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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 also are published.

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# GUEST EDITORIAL

# The Death of a Professional School: Does Anyone Really Care?

Northwestern University joins other universities in the United States in closing schools of dentistry. The 106-year-old school will close its doors in 2001. It was decided that dentistry was no longer worthy of being part of higher education, and that the "university must concentrate on research in the life sciences." Does this mean that the mouth is no longer to be considered a part of the body?

It seems to be a trend in the United States, and in much of the world, to fail to recognize dentistry as part of medicine. Dentistry struggled to gain recognition and demonstrate that it is more than a trade practiced on a street corner or in a barber shop. Now we find the slogan "It costs too much to educate a dentist." This is the driving force to close one school after another. No matter how much the dentition affects the overall health of the body, the dental practitioner fails to be respected in the same way as other medical practitioners.

In the USA when dentistry finally achieved a position in higher education, it was established as a separate profession and not a part of medicine. Instead of moving into the medical school, it became a distinct profession. It even split into specialties, trying to mimic the medical model. This failure to insist on being part of the medical education model is now coming to haunt the profession. Dentistry—stomatology—should be a part of a medical education. Professionals who are highly educated in the diseases affecting the oral cavity and the surrounding structures and in their treatment are the future of dentistry. We need to remember that teeth are living tissues, and we cut into tissues the same as any medical practitioner does. Maybe tooth decay and some periodontal disease can be reduced to some degree, but I doubt if the world will ever be totally free from all the diseases that affect the mouth and the surrounding structures.

To dentists throughout the world: It's time to realize what is happening to our profession. If you have not had a concern, let this most-recent death of a dental school stimulate you to look at dental education in your part of the world. If you do not care, then maybe dentists are just tooth mechanics. When you graduate from dental school, you are supposed to be able to perform all the procedures that those who become "specialists" perform. All of dentistry needs to be concerned, because without the dental schools, will there be a need for dental specialties?

The definition of "operative" is: procedures involving surgical operations. Operative Dentistry is the mother of the profession.

WILLIAM E HAWKINS
President
Academy of Operative Dentistry

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# LITERATURE REVIEW

# Fluoride-releasing Dental Restorative Materials

F C EICHMILLER • W A MARJENHOFF


## SUMMARY

**In the 1940s, dentists observed that secondary caries was rarely associated with silicate cement restorations. While the relatively stable dimensional properties of those restorations were undoubtedly a factor in this fortunate circumstance, the fact that fluoride was inherent in the composition of the material received the greater credit. By the mid-1980s, a wide variety of fluoride-releasing dental restorative materials were available to dentists and dental consumers, and the cariostatic effect of fluoride ions on enamel caries had been demonstrated in many studies. This paper reviews much of the fluoride-related research conducted on fluoride-releasing amalgam, glass-ionomer cements, composites, primers, sealants, liners, acrylic resins, and orthodontic bracket bonding materials. The need for standardization of test methods is addressed, as is the need for more controlled clinical trials and additional research.**



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## INTRODUCTION

In the 1940s, dentists observed that secondary caries was rarely associated with silicate cement restorations. While the relatively stable dimensional properties of those restorations was undoubtedly a factor, the fact that fluoride was inherent in the composition of the material received the greater credit. The observation, accordingly, heralded the development and increasing use of delayed-reaction and controlled-release therapeutic materials in dentistry (Colton & Ehrlich, 1954).

By the mid-1970s, some fluoride-releasing amalgams and luting cements were commercially available in Europe (Forsten,1976). While the initial release of fluoride from amalgam was found to be significant, the material was found to release only minor amounts after 1 week (Forsten, 1976). An early finding regarding fluoride-releasing luting cements also indicated that significant amounts of fluoride were released initially, and the release continued over a 5-week observation period to an extent equal to that of silicates in earlier studies (Forsten, 1976).

The "burst effect" associated with this high initial release of fluoride from nearly all fluoride-releasing dental restorative materials continues to be a concern regarding their long-term efficacy. There is mounting evidence that frequent applications or constant release of relatively low concentrations of fluoride are most efficacious for caries reduction, even where caries challenges are high. But sustained fluoride release from most dental restorative materials is

relatively short-lived compared to that from experimental copolymer membranes and glass controlled-release devices (Toumba & Curzon, 1993). It would be much more efficient and convenient, however, to deliver fluoride from the dental restorative material rather than from these additional devices that must be attached to teeth.

The use of dental restorative materials for preventive purposes has, accordingly, received increasing emphasis with time. The concept of combining the strength, rigidity, and fluoride-release properties of silicate cements with the biocompatibility and adhesive qualities of polyacrylic cements led to the introduction of glass-ionomer cements to the dental profession in the early 1970s (Wilson & Kent, 1972; Wilson & McLean, 1988).

By the mid-1980s, a wide variety of fluoride-releasing dental restorative materials were available to dentists and dental consumers, and the cariostatic effect of fluoride ions on enamel caries had been demonstrated in many studies. Some acrylic resin dental appliances (Zitz, Gedalia & Grajower, 1981) and acrylic restorative products (Rawls, 1987) were developed that were fluoride-releasing. As early as the 1970s, some composite resins incorporated fluoride and were shown to release fluoride (Forsten & Paunio, 1972). The effect of fluoride-releasing restorative materials on dentin also began to receive attention. Secondary root caries around fluoride-releasing composite, amalgam, glass ionomer, and silicate cement, as well as zinc oxide-eugenol cement, was studied by Dérand and Johansson in 1984. Their results on in vitro root caries showed inhibition by all of the fluoride-releasing materials when compared to materials that did not contain fluoride. Indeed, researchers have found deeper penetration depths of fluoride in dentin than in enamel on cavity walls adjacent to a variety of fluoride-containing restorative materials (Skartveit & others, 1990).

So convincing was the evidence that fluoride-releasing restorative materials and devices prevent caries—enamel caries, in particular—that scientists were encouraged in the 1980s to investigate the efficacy of releasing other bioactive agents from restorative materials. Some, for example, evaluated the therapeutic applicabilities of fluoride-releasing dental materials, as well as those of antimicrobial agents like antibiotics, chlorhexidine, eugenol, o-ethoxybenzoic acid, nystatin, and anti-inflammatory agents like benzydamine HCl and steroids (Masuhara, Kadoma & Fujisawa, 1985). Delayed-action preparations for the treatment of oral disease were also reviewed by Mirth (1987) and fluoride-releasing restorative materials, in particular, by others (Swift, 1988a; Dionysopoulos & others, 1988).

Forsten (1990) exposed a fluoride-containing amalgam, composite, pit and fissure sealant, and seven glass-ionomer filling materials to running water for 2 years, measuring long-term fluoride release periodically. Fluoride release from the glass ionomers reached a constant level of 0.5 µg/mL (ppm) to 1.0 µg/mL, an amount that was clearly greater than that from the amalgam and composite where release did not exceed 0.3 µg/mL, and release was found in all cases to increase by lowering the pH of the storage solution.

A useful review for researchers seeking to develop controlled-release delivery systems for treating dental diseases, including the incorporation of fluoride into sustained-release polymeric matrices, was written by Friedman and Steinberg in 1990. Other useful guides for the selection and use of fluoride-releasing restorative materials were written by Burgess (1995) and Burgess and others (1996).

## AMALGAM

Although not available in the United States, fluoride-containing amalgams have been shown in vitro to have anticaries properties sufficient to inhibit the development of caries in cavity walls (Tveit & Hals, 1980; Tveit & Totdal, 1981) and recurrent caries at enamel and dentin restoration margins (Skartveit, Wefel & Ekstrand, 1991; Donly, 1994). One study found salivary fluoride concentrations at more than 20 times baseline concentrations for the first few days after placement of the restorations (Skartveit, Tveit & Ekstrand, 1985). The release declined exponentially to baseline levels after 30 days. The authors postulated that since the salivary fluoride concentrations registered in their study seemed to be sufficient to enhance remineralization, fluoride-releasing amalgam restorations may have a favorable effect on any initial demineralization in the mouth.

