 30 seconds.

In the second group (Beautifil group), the cavity was treated with a mixture of the FB Primer A and B for 10 seconds. Next, excess primer was removed with gentle air drying, then the low viscosity FB Bond was placed and light-cured for 10 seconds, and the cavity was restored with Beautifil and light-cured for 40 seconds.

After three, 30 and 90 days, the experimental animals were sacrificed with an overdose of thiopental sodium (Ravonal, Tanabe Pharmaceutical Co, Osaka, Japan). The teeth were immediately removed from jaws using a diamond disk, then fixed with 10% neutral buffered formalin solution for one week. Before immersion, the mesial and distal approximal surfaces of the teeth were reduced with a high-speed diamond bur under spray coolant until the pulp became

almost visible through the remaining dentin to facilitate the penetration of the fixative solution. The teeth were demineralized with Plank-Rychlo's demineralizing solution at 4°C for four-to-five days, and each tooth was then embedded in paraffin. Serial sections 5 µm thick were prepared through the cavities and pulp, obtaining approximately 80-100 sections per cavity. They were placed on glass microslides and stained with either hematoxylin-eosin for routine histological evaluation or Taylor's modification of Gram's staining technique for detecting micro-organisms (Taylor, 1966).

The pulpal response and the presence of bacteria in the cavity

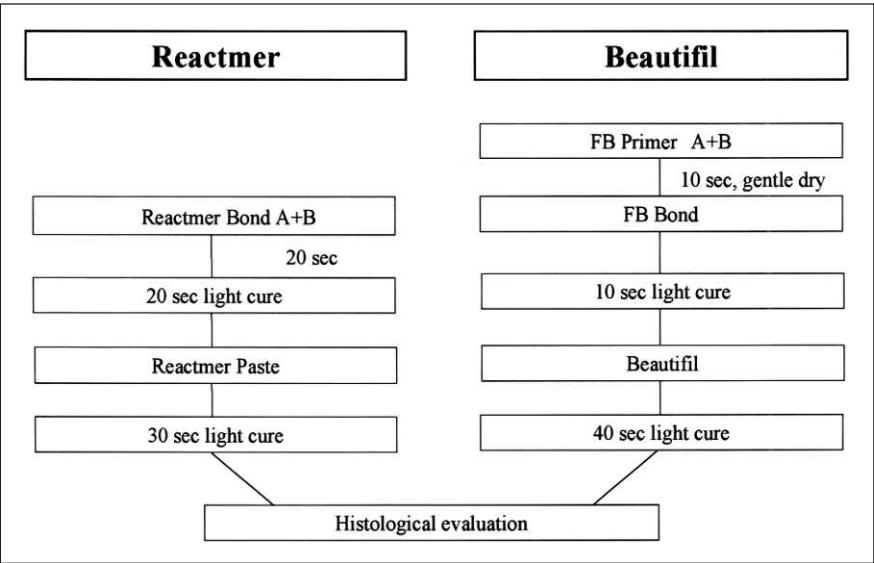


Figure 1. Experimental procedure.

Table 2: Evaluation Criteria	
Odontoblastic Change	
None (-):	Remarkable change was not observed in the pulp.
Slight (±):	Disarrangement of odontoblasts was noted slightly below the cut dentinal tubules.
Moderate (+):	Disarrangement of odontoblasts was seen most of the cut dentinal tubules.
Severe (++):	Disarrangement of odontoblasts was noted below the remaining dentin.
Inflammatory Cell Infiltration	
None (-):	None or a few inflammatory cell was observed through the pulp.
Slight (±):	A few inflammatory cell infiltration was noted below the cut dentinal tubules.
Moderate (+):	Inflammatory cell remarkably observed below the remaining dentin.
Severe (++):	Severe inflammatory cell infiltration was seen through the pulp.
Irritation Dentin Formation	
None (-):	No abnormal or reparative dentin observed.
Slight (±):	A small amount of irritation dentin was noted.
Moderate (+):	Irritation dentin was observed below the almost cut dentinal tubules.
Severe (++):	Complete and large bulk of irritation dentin was noted.



were evaluated and measured using a light microscope. The thickness of the remaining dentin underneath each cavity was measured following the course of the dentinal tubules. The thinnest tissue section of each tooth sample was selected as the representative section of the tooth. Odontoblastic change, inflammatory cell infiltration and irritation dentin formation were scored in four degrees of none (-), slight ( $\pm$ ), moderate (+) and severe (++). These criteria are shown in Table 2 (Sasafuchi & others, 1999). These specimens, where bacterial penetration was detected along the cavity walls and fixed inadequately, were omitted. The results of odontoblastic change, inflammatory cell infiltration and irritation dentin formation were statistically analyzed using the Mann-Whitney *U* Test at the 95% level of confidence ( $n=8$  for each time period). Parametric one-way analysis of variance (ANOVA) and Fisher's protected least significant difference (PLSD) test at the 99% level of confidence ( $n=8$  for each time period) were used for analyzing the results of the remaining dentin thickness in each group.

## RESULTS

Mean, maximum and minimum values of remaining dentin thickness (RDT) in each group are shown in Table 3. The RDT observed in this study ranged between 0.05 and 1.30 mm with the mean for the groups ranging from 0.18 to 0.66 mm. Significant differences were not found between the average value of RDT among all test groups ( $p>0.01$ ).

The graded scores for observed odontoblastic change, inflammatory cell infiltration and irritation dentin formation are summarized in Table 3 and Figure 2. Stained sections are seen in Figures 3 and 4. Severe inflammatory reactions of the pulp, such as necrosis and abscess formation, were not observed. Pulpal changes were located adjacent to the cavity.

### Odontoblastic Change

The disappearance of primary odontoblasts of the remaining dentin underlying the cavity preparation and aspiration of cell nuclei into these same dentinal tubules were observed in both the Reactmer group and the Beautifil group. In the Reactmer group, one of eight

Table 3: Results of Histopathological Findings

Time Intervals		3 Days		30 Days		90 Days	
Experimental Groups		Reactmer		Beautifil		Reactmer	
# of Specimens							
Odontoblastic Change	none	7	6	7	8	8	8
	slight	1	1	1	0	0	0
	moderate	0	1	0	0	0	0
	severe	0	0	0	0	0	0
Inflammation Cell Infiltration	none	6	7	7	8	8	8
	slight	2	1	1	0	0	0
	moderate	0	0	0	0	0	0
	severe	0	0	0	0	0	0
Irritation Dentin Formation	none	8	8	7	7	5	4
	slight	0	0	1	1	3	3
	moderate	0	0	0	0	0	1
	severe	0	0	0	0	0	0
Remaining Dentin Thickness (mm)	mean	0.66	0.65	0.18	0.41	0.38	0.43
	minimum	0.25	0.10	0.05	0.15	0.15	0.05
	maximum	1.10	1.30	0.44	1.10	0.60	1.20

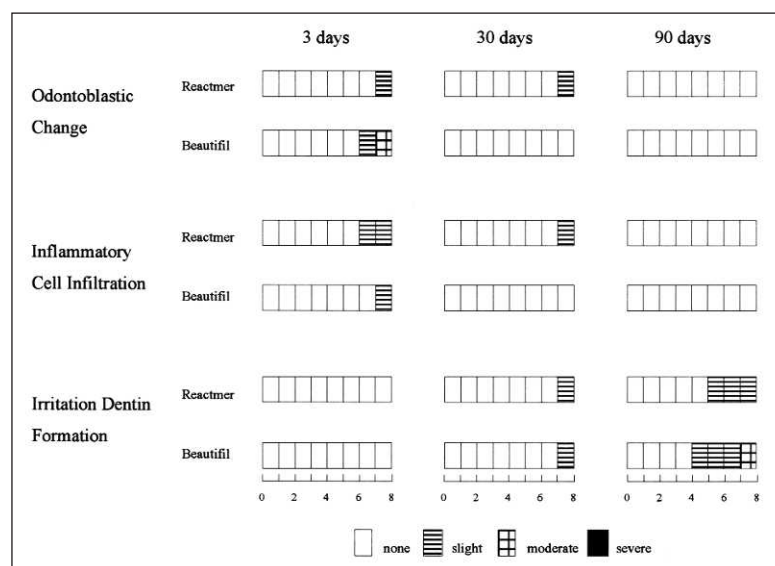


Figure 2. Pulpal responses of the two groups.

specimens showed slight change in the odontoblastic layer at three and 30 days after operation, and no change was shown at 90 days. In the Beautifil group, one slight and one moderate change were noted at three days, and no change was observed at 30 and 90 days. There were no statistically significant differences in odontoblastic changes between the two groups for the same time intervals.

### Inflammatory Cell Infiltration

In the Reactmer group, slight inflammatory cell infiltration was detected in two of eight specimens at three days

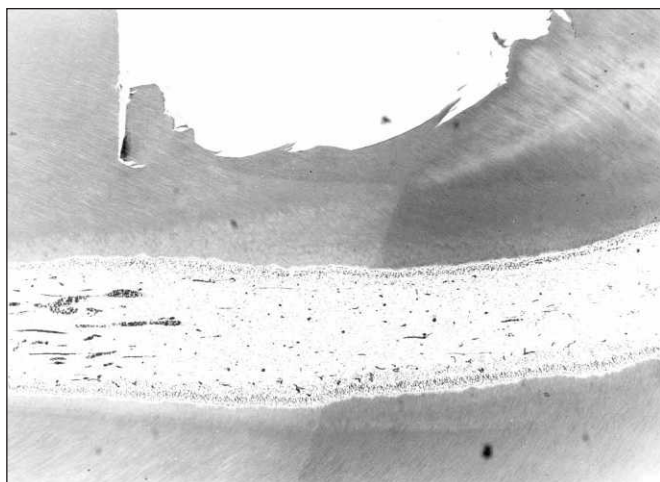


Figure 3. Light micrograph of restored pulps with Reactmer Bond and Reactmer Paste at 90 days. The RDT beneath the cavity is approximately 0.52 mm. Remarkable change is not observed. (stained with hematoxylin and eosin; original magnification x10).

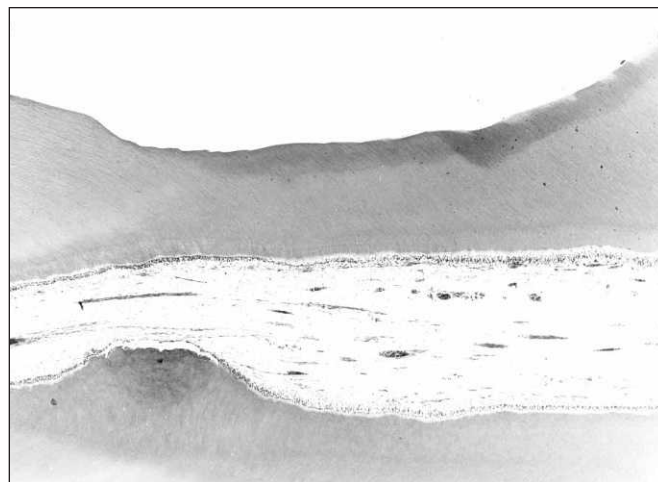


Figure 4. Light micrograph of restored pulps with Imperva Fluorobond and Beautifil at 90 days. The RDT beneath the cavity is approximately 0.55 mm. Remarkable change is not observed. (stained with hematoxylin and eosin; original magnification x10).

and in one of eight specimens at 30 days, and no inflammatory cell infiltration was detected at 90 days. In the Beautifil group, slight inflammatory cell infiltration was detected in one of eight specimens at three days, and no pulpal inflammation was observed in any specimen at 30 and 90 days. In both groups, no specimen was observed to have more than moderate inflammatory cell infiltration over the experimental periods, and there were no statistically significant differences in the incidence of inflammatory cell infiltration between the two groups for the same time intervals.

### Irritation Dentin Formation

At three days in both groups, there was no indication of any irritation dentin, nor were there any reorganized odontoblastoid cells below the cut tubules of the remaining dentin. In the Reactmer group, only slight irritation dentin formation was shown in one of eight specimens at 30 days, and three of eight specimens at 90 days. Slight or moderate irritation dentin formation was seen in the Beautifil group, one slight formation of eight specimens at 30 days, and three slight and one moderate formation at 90 days. There were no statistically significant differences in irritation dentin formation within any of the two groups at 30 and 90 days.

### Bacterial Penetration

There was no bacterial presence except for one case, and the specimen where bacterial penetration was detected along the cavity wall was omitted.

### DISCUSSION

In this study, only three of 24 pulps showed a slight inflammatory cell infiltration in the Reactmer group. Inokoshi, Iwaku & Fusayama (1982) reported that

total-etched cavity preparations restored with Clearfil bond system-F showed only slight pulpal response and no bacterial penetration. Hosoda & others (1991) reported at the same observation periods that zinc oxide-eugenol cement, which was used as a control material, showed slight-to-moderate inflammatory reactions with two cases of a severe reaction at 30 days without bacterial invasion. Otsuki & others (1999) reported that Clearfil Linerbond 2V system using a self-etching primer showed slight pulpal irritation. This study's authors demonstrated that their Reactmer data was comparable with that of Kitasako & others (2000), who investigated an experimental one-application resin bonding system, TOF-1, showing a slight pulpal response in only two of 30 cases. In general, the thinner the remaining dentin thickness, the more severe the pulpal response (Cox & others, 1992). Among all groups in this study, there were no statistical differences in remaining dentin thicknesses. This study's data demonstrated that the Reactmer system was biologically compatible with vital pulps.

It is clear that marginal sealing protects the pulp by preventing invasion by bacteria. Bacteria are perhaps the most useful biological tracers for the detection of marginal microleakage *in vivo*. Tarim & others (1997) reported that compomers failed to provide a complete biological seal, as bacterial staining was observed at grade 2 in 44 teeth. In this study, there was no bacterial presence except for one case, and the specimen where bacterial penetration was detected was omitted. Bacterial-staining data indicated that the Reactmer system provided an almost complete seal against bacterial microleakage through all time intervals. The optimum sealing capacity of Reactmer was its biological capacity to prevent bacterial microleakage.

The presence of gaps along the dentin cavity walls may allow bacterial microleakage and eventual pulp irritation. Recently, many studies (Inokoshi & others, 1982; Harnirattisai & Hosoda, 1991; Fujitani, Inokoshi & Hosoda, 1992) have demonstrated that the presence of bacteria within the dentinal tubules and the dental pulp was the major factor that led to pulpal irritation, and a significant relationship between bacterial penetration and pulpal response was confirmed. Sasafuchi & others (1999) detected some bacteria with the same bacterial stain technique as well in this study when evaluating the correlation between bacterial penetration due to microleakage and pulpal response. Kitasako & others (2000) showed that TOF-1 did not permit bacterial growth from the tooth surface due to its bactericidal properties. There was only a slight pulpal response in this study since the Reactmer system provided an excellent biological seal. This acceptable pulpal response was dependent upon the exclusion of bacterial penetration or the lack of toxicity of Reactmer.

Imperva Fluorobond, which is a self-etching priming system, requires two steps (etching/priming and bonding) during the bonding procedure. Reactmer Bond is a self-etching/priming/bonding resin system and requires only one step during the bonding procedure. Sano & others (1998) pointed out that reducing the steps of the bonding procedure was an important factor in obtaining a more reliable, strong bond between the resin and dentin. Many adhesive systems have been reported to be technique-sensitive (Fundingsland & others, 1996), however, this one-step bonding procedure was easy to use. The Reactmer system was expected to reduce the pulpal irritation because of its fewer steps and no rinsing. Although the number of bonding steps of Reactmer Bond were less than Imperva Fluorobond, in the present study, there were no significant differences in pulp inflammation of both groups at all time periods. However, the Reactmer system required only one step and therefore reduced chairside time and was easy to use for operators not thoroughly familiar with current adhesive systems.

Ikemura & others (2000) reported that the mean shear dentin bond strength after one day Reactmer Bond specimens was 16.3 MPa, and Imperva Fluorobond specimens was 22.2 MPa. Koda (2000) reported the mean shear dentin bond strength after one day Reactmer Bond specimens was 12.5 MPa, and Imperva Fluorobond specimens was 21.3 MPa. The mean dentin bond strength of Reactmer was less than that of Imperva Fluorobond, however, there was no pulp inflammation in both groups at 90 days. The adhesive system of Reactmer might show high bond strength over the long-term on the enamel and dentin in cavities. Reactmer, therefore, may be able to provide a long-term hermetic seal, preventing bacterial microleakage. However, Tarim & others (1997) reported that high bond

strengths of laboratory studies should not be equated with long-term *in vivo* resistance to bacterial microleakage, and high bond strength should not be misconstrued with long-term clinical success. Further studies are needed to characterize *in vivo* and laboratory dentin bond strengths of these materials and to evaluate the long-term marginal seal.

Fujitani & others (1992) reported that total-etched dentin showed a greater reduction of primary odontoblasts than non-etched cavities, and pulpal irritation originated from initial chemical and mechanical irritation of acid etching, rinsing and air drying. Kitasako & others (2000) reported the adhesive layer was intentionally applied thick in their study to obtain a stress-absorbing effect, and the bonding material was not air-thinned after application because the adhesive layer thickness could not be controlled *in vivo*. In this study, irritation dentin formation at 90 days showed only slight presence in three of eight cases for Reactmer. Since the Reactmer system needed no rinsing and Reactmer Bond was not air-dispersed after application, there was little initial chemical and mechanical irritation. That might be the reason that irritation dentin formation at 90 days was slight.

Fluoride has been shown to promote an anticariogenic effect and inhibiting caries. Reactmer Bond and Imperva Fluorobond are fluoride-containing restorative materials which have been developed with the attempt to prevent recurrent caries. Okuda & others (2000) reported that the thickness of the inhibition zone around Reactmer in bovine root dentin was comparable to that of a resin-modified glass polyalkenoate cement, Fuji II LC improved. Sonoda & others (2001b) reported that the Reactmer system appeared to inhibit demineralization of monkey root dentin *in vivo*. In this study, there was only a slight pulpal response at all time periods in both groups. From a clinical standpoint, this slight reaction might be effected by fluoride release from the restorative materials (Six & others, 2000) over the long-term, but further study is needed to confirm this.

## CONCLUSIONS

The fluoride-releasing all-in-one resin bonding system that was tested provided an almost complete seal against bacterial microleakage through all time intervals.

The pulpal response of the Reactmer group was marginally different from that of the control group. Only three of 24 pulps showed a slight inflammatory cell infiltration in the Reactmer group, therefore, the Reactmer system is believed to be biologically compatible with vital pulps.

There was little initial chemical and mechanical irritation from Reactmer, providing a slight reason for irritation dentin formation at 90 days.



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# Influence of Adhesive Application Duration on Dentin Bond Strength of Single-Application Bonding Systems

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## Clinical Relevance

The application duration of adhesives of single-application bonding systems was not a crucial factor for determining dentin bond strength, even though morphological change was observed on the dentin surface.