Bercy and Vreven (1980) found that stannous fluoride liberated from alloys containing mass fractions of 0.5 % or 1% of the compound was incorporated into the enamel of cavity walls in contact with the sample. No significant difference in fluoride enrichment appeared between alloys fluoridated at the higher and lower percentages; the amount of fluoride bound by fluorapatite was significant. Tveit and Lindh (1980) found that the greatest concentrations of fluoride in enamel surfaces exposed to fluoride-containing amalgam, about 4000 µg/mL, were found in the outer 0.05 µm of the tissue. In dentin, the greatest concentrations, about 9000 µg/mL, were found at a depth of 11.5 µm.

The amount of fluoride release relative to the initial starting content of fluoride in test specimens of fluoride-containing amalgam has been shown to be greater over 7 weeks than that released from glass-ionomer cement samples (Tveit & Gjerdet, 1981). The total amount of fluoride released into solution,

however, was highest for silicate cement samples followed by the glass ionomer and was lowest for the amalgam. This difference was due to the very large starting content of fluoride available from both silicate cement and glass-ionomer samples when compared to the amalgam. Forsten (1990) also found the total fluoride release from glass ionomers to be greater than from fluoride-containing amalgams. These results were supported by studies on restorations in extracted teeth where glass ionomer gave the better protection against demineralization than fluoridated or unfluoridated amalgam (Valenzuela & others, 1994).

## GLASS-IONOMER CEMENT

Glass-ionomer cements, the modern version of silicates, are perhaps the best known fluoride-releasing restorative material and, like silicates, have been shown to have anticariogenic properties due to their significant release of fluoride (Muzynski & others, 1988; Benelli & others, 1993); the uptake of fluoride (and aluminum) in cavity walls (Wesenberg & Hals, 1980a), enamel and plaque (Benelli & others, 1993); and the enhanced reprecipitation of calcium and phosphate promoted by the fluoride release (Wesenberg & Hals, 1980b).

Forsten (1977) found glass ionomers to release slightly more fluoride than silicate cement, while Tveit and Gjerdet (1981) found that the fluoride release from silicate cements was about five times greater than from glass ionomers. Whatever the case, glass ionomers are certainly more versatile than silicates, having found use not only as an esthetic filling material, but as a luting agent for dental prostheses (Muzynski & others, 1988), as an orthodontic bracket bonding material, and as a cavity liner. There is no need to place a protective liner under glass-ionomer restorations, and the presence of fluoride in the material helps inhibit plaque for-mation (Mount, 1995). Glass-ionomer cermets (sintered silver particles to glass-ionomer powder) and metal powder admix materials have demonstrated fluoride release and caries inhibition at enamel (García-Godoy & others, 1990; Forss & Seppa, 1990) and dentin restoration margins in vitro.

Both resin-modified glass-ionomer cements (Forsten, 1995) and conventional glass ionomers may have synergistic effects when used with extrinsic fluorides (Creanor & others, 1995), including topical APF (acidulated phosphate fluoride) treatments (Kupietzky & others, 1994), and with fluoride rinses and fluoridated dentifrices in the remineralization of incipient enamel caries (Donly, 1994; Hatibovic-Kofman & Koch, 1991). The mechanism of this synergy is thought to be a recharging effect, where extrinsic fluoride is deposited back into the ionomer, resupplying the release from the ionomer into the surrounding environment.

Glass-ionomer cements have been found to be antimicrobial against *Streptococcus mutans* (Koch & Hatibovic-Kofman, 1990) and other oral bacteria (Palenik & others, 1992). The mechanism of antimicrobial action in glass ionomers has been shown to be a function of both fluoride and pH (DeSchepper, White & von der Lehr, 1989; Fischman & Tinanoff, 1994). The antimicrobial activity of the liquid components in their study was totally lost when the pH was raised to 5.

As is the case with other fluoride-releasing restorative materials, all glass ionomers have been shown to have a burst effect, releasing considerably more fluoride in vitro soon after restoration placement than later (Forsten, 1994). DeSchepper and others (1991) found that all 11 commercially available glass-ionomer cements released the greatest proportion of their cumulative total fluoride in the first 24 hours after mixing. The fluoride levels varied for different types of ionomers during this early release, but stabilized after 2 weeks to comparable low release levels of 0.16 μg/mm² to 0.42 μg/mm² of material. Creanor and others (1994) found the same effect in five commercial glass ionomers. After 60 days, the concentration of fluoride released had slowed from 15.3-155.2 μg/mL at day 1 to 0.9-3.99 μg/mL. Forsten found the burst effect to be true over a period of several days (1991) to 2 weeks (1977). In the more recent study, he found that the release eventually reached a constant level of approximately 0.5 μg/mL to 1.0 μg/mL for all tested glass ionomers, other than cermets, during the second year. Fluoride concentrations in unstimulated saliva following in vivo placement of glass-ionomer restorations were found to decrease by about 35% after 3 weeks and another 30% after 6 weeks (Koch & Hatibovic-Kofman, 1990). After 6 weeks, however, the fluoride level in saliva was still 10 times the 0.038 μg/mL to 0.050 μg/mL baseline concentration. Wilson, Groffman, and Kuhn (1985) also found the rate of fluoride release to be much diminished but still present over a study period of 598 days. Fluoride release rates have not been found to be proportional to fluoride concentrations in glass-ionomer products (McKnight-Hanes & Whitford, 1992), and commercially available cements vary in the amounts of fluoride released (Muzynski & others, 1988; Olsen & others, 1989; DeSchepper & others, 1991). Fluoride release from a silver cermet was found to be significantly less than the release from a standard glass ionomer throughout a 12-month period; the in vitro cariostatic effect of the cermet was also significantly less (Swift, 1989). The steady state fluoride release from 6 to 12 months was approximately 1.5 μg/mL for standard glass ionomer and 0.5 μg/mL for the cermet. Both materials,

however, had significantly higher in vitro caries inhibition than the composite and amalgam controls. This indicated that caries inhibition was fluoride-dose dependent even at these low release levels.

Handling, as well as the composition of glass ionomers, has been found to affect release rates (Hörsted-Bindslev & Larsen, 1990; Swift, 1988b; Davies, Sefton & Wilson, 1993; Muzynski & others, 1988). Clinical factors, such as powder-to-liquid ratio and maturity of the cement matrix have a larger effect on fluoride release than the cement composition. Studies have shown that cements with lower powder-to-liquid ratios demonstrated greater fluoride release. McKnight-Hanes and Whitford (1992) also found that the release rate of fluoride in a glass-ionomer cement was inversely proportional to the powder-to-liquid ratio used to prepare experimental disks. This finding is probably due to the composition, amount, and maturity of the reaction matrix forming within the cement. Hand-mixed glass ionomers have been shown to release significantly less fluoride than mechanically triturated glass ionomers (Miller & others, 1995). All of these studies demonstrate the importance of establishing well-defined material preparation and handling procedures to produce optimal and consistent fluoride release.

The varnishing of disks made from glass-ionomer products has been found to sharply reduce fluoride release, while finishing varnished disks produced a significant increase in the fluoride release of one glass-ionomer product in a three-product study (McKnight-Hanes & Whitford, 1992). Likewise, Kupietzky and his research team (1994) found a significant reduction in fluoride release from glass-ionomer restorations covered with a sealant. This finding is important when considering that the objective of coating an ionomer to provide protection during maturation may be counterproductive to the subsequent fluoride release.

Fluoride release from four brands of resin-modified glass-ionomer cement over an 11-month test period was found to be comparable to that from a chemically cured glass ionomer (Forsten, 1995). These resin-modified materials were also found to possess the fluoride-recharging capability previously demonstrated in unmodified ionomers. Caries inhibition for resin-modified glass ionomer was also demonstrated on extracted teeth and found to be comparable to a conventional ionomer cement (Dunne & others, 1996).

## COMPOMERS

A relatively new class of fluoride-releasing restorative materials has been introduced that combines the one-part composition and handling characteristics of a light-cured composite with the fluoride-releasing properties of a glass ionomer. These compomers are combinations of glass-ionomer glass powder or prereacted glass ionomer with a polymerizable acidified monomer (Barnes & others, 1995; Swift & Vann, 1995; Denehy & Vargas, 1996).

A comparative study of the fluoride-releasing characteristics of the compomer Dyract (L D Caulk, Milford, DE 19963) to several conventional and resin-improved ionomers indicated that the initial release was comparable to the resin-improved materials at approximately 25 μg/mL, and that the release rate after 28 days was still maintaining at approximately 6 μg/mL (de Araujo & others, 1996). These materials appear to have bonding and margin-sealing ability equivalent to composite resin (Yap, Lim & Neo, 1995; Sjodin, Uusitalo & van Dijken, 1996). A study by Krejci and others (1994) using Dyract compomer in class 2 deciduous restorations showed that margin degradation and wear were excellent after 6 months, and the authors suggested that this compomer may be acceptable as an amalgam substitute in deciduous teeth. While the initial data for these materials are encouraging, there is precious little clinical evidence to support a claim for caries inhibition at this time.

## COMPOSITES

Polymeric materials are among the most popular experimental materials used for the release of various therapeutic agents, not only for cariostatic applications, but for anesthetic, endodontic, prosthodontic, and periodontal applications as well. Rawls (1991) reviewed several fluoride-releasing filling and adhesive resins and predicted that they represent the leading edge of a new class of controlled-release and site-specific preventive materials.

The release of fluoride from composite resins has been postulated to protect against secondary caries in enamel and dentin. Fluoride-releasing composite has been shown in vitro to inhibit enamel demineralization (Arends & van der Zee, 1990; Arends, Ruben & Dijkman, 1990; Dijkman & Arends, 1992). Dijkman and others (1993) were able to show a dose-related response to this release with 200-300 μg/cm$^2$ completely inhibiting in situ secondary caries. Donly and Gómez (1994) have also demonstrated the remineralizing effects of a fluoride-releasing composite. Their results showed a statistically significant area reduction in the body of artificial caries-like lesions exposed to the fluoridated composite at 2-week and 3-month intervals, as compared with restorations composed of a resin containing no fluoride. Although fluoride-releasing composites have consistently demonstrated recurrent caries inhibition at enamel margins, there are conflicting results regarding caries inhibition at dentin margins (Donly, 1994).

As is the case with glass ionomers, there may well

be a synergistic effect between fluoride-releasing composites and fluoride rinses or fluoridated dentifrices in inhibiting demineralization and even in remineralizing incipient enamel caries in adjacent tooth structure (Donly, 1994). When exposed to additional external fluoride, the material surface undergoes an increase in fluoride, which is subsequently released.

## ADHESIVE PRIMERS

Kerber and Donly (1993) studied the effect on demineralization of adding ammonium fluoride (mass fraction of 10%) to two different dentin primers. Results showed that primers containing fluoride demonstrated significantly less demineralization 0.25 mm from the dentin margins of experimental restorations than control primers without the fluoride.

## ORTHODONTIC BONDING MATERIALS

Decalcification has always been a problem during orthodontic treatment with fixed appliances. Early in the history of fluoride-releasing restorative materials, it was suggested that bracket bonding agents that release fluoride could supply the component to the tooth area most prone to decalcification.

In the late 1970s and early 1980s, laboratory and clinical evidence indicated the greater solubility reduction and rehardening effectiveness of NaF over $SnF_2$, when incorporated into zinc oxyphosphate orthodontic cement (Shannon, 1980). Beginning in the late 1980s, wider varieties of orthodontic bonding materials were investigated. Underwood, Rawls, and Zimmerman (1989) demonstrated the durability and caries-inhibition potential of a fluoride-exchanging resin for orthodontic adhesion. The first fluoride-containing commercial orthodontic bonding composite was introduced in the late 1980s; it was found to release only very small amounts of fluoride, however, and at least one researcher (Fox, 1990) thought it unlikely that the material would have a therapeutic effect. Fluoride levels in enamel adjacent to and under orthodontic brackets were found to be increased by one fluoride-releasing bracket adhesive (Ohshita & others, 1991). As late as 1994, some researchers were reporting that fluoride-releasing composite adhesives may have a caries-preventive effect around orthodontic brackets (Ghani & others, 1994a), even though the adhesives failed to demonstrate any potential long-term fluoride release, and that in the short term the amount of fluoride released was very small (Ghani & others, 1994b).

Research reports on resin-based orthodontic adhesives, while generally favorable, have been nonetheless somewhat less effusive than reports on glass ionomers. Glass ionomer-based materials have generally been found to release more fluoride than resin-based materials (Chadwick & Gordon, 1995a). Ogaard and others (1992), while investigating the cariostatic potential in vivo of a visible light-cured composite adhesive for bonding orthodontic brackets, determined that regular use of fluoride toothpastes was insufficient to inhibit lesion development around orthodontic brackets. The study showed that the fluoride adhesive reduced lesion depths by about 48% over the nonfluoride adhesive control. Although a burst effect peaking at a fluoride content of 8 μg/mL occurred between 24 hours and 1 week, the study showed that more than 2 μg/mL of fluoride was still being released from the fluoride adhesive after 6 months. Wiltshire and Janse van Rensburg (1995) also reported a burst effect from two light-cured orthodontic adhesives, with one adhesive releasing measurable fluoride for 22 days and the second adhesive continuing to release fluoride at a level of 0.5 μg/mL for up to 85 weeks. They concluded that fluorapatite formation resulting from fluoride release from the tested adhesives could be more advantageous in reducing decalcification during fixed appliance treatment than other preventive measures.

Turner (1993) compared bond failure rate, plaque score, gingival health, and enamel decalcification using a conventional orthodontic adhesive compared to a fluoride-containing composite adhesive and found no significant difference between the materials for each of the variables examined, although there was a reduction in the number of white spot lesions with the fluoride-containing adhesive. Dubroc, Mayo, and Rankine (1994) reported positive results in the Sprague-Dawley rat model for a fluoride-releasing resin adhesive, finding that it reduced demineralization around orthodontic brackets and reduced caries at distant sites as well.

Glass-ionomer cement was also investigated in the late 1980s for its potential as a fluoride-releasing orthodontic bonding material. Hallgren, Oliveby, and Twetman (1990) found a significant increase in salivary fluoride concentration the day after cementation of brackets with glass-ionomer cement. However, after 7, 14, and 28 days, salivary fluoride levels were not statistically different from baseline values. The total ingested fluoride dose during the first day was estimated to be only 0.02 mg. Fischer-Brandies and others (1991) measured the fluoride content of enamel in vitro after bonding orthodontic fixtures with glass-ionomer cement. A rise of more than 120% in enamel fluoride was observed after the first 10 days, and a saturation value was reached after 40 days. The fluoride penetrated more than 20 μm into the surface of the enamel and more than 3 mm laterally from the edge of the cement.