## SUMMARY

This study examined the relationship between the bond agent application duration and the dentin bond strength of several single-application bonding systems. The restorative material/bonding systems used were Reactmer, with its single-application bonding system Reactmer Bond (RB, Shofu Inc, Kyoto, Japan); Palfique Estelite, with its single-application bonding system One-Up Bond F (OU, Tokuyama Co, Tokyo, Japan) and F2000 Compomer, with its bonding system Primer/Adhesive in Clicker (F2, 3M Dental Products, St Paul, MN 55144, USA). Bovine mandibular incisors were mounted in self-curing resin and wet ground with #600 SiC to expose labial dentin. Adhesives were applied for 5, 10, 20, 30 and 60 seconds, and restorative materials were condensed into a Teflon mold (Ø4 x 2 mm) on the

dentin and light activated. Fifteen samples per test group were stored in 37°C water for 24 hours, then shear tested at a crosshead speed of 1.0 mm/min. One-way ANOVA followed by Duncan test ( $p=0.05$ ) was done. SEM observations of the treated dentin surface were also conducted. The dentin bond strength ranged from  $6.9 \pm 2.4$  to  $11.2 \pm 2.8$  MPa for RB,  $8.9 \pm 2.2$  to  $12.2 \pm 1.9$  MPa for OU, and  $7.8 \pm 3.1$  to  $11.4 \pm 2.6$  MPa for F2. No significant differences were found among the 10-60 second application duration groups for the systems used. From the SEM observations, demineralization of the dentin surface was more pronounced with longer application duration. The data suggest that the duration of single-application bonding systems was not a crucial factor for determining dentin bond strength, even though morphological changes were observed on the dentin surface.

## INTRODUCTION

Since the introduction of dentin primers (Munksgaard & Asmussen, 1984), three-step bonding systems have increased the acceptance and reliability of dentin bonding (Tyas, 1992; Triolo Jr & Swift Jr, 1992; Jordan, Suzuki & Davidson, 1993; Senda & others, 1995). Because these systems require dentin conditioning and priming steps prior to adhesive application (Erickson, 1992; Van Meerbeek & others, 1998), clinical success with these bonding systems is thought to depend on technique-sensitive and material-related factors (Sano & others, 1998;

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Finger & Balkenhol, 1999, Miyazaki, Onose & Moore, 2000). To overcome these shortcomings, single-application bonding systems that combine the functions of self-etching primer and adhesive have been developed (Kitasako & others, 2000; Miyazaki & others, 2001). These products are simply applied on the dentin surface for the prescribed period of time and light activated.

Micromechanical interlocking through creating a hybrid layer (Nakabayashi, Kojima & Masuhara, 1982; Van Meerbeek & others, 1992; Inokoshi & others, 1993) is currently accepted as a major mechanism of resin bonding to dentin. After applying the single-application adhesives, the smear layer is dissolved and the superficial dentin is demineralized to facilitate penetration of resin monomers. Etching the dentin and facilitating the monomers are the diffusion control phenomenon that requires time to penetrate into dentin. The duration that a dentin primer is applied to etched dentin surfaces has been reported to have an effect on the dentin bond strength of three-step bonding systems (Miyazaki & others, 1996).

The hypothesis to be tested here is that applying the adhesive for longer duration times would improve the dentin bond strength of commercially available single-application bonding systems. This study examined the relationship between the adhesive application duration and dentin bond strength of several single-application bonding systems.

METHODS AND MATERIALS

Two commercial single-application systems, One-Up Bond F and Reactmer Bond, and F2000 compomer as a control material, were used (see Table 1). All adhesive systems were used with the combination of manufacturers' instructed restoratives. An Optilux 400 (Kerr/ Demetron, Danbury, CT 06810, USA) curing unit was connected to a variable voltage transformer in order to adjust the light intensity to 600 mW/cm<sup>2</sup>.

Mandibular incisors extracted from 2-to-3 year old cattle and stored frozen (-20°C) were used as a substitute for human teeth. After removing the roots with an Isomet low-speed saw (Buehler Ltd, Lake Bluff, IL 60044, USA), the pulps were removed and the pulp chamber of each tooth was filled

with cotton to avoid penetration of the embedding media. The labial surfaces of the bovine incisors were ground on wet 240-grit SiC paper to make a flat surface in the dentin. The tooth was then mounted in cold-curing acrylic resin to expose the flattened area and stored in tap water to minimize the temperature rise from the exothermic polymerization reaction of the acrylic resin. Final finish was accomplished by grinding on wet 600-grit SiC paper to expose an area of dentin approximately 6-to-8 mm in diameter, which is sufficient for bond strength testing. After ultrasonic cleaning in distilled water for one minute to remove any debris, these surfaces were washed and dried with a three-way syringe. The mounted teeth with dentin exposed were randomly assigned to the three restorative materials with a sample size of 15 per experimental group.

Adhesive tape was used to define the area of the tooth for bonding and a Teflon mold 2.0 mm high and 4.0 mm in diameter was used to form and hold the materials to the tooth surface. The adhesives were applied to the dentin surface for 5, 10, 20, 30 and 60 seconds followed by light irradiation. The restorative was condensed into the mold and light activated for 40 seconds. The mold and adhesive tape were removed from the specimens 10 minutes after light irradiation. Then the specimens were stored in 37°C distilled water for 24 hours and tested in shear mode using a knife-edge shear testing apparatus in an Instron testing machine (Type 4204, Instron Corp, Canton, MA 02021, USA) at a crosshead speed of 1.0 mm/minute. Shear bond strength values in MPa were calculated from the peak load at failure divided by the specimen surface area.

After testing, the specimens were examined in an optical microscope at a magnification of 10x to determine the location of the bond failure. The test area on the tooth was divided into eight segments, and the percentage that was free of adhesive or restorative material was estimated. The types of failures were determined based on the predominant percentage of substrate free

Table 1: Bonding Systems Used in This Study

Code	System (Manufacturer)	Adhesive (Main components)	Lot #	Restorative	Lot #
RB	Reactmer (Shofu)	Reactmer Bond (4-AET, 4-AETA, UDMA,HEMA, PRG filler, F aluminosilicate glass, acetone, water, initiator)	A: 099900 B: 099900	Reactmer	040001
OU	One-Up Bond F (Tokuyama)	One-Up Bond F (MAC-10, HEMA, F aluminosilicate glass, water, initiator)	A: 001 B: 501	Palfique Estelite	219
F2	F2000 Compomer (3M)	Primer/Adhesive (Vitremer copolymer, Bis-GMA, HEMA maleic acid, water, initiator)	ALAR	F2000	OBN

material as adhesive failure, cohesive failure in resin composite, cohesive failure in adhesive and cohesive failure in dentin.

The results were analyzed by calculating the mean shear bond strength (MPa) and standard deviation for each group. The data for each group were tested for homogeneity of variance using Bartlett's test, and then subjected to an ANOVA followed by the Duncan multiple range test at  $p=0.05$ . The statistical analysis was carried out with the Sigma Stat software system (SPSS Inc, Chicago, IL 60611, USA).

The treated dentin surface and the restorative/dentin interface were observed by scanning electron microscopy (SEM). For the treated dentin surface observation, the dentin surfaces were treated with adhesive according to each manufacturer's instructions, then rinsed with acetone and water. For the ultra structure observation of the restorative/dentin interface, bonded specimens stored in 37°C distilled water for 24 hours were embedded in epoxy resin, then longitudinally sectioned with a diamond saw. The sectioned surfaces were polished with abrasive discs and diamond pastes down to a 0.1  $\mu\text{m}$  particle size. All the SEM specimens were dehydrated in ascending concentrations of *tert*-butanol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes and 100% for two hours), then transferred to a critical-point dryer for 30 minutes. The polished surfaces were subjected to argon-ion beam etching for 30 seconds with the ion beam (Elionox Ltd, Tokyo, Japan), with accelerating voltage of 1.0 kV and ion current density of 0.4 mA/cm<sup>2</sup> directed perpendicular to the polished surface (Inokoshi & others, 1993). The surfaces were coated in a vacuum evaporator with a thin film of Au. The specimens were observed in a scanning electron microscope (JSM-5400, JEOL Ltd, Tokyo, Japan).

RESULTS

The mean shear bond strengths to bovine dentin and fracture modes are shown in Table 2. The dentin bond strength ranged from 6.9  $\pm$  2.4 to 11.2  $\pm$  2.8 MPa for RB, 8.9  $\pm$  2.2 to 12.2  $\pm$  1.9 MPa for OU, and 7.8  $\pm$  3.1 to 11.4  $\pm$  2.6 MPa for F2. Bond strength tended to increase with

longer application duration of adhesives, but no significant differences were found for the groups above 10-second application durations. With the shortest application duration, a significant decrease of bond strength was found.

The predominant failure was cohesive failure in adhesive for groups above 10-second application in duration. For the five-second application duration groups, failure occurred between the dentin and adhesive for all materials tested.

Figure 1 shows the representative SEM observations of treated dentin surfaces. The smear layer was removed and the collagen fibers observed to some extent. Demineralization of the dentin surface was more pronounced with the longer adhesive application duration.

Figure 2 shows representative SEM micrographs of the dentin/resin interface after argon-ion beam etching. A thin layer with low resistance to argon-ion etching was identified as the hybrid layer, and the width of this layer was about 0.5-1.0  $\mu\text{m}$  for all systems employed.

DISCUSSION

Although many intact, extracted teeth are required for conducting bond strength tests, it is difficult to obtain enough extracted human teeth in Japan. Adhesion to the superficial layer of dentin has been reported to show no significant differences between human and bovine dentin, and dentin bond strength has been shown to decrease with the depth of dentin due to the lower density of dentinal tubules (Schilke & others, 1999). Because differences in tubule diameters and the number of lateral branches may have some effect on dentin bond strength (Ferrari & Davidson, 1996), in this study, bovine superficial dentin was used as a substitute for human dentin, as reported in previous studies (Nakamichi, Iwaku & Fusayama, 1983; Fowler & others, 1992). The bond strength values measured depend on the bonding systems used, the site of the tooth and the type of tooth structure (Suzuki & Finger, 1988; Ishioka & Caputo, 1989, Eick & others, 1991). Care should be taken when drawing conclusions from bond strength data since many factors affect bond values (Miyazaki, Oshida & Xirouchaki, 1996).

The dentin bonding mechanism of resin composite is believed to consist of three steps: dentin conditioning, priming and adhesive application (Van Meerbeek & others, 1998). It is generally accepted

Table 2: Shear Bond Strength (Mean $\pm$ SD) to Bovine Dentin					
Code	Adhesive Application Duration (sec)				
	5	10	20	30	60
RB	8.9 $\pm$ 2.2 <sup>a</sup>	11.3 $\pm$ 1.9 <sup>b</sup> [8/2/5/0]	12.0 $\pm$ 1.5 <sup>b</sup> [3/2/8/2]	12.1 $\pm$ 2.2 <sup>b</sup> [2/3/7/2]	12.2 $\pm$ 1.9 <sup>b</sup> [2/2/7/4]
[2/4/5/4]					
OU	6.8 $\pm$ 1.6 <sup>c</sup>	8.7 $\pm$ 2.2 <sup>d</sup> [10/0/5/0]	10.2 $\pm$ 2.3 <sup>d</sup> [6/0/9/0]	10.6 $\pm$ 2.4 <sup>d</sup> [3/4/6/2]	11.2 $\pm$ 2.6 <sup>d</sup> [5/2/6/2]
[2/5/6/2]					
SD: standard deviation					
Values with the same letter are not significantly different at $p>0.05$ .					
Failure mode: [adhesive failure/cohesive failure in resin/cohesive failure in adhesive/cohesive failure in dentin].					



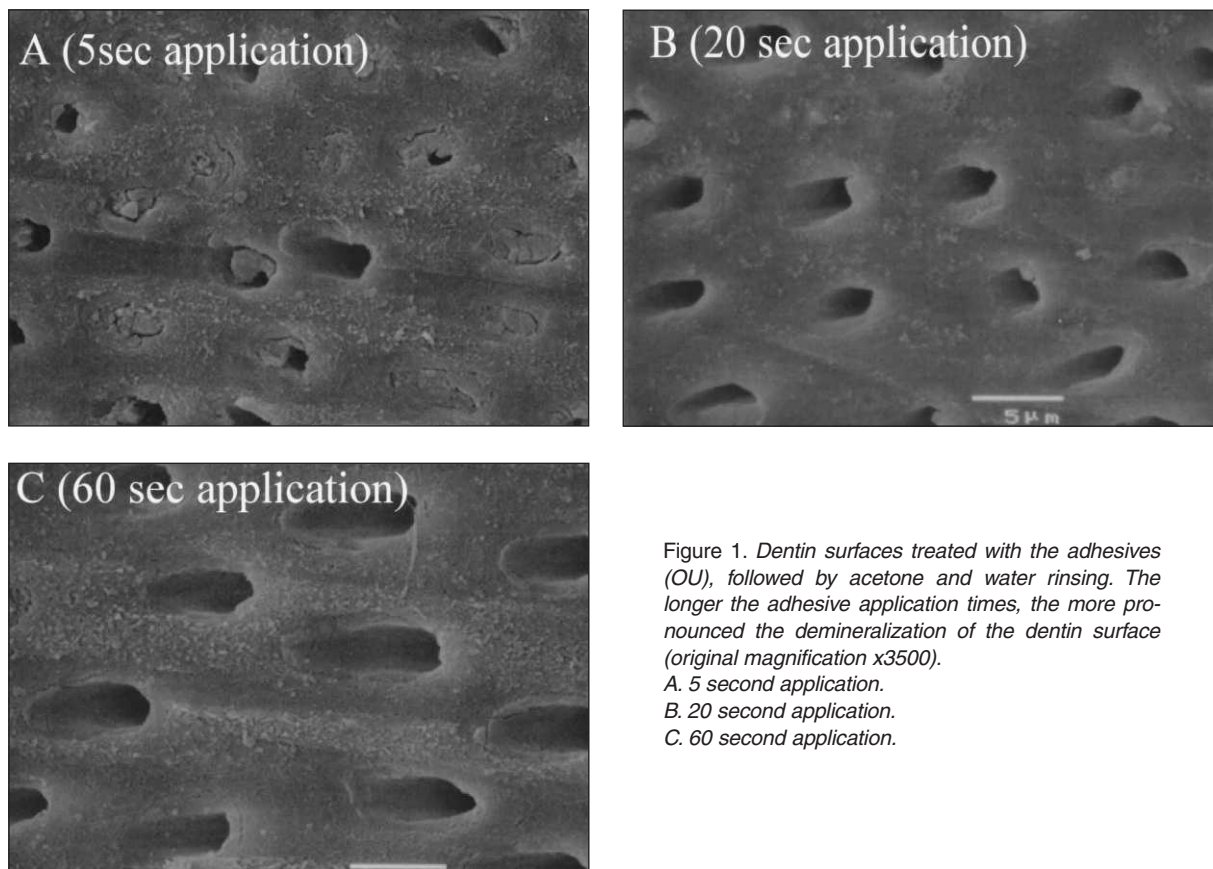


Figure 1. Dentin surfaces treated with the adhesives (OU), followed by acetone and water rinsing. The longer the adhesive application times, the more pronounced the demineralization of the dentin surface (original magnification  $\times 3500$ ).

A. 5 second application.

B. 20 second application.

C. 60 second application.

that the smear layer that forms on ground dentin should be removed or altered with acidic conditioners to achieve good adhesion between demineralized dentin and an applied bonding system (Prati & others, 1998; Pashley, 1991; Nakabayashi & Saimi, 1996). After removing the smear layer, the conditioned dentin surface should be wetted by hydrophobic resin monomers. Single-application bonding systems rely on their adhesives for wetting in order to create good adaptability to dentin.

A precursor of the single-application adhesive is a self-etching primer that contains acidic functional monomers such as 4-AET (Ikemura, Kouro & Endo, 1996), phenyl-p and MDP (Barkmeier, Los & Triolo, Jr, 1995; Gordan & others, 1997). The self-etching primer enables the simultaneous etching and priming of tooth surfaces. The depth of demineralization during adhesive application depends on the type of acidic monomers, the concentration, the duration of application and the composition of the dentin. The adhesive used in single-application bonding systems is a hydrophilic solution that is extremely effective in wetting the tooth surface. The etching effect of these systems is related to the acidic monomers or organic acid solutions that may interact with the mineral compo-

nent of dentin and enhance monomer penetration (Ikemura & others, 1996). These single-application adhesives may form a continuum between the tooth surface and the adhesive by the simultaneous demineralization and resin penetration followed by polymerization. Penetration of acidic monomers into the dentin surface creates resin tags and a hybrid layer. For single-application bonding systems, applying adhesive allows mineralized tissue to be demineralized and roughened. In this study, based on morphological observations made using SEM, single-application products produce a demineralized surface similar to that of products using separate self-etching primers. However, specific demineralization of dentin may not be a critical factor in determining dentin bond strength. From the results of this study, dentin bond strengths were not affected by the duration times of adhesive application, although the demineralization patterns observed with SEM were quite different.

The relationship between the depth of demineralization and the extent of resin monomer penetration is the key to creating a high quality hybrid layer. Poor infiltration of adhesive resin into the demineralized dentin leaves nano-spaces in the hybrid layer (Sano & others, 1995; Spencer & Swafford, 1999), and such a



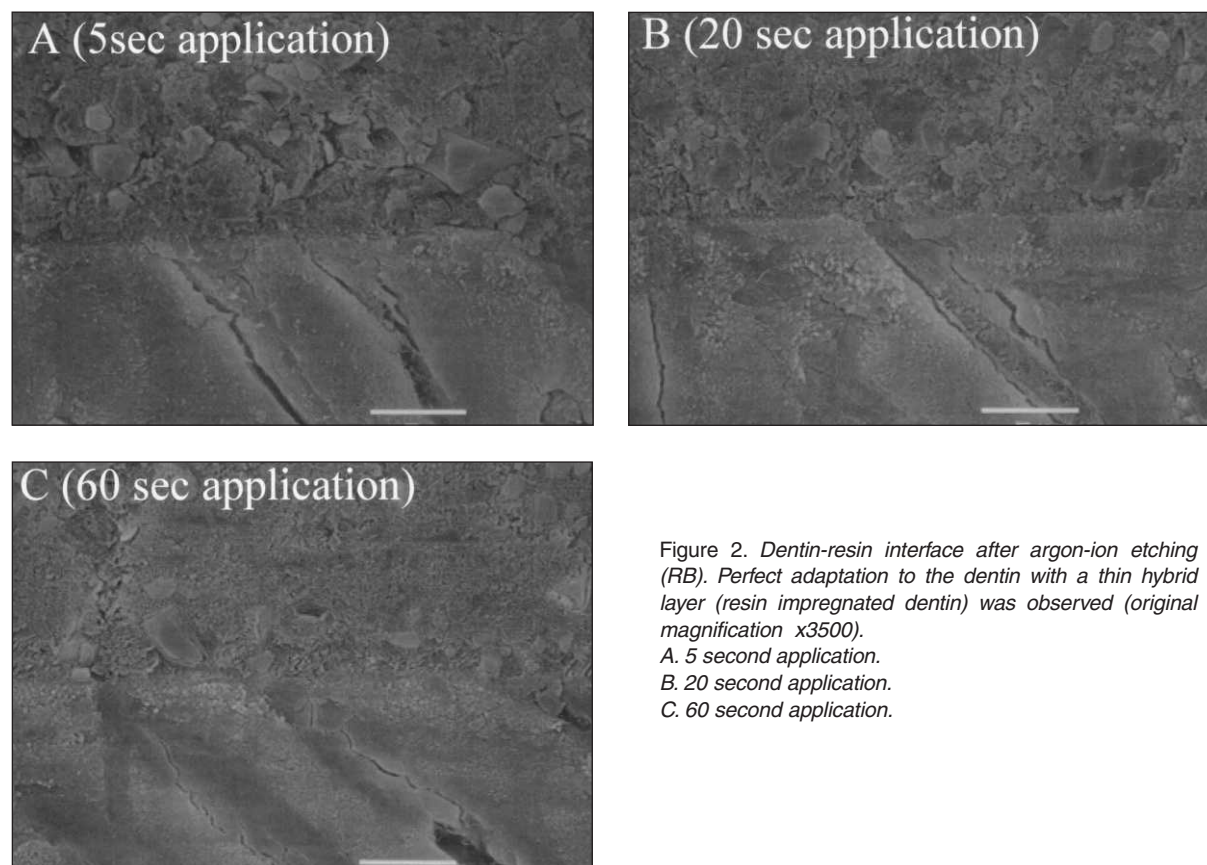


Figure 2. Dentin-resin interface after argon-ion etching (RB). Perfect adaptation to the dentin with a thin hybrid layer (resin impregnated dentin) was observed (original magnification  $\times 3500$ ).

A. 5 second application.

B. 20 second application.

C. 60 second application.

region may be susceptible to degradation from oral fluids (Sano & others, 1999). In the case of a single-application bonding system, penetration of the adhesive into dentin occurs simultaneously with demineralization of the mineral component of the dentin. When the resin fails to completely infiltrate deeper portions of the demineralized dentin, the bond between resin and dentin might be weakened. With single-application bonding systems, the possibility of monomers not infiltrating demineralized dentin is reduced. Longer adhesive application time might create a deeper demineralized dentin layer, with the resin monomer penetrating into the total depth of etched, demineralized dentin.

From the results of this study, no correlation between layer thickness and bond strength was found with single-application bonding systems as reported by previous studies (Uno & Finger, 1996; Vargas, Cobb & Denehy, 1997; Prati & others, 1998). One reason for this small difference in bond strengths might be related to the site of failure after the bond strength test. Observing cohesive failure within the adhesive layer after bond strength testing indicates a bond to dentin in excess of the cohesive strength of the adhesives.

## CONCLUSIONS

Current developments in adhesive systems have focused on simplifying the application methods by decreasing the steps required for placement. Dentists can expect a reduction in the clinical steps for adhesive materials with stable bond and hermetic seal. Based on the results of this study, the duration of single-application bonding systems may not be a crucial factor for determining dentin bond strength, even though morphological changes were observed on the dentin surface. Further research with clinical studies will be required to establish the performance of the single-application bonding systems.

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# The Effect of Resin Composite Pins on the Retention of Class IV Restorations

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HW Roberts • DF Murchison

## Clinical Relevance

The use of an intracoronal resin composite pin does not improve the retention of Class IV resin-based composite restorations.

## SUMMARY

Standardized Class IV cavity preparations were made in 48 human incisors. They were then divided into three groups of 16 teeth each. Group 1 was prepared with no internal retentive features. Groups 2 and 3 included an internal pin channel prepared with a #330 and a #329 bur, respectively. All specimens were restored with a micro-hybrid resin-based composite restorative material and a fifth generation dentin bonding system. Specimens were loaded to failure in an Instron Universal Testing Machine perpendicular to the long axis of the teeth. There was no evidence to

suggest a difference in force required to dislodge the restoration among the three groups tested ( $p=0.185$ ). Resin composite pins do not increase the retention of Class IV resin composite restorations.

## INTRODUCTION

Resin composite is the restorative material of choice for esthetic anterior restorations. However, resin composites possess limitations. One significant problem is retention of large Class IV bonded restorations (Browning & Dennison, 1996; Potoky & Rothfuss, 1993). Browning & Dennison (1996) surveyed 108 dentists who routinely placed anterior composites and reported that the median survival rate of Class IV restorations was five years of clinical service, half the Class III median survival rate of 10 years. Furthermore, these authors reported that approximately 35% of Class IV composite restorations were replaced within three years, while less than 13% of Class III restorations required replacement in the same timeframe (Browning & Dennison, 1996; Potoky & Rothfuss, 1993). To overcome some of the limitations of large Class IV restorations, some dental schools advocate indirect porcelain laminate veneers as the treatment of choice instead of extensive Class IV restorations (Potoky & Rothfuss, 1993).

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Several techniques have been used to assist in the retention of Class IV composite restorations, including dentin undercuts, grooves, dovetail extensions and slots (Sturdevant & others, 1994). Historically, intracoronal threaded pins were used to augment resistance and retention form with large composite restorations (Sturdevant & others, 1994; Attin & others, 1994; Frederick, 1987; Tjan, Dunn & Grant, 1992). In fact, some intracoronal pins, such as Bondent (Coltene/Whaledent, Mahwah, NJ 07430, USA) are marketed specifically for use with resin-bonded restorative materials (Attin & others, 1994; Butchart, Grieve & Kamel, 1988). However, intracoronal threaded pin retention has some disadvantages, including decreased esthetics due to pin visibility through translucent teeth and/or restorations, pulpal exposure and/or root perforation into the periodontal ligament, decreased restoration strength, production of crack or craze lines in the dentin and enamel, and pulpal inflammation (Sturdevant & others, 1995; Janis & Lugassy, 1972; Dilts & others, 1970; Standlee, Collard & Caputo, 1970; Shavell, 1980; Felton & others, 1991).

The retentive properties of intracoronal resistance features have been shown to be comparable with self-threading pins for some dental materials (Outhwaite, Garman & Pashley, 1979). In addition, it has been suggested that slot-retained composite restorations had higher retentive strength compared to pin-retained composite resins (Chan & Chan, 1987). In 1980, the amalgapin retention technique was first described by Shavell as assisting in the retention of complex amalgam restorations (Shavell, 1986). Roddy & others (1987) further modified the amalgapin concept, suggesting that the depth required for the intracoronal retentive channel feature be reduced.

The retention and resistance features of intracoronal composite pins for complex Class IV resin composite restorations have been investigated in bovine teeth (Roberts, Hermes & Charlton, 2000). The results from this study suggested that complex Class IV restorations using an intracoronal composite pin feature in the incisal aspect of the preparation had significantly more resistance to dislodgement than similar restorations that solely relied on enamel and dentin bonding. The relevance of bovine teeth as a suitable substrate is well documented (Retief, 1991; Retief & others, 1990). Also, the authors in the study involving bovine teeth noted the size discrepancy between bovine and human teeth, and the concern for tooth structure conservation in human teeth could necessitate smaller intracoronal retentive features.

This study evaluated the relationship between intracoronal retention features and fracture resistance of Class IV resin-based composite restorations in human incisors.

## METHODS AND MATERIALS

Human incisors extracted within six months were collected and stored in 0.5% Chloramine-T solution. The teeth were debrided of all soft tissue and the enamel surfaces were cleaned with a non-fluoride slurry of pumice flour (Moyco Union Broach, York, PA 17402, USA) using a rubber cup in a slow speed dental hand-piece (KaVo, Lake Zurich, IL 60047, USA). The teeth were rank-ordered by size, then divided into three treatment groups (n=16) of similar-size. Roots were notched and mounted into cylindrical molds with autopolymerizing acrylic resin (Dentsply/LD Caulk, Milford, DE 19963, USA) with the long axis of the tooth parallel to the long axis of the cylinder.

One operator (J-M) prepared standardized Class IV preparations of approximately 3 mm in mesial-distal width by 4 mm inciso-gingival height (Figure 1). The peripheral enamel margins were prepared with a 1-mm wide, 45-degree bevel as described by Summitt, Robbins & Schwartz (2001). Teeth in Group 1 (control) were prepared for the traditional Class IV composite restorations with no internal retentive features. Group 2 was prepared using a traditional Class IV prepara-

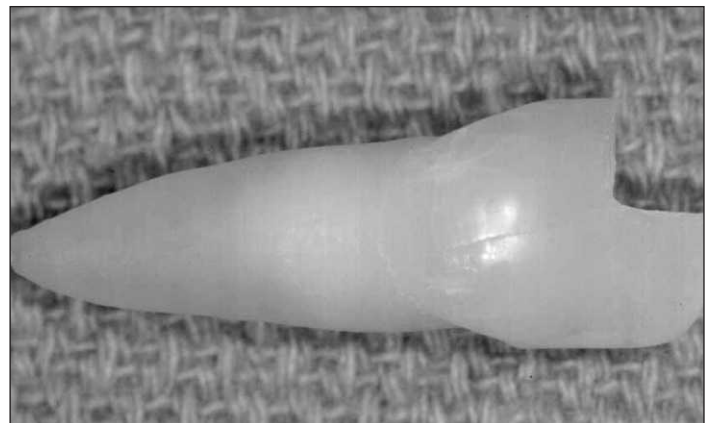


Figure 1. Class IV preparation, 3 x 4 mm.

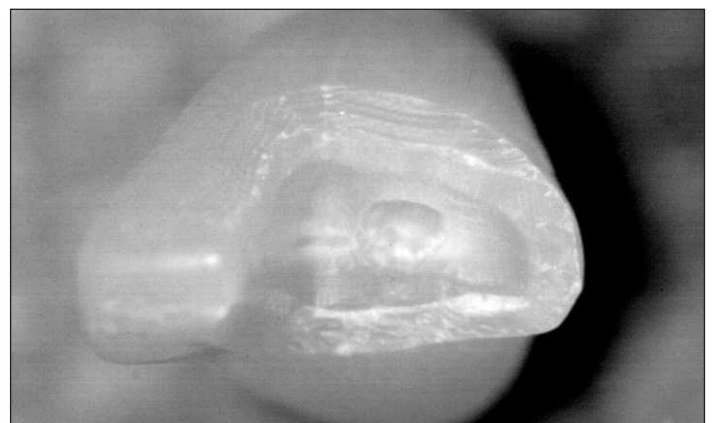


Figure 2. Cervically placed intracoronal retentive feature.





Figure 3. Load testing at 90° degrees with Instron Universal Testing Machine.

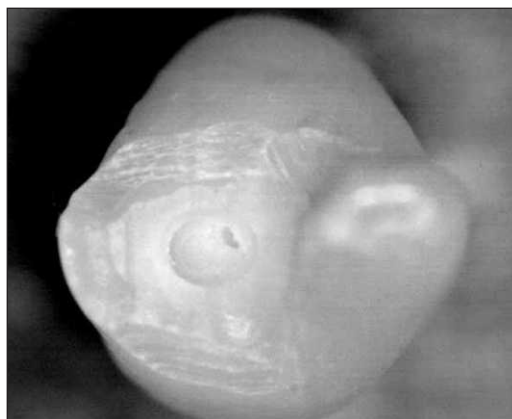


Figure 4. Fractured specimen revealing void in composite pin channel.

tion with a cervical pin channel placed in dentin 1 mm from the dentinoenamel junction (Figure 2) using a #330 carbide bur (SS White, Piscataway, NJ 08812, USA) to a depth of 1 mm with a high speed dental handpiece (KaVo, Lake Zurich, IL 60047, USA) as described by Roddy & others (1987). The teeth in Group 3 were prepared for Class IV composite restorations identical to those in Group 2 except that a #329 carbide bur (SS White) was used to prepare the intracoronar retentive feature. The enamel and dentin of the preparations were etched simultaneously with 34% phosphoric acid gel for 20 seconds, then rinsed with water for 30 seconds. The teeth were briefly air-dried and re-examined to ensure that the moist dentin remained. Optibond Solo Plus (SDS Kerr Corporation, Orange, CA 92867, USA) was applied following the manufacturer's directions and photo-polymerized for 40 seconds using an Optilux 400 visible light curing unit (Kerr/Demetron, Orange, CA 92867, USA). The preparations were restored with shade B-1 Point 4 resin composite (SDS Kerr Corporation). Composite was first placed into prepared intracoronar retention features with a #8A anterior composite placement instrument (Cosmedent, Chicago, IL 60611, USA), condensed to avoid air entrapment and photo-polymerized for 40 seconds. The preparation was then restored to anatomical form in 2-mm incremental layers polymerized for 40 seconds each. A minimum output of at least 400 mW/cm<sup>2</sup> was verified before each photo-polymerization cycle with a curing radiometer (Kerr/Demetron). The composite restorations were immediately finished to contour with Sof-Lex XT Disks (3M Dental Products, St Paul, MN 55144, USA) sequentially using medium (360), fine (600) and finishing superfine (1200) grit. Immediately after finishing, Optiguard FI (SDS Kerr Corporation) surface glaze was applied to each specimen using acid-etch preparation. The restored teeth were stored in distilled water at room temperature when not in use.

Specimens were thermocycled for 500 cycles at 5° and 55°C with a dwell time of 30 seconds and a transfer time of five seconds. Specimens were stored in distilled water at room temperature for one week. Prior to load testing, a round, 12-fluted carbide finishing bur (Brasseler USA, Savannah, GA 31419, USA) in a high-speed handpiece was used to place a dimple on the middle third of the lingual

surface of each restoration to assist in placement of the testing device. The specimens were mounted in an Instron Universal Testing Machine (Instron Corp, Canton, MA 02021, USA) and loaded at a 90-degree angle to the long axis of the tooth using a crosshead speed of 0.5 mm per minute until the specimen fractured (Figure 3). The force required to cause failure of the restoration was recorded for each sample with resultant data subjected to analysis of variance (ANOVA) at the 0.05 level of significance.

## RESULTS

Table 1 summarizes the mean fracture strength for the groups tested. Statistical analysis revealed that there was no difference in the force required to cause restoration failure among the three groups ( $p=0.185$ ).

One specimen from Group 1 was omitted due to fracture during experiment set-up. The data for one specimen each from Groups 2 and 3 were omitted because of root fracture occurring during the test.

## DISCUSSION

This study investigated the effect of using intracoronar composite pin retentive features and restoration resistance to the dislodgement of Class IV composite restorations in human teeth. Under the conditions of this study, there was no evidence to suggest a difference between groups with intracoronar retentive features and the control. The results of this study disagree with a previous study performed on bovine teeth (Roberts & others, 2000). However, the findings of this study may not totally relate with the previous study using bovine teeth. The investigation of Roberts & others (2000) investigated intracoronar retentive composite pin features in the incisal aspect of the preparation, whereas this study featured retentive features in the cervical region of the Class IV preparation. Both this study and the one on bovine teeth reported a high variability of data that can be explained by inherent differences in dentin, operator

Table 1: . Comparison of Fracture Strength in Newtons of Class IV Composite Restorations

Group	n	Mean	SD	Duncan's Grouping
1 (no internal retention)	15	633.9	234.3	A
2 (internal pin # 330)	15	759.5	400.6	A
3 (internal pin # 329)	15	532.2	305.7	A

Groups with the same letter are not statistically different from each other (p=0.185)

characteristics and technique sensitivity of dental materials. One of the assumptions necessary to make valid conclusions from numerical data is that a normal distribution of data exists and that standard deviation among groups is similar. These assumptions were satisfied in the current study.

Relating laboratory study results using bovine teeth to human teeth produce divided opinions (Retief, 1991). Nakamichi, Iwaku & Fusayama (1983) reported that the bond strengths of glass ionomer cements and resin composites to bovine enamel and dentin were not significantly different from human enamel and dentin. Saunders (1988) evaluated the bond strength of four dentin bonding agents to human premolars and bovine incisors and concluded that bovine dentin was suitable as a substitute for human dentin. Retief & others (1990), however, reported that the shear bond strength of composite to human dentin was substantially greater and microleakage was significantly less than to bovine dentin. It has also been reported in ultrastructural studies that the dentin tubular distribution is more compact in bovine dentin than in human specimens (Retief, 1991). Certainly, the use of human teeth in studies is ideal, but obtaining adequate numbers of intact, non-carious extracted human incisors is problematic.

Mechanical intracoronal retentive features for Class III preparations with adequate enamel have been reported to not significantly augment restoration retention in laboratory studies. Caplan, Denehy & Reinhardt (1990) demonstrated that retention grooves did not enhance failure resistance under load in composite restorations. Strassler & Buchness (1990) suggested that Class III preparations using solely etched enamel provided retention similar to preparations that used undercut retention. In addition, Summitt, Chan & Dutton (1992) did not detect a difference when evaluating Class III restorations in the failure load between groups of teeth that used intracoronal retentive grooves placed versus groups without grooves.

It has been suggested that using intracoronal resin composite pins analogous to the amalgapin technique may be somewhat beneficial in clinical situations for complex Class IV preparations that are particularly large and where less enamel is available for bonding (Roberts & others, 2000). As previously discussed, placing retentive channels is not without its hazards. Unlike the

study by Roberts & others (2000), the smaller size of human incisors used in this study necessitated placing intracoronal pin channels in the cervical aspect of the preparations. In human incisors, an incisally-placed pin channel using a #329 or #330 bur may result in unsupported enamel, while a pin channel placed apically into dentin may increase the risk of pulpal exposure. One touted advantage of using intracoronal composite pin retention was that retentive features could be placed without the need for additional armamentarium. However, fabricating a pin channel with small diameter burs could require significant manual dexterity and could be assisted with self-limiting, depth cutting burs. Other technical difficulties to consider include the application and polymerization of dentin adhesive into the pin channel and the problem of adequate condensation of composite into a small channel without voids (Figure 4). Clinicians should thoroughly consider the risks related to placing intracoronal retentive features in anterior teeth.

CONCLUSIONS

Under the conditions of this study, Class IV preparations using intracoronal composite pin retentive features in the cervical aspect of the preparations did not significantly improve restoration resistance to failure.

Disclaimer

The views and opinions expressed in this article are those of the authors and do not reflect the official policy or position of the United States Air Force, Department of Defense or the United States Government.

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# Long-Term Durability of Resin Dentin Interface: Nanoleakage vs Microtensile Bond Strength

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## Clinical Relevance

Self-etching primer adhesive systems may exhibit a decrease in bond strength over time due to nanoleakage-induced hydrolytic degradation.

## SUMMARY

This study tested the hypothesis that long-term durability of resin bonds to dentin is directly related to the nanoleakage of dentin bonding systems. Extracted human third molars were ground flat with 600-grit SiC paper under running water to expose middle dentin. Clearfil Liner Bond 2V (LB2V) or Fluoro Bond (FB) was applied to dentin surfaces according to the manufacturer's instructions. A crown was built-up with Clearfil AP-X resin composite, and the spec-

imens were stored in water for 24 hours at 37°C. The bonded assemblies were vertically sectioned into approximately 0.7 mm thick slabs and trimmed for microtensile bond test. All slabs were immersed in individual bottles of water at 37°C, which was changed every day. Specimens were incubated for one day, and three, six, and nine months, and at the specified time period, they were randomly divided to two subgroups: 50% AgNO<sub>3</sub> and the control. In the 50% AgNO<sub>3</sub> subgroup, the slabs were immersed for one hour in 50% AgNO<sub>3</sub>, followed by exposure in a photo-developing solution for 12 hours just prior to debonding. The specimens in the control subgroup were soaked in water until debonding. Then, all specimens were subjected to microtensile bond testing. The debonded specimens of the AgNO<sub>3</sub> subgroup had micrographs subjected to image analysis by NIH Image PC (Scion, Fredrick, MD, USA), and the area of silver penetration was quantitated. The bond strength data and silver penetration areas were subjected to two- and three-way ANOVA and Fisher's PLSD test at the 95% level of confidence. Regression analysis was used to test the relationship between bond strengths and the silver penetration area at each time period. For both adhesive

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systems, the bond strengths gradually decreased over time, although there were no statistically significant differences in the FB bond strength among the four time periods tested ( $p>0.05$ ). Silver penetration in specimens bonded with LB2V and FB gradually increased over time. Regression analysis showed a higher correlation between bond strength and silver penetration at 9 mm for specimens bonded with LB2V ( $R^2=0.844$ ) than at shorter time periods. The authors speculate that hydrolytic degradation within the hybrid layer gradually increased due to water penetration through nanoleakage channels, resulting in lower bond strengths and interfacial failure after as little as nine months.