The efficacy of using glass-ionomer cement as a bracket bonding material has been confirmed in

other studies (Hallgren, Oliveby & Twetman, 1993; Marcushamer, García-Godoy & Chan, 1993). While most studies have shown that glass ionomers inhibit demineralization, one has shown remineralization in demineralized enamel adjacent to glass ionomer-bonded orthodontic bands (Donly, Istre & Istre, 1995). Hallgren, Oliveby, and Twetman (1992) investigated microflora associated with caries in dental plaque adjacent to brackets bonded with a glass-ionomer cement and a composite containing no fluoride. They found an increasing prevalence of *Streptococci mutans* and lactobacilli in plaque from both retaining materials but significantly less colonization around the glass ionomer-retained brackets.

## PIT AND FISSURE SEALANTS

In 1984, Roberts, Shern, and Kennedy evaluated an autopolymerizing pit and fissure sealant as a vehicle for the slow release of fluoride. Sodium fluoride was added to the sealant at several concentrations up to mass fraction of 2.5%, and release of approximately 0.3 μg/mL was measured for the period from 31 days to 90 days at this highest concentration. When the authors considered the dilution factor due to average salivary flow, however, they concluded that this level of release would be below any known level of physiological significance. A fluoride-containing sealant (FluroShield, L D Caulk/Dentsply) was introduced to the dental materials marketplace in the late 1980s, was evaluated in vitro (Cooley & McCourt, 1990), and found to release fluoride over a 7-day evaluation period, beginning at a level of 3.5 μg/mL on the first day and declining to a level of at 0.41 μg/mL on the last 2 days. This same product was clinically compared to a conventional glass-ionomer sealant, where it was found that retention of the fluoride-releasing resin was much higher and caries incidence much lower than the glass ionomer (Rock & others, 1996). What could not be resolved in this study was whether this lower incidence of caries was due to the fluoride release or the greater retention of the resin. Hicks and Flaitz (1992) studied surface lesion depth and progression around in situ class 5 preparations restored with glass ionomer, fluoride-releasing sealant, and conventional sealant materials. They found that the glass ionomer provided the greatest degree of caries protection: 7.5% of the specimens had enamel wall lesions, while the conventional sealant group had 17.5% of the specimens with caries-like wall lesions. In another in vitro study (Jensen, García-Godoy & Wefel, 1990), a fluoride-releasing pit and fissure sealant was found to substantially reduce the amount of enamel demineralization adjacent to the material, compared with a conventional sealant. Seppa and Forss (1991) found that fissures sealed with a glass-ionomer sealant were more resistant to demineralization than were unsealed controls, even after macroscopic sealant loss. They suggested that the result may be the combined effect of fluoride release and residual material in the bottom of the fissures.

In an all-too-rare clinical trial, Songpaisan and others (1995) studied the prevention of fissure caries in 1264 Thai children by using (1) glass-ionomer cement applied by (a) dentists and (b) teachers who had received short-term training, (2) three applications of hydrofluoric acid solution (HF), mass fraction of 0.5%, or (3) a conventional autopolymerized resin-based sealant. There was a 74% reduction in fissure caries with the use of dentist-applied glass ionomer in relation to the control group and a 52% reduction with teacher-applied glass ionomer. There was a 33% reduction in the HF group. Almost no occlusal lesions were found in the sealant group where there was a 93% reduction in fissure caries incidence. These results suggest that the superior sealing capability of the nonfluoride resin material may be more important in preventing pit and fissure lesions than the caries inhibition effects of the ionomer fluoride release.

## LINERS/BASES AND CAVITY VARNISHES

Extensive research has been done to study the relationship between bacteria and subsequent pulpal damage following restorative procedures. Some researchers have claimed that the damage is caused solely by bacteria, while others postulate some combination of bacteria, operative trauma, and material toxicity (Hörsted & Eriksen, 1986). Cavity liners/bases, particularly calcium hydroxide materials, are routinely used to provide pulpal protection under deep restorations. Varnishes may also be used in shallow cavities or to supplement liners in deeper cavity preparations.

Brännström and Nyborg (1973) studied the effects of a microbicidal fluoride solution as a cavity cleanser, which, unlike the cleansers then commercially available, would not have a demineralizing effect. They recommended the use of a suitable liner to prevent leakage and to eliminate bacteria from cavity walls before restoration. Research by Beltrami and Nery de Lima Moro (1990) demonstrated the efficacy of sodium fluoride solution (mass fraction of 2%) application to cavity walls before lining with conventional cavity varnish and restoring with amalgam. In another study, amine fluorides were added to a copal resin liner and a chlorine caoutchouc cavity varnish to determine permeability and fluoride-release qualities (Nordbø & Eriksen, 1976). The liner films released high initial levels of fluoride, but within 2 weeks the release had decreased to levels

below 2 µg/mL. The permeability of the copal liner was only slightly increased, whereas the addition of fluoride destroyed the film-forming qualities of the chlorine caoutchouc varnish.

There are currently a half dozen or more fluoride-releasing liners on the market. Some have been found to significantly reduce lesion areas under amalgam restorations, as compared to amalgam alone or two layers of copal varnish and amalgam (Jensen & others, 1990). Most have been found to have a burst effect in the release of fluoride, with the largest proportion of total fluoride release occurring in the first days or weeks of a study, followed by dramatic reductions in the rate of release (DeSchepper & others, 1990; McCourt, Cooley & Huddleston, 1990; Cooley & McCourt, 1990; Horsted-Bindslev & Larsen, 1991; García-Godoy & others, 1990). Most of these studies found that some brands released more fluoride than others and that the long-term release varied over a range of 0 µg/mL to 7 µg/mL.

Glass-ionomer cements have also been used as a liner material under amalgam restorations. They have been shown to continue releasing measurable amounts of fluoride in the range of 0.3 µg/mL to 1.1 µg/mL after 1 year (García-Godoy & Chan, 1991), and to reduce artificial recurrent caries in vitro when placed under amalgam restorations (García-Godoy & Jensen, 1990). A light-cured and a chemically cured glass-ionomer cement liner were found to have a similar effect in inhibiting demineralization, and both demonstrated significantly less demineralization than a nonfluoride-releasing control liner (Souto & Donly, 1994). In an antimicrobial study of two glass-ionomer cavity liners and four glass-ionomer filling materials, the liners and two of the restorative-class materials were found to produce the largest growth inhibition zones by direct contact (Palenik & others, 1992). One resin-bonded liner, however, despite the fact that it was filled with a high-fluoride ionomer glass, did not release sufficiently inhibitory concentrations of fluoride to be active against *Streptococcus mutans*. The fluoride concentration of agar surrounding the resin-bonded liner was approximately 5 µg/mL compared to more than 100 µg/mL for the true glass-ionomer liners (DeSchepper & others, 1989).

## CONCLUSION

Numerous laboratory studies have examined the release of fluoride from restorative materials, uptake of fluoride in enamel and dentin, and the inhibitory effect of fluoride release on demineralization (Hörsted-Bindslev, 1994). Chadwick and Gordon (1995b), while noting a significant increase in the concentration of fluoride in enamel adjacent to a fluoride-releasing hybrid orthodontic adhesive, conclude that the clinical significance of the increase and the mechanism by which fluoride moves from the material into the enamel remain unclear. While in vitro studies have unquestionable value, Ripa (1991) is convincing in his call for controlled clinical trials to determine unequivocally if, when fluoride is introduced into dental materials, it inhibits dental caries. Such trials are, indeed, rare.

A preliminary report on the clinical performance of glass-ionomer restorations by Mjör (1996) indicated that about 50% of ionomer restorations were replaced due to secondary caries. This replacement figure was equal to that found for amalgam and was higher than the 33% of composite restorations replaced due to secondary caries. However, glass ionomers were initially placed in carious lesions more than twice as often as both composite and amalgam, and this may indicate that those restorations could have been at a higher risk for recurrence than either the composite or amalgam restorations.