INTRODUCTION

The durability of bonds between adhesive resins and dentin is of critical importance for the longevity of bonded restorations. Several reports that evaluate the durability of dentin bonds *in vitro* (Kiyomura, 1987; Burrow, Tagami & Hosoda, 1993) are available. The reports show that dentin bond strength decreased in water storage after several years. This degradation of bond strength may result from the plasticizing effects of water on resin and collagen (Maciel & others, 1996), to water sorption and/or hydrolysis of adhesive resin (Gwinnett & Yu, 1995; Burrow, Satoh & Tagami, 1996; Sano & others, 1998) or to hydrolysis of collagen fibrils at the base of the hybrid layer (Kiyomura, 1987; Burrow & others, 1996).

Recently, Okuda & others (2001) evaluated the long-term durability of dentin bonds of two total-etch single-bottle adhesive systems using an accelerated *in vitro* test by reducing the specimen size to a cross-sectional area of about 1 x 1 mm prior to storage (Shono & others, 2000) and by changing the storage medium daily (Kitasako & others, 2000) to maximize hydrolysis over time. They reported that the tensile bond strengths for total-etch single bottle adhesive systems dramatically decreased after a relatively short storage time (three months). They also compared the long-term durability of dentin bonds with silver uptake on the same specimens as a measure of nanoleakage. However, that study failed to show a relationship between bond strength and nanoleakage after water storage for nine months (Okuda & others, 2001).

On the other hand, Sano & others (1999) reported that although *in vivo* bond strengths were relatively stable over one year, porosity of the hybrid layer increased over time for a self-etching primer adhesive system

(Clearfil Liner Bond 2). This increase in porosity within the hybrid layer would form nanoleakage pathways that were thought to permit fluid penetration within the hybrid layer. They speculated that fluid penetration through nanoleakage pathways might cause degradation of the dentin bond with the self-etching primer adhesive system (Sano & others, 1999) although they had no direct evidence. However, the relationship between degradation of the resin-dentin bond and nanoleakage is unclear. Moreover, concern remains regarding the long-term durability of resin bonds since degradation of the resin-dentin interface may result from hydrolytic attack of resin through ever enlarging nanoleakage pathways.

This study tested the hypothesis that long-term durability of dentin bonds is directly related to nanoleakage using self-etching primer adhesive systems. The null hypothesis was that there is no correlation between resin-dentin bond strengths of self-etching primer systems and nanoleakage over time.

METHODS AND MATERIALS

Twenty extracted human third molars stored frozen were used in this study. The teeth were thawed, cleaned of debris and their occlusal surface was ground flat to remove the enamel and exposed middle dentin. The dentin surfaces were inspected to ensure that there were no remnants of enamel, then polished with 600-grit silicone carbide paper under running water.

The bonding procedures followed the manufacturers' recommendations. The adhesive systems used in this study were Clearfil Liner Bond 2V (LB2V; Kuraray Co, Tokyo, Japan) and Fluoro Bond (FB; Shofu Co, Kyoto, Japan). Both used self-etching primers (Table 1). Ten teeth were prepared for each material. For LB2V, dentin surfaces were treated with a mixture of Primer liquid A and B for 30 seconds. After gentle air-drying, Bond liquid A was applied to the surface, thinned with a gentle stream of air and light cured for 10 seconds.

Table 1: Materials Used in This Study

System	Ingredient	Code/Batch #
Liner Bond 2V (Kuraray)		
	Primer A	MDP, HEMA, H2O, Photoinitiator Accelerators
	Primer B	HEMA, H2O, Accelerators
	Bond A	MDP, Dimethacrylates, Photoinitiator Accelerators
Fluoro Bond (Shofu)		
	PrimerA	Water, Acetone, Catalyst
	Primer B	HEMA, 4-MET, 4AETA, Catalyst
	Bonding	Full reacted-glass ionomer, UDMA, HEMA, 4-AET, Photoinitiator

The crown was then built-up incrementally over the adhesive resin using Clearfil AP-X (Kuraray Co, Osaka, Japan) resin composite. The bonded assemblies were stored in tap water at 37°C for one day, then sectioned perpendicular to the bonded interface into approximately 0.7 mm thick slabs with a diamond saw (Figure 1). The adhesive-dentin interface was trimmed to an approximate width of 1.4 mm with a fine diamond bur to produce a cross-sectional surface area of approximately 1 mm<sup>2</sup> for micro-tensile bond testing. Eight or nine slabs were cut perpendicular to the bonded interface from each tooth. After pooling all the slabs from each bonding system, they were immersed in individual bottles containing water at 37°C. The water was changed daily until testing.

The specimens were randomly assigned to four groups with specified time periods (one day, and three, six and nine months). At the specified time period, the specimens to be tested were randomly divided into two subgroups: in one subgroup, the slabs were coated with fingernail varnish except for approximately 0.5 mm around the trimmed bonded interface (Figure 1) and immersed in 50 wt% AgNO<sub>3</sub> for one hour followed by immersion in a photo-developing solution for 12 hours. The specimens in the other subgroup were soaked in water for 13 hours. Then, all specimens were subjected to microtensile bond testing using a table-top material tester (EZ-test, Shimadzu Co, Kyoto, Japan) at a crosshead speed of 1 mm/minute (Figure 1). All debonded specimens were fixed in 10% neutral formalin for at least eight hours.

The debonded specimens in the AgNO<sub>3</sub> subgroup had micrographs taken of the fractured surfaces using light microscopy at x64 magnification. These images were scanned to convert them from analog to digital format. They were then subjected to image analysis by NIH Image PC (Scion), and the area of silver penetration (black stain) was quantitated automatically with a standardized level in black/white digital image. Means and standard deviations of silver penetration surface areas were calculated for specimens from each time period (one day, and three, six and nine months).

Bond strength data and the silver penetration areas were subjected to two and three-way ANOVA (material, time,  $\pm$  AgNO<sub>3</sub>) and Fisher's PLSD test at the 95% level of confidence. Regression analysis was used to test the relationship between the bond strength and silver penetration surface area at each time period. All fractured specimens were

placed on SEM stubs and gold sputter-coated after air-drying. Using scanning electron microscopy (JSM-5310LV, JEOL, Tokyo, Japan), their fracture patterns were assessed by three investigators under the micrographs (x70), and the morphology of the representative areas within the debonded surfaces were observed at x10,000 magnification.

## RESULTS

Table 2 (Control group) and Table 3 (AgNO<sub>3</sub> group) summarize the means and standard deviations of the microtensile bond strengths for LB2V and FB. The tensile bond strengths of LB2V gradually decreased over time. The ANOVA revealed significant differences in materials (LB2V vs FB) ( $p < 0.001$ ) and time periods ( $p = 0.008$ ), while bond strength of the control group was not significantly

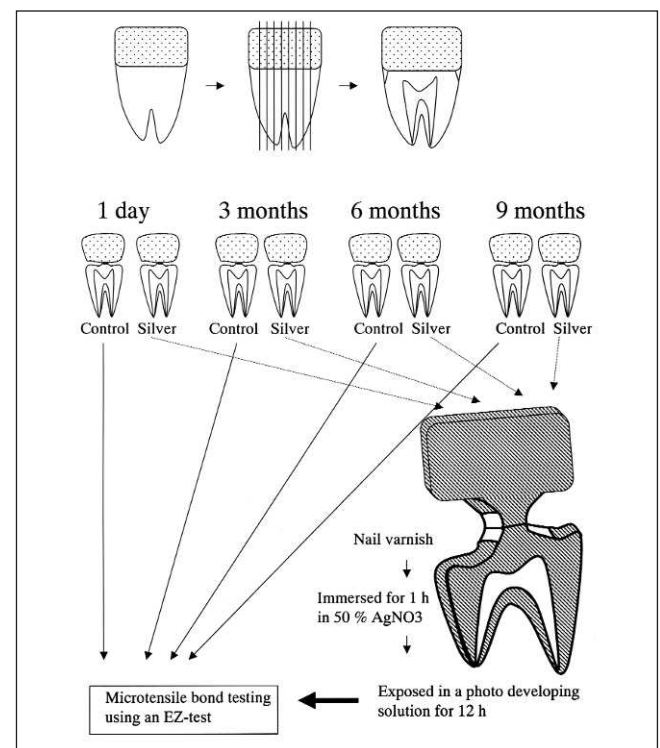


Figure 1. Schematic showing how bonded composite buildups were vertically serially-sectioned into 0.7 mm thick slabs that were then trimmed to hour-glass shapes giving a 1.4 mm<sup>2</sup> bonded cross-sectional area. These were incubated in 37°C water for one day, three, six or nine months prior to immersion in water (control) or silver nitrate (exp) and then the bond strengths tested.

Table 2: Microtensile Bond Strengths of Self-Etching Primer Adhesives Over Time

(Control group)				
Periods	One Day	Three Months	Six Months	Nine Months
Adhesive				
LB	52.2 $\pm$ 12.0 <sup>a</sup> (12)	47.3 $\pm$ 13.1 <sup>ab</sup> (10)	40.0 $\pm$ 9.1b, <sup>c</sup> (10)	32.3 $\pm$ 12.8 <sup>c</sup> (10)
FB	35.6 $\pm$ 9.1 <sup>a</sup> (14)	30.3 $\pm$ 14.8 <sup>c</sup> (9)	28.5 $\pm$ 10.2 <sup>c</sup> (8)	25.8 $\pm$ 9.7 <sup>c</sup> (7)

Values are mean  $\pm$  SD (N). Groups identified by different superscript lower case letters are significantly different ( $p < 0.05$ ).

different from that of the AgNO<sub>3</sub> group ( $p=0.2374$ ). The ANOVA indicated that there were no interactions between materials with or without silver penetration and time periods ( $p>0.05$ ). Tensile bond strengths in the LB2V control subgroup became significantly lower after six and nine months ( $p<0.05$ ) than after one day, and the bond strengths after nine months were about two-thirds of the value of the one day group. In the FB control subgroup, there were no significant differences among the four time periods tested ( $p>0.05$ ) due, in part, to the lower initial bond strength. The tensile bond strength of this subgroup also gradually decreased over time. Within the subgroups exposed to AgNO<sub>3</sub>, the tensile bond strength of both materials was similar to the control subgroups. The tensile bond strengths in the AgNO<sub>3</sub> subgroup bonded with LB2V decreased over time, while the tensile bond strengths in the AgNO<sub>3</sub> subgroups that bonded with FB did not change among the testing periods except for three months ( $p>0.05$ ). There were no significant differences between the bond strengths of the control and the AgNO<sub>3</sub> subgroups within either material at each time period ( $p>0.05$ ).

Table 4 summarizes the means and standard deviations of silver penetration areas for specimens bonded with LB2V and FB. Two-way ANOVA revealed significant differences in the material (LB2V vs FB) ( $p=0.0025$ ) and the time periods ( $p<0.0001$ ). The silver penetration of specimens bonded with LB2V gradually increased over time, and the silver penetration at nine months was significantly greater than that seen at one day ( $p<0.05$ ). The penetration area of silver for specimens bonded with FB also gradually increased over time. The silver penetration at three and nine months was significantly greater than that of the one-day ( $p<0.05$ ) values. However, there were no significant differences between one-day and three month values and between the three and nine month values ( $p>0.05$ ).

Regression analysis for the relationship between microtensile bond strength and silver penetration for specimens bonded with LB2V showed an inverse relationship that grew stronger with time (Figure 2). In the one-day group, the R<sup>2</sup> value was very low (R<sup>2</sup>=0.007). After three and six months, the R<sup>2</sup> values increased but remained relatively low (R<sup>2</sup>=0.519 and 0.555, respectively). In contrast, after nine months the correlation was much higher (R<sup>2</sup>=0.844). However, regression analysis for the relationship between microtensile bond strength and nanoleakage of FB failed to produce any consistent significant correlation. That is, although there was a negative correlation between FB bond strengths and silver penetration at one day, three and six months, by nine months the correlation became positive (Figure 3).

SEM observations of fractured sites of specimens bonded with LB2V showed morphological changes over time. Failures occurred within the resinous component at the adhesive resin in the one-day group (Figure 4A). In the three and six month groups, there were a mixed failures within the adhesive resin and hybrid layer, although most of the surface area of the failure pattern was still in the adhesive resin. In the nine-month group, SEM observations showed that failures were predominant involving the hybrid layer. In the area of failures within the hybrid layer, the fibrous structures were observed more than intact dentin (Figures 4B and 4C). SEM observations of fractured sites of specimens bonded with FB showed nearly identical appearances over time. That is, all fracture modes were mainly classified as a mixed cohesive failure within adhesive resin and in the hybrid layer. The fractured area within the hybrid layer in specimens of FB was more fibrous than in LB2V (Figures 5A, 5B and 5C).

Table 3				
(AgNO <sub>3</sub> group)				
Periods	One Day	Three Months	Six Months	Nine Months
Adhesive				
LB	52.7 ± 7.3 <sup>a</sup> (8)	42.9 ± 8.4 <sup>a,b</sup> (8)	45.5 ± 9.6 <sup>a,b</sup> (9)	36.4 ± 1.35 <sup>b</sup> (9)
FB	35.7 ± 11.0 <sup>b</sup> (12)	24.9 ± 12.4 <sup>c</sup> (8)	39.1 ± 15.2 <sup>b</sup> (7)	33.0 ± 13.0 <sup>b,c</sup> (8)
Values are the mean ± SD (N). Groups identified with the same superscript letters are not significantly different ( $p>0.05$ ) using Fisher's PLSD test. Groups identified by different superscript letters are significantly different ( $p<0.05$ ).				

Table 4: The Area of Silver Penetration				
Periods	One Day	Three Months	Six Months	Nine Months
Adhesive				
LB	0.056 (0.021) <sup>a</sup> (8)	0.079 (0.025) <sup>a,b</sup> (8)	0.077 (0.022) <sup>a,b</sup> (9)	0.090 (0.027) <sup>b</sup> (9)
FB	0.075 (0.015) <sup>a,b</sup> (12)	0.105 (0.028) <sup>c</sup> (8)	0.078 (0.019) <sup>b</sup> (7)	0.113 (0.018) <sup>b,c</sup> 8
Values are mean mm <sup>2</sup> (SD), N. Mean figures identified by the same superscript letter are not significantly different ( $p>0.05$ ) using Fisher's PLSD test.				



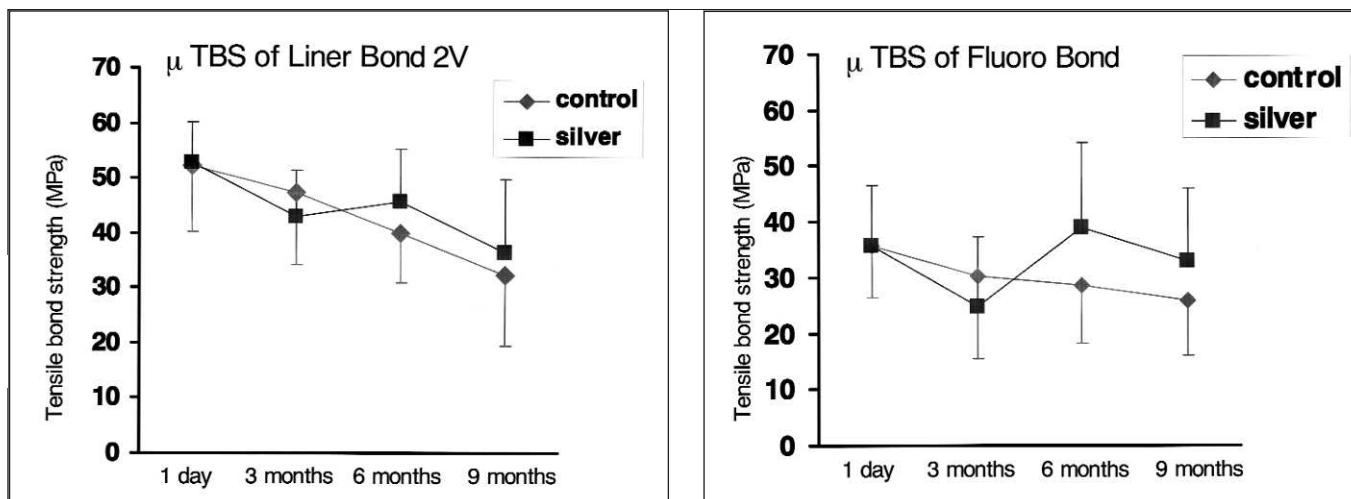


Figure 2. Regression analysis of  $\mu$ TBS of LB-bonded specimens versus silver uptake, as a function of incubation time. A significant negative correlation developed after three, six and nine months of incubation.

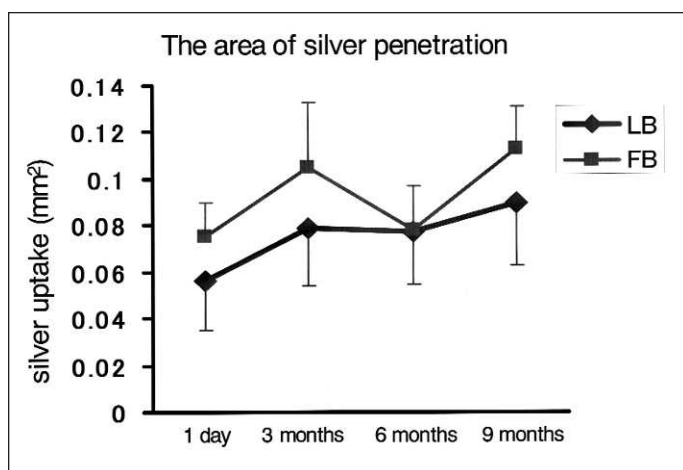


Figure 3. Regression analysis of  $\mu$ TBS of FB-bonded specimens versus silver uptake, as a function of incubation time. None of the regressions were statistically significant. NS=not significant.

## DISCUSSION

Recently, the term nanoleakage has been suggested to distinguish silver uptake into gap-free resin bonded interfaces from microleakage via gaps (Sano & others, 1995). Nanoleakage was defined as the diffusion of silver ions through nanometer-sized channels within gap-free restoration. The most important pathway for nanoleakage is within the hybrid layer that has not been fully penetrated by resin, leaving spaces for fluid penetration. Fluid penetration through nanoleakage pathways may cause degradation of the dentin bond (Sano & others, 1999). Sano & others (1999) demonstrated that fractured interfaces of resin-dentin bonds obtained using a self-etching primer after one year of function exhibited an increased porosity over time, whereas bond strength remained unaltered. They speculated that the increased porosity at the bonded inter-

face over time might have occurred via nanoleakage pathways that developed within the hybrid layer. Hashimoto & others (2000) investigated *in vivo* degradation of resin-dentin bonds in humans over one-to-three years using Scotchbond Multi-Purpose on cavities etched with 10% maleic acid. They reported that bond strengths decreased significantly over time. Their SEM observations of the resin-dentin interface revealed the complete loss of resinous material between the collagen fibrils or depletion of collagen fibrils within the degraded hybridized dentin. They speculated that nanoleakage in the oral cavity was caused by deterioration of the hybrid layer (Hashimoto & others, 2000), creating nanometer-sized diffusion channels.