Whether the discussion centers on dentin adhesives, posterior composites, or, for that matter, almost any biomedical device or agent, contradictions in research results presented by different investigators and, occasionally, by the same investigator, become contentious and divisive issues. A concern, therefore, for scientists studying fluoride-releasing dental materials is the lack of standardization in test methods. Any attempt to standardize testing of fluoride release must address specific specimen-fabrication techniques, immersion test conditions such as solution pH and temperature, standardized immersion times, and the units and methods of measure.

Even with apparent ambiguities in the literature, several facts remain. Fluoride release can be tested relatively easily and has been adequately demonstrated with most fluoride-containing materials. All fluoride-containing materials release fluoride in an initial burst and then reduce exponentially to a much lower steady-state level of release. Steady-state release of fluoride is reached after approximately 30 days for most materials. Caries inhibition and remineralization potential have been shown in vitro by all of these materials when release levels have been equal to or exceeding approximately 1 µg/mL. These facts should serve as a basis for the development of criteria for which dental restorative materials may claim effective fluoride release.

Few clinical studies appear to support the proposition that low levels of fluoride release can inhibit in vivo demineralization and caries formation. The ultimate goal of correlating fluoride release with actual caries reduction is an objective that can only be met by completing controlled clinical studies on materials with well-characterized kinetics of fluoride release.

## 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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# ORIGINAL ARTICLES

# Effects of Sealers and Liners on Marginal Leakage of Amalgam and Gallium Alloy Restorations

B P NG • J A A HOOD • D G PURTON

**Clinical Relevance**
The use of a varnish, glass ionomer, or adhesive resin sealer or liner signicantly reduced marginal leakage around amalgam and gallium alloy restorations.


## SUMMARY

**In an in vitro study, the use of sealers and liners (Fuji varnish, Vitrabond, Vitremer, Paama 2, All-Bond 2, or Resinomer) significantly reduced the amount of marginal leakage around amalgam (Permite C or Lojic Plus) and gallium (Galloy) alloy restorations. This reduction in marginal leakage was produced by all sealers and liners tested, and there were no statistically significant differences between these materials. Unlined restorations of Permite C had significantly less marginal leakage than Galloy or Lojic Plus. Unlined Lojic Plus restorations had the greatest amount of marginal leakage. The experimental method used in the present study proved to be suitable for quantitative comparison of marginal leakage of different dental materials.**


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## INTRODUCTION

Amalgam has served effectively as a dental restorative material for over a century. It is widely recognized that there is significant marginal leakage initially after placement of amalgam, but that it diminishes with time due to the formation of corrosion products. Recently, attention has turned toward the use of adhesive materials, such as cavity sealers and liners, for reducing leakage. There are two main groups of adhesive resin liners, some based on the bisphenyl-A glycidyl methacrylate (BIS-GMA) monomer, and others based on methyl methacrylate. Some studies have suggested that glass-ionomer cements during their early reaction phase can bond to base metals, especially tin (Hotz & others, 1977) and silver (Negm, Beech & Grant, 1982). This provides an alternative mechanism for reducing interfacial leakage and enhancing bonding.

The significance of dimensional changes in amalgam restorations during setting and their effects on marginal leakage has been controversial. Swartz and Phillips (1962), in an in vitro radioisotope leakage

study, found comparable marginal leakage among different alloys. This was in agreement with the results of an in vitro study using an air-pressure technique (Granath, 1971). However, Rupp, Paffenbarger, and Manuszewski (1977), in an in vitro study using porcelain teeth and an air-pressure technique, found greater marginal leakage associated with restorations made from contracting amalgam alloys compared with restorations made from balanced or expanding amalgam alloys.

Research suggests that different amalgam alloys may adapt differently to the cavity walls as a result of factors other than dimensional changes on setting. A number of in vitro studies have found no significant differences between marginal leakage and amalgam composition (Andrews & Hembree, 1978; Symer & Wing, 1981; Fanian, Hadavi & Asgar, 1983). The results of alloy particle-shape studies have been conflicting, possibly due to the variation in experimental methods.

In recent years, some patients have perceived amalgam as a health hazard because of the supposed mercury toxicity. This has led to the search for an alternative mercury-free restorative alloy. Restorative alloys containing gallium have been developed recently. Smith and Caul (1956) and Smith, Caul, and Sweeney (1956) showed that gallium-copper-tin alloys exhibited little change in dimension during hardening, and that their physical and mechanical properties were superior to silver amalgam. In a study by Okamoto and Horibe (1991), Ga-In-Zn, which is a liquid at room temperature, was mixed with Sn-Pd-Cu-Zn-Ag powder. The two components mixed readily and hardened relatively rapidly at mouth temperature. The strength of this alloy increased markedly immediately after mixing, while polishing was possible on the same day.

Traditionally, varnish has been used as a sealer for amalgam restorations. The application of varnish to the cavity walls and margins prior to the condensation of amalgam has been shown to reduce marginal leakage around new amalgam restorations (Swartz & Phillips, 1962; Barber, Lyell & Massler, 1964). However, it is not effective in totally eliminating marginal leakage (Ben-Amar & others, 1990; Grossman & Matejka, 1993; Berry & Tjan, 1994). In fact its sealing effect appears to be temporary, with reported effectiveness of 6 to 12 months (Sneed, Hembree & Welsh, 1984; Fitchie & others, 1990). A study by Tjan and Li (1992) showed that All-Bond 2 was more effective than varnish in reducing marginal leakage. The nature of the bonding mechanism between adhesive resin liners and amalgam alloys has yet to be established.

Glass ionomer has gained popularity for its use as a liner under amalgam restorations due to its ability to release fluoride (Maldonado, Swartz & Phillips, 1978; McCourt, Cooley & Huddleston, 1990), the ability to bond to both enamel and dentin (Maldonado & others, 1978), and by having a low coefficient of thermal diffusivity (Watts & Smith, 1981). In one study with glass-ionomer liner, less marginal leakage was found than with copal varnish even after thermocycling and mechanical loading (Jeblinger & Lutz, 1987). The more recent light-cure glass-ionomer materials offer more convenient handling properties and may overcome the problem of initial high water absorption of these aluminosilicate-based liners (Crisp, Lewis & Wilson, 1980).

In an in vitro study by Youngson, Grey, and Jones (1990), using a dye-penetration technique, there was significantly less marginal leakage associated with amalgam restorations lined with a glass-ionomer liner (Vitrabond) than with amalgam restorations sealed with varnish.

In recent years, techniques have been introduced for bonding amalgam restorations to tooth tissues by using adhesive resin sealers and liners. The possible advantages of this include preservation of tooth structure, resistance to dislodgment, reduction of microleakage, and increased resistance of restored teeth to fracture (Staninec, 1989).

Various techniques have been used by researchers to assess the marginal leakage around dental restorations. These include penetration by various dyes, bacteria, or radioisotopes; the passage of air under pressure; neutron activation analysis; and artificial caries (Kidd, 1976). These techniques are sometimes used in conjunction with thermocycling; however, there is no scientific evidence to indicate that thermocycling is essential for leakage studies. Using gas pressure as a laboratory test method allows quantitative comparison of leakage. An experimental technique using gas pressure to assess marginal leakage around amalgam restorations was developed by Fanian and others (1983). The apparatus allowed a pressured gas to be forced through the interface between an amalgam restoration and a methyl methacrylate disk. The marginal leakage was then measured by the volume of gas leaked over a specific period of time. The higher the recorded value, the poorer the marginal seal of the restoration.

The present experiment used a gas-pressure technique to study the effectiveness of glass-ionomer liners and adhesive resin sealers and liners in reducing marginal leakage of alloy restorations.

## METHODS AND MATERIALS

Standard cavities were prepared centrally in 210 methyl methacrylate disks (4.0 mm thick), using an 8-degree taper conometric cutter (Komet ISO-021, Brasseler, Lemgo, Germany) in an engineering lathe. The nominal dimensions of the prepared cavity are

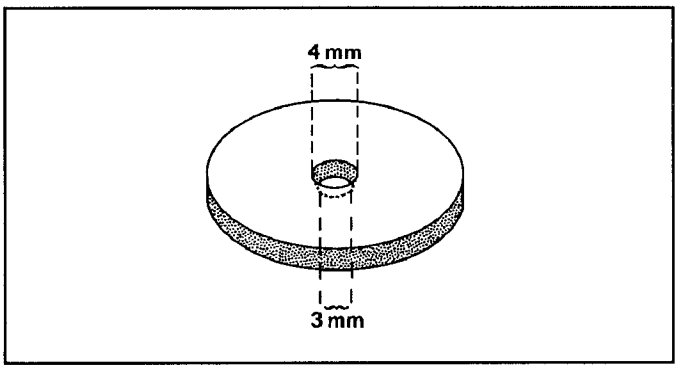


Figure 1. *The nominal dimensions of the finished cavity preparation in the methyl methacrylate disk*

illustrated in Figure 1. The prepared disks were allocated to 21 groups of 10, in preparation for testing seven sealers and liners in combination with three restorative alloys.

In the present experiment, Vitrabond (3M Dental Products, St Paul, MN 55144) and Vitremer (3M Dental Products) were the glass-ionomer liners tested. Vitrabond is a light-cure glass-ionomer liner/base. Vitremer is a liner that consists of a glass-ionomer primer, a glass-ionomer powder, and a tri-cure glass-ionomer liquid.

The test sealers or liners were applied to the cavity walls following the manufacturers' instructions. The adhesive resin sealers and liners used in the present study included Paama 2 (SDI, Melbourne, Australia), All-Bond 2 (Bisco, Itasca, IL 60143), and Resinomer (Bisco). Paama 2 is a light-cure dentin and enamel adhesive. All-Bond 2 is a fourth-generation dual-cure dentin and enamel adhesive resin sealer system that bonds micromechanically to both enamel and dentin. Resinomer is a dual-cure, glass-filled, fluoride-releasing composite. Resinomer was used together with the All-Bond 2 primer. The Fuji varnish (GC, Tokyo, Japan) was applied in two layers to the walls of the tapered cavities. Vitrabond, Vitremer, and Resinomer liners were not light cured prior to the placement of alloy restorations. In the control groups, no lining material was placed.

The restorative alloys tested were Permite C (admixed amalgam alloy, SDI), Lojic Plus, and Galloy. Galloy (SDI) is a gallium-based amalgam type restorative material that is essentially similar to the Lojic Plus (60 wt% silver) spherical alloy (amalgam, SDI), with mercury being substituted by gallium alloy liquid. The alloys were triturated according to the manufacturers' instructions using an amalgamator (Silamat type S3, Vivadent, Schaan, Liechtenstein). The cavities were packed with the restorative alloys immediately after lining, using a hand-held plugger (FE0264, American Dental Mfg Co, Chicago, IL 60618). The alloy was condensed onto the uncured (oxygen-inhibited) Resinomer layer, leading to a mechanical bond with the restoration. One end of the plugger was 1.5 mm and the other 2.0 mm in diameter. Both Lojic Plus and Galloy are spherical alloys that offered less resistance to condensation forces, therefore only the 2.0 mm end was used. With Permite C, both 1.5 and 2.0 mm ends were used. All the alloys were condensed with a force of approximately 5.0 kg, the force being monitored by a force cell (Transducer/Converter Type SE905, Sample Electronics Laboratories Ltd, Feltham, England). The rate of condensation was 40-45 thrusts per minute, and each restoration was packed for 1 minute before the excess alloy was carved. For Galloy restorations, sealer was applied to the surfaces according to the manufacturer's recommendation. The restored disks were stored in a water bath at 37 °C for 7 days.

The leakage apparatus used in the experiment is illustrated in Figure 2. It consisted of a specimen holder connected to two tubes. The specimen holder consisted of two compartments forming a chamber that held the disk and restoration when the compartments were screwed together. The specimen holder is illustrated in Figure 3. Two O-rings were used to ensure that the specimen chamber was sealed. One of the tubes connected the specimen holder to a dry nitrogen gas tank. The pressure selected for the marginal leakage test was 410 KPa, similar to that used by Fanian and others (1983). The other tube carried any gas leaked from the chamber to an inverted graduated burette.

Within the chamber, the restoration was arranged so that the gas was forced from the larger-diameter side through to the side with the smaller diameter. Any gas that leaked through the disk/restoration interface

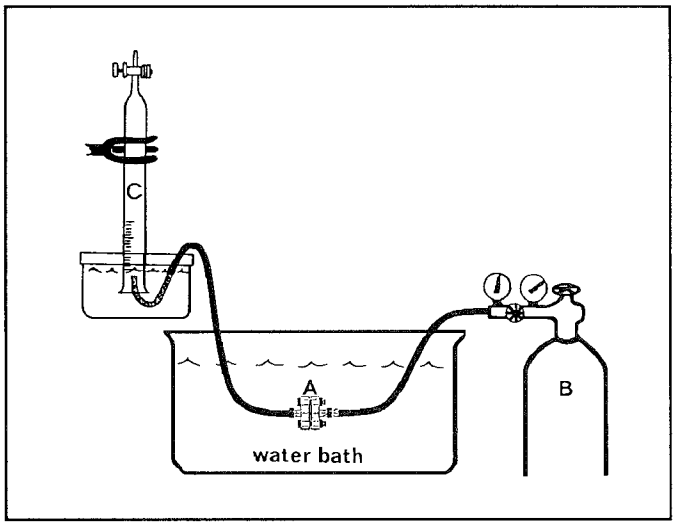


Figure 2. *The leakage apparatus. A = specimen holder; B = dry nitrogen gas tank; C = inverted graduated burette.*

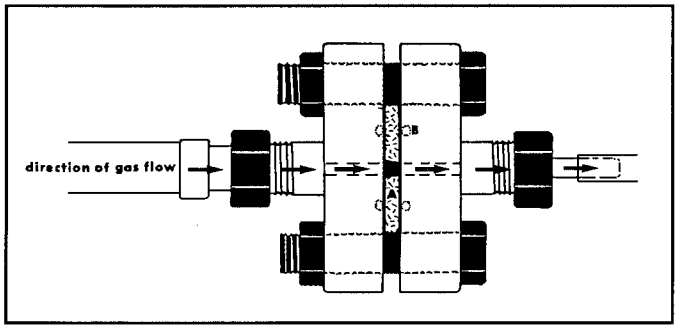


Figure 3. *The specimen holder. A = methyl methacrylate disk containing the test restoration; B = O-rings.*

displaced the water in the burette and thereby lowered the water level, which was recorded after 15 minutes. However, for restorations that exhibited substantial leakage, the time taken to collect 90 ml of gas was recorded. The entire specimen holder was immersed in water during the test period to allow detection of any leakage of gas from the specimen chamber.

A two-way analysis of variance (ANOVA) statistical test was used, followed by a one-way ANOVA and the Duncan New Multiple Range test to analyze the statistical significance of the differences of the means among the alloys used with the seven sealers and liners.

## RESULTS

### Marginal Leakage of the Sealed and Lined Restorations

With the two-way ANOVA test, no statistically significant differences were found. With the use of one-way ANOVA and Duncan New Multiple Range test, the results showed that the use of a sealer or liner reduced the marginal leakage between the restorations and the cavity walls when compared to those specimens with no lining. For all three restorative alloys, the placement of a sealer or liner resulted in a statistically significant reduction in marginal leakage when compared with the unlined specimens ($P < 0.05$). There was no statistically significant difference in the ability to reduce marginal leakage among varnish, the glass-ionomer liners, and the adhesive resin sealer or liner. Results are illustrated in Tables 1, 2, and 3, and Figure 4.