In this study, the bond strength of both self-etching primer adhesive systems gradually decreased over time. This was associated with a gradual increase in silver penetration of specimens bonded with both self-etching primer adhesive systems over time. In our previous *in vitro* study (Okuda & others, 2001), although the bond strength of two total-etch, single-bottle adhesive systems gradually decreased over time in a manner similar to the current study, silver uptake (nanoleakage) did not increase during the testing periods. That is, the silver uptake began at a high level and did not change over time. In contrast, the silver uptake by the two self-etching primers used in this study began at values less than half that of the total etched specimens in the previous study. Even the maximum silver uptake in LB2V at nine months in the current study was less than the initial silver uptake by the total etched single-bottle adhesive systems (One-Step and Single Bond) reported by Okuda & others (2001). These observations also agree with Pereira & others (2001), who reported that the penetration of silver was greater in hybrid layers created using Single Bond than in those created by Clearfil Liner Bond 2V. Presumably, this was due to



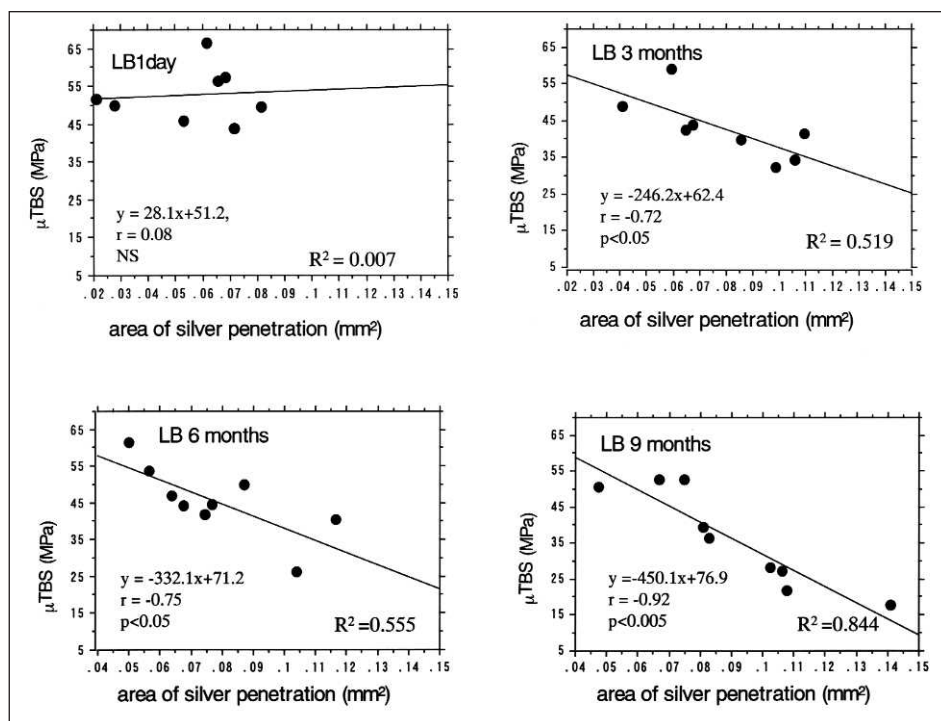


Figure 4. Secondary electron images of the dentin side of failed LB resin-dentin bonds. A. 1-day specimen showing cohesive failure in the adhesive. No tubules are evident. B. Six-month specimen exhibiting mixed cohesive failure of the hybrid layer and the adhesive layer. The presence of patent tubule orifices surrounded by peritubular dentin matrix indicates that this specimen fractured near the base of the hybrid layer. Few collagen fibrils are exposed. C. Nine-month specimen, exhibiting more collagen fibrils.

the fact that the hybrid layer created by Single Bond (ca 3  $\mu\text{m}$ ) was thicker than LB2V (ca 0.5  $\mu\text{m}$ ) (Pereira & others 2001). The deeper the dentin matrix is demineralized (that is, the thicker the hybrid layer), the greater the potential for nanoleakage of silver ions within partially or fully demineralized dentin, the hybrid layer or within partially polymerized adhesive resin (Pereira & others, 2001).

When a self-etching primer is applied to a dentin surface covered with smear layer, the acidic primer etches through the smear layer into the underlying mineralized dentin (Watanabe, Nakabayashi & Pashley, 1994). The difference in acidity of self-etching primer would reflect the difference in the demineralization effect so that it would be associated with the ability to remove the smear layer and smear plug. The SEM observation demonstrated that the debonded surface of the LB2V specimens showed the absence of resin tags in the dentinal tubules, while the resin tags of FB could be seen in dentinal tubules. This indicates that the FB primer would have a higher removal ability of smear plugs than the LB2V primer. In this study, FB had significantly greater nanoleakage than LB2V. However, this might not result from the higher demineralization ability of the FB primer because a self-etching primer simultaneously demineralizes and penetrates into dentin.

The distribution of silver nitrate as an index of nanoleakage was limited to its penetration from the periphery. As this distance is controlled by diffusion, it should vary with the square root to time (Pashley, Horner & Brewer, 1992), which was standardized in this and in our previous studies at one hour. In our previous study using total-etch single-bottle adhesive systems, nanoleakage did not change during the testing periods (Okuda & others, 2001). The maximum penetration of silver ion into peripheral nanoleakage spaces in that study was obtained by immersion of 50%  $\text{AgNO}_3$  solution for one hour. If immersion time in  $\text{AgNO}_3$  is changed, different results may be obtained. We had speculated that nanoleakage of total-etch single-bottle adhesive systems would increase over time. The fact that it was higher than that found with self-etching primers, and the fact that it did not change over time indicates that there was a discrepancy between the depth of etch and the depth of resin penetration that remained constant with time.

In the current study, as the bond strength of LB2V gradually decreased over time, there was a corresponding increase in silver penetration. Under regression analysis, the relationship between microtensile bond strength of LB2V bonds and silver penetration showed a gradual increase in negative correlation over time. Failures in the one-day group occurred within the resinous component at the bond. That is, the adhesive bond to the hybrid layer exceeded the cohesive strength of the resin. This indicated that the tensile bond strength data did not reflect the quality of the hybrid layer or the amount of nanoleakage. Therefore, the correlation coefficient ( $r$ ) value for the relationship between microtensile bond strength and silver penetration was very low ( $r=0.08$ ) and not statistically significant ( $p>0.8$ ). After three and six months, the proportion of interfacial failures, including portions of the hybrid layer at the fractured surface, was greater. However, most of the failures still occurred within the resinous component. This was associated with more negative correlation coefficient values for the three- and six-months specimens of -0.72 and -0.75, both of which were highly statistically significant ( $p<0.05$  and 0.005, respectively). In contrast, after nine months, SEM observations of LB2V bonded specimens showed

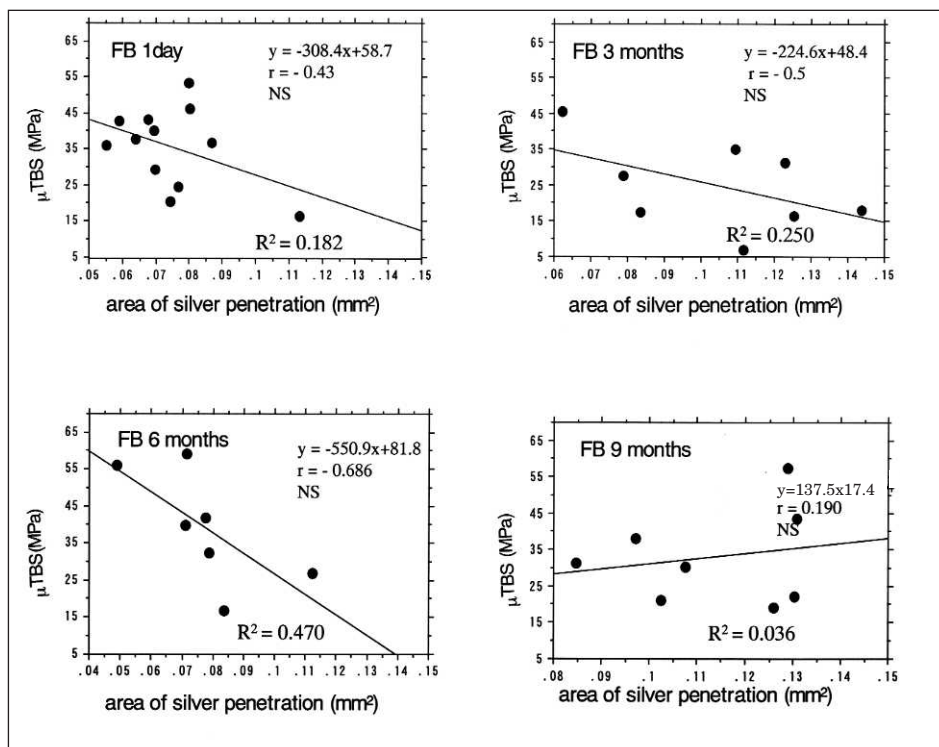


Figure 5. Secondary electron images of the dentin side of failed FB resin-dentin bonds. A. One-day specimen exhibiting mixed cohesive failures of the hybrid layer and adhesive layer. B. Six-month specimen showing cohesive failure at the base of the hybrid layer and cohesive failure of resin tags in tubules. C. Nine-month specimen showing failure patterns similar to B.

that most of the failures were predominated involving the hybrid layer. Moreover, regression analysis showed a very high correlation coefficient ( $r = -0.92$ ) between tensile bond strength and silver penetration at nine months. The high  $R^2$  value, 0.844 indicates that the equation of the line is very predictive ( $p < 0.005$ ). We speculate that the highly significant correlation between decreases in bond strength and increases in silver uptake reflect degradation at the resin-dentin interface. Considering the effects of the lack of water storage on the mechanical properties of demineralized dentin matrices over a four-year period (Carvalho & others, 2000), we propose that the fall in bond strength was due to hydrolysis of ester bonds of the polymerized resin within the hybrid layer that gradually increased as water diffused through nanoleakage channels that became larger over time, resulting in low bond strengths and interfacial failure after nine months.

For FB, although the bond strengths gradually decreased over time, there were no statistically significant differences in the bond strength among the four time periods tested ( $p > 0.05$ ). The silver penetration at three and nine months were significantly greater than the one-day ( $p < 0.05$ ) values. Increased silver penetration indicated that the hydrolytic degradation of the resin-dentin interface progressed. However, the bond

strengths were relatively stable. In the case of FB, the fracture pattern showed mixed failure at the interface throughout the testing periods. That meant that the tensile bond strength data did not reflect degradation of the resin-dentin interface through the testing periods. Therefore, regression analysis of FB bonded specimens failed to produce any consistent, significant correlation between microtensile bond strength and silver penetration ( $R^2 = 0.04 \sim 0.47$ ).

Recently, manufacturers have developed various fluoride-releasing restorative materials because fluoride is thought to exert its anti-cariogenic activity by increasing enamel and dentin resistance to subsequent acid attack (Corpron & others, 1986; Dionysopoulos, Kotsanos & Papadogiannis, 1990) or by inhibiting the carbohydrate metabolism in dental plaque (Norman & others, 1972). FB adhesion contains fluoride in the form of a GIC filler. The manufacturer claims that the fluoride contained in FB prevents secondary caries.

On the other hand, Saito (1996) demonstrated that fluoride contained in MMA-TBB resin modified the durability of dentin bond. It was reported that the tensile bond strengths of MMA-TBB resin containing fluoride did not decrease after long-term (18 months) water immersion, while the tensile bond strengths of the same resin system without fluoride decreased during the same immersion time (Saito, 1996). It was speculated that the fluoride somehow prevented the degradation of dentin, resulting in improvement of the long-term stability at the dentin interface (Saito, 1996). In the current study, the nanoleakage of FB did not increase from three months to six months although it increased until three months, and the bond strengths were relatively stable during testing periods. This might indicate that the hydrolytic degradation of the resin-dentin interface of FB did not progress from three to six months. This might be due to the effect of fluoride release from the FB adhesive. Further research is needed to clarify the effect of fluoride-containing adhesives on the durability of resin-dentin bonds.

## CONCLUSIONS

Within the limitations of this *in vitro* study, nanoleakage was concluded to gradually increase at the dentin

interfaces of the self-etching primer adhesive systems tested. As a result, the bond strength of both adhesive systems was gradually decreased over time. There was a highly significant negative correlation between tensile bond strength and nanoleakage within LB2V after three, six and nine months of storage, presumably due to the degradation of resin at the resin-dentin interface.

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# Effect of Interfacial Bond Quality on the Direction of Polymerization Shrinkage Flow in Resin Composite Restorations

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## Clinical Relevance

If a good bond between dentin and resin composite can be established, the shrinkage flow will be directed toward a center located near the bonded interface rather than toward the incident light.

## SUMMARY

Shrinkage in light curing resin composites is assumed to be directed toward the light source. However, the strong bond at the dentin-resin interface achieved by newer generation dentin bonding systems may affect the direction of polymerization shrinkage. In this study, various curing modes of adhesive resin simulating different bond qualities were applied to determine the extent of interfacial gap formation with a scanning electron microscope. We also measured the free surface depression with a profilometer. The direction of polymerization shrinkage was

inferred from the ratio of the interfacial gap measurement at the floor to the free surface depression. Various curing modes used in this study include Group 1: light curing of resin composite without the bonding agent as the negative control; Group 2: simultaneous light curing of the bonding agent and resin composite; Group 3: start of the chemical cure of the dual-cured bonding agent before light curing the resin composite; Group 4: curing the light-initiated bonding agent before insertion and light curing of the resin composite. When the bonding agent was light cured prior to inserting the resin composite (Group 4), the free surface depression was the greatest and the interfacial gap smallest among those in all groups. Therefore, if a good bond between dentin and resin composite can be established, the shrinkage flow will be directed toward a center located near the bonded interface rather than toward the incident light, thus reducing detrimental shrinkage stress.

## INTRODUCTION

Due to the nature of polymerization of dimethacrylate resin systems in which single monomer molecules polymerize to form a cross-linked polymer network, the inherent polymerization shrinkage of resin composites cannot be avoided. This volume shrinkage of 2–4 vol %; (Feilzer, de Gee & Davidson, 1988; Ferracane, 2001) results in a number of problems related to restoration

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of the resin composite, such as microleakage, hypersensitivity, marginal discoloration, secondary caries and pulpal pathology (Retief, 1994). To minimize these shrinkage-related complications, many innovative approaches, such as development of new resin monomers and improvement of filler systems, were tried in the field of material science (Asmussen, 1975; Liu, Collard & Armeniades, 1990; Stansbury, 1992; Eick & others, 1993) and a variety of meticulous techniques, such as the incremental filling technique (Lutz, Krejci & Oldenburg, 1986a), applying the "C-factor" concept (Feilzer, de Gee & Davidson, 1987), the three-sided light-cure technique (Lutz & others, 1986b), controlled (Uno & Asmussen, 1991) or soft-start polymerization (Davidson & Feilzer, 1997), additional steps with flexible liners (Kemp-Scholte & Davidson, 1988) and the rebond technique (Torstenson, Brännström & Mattsson, 1985) were introduced into routine clinical procedures.

To improve the marginal adaptation of resin composite using these clinical remedies, there needs to be an understanding of the direction of polymerization shrinkage of resin composite. Traditionally, shrinkage has been assumed to occur toward the center of the bulk in self-curing resin composites and toward the light source in light curing resin composites (Lutz & others, 1986b; Fusayama, 1992). However, different factors have been suggested to affect the direction of polymerization shrinkage of the light-curing resin composite in clinical situations. These factors include cavity design (C-factor), the polymerization reaction rate, thickness, translucency and rheological properties of the resin composite and the intensity and direction of the light (Hansen, 1982; Hansen, 1986; Feilzer & others, 1987; Uno & Asmussen, 1991; Unterbrink & Muessner, 1995; Asmussen & Peutzfeldt, 1999). Interfacial bond strength was also considered a contributing factor in a theoretical finite element analysis (FEA) (Versluis, Tantbirojn & Douglas, 1998).

Among these factors, interfacial bond strength is the most likely candidate to affect shrinkage direction, as the apparent interfacial bond strength of newer generation dentin bonding systems has rapidly exceeded the stresses resulting from polymerization shrinkage of the resin composite (Triolo, Swift & Barkmeier, 1995; Swift & Bayne, 1997; Wakefield & others, 1998; Pashley & others, 1999). To obtain a strong bond in current light cured dentin bonding systems, sufficient polymerization of the bonding agent before insertion and light-curing of the resin composite is recommended (Davidson & de Gee, 1984b; Imai & others, 1991; Feilzer, de Gee & Davidson, 1993). Adequate polymerization of the bonding agent at the interface may improve the bond quality of the adhesive layer, turn the direction of the shrinkage towards the interface and minimize the undesirable effect of the polymerization shrinkage. Theoretically, if

the bond between tooth structure and resin composite is assumed to be perfect, as in FEA, the shrinkage vector can even be directed entirely towards the fixed boundaries and to the composite-resin interface (Katona & Winkler, 1994; Versluis & others, 1998).

Therefore, it was hypothesized that the direction of polymerization shrinkage of the overlying light-curing resin composite would be affected if the quality of the interfacial bond between the tooth structure and resin composite was altered by modifying the mode and time of curing of the dentin bonding agent. For this purpose, the quality of the interfacial bond was modified by applying various curing modes to the dentin bonding system and the interfacial gap between the cavity wall and restoration and the maximum depression of the restoration at the free surface were measured quantitatively. From these measurements, the direction of polymerization shrinkage was inferred.

## METHODS AND MATERIALS

### Experimental Groups and Material Selection

Four different experimental setups were used (Table 1) to simulate different quality levels of the interfacial bond. They consisted of a negative control without any bonding resin present and three experimental groups where the dentin bonding systems were varied when light curing the overlying resin composite.

Group 1—Negative control: the resin composite was filled and light cured without bonding agent.

Group 2—Late cure: the light cured bonding agent was applied but not cured until after inserting the resin composite.

Group 3—Intermediate cure: the dual-cured bonding agent was used so that the bonding agent had already been somewhat cured chemically before inserting and light-curing the resin composite.

Group 4—Early cure: the light cured bonding agent was applied and cured before inserting the resin composite.

To distinguish the different qualities of interfacial bonds when curing the resin composite, peak cure times of the bonding agent in each experimental group were distinctly different. The peak cure time was measured with a differential scanning calorimeter (DSC; DSC 7 system; Perkin-Elmer LLC, Norwalk, CT 06859, USA). Table 1 shows the combinations of experimental materials and curing modes. Peak cure times were determined by irradiating the bonding agents for 20 seconds and the resin composites for 40 seconds at 500 mW/cm<sup>2</sup>. All-Bond 2 (Batch #069268; BISCO Inc, Schaumburg, IL 60193, USA) was used as the bonding agent because it could be cured by light (E/D Bonding Resin only) or in the dual-curing mode by simply mixing the E/D Bonding Resin with Pre-bond. The cavities

were filled as described below with a high modulus composite (HMC; Z100, Batch #5095; 3M Dental Products, St Paul, MN 55144, USA) or a flowable resin composite (FC; *Æliteflo*, Batch #H-671; BISCO Inc).

### Measurements of Interfacial Gap and Free Surface Depression

Twenty-eight human molars free of visible defects were stored in 0.5 mass fraction % aqueous chloramine-T solution until used. Within one month of extraction, they were embedded in self-curing epoxy resin and the middle third of the buccal and lingual surfaces were ground to expose flat enamel surfaces. Class I conventional 90° butt-joint type cavities with dimensions of 2 mm x 2 mm x 2 mm were prepared so that at least 0.5 mm of enamel remained along the entire margin. The teeth were randomly divided into four groups. In each group, the seven buccal and seven lingual cavities were treated following the assigned protocol shown in Table 1. For groups using the bonding agent (Groups 2, 3 and 4), the cavities were etched for 15 seconds with 32% phosphoric acid gel (Uni-Etch; BISCO Co) and rinsed with copious amounts of water. After gently removing excess water, five coatings of the primer solution were applied and dried for 10 seconds, followed by applying and thinning the bonding agent. In Group 4, the bonding agent was light cured for 20 seconds. All cavities were filled in one increment, the buccal cavities with HMC and the lingual cavities with FC. After removing the excess material, the cavity and restorative material were covered with a Mylar strip and a slide cover glass to reduce irregularities and overhangs at the margin. The resin composites were then light cured.

After one day, the maximum free surface depression was measured with a profilometer (Alpha-step 200; Tencor Instrument Co, San Jose, CA 95134, USA). Twenty-four hours later, the specimens were re-embedded and sectioned bucco-lingually with a diamond saw through the center of the restorations. The surfaces were cleaned with phosphoric acid to remove

the smear layer. Silicone impressions (*Examix*; GC Co, Tokyo, 174, Japan) were used to prepare epoxy resin replicas. Images of the gold sputter-coated replicas obtained with a scanning electron microscope (SEM; JSM-840A; JEOL Ltd, Tokyo, 196-8558, Japan) were analyzed with Sigma Scan (Image ver 1.20; Jandel Scientific, Chicago, IL 60606, USA). To obtain a more accurate interfacial gap measurement, the “Maximum Gap” (MG) and “Marginal Index” (MI) parameters developed by Hansen & Asmussen (1988) were modified into Average Maximum Gap (AMG) and Average Marginal Index (AMI). For this purpose, each cavity wall was divided into five equal segments and a SEM image (about 230 µm wide) was taken at x500 magnification at the center of each segment. In each image, the width of the widest portion of the gap was measured and the AMG of a wall was calculated as the mean of the five measurements along that wall. Images of each wall at x100 magnification were used to calculate the Gap Proportion (GP) from the ratio of the debonded perimeter to the total wall length. From AMG and GP, the AMI of each wall was calculated as  $AMI = AMG \times GP / 100$ . The usefulness of the AMI to represent the gap dimension at a cavity wall was verified using Pearson Product Moment Correlation between AMG and AMI. The direction of polymerization shrinkage was inferred from the ratio of AMI at the cavity floor ( $AMI_F$ ) to the free surface depression. One-Way ANOVA and Duncan’s all pairwise multiple comparison methods compared the AMI parameters, surface depression values and ratios among walls and groups. Student t-test determined the statistical differences between AMI values of the HMC-filled buccal and the FC-filled lingual cavities for each group.

### RESULTS

Table 1 shows the peak cure times measured by DSC. This data shows that the cure time of the bonding agent in each group differed from other groups. Figure 1 displays SEM photographs demonstrating typical interfa-

Table 1: .The Combinations of Experimental Materials and Curing Modes in the Experimental Groups and Their Peak Cure Times (Unit: second, mean  $\pm$  SD, n=3)

	Etching <sup>a</sup>	Primer <sup>b</sup>	Bonding Agent <sup>b</sup>		Peak Cure Time
		NL <sup>c</sup>	Light-Cure	Dual-Cure	
Group 1 (no bond)	-	- <sup>a</sup>	-	-	-
Group 2 (late cure)	(+)	(+) <sup>a</sup>	(+)/NL	-	$\alpha^d$
Group 3 (intermediate cure)	(+)	(+)	-	(+)/NL	142 $\pm$ 6
Group 4 (early cure)	(+)	(+)	(+) / LC <sup>c</sup>	-	8 $\pm$ 2
Composite Resin (Z-100)		LC <sup>c</sup>			7 $\pm$ 0

a: (+) means that the materials or bonding steps were used, but ‘-’ means that they were not used.

b: The Primer and E/D Bonding resin of All-Bond2 were used as dentin bonding system throughout this experiment.

c: NL means “no light-cure” and LC means “light-cure”(20 seconds for bonding agent and 40 seconds for resin composite).

d: the materials did not polymerize.

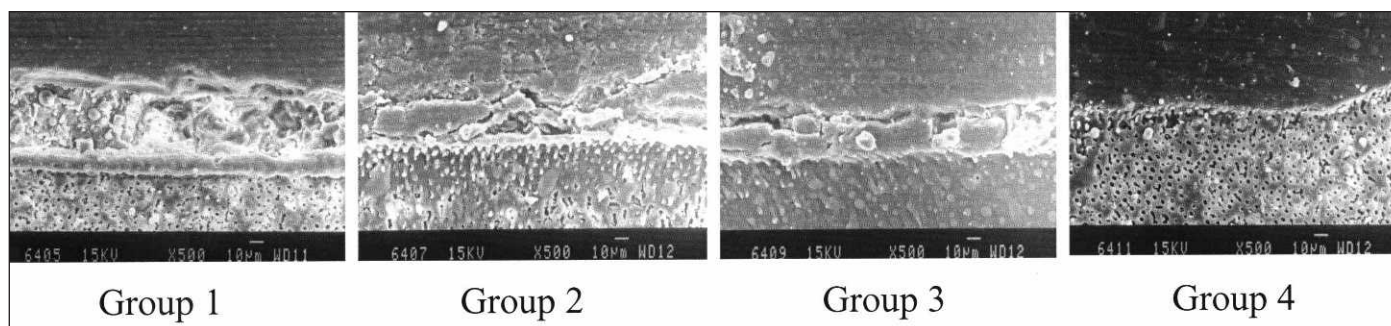


Figure 1. Typical SEM photographs that show the interfacial gaps in the four experimental groups of the high modulus composite (Z-100) at x500 magnification. Using these SEM images and image analysis software, the accuracy of the measurement was determined to be 0.2  $\mu\text{m}$  per pixel.

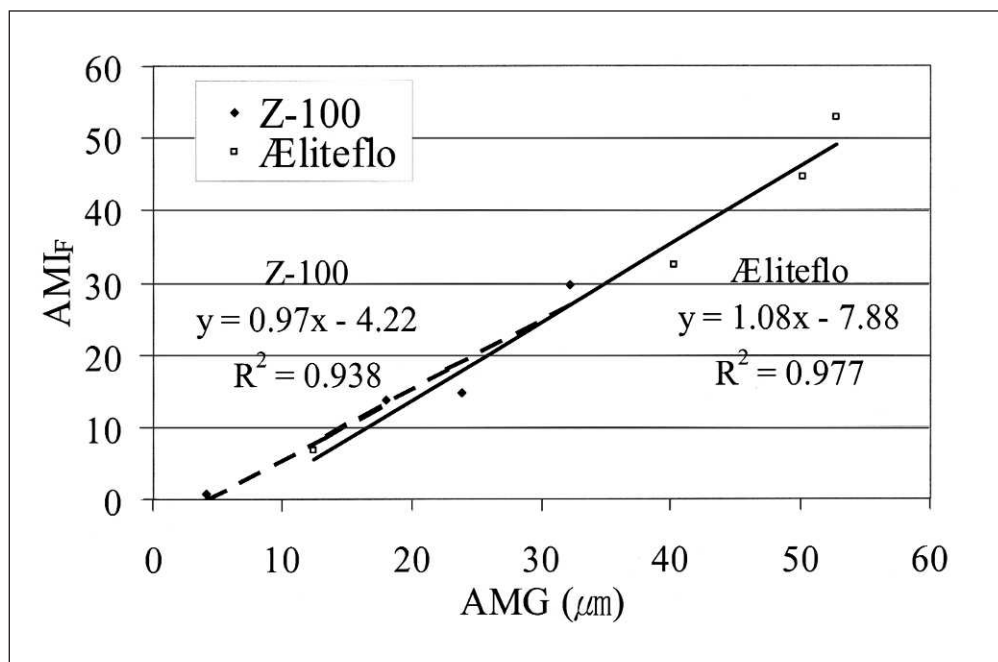


Figure 2. Positive Pearson Product Moment Correlation between the AMI at the floor of the cavities ( $\text{AMI}_F$ ) and the AMG of all groups indicating that the  $\text{AMI}_F$  can represent the gap dimension of the cavity floor.

cial gaps in each group. The correlation between the AMG and the AMI was statistically significant (Pearson Product Moment Correlation;  $p < 0.05$ ; Figure 2) except at the floor of the HMC-filled cavities of Group 2 ( $r = 0.73$ ,  $p = 0.063$ ).

For the HMC-filled (buccal) and FC-filled (lingual) cavities, the  $\text{AMI}_F$  values were greatest in Group 1 and smallest in Group 4 (Table 2). The  $\text{AMI}_F$  in Group 4 were significantly smaller than in the other groups (One-way ANOVA;  $p < 0.05$ ). For both types of composites, the AMI values for the lateral walls ( $\text{AMI}_L$ ) were significantly less than the  $\text{AMI}_F$  values ( $p < 0.05$ ) except in Group 4 of the HMC-filled cavities, where the difference was not significant ( $t$ -test,  $p = 0.070$ ). However, for both composites, the  $\text{AMI}_L$  values showed no statistical differences among Groups 2, 3 and 4 ( $p > 0.05$ ), while Group 1 had significantly higher AMI and AMG values

( $p < 0.05$ ). As expected for both types of composites, the maximum free surface depression was significantly less in Group 1 than in the other groups (One-way ANOVA,  $p < 0.05$ ).

Comparing HMC and FC, the values of  $\text{AMI}_F$  and the free surface depression in all groups were statistically different, but the  $\text{AMI}_L$  values showed no statistical differences (Table 3). For the HMC in Group 1, the ratio of  $\text{AMI}_F$  to free surface depression reached  $13.6 \pm 7.4$ . However, the ratio in Group 4 changed to a fraction, indicating an inverse relationship caused by a small  $\text{AMI}_F$  and a large free surface depression. In Groups 2 and 3, the ratios were not significantly different from Group 4 ( $p > 0.05$ ). For the FC, the ratios showed similar

trends and values to those of the HMC, but the  $\text{AMI}_F$  values in all groups were significantly greater than the HMC (Table 2). The data also indicate a strong inverse relationship between the  $\text{AMI}_F$  and free surface depression for the HMC, with a higher correlation coefficient than for the FC (Figure 3).

## DISCUSSION

Although it was previously suggested that the ongoing reaction of an auto-cured adhesive directs the shrinkage of an overlying resin composite toward the cavity margins, there was no further research to support the suggestion (Hilton, Schwartz & Ferracane, 1997). To evaluate the effect that adhesive resin may have on the direction of shrinkage flow, the extent of polymerization of the bonding agent at the time of polymerizing the overlaid light-curing resin composite in



Table 2: AMG and AMI Values at the Cavity Walls, Surface Depression at the Free Surface, and Ratio of AMI at the Floor ( $AMI_F$ ) to Surface Depression in the HMC-Filled Buccal (Z-100) and FC-Filled Lingual (Æliteflo) Cavities (Vertical bars indicate that the connected groups are not significantly different,  $p>0.05$ )

	Group	Lateral Walls		Floor		Free Surface	Ratio of
		AMG ( $\mu\text{m}$ )	AMI	AMG ( $\mu\text{m}$ )	AMI	Depression Max Depth ( $\mu\text{m}$ )	$AMI_F$ /Free Surface Depression
Buccal cavities (Z-100, HMC)	1	11.7 $\pm$ 4.6	6.0 $\pm$ 3.1	32.2 $\pm$ 15.2	29.6 $\pm$ 17.2	2.1 $\pm$ 0.2	13.6 $\pm$ 7.4
	2	2.5 $\pm$ 1.3	0.5 $\pm$ 0.5	23.9 $\pm$ 5.8	14.8 $\pm$ 5.1	6.2 $\pm$ 1.6	-2.5 $\pm$ 0.9
	3	1.7 $\pm$ 1.6	0.5 $\pm$ 0.5	18.0 $\pm$ 10.9	13.6 $\pm$ 11.7	5.7 $\pm$ 0.8	-2.5 $\pm$ 2.3
	4	0.6 $\pm$ 1.0	0.1 $\pm$ 0.2	4.2 $\pm$ 2.6	0.8 $\pm$ 1.0	7.3 $\pm$ 0.6	-0.1 $\pm$ 0.1
Lingual cavities (Æliteflo, FC)	1	14.5 $\pm$ 3.6	7.5 $\pm$ 3.7	52.7 $\pm$ 12.4	52.7 $\pm$ 12.4	6.3 $\pm$ 1.1	-8.6 $\pm$ 2.8
	2	6.6 $\pm$ 6.4	2.1 $\pm$ 2.7	50.2 $\pm$ 19.1	44.7 $\pm$ 20.8	13.0 $\pm$ 3.1	-3.5 $\pm$ 1.6
	3	3.2 $\pm$ 3.4	0.7 $\pm$ 0.9	40.2 $\pm$ 14.3	32.3 $\pm$ 18.7	10.7 $\pm$ 1.3	-3.1 $\pm$ 2.0
	4	0.6 $\pm$ 0.7	0.1 $\pm$ 0.1	12.4 $\pm$ 11.3	6.6 $\pm$ 7.0	13.0 $\pm$ 3.3	-0.5 $\pm$ 0.4

this study was varied to represent a range of interfacial bond qualities of the bonding agent. The shrinkage direction of the light-curing resin composite was inferred from the interfacial gap and free surface depression measurements. The term “bond quality” was used to qualify the relationship between tooth-to-restoration bond strength and the stresses resulting from polymerization shrinkage (Versluis & others, 1998). Before the advent of fourth generation dentin bonding systems, the interfacial bond strength was lower than the contraction stresses that developed during light curing of an overlying resin composite. This resulted in a misinterpretation of the direction of polymerization shrinkage (Lutz & others, 1986b; Fusayama, 1992). However, the apparent interfacial bond strengths of the current generation dentin bonding systems were reported to exceed the level of polymerization shrinkage stresses and might affect the vector of polymerization shrinkage of the resin composite (Versluis & others, 1998).

During polymerization of a resin composite, the contraction stress before the moment of gelation can be compensated for by flow within the structure (Davidson & de Gee, 1984a). At the gel point and beyond, the

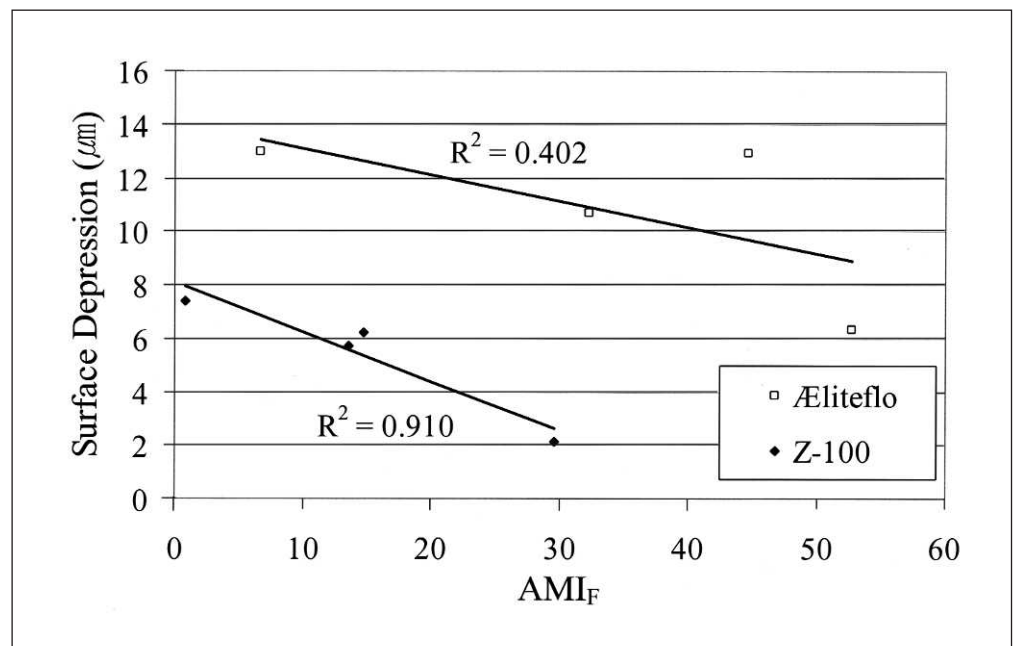


Figure 3. Inverse correlation between the Free Surface Depression and the  $AMI_F$ . The high-modulus resin composite (Z-100) showed less shrinkage and better adaptation of the restoration.

material develops stiffness that is reflected in the modulus of elasticity and flow can no longer compensate for contraction stresses (Sakaguchi & others, 1992). Clinically, the gel point can be considered the moment at which the setting materials start to contract rigidly. Although peak time in DSC exotherm, where the temperature reaches maximum value and begins to fall again, does not precisely coincide with gelation time, clinically, it can be used to estimate the setting time of quick-setting restoratives such as resin composites or dentin bonding agents (Griggs, Shen & Anusavice,



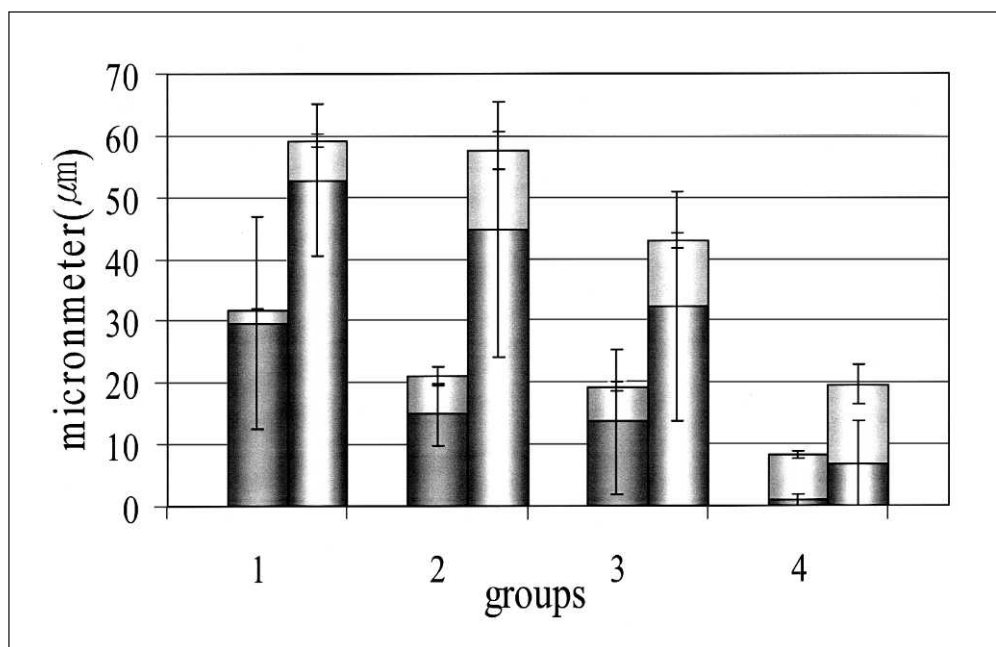


Figure 4. Maximum depression at the free surface (top) and AMI at the cavity floor (bottom) of the high modulus resin composite (Z-100; left column) and the flowable resin composite (Ælitedflo; right column) for the control (Group 1) and the experimental groups (Groups 2, 3 and 4).