Because large variations were found in the results within some lined test groups, arbitrary groupings

*Table 1. Mean Marginal Leakage of Permite C/Liner Interface (ml/min)*

| | Mean | SD | Duncan New Multiple Range Test at $P < 0.05$ |
|---|---|---|---|
| No lining | 1.721 | 1.819 | a* |
| Varnish | 0.009 | 0.021 | b |
| Paama 2 | 0.011 | 0.023 | b |
| Vitrabond | 0.005 | 0.007 | b |
| Vitremer | 0.003 | 0.005 | b |
| All-Bond 2 | 0.003 | 0.005 | b |
| Resinomer | 0.003 | 0.006 | b |

n = 10 restorations/group; * = categories with same letters are not significantly different.

*Table 2. Mean Marginal Leakage of Lojic Plus/Liner Interface (ml/min)*

| | Mean | SD | Duncan New Multiple Range Test at $P < 0.05$ |
|---|---|---|---|
| No lining | 31.513 | 7.390 | a* |
| Vitrabond | 0.939 | 1.598 | b |
| Varnish | 0.663 | 1.413 | b |
| All-Bond | 0.090 | 0.141 | b |
| Paama 2 | 0.083 | 0.219 | b |
| Vitremer | 0.029 | 0.052 | b |
| Resinomer | 0.003 | 0.004 | b |

n = 10 restorations/group; * = categories with same letters are not significantly different.

*Table 3. Mean Marginal Leakage of Galloy/Liner Interface (ml/min)*

| | Mean | SD | Duncan New Multiple Range Test at $P < 0.05$ |
|---|---|---|---|
| No lining | 19.475 | 8.181 | a* |
| Paama 2 | 0.409 | 0.880 | b |
| Varnish | 0.367 | 1.157 | b |
| Vitremer | 0.232 | 0.509 | b |
| All-Bond 2 | 0.037 | 0.100 | b |
| Vitrabond | 0.017 | 0.026 | b |
| Resinomer | 0.001 | 0.003 | b |

n = 10 restorations/group; * = categories with same letters are not significantly different.

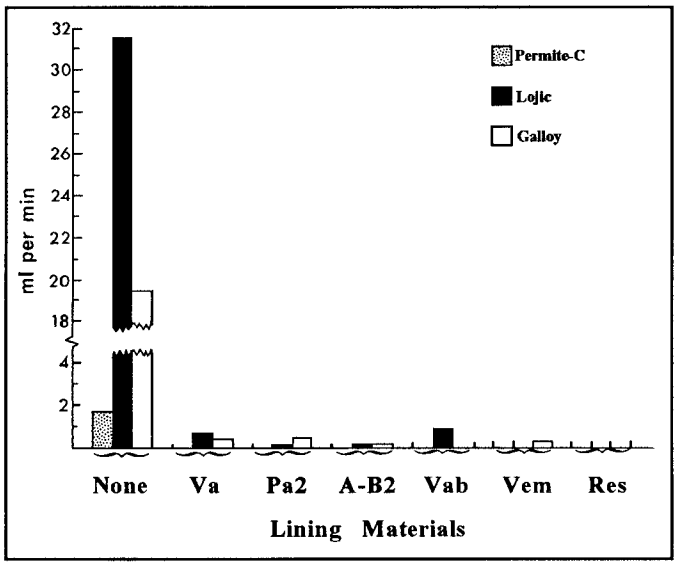


Figure 4. *Mean marginal leakage of the different restorative alloys with different sealers and liners. Va = varnish; Pa2 = Paama 2; A-B2 = All-Bond 2; Vab = Vitrabond; Vem = Vitremer; Res = Resinomer.*

were chosen to illustrate the distribution of results within each test group (Figure 5).

## Marginal Leakage of Unlined Alloy Restorations

For restorations with no lining material, there were differences in marginal leakage among the three different alloys. Significantly less marginal leakage was found with the unlined Permite C (admixed amalgam alloy) restorations compared with the unlined Lojic Plus (spherical amalgam alloy) restorations and the unlined Galloy (gallium alloy) restorations ($P < 0.05$). There was also significantly less marginal leakage with the unlined Galloy restorations compared with the unlined Lojic Plus restorations ($P < 0.05$). The results are illustrated in Table 4.

*Table 4. Mean Marginal Leakage of the Different Restorative Alloys with No Lining (ml/min)*

| | Mean | SD | Duncan New Multiple Range Test at $P < 0.05$ |
|---|---|---|---|
| Permite C | 1.721 | 1.819 | a* |
| Galloy | 19.475 | 8.181 | b |
| Lojic Plus | 31.513 | 7.390 | c |

n = 10 restorations/group; * = different letters indicate a significant difference.

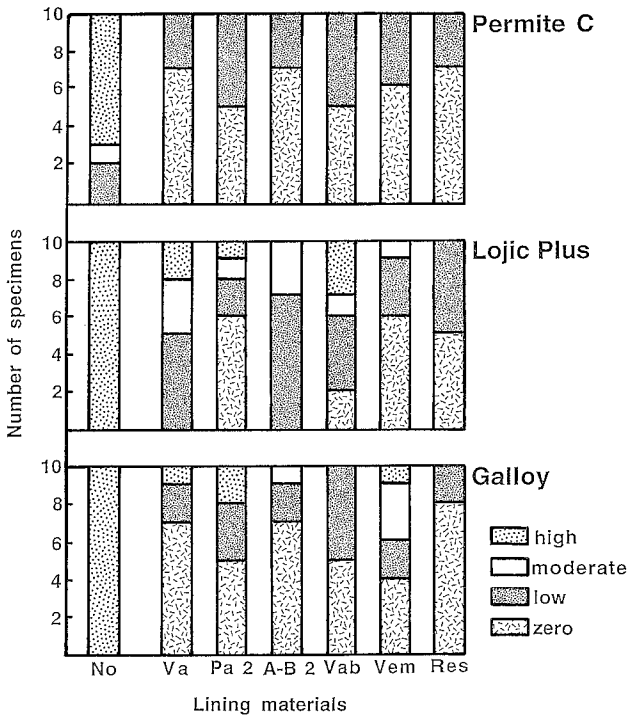


Figure 5. *Arbitrary groupings of the distribution of marginal leakage within each test group. Zero = 0 ml/min; 0 < low < 0.1 ml/min; 0.1 < moderate < 0.5 ml/min; high > 0.5 ml/min. No = no lining; Va = varnish; Pa2 = Paama 2; A-B2 = All-Bond 2; Vab = Vitrabond; Vem = Vitremer; Res = Resinomer.*

# DISCUSSION

## Marginal Leakage of Sealed and Lined Alloy Restorations

Amalgam and gallium alloys do not adhere to cavity walls. Consequently, leakage will occur around the margins of the restorations. In the present study, sealed or lined alloy restorations leaked significantly less than unlined restorations. No significant differences were found among the different sealers and liners used. It has been clearly established that with time amalgam restorations corrode, and the corrosion products subsequently reduce marginal leakage. In the short term, there is leakage, and the clinical manifestation of this is sensitivity. Traditionally, varnish has been used to seal the interface to reduce both the leakage and sensitivity. Varnish has the ability to cover the cavity wall and provide a physical barrier to marginal leakage. As shown in the results, the use of varnish does not completely eliminate marginal leakage. This is probably due to voids present within the layer of varnish. For glass-ionomer liners and adhesive resin sealers or liners, the possible

bonding mechanisms to tooth structures have been described earlier. However, bonding to restorative alloys has not been demonstrated. Therefore the ability of these sealers and liners to reduce marginal leakage is probably due to their providing physical barriers to fluid penetration.