Table 3: *P-Values of T-Tests Used to Compare the AMI Values Between HMC-Filled Buccal (Z-100) and FC-Filled Lingual (Ælitedflo) Cavities*

	Lateral	Floor	Depression
Group 1	0.4216	0.0138	<0.0001
Group 2	0.1593	0.0030	<0.0003
Group 3	0.4871	0.0446	<0.0001
Group 4	0.7653	0.0485	<0.0008

1994). Therefore, in this study, the peak cure time was used to differentiate the quality of the interfacial bond of three experimental groups according to the extent of cure of the adhesive at the time of curing the overlying resin composite (Table 1).

For quantitative measurement of the interfacial gap, Hansen & Asmussen (1988) used a single-section technique to measure MI and GP under a light microscope. While this technique gives valuable information about interfacial bond quality, it is inferior to three-dimensional techniques for evaluating marginal leakage. The latter demonstrated significantly greater leakage values than those found in the two-dimensional evaluation of single-sections (Gwinnett & others, 1995; Hilton & others, 1997). However, since three-dimensional techniques could not be used for quantitative measurements of the interfacial adaptation, especially when using SEM, this study attempted to improve assessment of the interfacial adaptation by averaging five measurements along the interface, resulting in param-

eters of an average marginal gap (AMG) and an average marginal index (AMI).

Both the ratios of  $AMI_F$  to free surface depression (Table 2) and the results in Figure 4 demonstrate that in Group 1, compensation for shrinkage flow occurred mostly in the portion of resin composite near the cavity floor, while that in Group 4 occurred near the free surface. From Group 1 to Group 4, as the interfacial bond quality improved, the total amount of linear shrinkage decreased. This was estimated by adding the contribution from the floor ( $AMI_F$ ) and from the free surface (surface depression). The ratio between  $AMI_F$  and surface depression also decreased. In this study, the resin composite near the free surface shrank

toward the interior of the resin composite bulk producing a concave surface. Even in Group 1, which was applied without bonding agent, a small measurable surface depression was observed (Table 2). This observation contrasted that made by Asmussen & Peutzfeldt (1999), who reported shrinkage toward the light source for a specimen thickness of 3 mm. This difference may be explained by the difference in the number of cavity walls contacting the materials. The higher surface depression of the resin composite with maximal attachment to the cavity floor, as in Group 4, demonstrates that the direction of shrinkage not only depends on the specimen thickness and the presence or absence of a flash (Asmussen & Peutzfeldt, 1999), but also on the extent and quality of the attachment of the resin composite that directs the shrinkage of the resin composite toward a center near the interface. Therefore, bond quality must be regarded as a crucial factor in evaluating the direction of the shrinkage. These results indicate that in order to obtain a bond quality high enough to overcome the shrinkage stress of the resin composite, the bonding agent of the current generation dentin bonding system must be polymerized before starting polymerization of the resin composite.

A flowable resin composite has been speculated to induce lower shrinkage stresses than the highly filled Z-100 resin composite because of its lower modulus of elasticity (Bayne & others, 1998). Ælitedflo is a flowable resin composite that has lower filler content than Z-100 and thus, the amount of linear polymerization contraction was consistently nearly twice that of the highly

filled resin composite (Table 2, Figure 4). In particular,  $AMI_F$  in Group 4 of the flowable resin composite was about eight times greater than that of the corresponding high modulus resin composite. This finding suggests that excessive polymerization shrinkage that develops in the light-curing flowable resin composite might be great enough to surpass the well-established interfacial bond and flow characteristics cannot compensate for such high shrinkage and its subsequent stress development.

Most of the FE analyses in the dental field assumed the connection between restorative and tooth substrate to be a perfect bond. However, as FEA is a mathematical simulation, its results can be greatly affected by such an assumption. In fact, in this experiment, the ratios of  $AMI_F$  to free surface depression in Group 1 were 13.6 and 8.6 in the HMC-filled buccal and FC-filled lingual cavities, respectively, but in Group 4, the ratios were 0.1 and 0.5. From these results, the center of the polymerization shrinkage flow in Group 1 can be suggested to occur eccentrically near the free surface and that of Group 4 near the bottom of the resin composite. This means that although the center of shrinkage flow moved with improvement of the interfacial bond quality, contraction stresses detrimental to the interfacial bond must have still occurred at the floor of the cavity, even in Group 4. This finding also contrasts other studies in which the vectors, that is, the magnitude and direction of polymerization shrinkage, pointed toward the bonded margins and were predominantly controlled by cavity shape and boundary condition used to designate the assumed restorative-tooth bond (Winkler, Katona & Paydar, 1996; Versluis & others, 1998). Therefore, this limitation in FE analysis must be considered when evaluating the results for the prediction of the shrinkage vector.

## CONCLUSIONS

In summary, if a good interfacial adaptation of resin composite and tooth substrate can be established, polymerization shrinkage will be directed towards a center located near the well-bonded cavity floor. However, even in a well-bonded group, detrimental contraction stresses still develop at the resin-dentin interface at the floor. Therefore, meticulous use of a dentin bonding system that aims at the highest possible cure of the bonding resin with well-established physical properties before curing the restorative is required to achieve a restoration with the best interfacial adaptation.

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## Disclaimer

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

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# Influence of Acidulated Phosphate Fluoride Agent and Effectiveness of Subsequent Polishing on Composite Material Surfaces

K Soeno • H Matsumura  
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## Clinical Relevance

Restoration with microfilled composites or polishing in conjunction with composite materials containing macro fillers and submicron fillers can be beneficial if patients are set to receive oral care including APF application.

## SUMMARY

This study examined the surface morphological changes of prosthodontic composite materials when exposed to an acidulated phosphate fluoride (APF) agent and the effectiveness of subsequent polishing on APF-treated material surfaces. Nine composite materials (Conquest Sculpture, Dentacolor Sirius, Diamond Crown, Estenia, Eye Sight, Gradia, New Meta Color Infis, Prywood Color and Vita Zeta) were assessed. After half the surface of each composite disk specimen was coated with a varnish, the entire surface was treated with an APF solution (Floden A). The varnish was removed and a surface analyzer helped to determine changes in the surface roughness value (Ra). A scanning elec-

tron microscope (SEM) was used to observe relief patterns for all specimens. The composite surfaces treated with the APF were then polished and examined using the SEM. Ra values for the Conquest Sculpture, Diamond Crown, Estenia, Gradia and Prywood Color materials (macro inorganic filler) showed a significant increase as a result of treatment with the APF solution, and the macro-inorganic fillers (Estenia, Prywood Color) demonstrated noticeable etched patterns. There were no significant statistical differences in Ra values between the treated and untreated halves for any of the microfilled materials. In addition, photographs of the microfilled materials indicated no changes in the surfaces and showed the effectiveness of polishing on the material surfaces. Microfilled material surfaces were not sensitive to the APF agent compared with macro-inorganic filled material surfaces. For the latter, the effectiveness of surface polishing was demonstrated.

## INTRODUCTION

The application of fluoride solution to tooth surfaces is reported to be effective in preventing dental caries. However, some fluoride solutions are also known to dissolve inorganic components in dental materials. Acidulated phosphate fluoride (APF) solution is fre-

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quently used for caries prevention, as it provides maximal fluoride uptake to enamel while causing minimal demineralization (Brudevold & others, 1963; Petersson, 1975). The APF solution that is used for the surface treatment of ceramics prior to resin bonding (Lacy & others, 1988; Kato, Matsumura & Atsuta, 2000) contains sodium fluoride and phosphoric acid. HF dissolves glass, and Gau & Krause (1973) reported the etching effect of an APF agent applied to a ceramic material with a glass constituent. A number of studies related to the influence of fluoride solution on the surface characteristics of dental materials containing inorganic components have been conducted. These include dental porcelains (Copp & others, 1984; Sposetti, Shen & Levin, 1986; Wunderlich & Yaman, 1986; Demirhanoglu & Sahin, 1992; Kula & Kula, 1995), resin composite (Yaffe & Zalkind, 1981; Kula, Nelson & Thompson, 1983; Kula & others, 1986; El-Badrawy, McComb & Wood, 1993; Kula, Webb & Kula, 1996; Papagiannoulis, Tzoutzas & Eliades, 1997; Kula, McKinney & Kula, 1997) and glass ionomer cements (Neuman & García-Godoy, 1992; García-Godoy & Leon de Perez, 1993; Triana & others, 1994; Diaz-Arnold, Wistrom & Swift, 1995; El-Badrawy & McComb, 1998; Çehreli, Yazici & García-Godoy, 2000).

Soeno & others (2000) reported the influence of an APF solution on the surface characteristics of restorative composite materials; the inorganic component of fillers in a restorative resin was dissolved after applying the APF solution. It was accordingly revealed that the surface roughness of macro-filled materials was significantly increased, such that the influence of APF agents on material surface degradation can be minimized with the use of microfilled and submicron composite materials.

Anterior teeth require either a ceramic or veneering composite material for esthetic restoration. Veneering composite material basically consists of dimethacrylate

monomers and inorganic fillers. However, little attention has been given to the influence of APF on prosthodontic composite materials containing inorganic components. Dissolution of inorganic filler and increased surface roughness also leads to staining and accumulation of plaque on the veneering composite surfaces. This study evaluated the influence of an APF agent on the surface characteristics of veneering composite materials. Subsidiary aims included establishing a system for preventing the influence of APF solution by means of polishing techniques, as well as investigation of methods for protecting the surface smooth of the materials and selecting prosthetic dental materials.

## METHODS AND MATERIALS

The prosthodontic composite materials examined in this study were Conquest Sculpture (Jeneric/Pentron, Inc, Wallingford, PA 06492, USA), Dentacolor Sirius (Heraeus Kulzer GmbH, Dormagen, 1242, Germany), Diamond Crown (DRM Laboratories, Inc, Branford, CT 06405, USA.), Estenia (Kuraray Co Ltd, Osaka, 530-8611, Japan), Eye Sight (Kanebo Ltd, Tokyo, 131-0031, Japan), Gradia (GC, Tokyo, 174-8585, Japan), New Meta Color Infis (Sun Medical Co Ltd, Moriyama, 524-0044, Japan), Prywood Color (Yamahachi Dental Mfg Co, Gamagori, 443-0105, Japan), and Vita Zeta (VITA Zahnfabrik GmbH, Bad Säckingen, 1338, Germany) materials. Table 1 summarizes the brand name, manufacturer, lot number and filler information obtained from the manufacturer for each material. The veneering particulate filler composites used in this study were classified into three groups according to the size of the inorganic filler as indicated in Table 1: Group 1, consisting of the Estenia and Prywood Color materials containing inorganic macro fillers (2-5 µm); Group 2, consisting of the Conquest Sculpture, Diamond Crown,

Table 1: *Materials Used*

Group	Trade Name	Lot #	Filler Content (wt%)	Filler Information
1	Estenia	00210B	92	Splintered glass (average 2 µm) Splintered glass (0.02 µm)
	Prywood Color	02017	73.3	Splintered glass (average 5 µm) Prepolymerized silica composite
2	Conquest Sculpture	25673	78.5	Splintered barium glass (0.7 µm)
	Diamond Crown	229901-1	*	*
	Eye Sight	21K03	82	Spherical glass (0.1–1.5 µm)
	Gradia	000201A	75	Prepolymerized silica composite (less than 0.05 µm) Splintered glass (average 1.0 µm)
3	Dentacolor Sirius	066021	74	Prepolymerized silica composite
	New Meta Color Infis	VG1	70	Prepolymerized silica composite (TMPT filler)
	Vita Zeta	5335	*	*

\*SEM analysis led to the classification of the Diamond Crown material into Group 2 and the Vita Zeta material into Group 3, although there are no specific claims in this regard by the manufacturers.

Eye Sight and Gradia materials containing inorganic fillers not greater than approximately 1.5  $\mu\text{m}$  (that is, submicron composite) and Group 3, consisting of the Dentacolor Sirius, New Meta Color Infis and Vita Zeta materials composed of prepolymerized silica composites that are categorized as microfilled composite and contain the smallest inorganic-filler (approximately 0.04  $\mu\text{m}$ ) of the nine materials. SEM analysis led to classification of the Diamond Crown material into Group 2 and the Vita Zeta material into Group 3 although there are no specific claims made by the manufacturers in this regard. Floden A (Sunstar, Osaka, 569-1195, Japan) was used as the APF agent, containing 2 g NaF and 1.73 g phosphoric acid per 100 mL of solution (pH 3.2-3.8). Protect Varnish (Kuraray Co Ltd, Osaka, 530-8611, Japan) was used as the varnish that was designed for preventing tooth surfaces surrounding prepared cavities from being decalcified by etching agents during resin filling (Hachiya & others, 1985).

Each of the nine veneering materials in Table 1 was packed into a metal mold 10 mm in diameter and 2.2 mm in thickness, and specimens were produced by photo-polymerizing the resin using a light curing unit (Dentacolor XS, Kulzer, Dormagen, 1242, Germany) for 270 seconds. Then, after storing the resin in water at 37°C in an incubator for 24 hours, the surfaces of the specimens were polished with a water-resistant abrasive paper series to #1500 under a water stream while pouring water, and buffing was performed with wet felt and 0.3  $\mu\text{m}$  alumina slurry.

Varnish was first applied to half the surface of each specimen. The APF solution was applied to the entire surface of each specimen for four minutes, then washed with water and dried. After this process was repeated eight times for each specimen, the varnish film on the untreated side was carefully peeled off in a single sheet using a pair of tweezers and the disks were dried in a desiccator. The mean surface roughness values (Ra) of the APF-treated and untreated (that is, previously varnish-coated) sides were measured using a profilometer (Surfcorder SE-30D, Kosaka, Tokyo, 101-0021, Japan) equipped with a detector having a diamond needle (R 2  $\mu\text{m}$ ). The length of the profile measurements was 0.8 mm and the cut-off of the profile was  $\lambda_c$  0.8 mm. For both properties, five measurements were performed in the measurement area of each specimen at intervals of 0.5 mm and averaged. The average surface roughness value (Ra) of five specimens for each condition was calculated. Regarding surface characteristics, the erosive conditions of inorganic components in each specimen were observed by means of scanning electron microscopy (SEM) (S-3500N, Hitachi, Tokyo, 100-8220, Japan).

For the Estenia and Prywood Color materials, the APF-roughened surfaces were polished using a rotary polishing instrument (Compomaster, Shofu, Kyoto,

605-0983 Japan), surface smooth recovery conditions were compared using SEM, and the results were evaluated.

## RESULTS

Table 2 shows the Ra values for the APF-treated and untreated surfaces, standard deviations and statistical analysis results. Two-factor ANOVA revealed that surface roughness (Ra) was affected by the type of prosthetic veneering composite materials ( $p < 0.0001$ ), by application of the APF ( $p < 0.0001$ ) and their interaction ( $p < 0.0001$ ). The results were therefore compared by one-way ANOVA and a *post-hoc* Duncan new multiple range test. Ra values for the Estenia and Prywood Color materials with inorganic macro-fillers showed a significant increase as a result of treatment with the APF solution ( $p < 0.05$ ). Three of four submicron composite materials (Conquest Sculpture, Diamond Crown, Gradia) with inorganic fillers (approximately 0.1-1.5  $\mu\text{m}$ ) exhibited significant differences in terms of the Ra value between the APF-treated and untreated halves ( $p < 0.05$ ). There were no significant statistical differences in the Ra values between the treated and untreated halves for any pre-polymerized silica composites categorized as microfilled ( $p = 0.05$ ).

Figure 1 shows representative surfaces for materials in each group. SEM observation confirmed that the macro-fillers contained in the Estenia and Prywood Color materials in Group 1 were strongly attacked. The erosion of inorganic fillers in the Group 2 materials was also observed. Dissolution of the fillers in the micro-filled type materials in Group 3 was not observed in the SEM images at this magnification. Figure 2 shows specimen surfaces for the Estenia and Prywood Color materials etched with the APF agent, with the surfaces on the left polished using the rotary polishing instrument. By comparing the left half of the surface to right half, it was confirmed that the surface smoothness on the left was substantially recovered.

## DISCUSSION

Application of an APF solution is effective in preventing dental caries and plays an important role in the oral health care of children and adults. However, APF solution contains sodium fluoride and phosphoric acid. The typical dissociation reaction in the solution is:



$\text{H}^+$  and  $\text{F}^-$  ions are therefore present in the solution and HF dissolves glass. The results shown in Table 2 suggest that applying the APF solution increases the surface roughness of some veneering composite materials. It was also suggested that the type of filler influ-



ences the surface characteristics of materials. SEM observation made it clear that inorganic macro fillers or submicron fillers were strongly attacked by the APF agent, and this was considered to be a cause of an increase in the Ra values for Groups 1 and 2 materials. As the micrographs indicate, material surfaces roughened by acid attack could be recovered to a smoother state by means of an appropriate rotary polishing device such as the Compomaster instrument, although surface characteristics prior to applying the APF solution could not be completely recovered.

It was revealed that the influence was smallest in microfilled-type materials. There have been reports that the siloxane bonds in the fillers and the matrices in materials containing macro-fillers or sub-micron-fillers were influenced by hydrolysis or acids over the course of long-term observation periods (Miettinen, Narva & Vallittu, 1999; Vallittu, 1998; Söderh  lm & others, 1984; S  derh  lm, Mukherjee & Longmate, 1996). Greater APF-induced influence can therefore be presumed, and the use of microfilled type materials that sustain less damage is highly desirable.

Clinically, if the inorganic filler in materials is dissolved and the surface roughness of the materials increases, plaque will accumulate on the surface. Gingivitis and periodontitis are likely in such cases. Stains also occur on the veneering composite surfaces, and it should be noted that maintenance of veneering composite is necessary in the context of long-term esthetics. Particularly with anterior veneering composite materials, it is

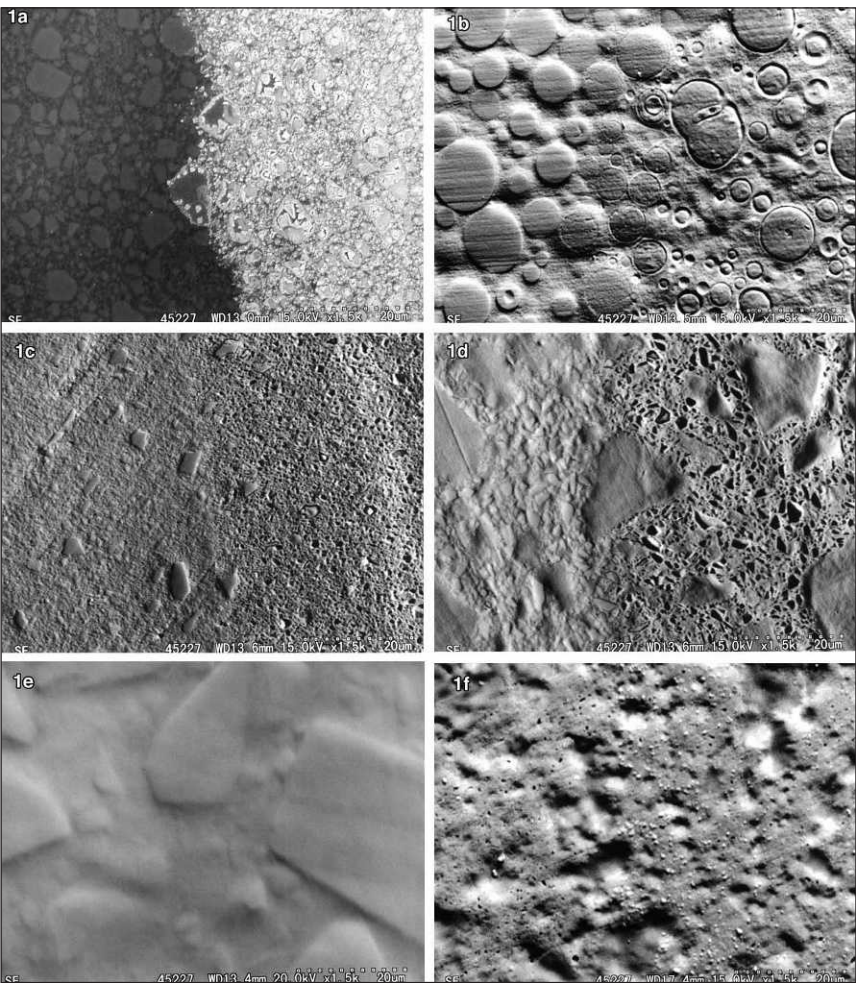


Figure 1. Microphotographs of representative surfaces of materials in each group (Table 1) observed with a scanning electron microscope (1500x). The right sides are APF-treated; the left sides are varnished and untreated.  
(a) The Estenia material; Group 1  
(b) The Prywood Color material; Group 1  
(c) The Diamond Crown material; Group 2  
(d) The Gradia material; Group 2  
(e) The New Meta Color Infis material; Group 3  
(f) The Vita Zeta material; Group 3

Table 2: Average Surface Roughness Values (Ra) and Statistical Analysis Results						
APF Treatment Material	None Ra (��m)	SD	Statistical Category*	Floden A Ra (��m)	SD	Statistical Category*
Conquest Sculpture	0.063	0.005	a	0.121	0.010	d
Dentacolor Sirius	0.152	0.005	e f g	0.158	0.007	f g
Diamond Crown	0.142	0.010	e	0.190	0.012	h
Estenia	0.083	0.006	b	0.141	0.003	e
Eye Sight	0.113	0.010	c d	0.118	0.011	d
Gradia	0.105	0.011	c	0.121	0.006	d
New Meta Color Infis	0.151	0.008	e f g	0.163	0.004	g
Prywood Color	0.120	0.017	d	0.141	0.016	e
Vita Zeta	0.147	0.004	e f	0.158	0.005	f g

\*Identical letters indicate that the values are not statistically different at  $p<0.05$ .

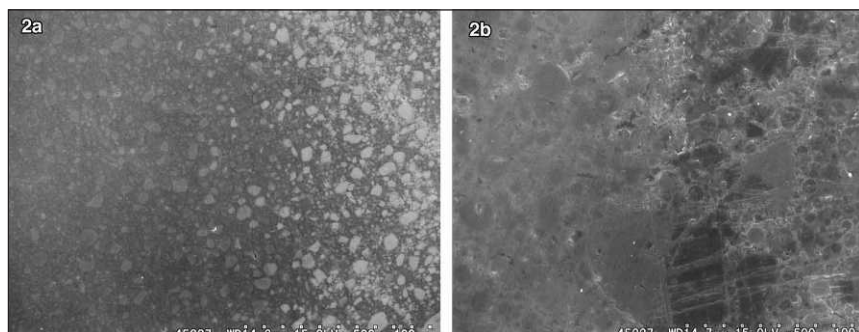


Figure 2. Microphotographs (500x) of polishing on surfaces roughened by APF treatment. The right sides are APF-treated; the left sides are polished using a rotary polishing instrument.

(a) The Estenia material

(b) The Prywood Color material

important to protect the material surface smoothness by selecting microfilled-type composite and the use of neutral fluoride agents (Kula & others, 1996; Kula & others, 1997) or varnish is recommended to prevent acid attack on the macro fillers and submicron fillers. In addition, the effectiveness of polishing on roughened material surfaces also deserves careful attention. Furthermore, to re-confirm the clinical importance of polishing, clinical trials are underway to further evaluate and confirm that polishing affects the recovery of the surface smoothness of materials eroded by the application of APF agent.

### CONCLUSIONS

1. Materials with inorganic macro-fillers (Group 1) and all submicron composite materials (Group 2), except for Eye Sight material, showed significant statistical differences in Ra values between APF treatment and non-treatment. However, the Ra values of microfilled composite (Group 3) were not affected by applying the APF agent.
2. SEM observation clearly showed that the materials containing macro-fillers (Group 1) were strongly attacked. The erosion of inorganic fillers in the Group 2 materials was also observed. There were no APF-induced changes in surfaces of the microfilled type materials (Group 3).
3. While macro inorganic fillers or submicron fillers in the composites demonstrated noticeable etched patterns generated by the APF solution, material surfaces roughened by acid attack could be recovered to a smoother state with a rotary polishing instrument.

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# Academy of Operative Dentistry Award of Excellence

Dr John W Reinhardt



It is a great honor and pleasure that I have been asked to present the Award of Excellence to my friend and mentor, Dr John W Reinhardt. I had the good fortune to be John's first graduate student in 1982. Like the number #001 on John's American Board of Operative Dentistry Certificate, I also hold the special honor of being #001 among his group of graduate students. Together with his wife Claudia and his mother-in-law Adrienne, all six of us save one are here today to congratulate John in receiving this prestigious award.

It is difficult to summarize John's illustrious academic career in such a short time. Chicago can certainly claim John as one of its own. John holds a BA in Biology from Illinois Wesleyan University and a DDS from Loyola University. He began his dental career in the US Army Dental Corps at Fort Carlson, CO, and then joined the University Of Iowa College of Dentistry in 1977, receiving an MS in Operative Dentistry in 1979. John took a leave of absence from 1986-1988 to pursue a MPH degree from Harvard University, sponsored by the Robert Wood Johnson Dental Service Research Scholars Fellowship. He returned to Iowa in 1988 to become the Department Head of Operative Dentistry, progressing to the rank of Professor in 1990 and as Acting Assistant Dean of Patient Services in 1997. He currently serves the University of Nebraska College of Dentistry as Dean.

John has been a member of this Academy since 1978 and has been active in all aspects of its activities. He has served on numerous committees of the Academy, including serving as President in 1997. John's memberships in other professional organization include the American Dental Education Association, Conference of Operative Dentistry Educators, OKU honor fraternity and both AADR and IADR. Since 1991, He has served as consultant to the American Dental Association, National Institute of Health and US Navy Postgraduate Dental School. John also serves on numerous editorial boards and grant review panels. He has published widely and is especially known for his early work on amalgam and mercury hygiene.

As an educator, John has taught both at the undergraduate and graduate levels. Those of you who know John, I am sure are all familiar with his warm smiles and genuine friendliness. To show you how well he is loved and respected by those who worked closely with him, I have gathered the following comments from John's graduate students:



John W Reinhardt

"I owe John a gallon of red ink for his relentless revision of my thesis. He was more nervous than I was during my thesis defense. In the end, it was through his wisdom and gentle guidance that I was able to go through the process."

"I was covering clinic as a graduate student and one of the sophomore students had a fractured restoration near closing time. John happened to walk by and offered to help and redid the restoration with my assisting. His compassion and willingness to help touched the patient, the student and myself. That's typical of Dr Reinhardt."

"With Dr Reinhardt, what you see is what you get. A truly honest and sincere individual. As my thesis advisor and mentor at Iowa, he always had time for me in spite of his many responsibilities. As a Dean and friend, I still seek his input and sage advice. Some things don't change...that's is good. He truly deserves to be honored with the Award of Excellence."

"John taught our gold foil seminars and laboratories. We ran a little late in lab one day and didn't have much time before our next class. Even though I'm sure he had to be somewhere else, John stayed, not to supervise, but to help us clean up the lab and put away our instruments so WE wouldn't be late. How many department chairman would do that?"

From his #001 graduate student:

“John and Claudia, I thank you for welcoming a total stranger into your family. You couldn’t imagine what that meant to a foreign student in an unfamiliar environment. Thank you for the Cornish hen barbecue at your home. Thank you for the tailgate party from a guy who can’t tell the difference between a “tailgate” and a “tailpipe.” Thank you for my first Iowa football game with seating at the 50-yard line. Thank you for the exciting basketball games on cold winter nights. Above

all, thank you for teaching us beyond the mechanics of operative dentistry. You are indeed a gentleman and scholar and an excellent role model. It is no coincidence that all of your former graduate students share this moment with you. We all want to be just like you.”

Dear colleagues of the Academy, families and friends, I present to you this year’s recipient of the Award of Excellence, Dr John W Reinhardt.

Dr Daniel Chan

# Academy of Operative Dentistry Hollenback Memorial Prize

Dr Nairn H F Wilson



George Hollenback



Nairn H F Wilson

**I**t is a great pleasure to introduce the recipient of the 2002 Hollenback Memorial Prize: Professor Nairn HF Wilson from London, UK. This is a prize given annually for research that has contributed substantially to the advancement of restorative dentistry. Dr Wilson is a dedicated restorative and operative dentist, a world-renowned researcher, a great administrator and a

hard working professional (to which his 120-page CV will attest). He is also a good friend, which makes this presentation a particularly pleasant task.

Dr Wilson's principle research, for which he is awarded the Hollenback Prize, is in the field of tooth-colored restorative materials. He is best known for his continuing involvement in clinical testing of dental restorative materials, having been principle investigator for various international multi-center studies and a contributor to expertise in clinical trial methodology. During the last few years he has also become involved in practice-based research, which I consider to be an upcoming and important part of clinically relevant studies in the field of operative dentistry. Dr Wilson is truly an international figure in the field of Restorative Dentistry, with a firm focus on Operative Dentistry and its application in enhancing quality dental practice.

Dr Wilson is a graduate of the University of Edinburgh. He served as a Lecturer there prior to moving to the University Dental Hospital of Manchester, England, where he was awarded the title of Professor of Restorative Dentistry in 1986. He served as Dean and Clinical Director from 1992-1995; Dean of the Faculty of Dental Surgery, Royal College of Surgeons of Edinburgh until 1998 and Pro-Vice Chancellor of the University of Manchester from 1997-1999. He has been President of the General Dental Council of the UK

Statutory Regulatory Body for Dentistry since 1999. Currently, he is Head (Dean) of the Guy's, King's and St Thomas' Dental Institute in London. He also holds visiting professorships in a number of dental schools in the US and Canada and serves as President of the European Section of our Academy of Operative Dentistry.

As I mentioned earlier, Dr Wilson is Head (Dean). You may not know what this means, but having a Scottish dental degree myself, I know that Nairn is Dean and Head of the merged Guy's, King's and St Thomas' Dental Institutes in London. The "Head" part of his title stems from the fact that he is the Head of the Dental Hospital, that is, the clinic section of the school, and he is Dean of the School, which is the academic





entity. Together, the School and the Hospital form the Institute. This division into academic and clinical parts is the way the British arrange their dental schools.

However, the most significant position he currently holds is President of the UK General Dental Council. This is a five-year appointment, and it is the most prominent and prestigious administrative position in dentistry in the UK. It is formally a half-time position, but everyone knows that it requires full-time attention.

To help you understand what a busy man Nairn really is, let me tell you a short story. After having spent three days in their house while we worked on a collaborative research project, I thanked Margaret, Nairn's wife, for putting up with me and for the hospitality extended to me. I was pleased to hear her response: "Ivar, please

come back soon," which made me feel really good, but then she added, "You and the work you have been doing with Nairn has kept him at home for three full days. It has been several years since I was able to keep him at home for that long."

Well, that is the price the family pays for individuals who are recipients of awards, such as the Hollenback Memorial Prize.

It is a high honor for the Academy of Operative Dentistry to present the 2002 Hollenback Memorial Prize to Dr Nairn HF Wilson.

Ivar A Mjör, Chair  
AOD Research Committee

## Departments

### Letters



Dear Editor,

Thank you for the outstanding journal. Since you asked for feedback in a recent editorial, I want to respond. I am pleased to see articles on bleaching in the journal.

In Vol 27 (1) January/February 2002, there was an article entitled "The Whitening Effect of Bleaching Agents on Tetracycline-Stained Rat Teeth" by Drs Shin and Summitt. I would like two items clarified.

Figure 2 represents images at baseline. If it is at baseline, and Delta E is the difference between baseline and the next value as indicated on page 70 of the article, that may be Delta L\* value...or am I missing something?

I also have some concern about Phase 2 of the study. For Group HE, human enamel was placed in close proximity to the rat enamel and the area was bleached. The underlying dentin did not bleach. The point was made that enamel inhibits the penetration of the bleach and it would be better to bleach after reducing the enamel for veneers. I believe an incorrect conclusion was postulated.

I have found that peroxide, whether in the form of hydrogen peroxide or carbamide peroxide, readily penetrates through the enamel, into the dentin. I postulate that perhaps the bleach did not penetrate to the deeper parts of the enamel because of voids between the two layers of enamel. I would be interested in learning whether the authors had considered that to be a reason for non-bleaching of the underlying dentin.

I look forward to the reply.

Sincerely,

Bruce A Matis, DDS, MSD  
Professor and Director of Clinical Research Section  
Indiana University School of Dentistry

### RESPONSE

I think Dr Matis' questions were really excellent. I had tried to solve those kinds of things when preparing this study. I would like to respond to his two questions as follows:

In regard to the first question about images at baseline and Delta E vs Delta L\*, although we tried to have uniform illumination, the quality of images might be unstable depending on the conditions of illumination around the microscope. In order to reduce the potential influence of variations in illumination, we thought it

would be better to compare each day's color difference in a set (estimated between stained and non-stained dentin area in the same image) rather than to compare the amount of change of the stained area among images taken serially.

As far as we know, delta E means a color difference between two points (areas) as well as a change in color itself.

In our paper, delta E was determined as follows:

delta E = root {[L\*(stained area)-L\*(non-stained area)]<sup>2</sup>

+ [a\*(stained area)-a\*(non-stained area)]<sup>2</sup>

+ [b\*(stained area)-b\*(non-stained area)]<sup>2</sup>}

As for the concern regarding our conclusion on penetration of the bleaching agent, we agree with Dr Matis' opinion. That was one of the most difficult points in this study, that there might be a space between human enamel and rat enamel that might hinder molecular diffusion.

In an attempt to overcome this problem, we thought about using a luting agent. However, we were concerned that this would also act as a barrier and, therefore, paper tape was chosen. We thought that if a bleaching agent has enough power to penetrate human enamel, the amount pooled in a space might bleach TC discolored dentin. However, the result did not show this pattern, so within the limits of this study, we postulated that the ability of penetration into human enamel was not enough in a case of TC discoloration. Obviously, this was an inappropriate conclusion and we appreciate Dr Matis' correction.

Thanks for the inquiries and the opportunity to respond.

DongHoon Shin, DDS, MS, PhD  
Associate Professor, Department of Conservative Dentistry, Danhook University School of Dentistry

Dear Editor:

I was greatly interested to read the January/February article in *Operative Dentistry* by Miyazaki, Iwasaki & Onose on "Adhesion of Single Application Bonding Systems to Bovine Enamel and Dentin," in which they found that one-step bonding systems are comparable to those of a compomer restorative system. They concluded that the enamel bond strengths of newly developed one-step bonding systems were not significantly different except for Prompt L-Pop (3M ESPE) that showed the highest value.

The authors appear not to be familiar with work carried out on one-step bonding systems in the 1950s at the Amalgamated Dental Company in conjunction with the Eastman Dental Hospital, London. I recorded the history of this work in a recent editorial in a chapter in Roulet and Degrange's book, *Adhesion. The Silent Revolution in Dentistry*, published in *Quintessence* in 2000. The idea of a single-step bonding agent is not new and originated in the patents for *Sevriton Cavity Seal*<sup>1</sup>. This invention by Hagger was revolutionary and almost unrecognized in current literature and was based on glycerophosphoric acid dimethacrylate. Work at the Eastman Dental Hospital<sup>2,3</sup> showed that *Sevriton Cavity Seal* increased adhesion to dentin by penetrating the surface and forming an intermediate layer that was detected because the dentin exhibited an intense affinity for hematoxylin staining with affinities similar to those of calcified dentin in an exaggerated form. Promp L-Pop uses similar chemistry but with improvements such as light curing.

As far as I am aware, the work quoted above was the first to describe bonding to tooth structure using organic acids. Due to the high shrinkage of self-curing acrylic resins, the work of Hagger and Buonocore on acid etching did not come to fruition for many years until the low shrinkage resin composites were developed. However, Hagger also deserves our recognition as one of the great pioneers in enamel and dentin bonding.

Yours truly,

John W McLean

## References

1. Hagger O (1951) British patent 687,299 and Swiss patent 278,946.
2. Kramer IRH & McLean JW (1952) Alterations in the staining reaction of dentine resulting from a constituent of a new self-polymerizing resin *British Dental Journal* (93) 150-153.
3. McLean & Kramer IRH (1952) A clinical and pathological evaluation of a sulphinic acid activated resin for use in restorative dentistry *British Dental Journal* (93) 255-269, 291-293.

## RESPONSE

Dear Prof Cochran:

Thank you for your taking the time to share Dr McLean's letter with me. While I do not believe the letter asks questions regarding the study that require clarification, I completely agree with Dr McLean and do realize that previous studies should be recognized and considered prior to undertaking a research project. I also believe all dental technologies used today are based on older techniques developed by many of our predecessors and that it is important to review all previous works and their impact on current research. I appreciate Dr McLean's time in reinforcing this principle.

Thank you again and best regards.

Masashi Miyazaki, DDS, PhD  
Department of Operative Dentistry  
Nihon University School of Dentistry

## LETTERS CORRECTION

The Editor would like to correct a possible misconception generated by the authors' addresses listed in the article "One-Year Clinical Performance of a Resin-Modified Glass Ionomer and a Resin Composite Restorative Material in Unprepared Class V Restorations" (27-2: March/April 2002). The authors' addresses listed at the beginning of each article are intended to represent their most current mailing address, and are not necessarily an indication that the research was actually conducted at the university affiliation listed in the address. The study presented was conducted at, and under the human subjects guidelines and rules of the University of Puebla in Puebla, Mexico. We would like to clarify that the corresponding author (Dr William W Brackett) was a visiting professor at that institution when the research was done. However, his current mailing address at The University of Nebraska Medical Center, College of Dentistry was listed in our journal. This in no way implies any affiliation or connection between the University of Nebraska Medical Center and the research presented. We apologize for any concerns or inconvenience generated by our method of listing authors' addresses.



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