In each of the lined groups of Permite C restorations, half recorded zero marginal leakage over the test period. The remaining lined Permite C restorations recorded low marginal leakage (< 0.1 ml/min). The consistently low marginal leakage is probably related to the properties of the Permite C alloy, which will be discussed in the next section. Zero marginal leakage was also recorded for half or more of the Resinomer-lined restorations. Resinomer is a paste lining material, and the restorative alloy is condensed onto it before it cures. The unset paste probably adapts more closely to the restorative alloy than a preset lining material could, and is therefore more effective in eliminating marginal leakage.

A small number of the sealed or lined Lojic Plus and Galloy specimens showed high marginal leakage values. As the restorations were condensed under ideal laboratory conditions, the authors suspect that under normal clinical conditions even larger variations may occur.

In the present study, methyl methacrylate disks were used as the substrate in preference to dentin because of easy access and constant quality. The sealers and liners tested (except varnish) contained components with methacrylate end groups. Subsequently, these same specimens were used in a shear bond test, and the sealers and liners were found to be attached to the methyl methacrylate disks following displacement of the restorations. This finding confirmed that the results obtained from the present tests related to leakage along the interface between the alloy and the sealer or liner. The results obtained from the shear bond test will be discussed in a separate article.

### Marginal Leakage of Unlined Alloy Restorations

In the present study, unlined Permite C (admixed amalgam alloy) restorations showed less marginal leakage than Lojic Plus (spherical amalgam alloy) restorations. This is in agreement with the findings of Mahler and Nelson (1984), who also used an in vitro air-pressure technique. However, the study of Fanian and others (1983) showed no significant difference in marginal leakage between admixed and spherical amalgam restorations. The present experimental technique is similar to that used by Fanian and others (1983). The discrepancy in results may be due to the differences in dimensional change during the setting of the alloys, since different amalgam alloys were used. The setting expansion is 0.03% for Permite C, 0% for Lojic Plus, and 0.01% for Galloy as described by each manufacturer's technical data sheet. A correlation between marginal leakage and dimensional change of alloys has previously been established (Øilo, 1976; Rupp & others, 1977).

According to the manufacturer, Galloy is a spherical alloy with the same powder composition as Lojic Plus, but with the distinction of having a mercury-free liquid component. In the present study, Galloy restorations had significantly less marginal leakage than Lojic Plus restorations. The difference probably resulted from two factors, the difference in composition of the set alloys and the difference in setting expansion. According to the manufacturer, free gallium can react with moisture for up to 18 hours after placement, forming corrosion products and causing expansion of the alloy. As the restorations were stored in water for 7 days prior to testing, it is likely that the reduced marginal leakage associated with unlined Galloy restorations was a result of alloy expansion.

## CONCLUSIONS

In the present in vitro study, the use of sealers and liners significantly reduced the amount of marginal leakage around alloy restorations ($P < 0.05$). This reduction in marginal leakage was produced by all the sealers and liners tested, and there were no statistically significant differences among these materials.

Of the unlined restorations, Permite C had significantly less marginal leakage than Galloy or Lojic Plus. Unlined Lojic Plus restorations had the greatest amount of marginal leakage.

The experimental method used in the present study proved to be suitable for quantitative comparison of marginal leakage of different dental materials.

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# Selective Caries Removal with Air Abrasion

S HORIGUCHI • T YAMADA
S INOKOSHI • J TAGAMI

**Clinical Relevance**
Air abrasion with particles of similar hardness to that of intact dentin could selectively remove carious dentin.

## SUMMARY

**The purpose of this study was to evaluate selective caries removal using an air-abrasive technique. Alumina powders, glass beads, crushed glass powders, and crushed powders of polycarbonate resin were applied to intact human enamel, dentin, and artificially demineralized dentin (caries-model dentin). Furthermore, the effect of the particle size of abrasives and air pressures on the abraded depths was examined.**

**When alumina powders and glass beads were used, the abraded depths of enamel, dentin, and caries model increased as the particle size and air pressure increased. Alumina powders and crushed glass powders abraded intact enamel and dentin more than the caries-model dentin, whereas glass beads abraded the caries-model dentin more than the intact enamel and dentin. Only crushed powders of polycarbonate resin abraded the caries-model dentin without reducing intact enamel and dentin.**

**With hard particles, such as alumina powders, glass beads, or crushed glass powders, selective caries removal by the air-abrasive technique appeared to be difficult to achieve, even if the particle size and the air pressure were changed. Crushed powders of polycarbonate resin that reduced only the caries-model dentin are harder than caries-model dentin, but softer than intact enamel and dentin. This study elucidated the possibility of selective carious dentin removal with the air-abrasive technique.**

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## INTRODUCTION

In modern dentistry, the adhesive resin bonding systems minimize the requirement for retention and resistance form for cavity preparations (Fusayama, 1980) that were needed in the past system (Black, 1899). This has enabled dentists to restore decayed teeth without reducing and extending into intact dentinal tissue. It is recommended currently that only

*Table 1. Abrasive Particles Used*

| Abrasive Particles | | Average Particle Diameter (μm) | Vickers Hardness (Hv) | Particle Shape | Density (g/cm³) | Manufacturer |
|---|---|---|---|---|---|---|
| Alumina powders | #360 | 49 | 2000 --- 2300 | angular | 3.9 | Fujimi Inc, Nagoya, Japan |
| Alumina powders | #150 | 74 | 2000 --- 2300 | angular | 3.9 | Fujimi Inc |
| Alumina powders | #100 | 125 | 2000 --- 2300 | angular | 3.9 | Fujimi Inc |
| Alumina powders | #80 | 177 | 2000 --- 2300 | angular | 3.9 | Fujimi Inc |
| Glass beads | #360 | 50 | 500 --- 550 | spherical | 2.5 | Pana Heraeus Dental Co, Tokyo, Japan |
| Glass beads | #150 | 91 | 500 --- 550 | spherical | 2.5 | Toshiba Ballotini Corp, Tokyo, Japan |
| Glass beads | #100 | 128 | 500 --- 550 | spherical | 2.5 | Toshiba Ballotini |
| Glass beads | #80 | 196 | 500 --- 550 | spherical | 2.5 | Toshiba Ballotini |
| Crushed glass powders | | 178 | 500 --- 550 | angular | 2.5 | Toshiba Ballotini |
| Polycarbonate resin | #80 | 215 | 40 --- 50 | angular | 1.3 | Toshiba Ballotini |
| Polycarbonate resin | #60 | 338 | 40 --- 50 | angular | 1.3 | Toshiba Ballotini |

carious lesions should be excavated for adhesive resin restorations (Fusayama, 1980). Current techniques of conservative cavity preparation use rotary cutting tools and caries-disclosing dye solution. However, caries removal can be time-consuming and troublesome (Takatsu & others, 1984). Therefore, new techniques and/or equipment that could ease caries removal and shorten the chair time could be of great benefit to the practicing dentist.

Air-abrasive technology was introduced into the dental field by R B Black (1945). Recently, several new air-abrasive cutting instruments have been introduced (Berry & Ward, 1995; Goldstein & Parkins, 1995; Laurell & others, 1995; Nikaido & others, 1996) to the dental profession. Air-abrasive systems use air pressure (7-11atm) with alumina powders (20-50 μm) to cut dental tissue. Devices with alumina powders were investigated as cutting tools for cavity preparation, and their potential efficiency for cutting hard tooth tissue was reported (Black, 1950; Myers, 1954; Goldstein & Parkins, 1995). However, several investigators (Epstein, 1951; Ohsawa & others, 1987; Goldstein & Parkins, 1995) claimed that the effectiveness of caries removal was low, compared to intact enamel and dentin. There has been no reported research concerning selective caries removal using an air-abrasive technique.

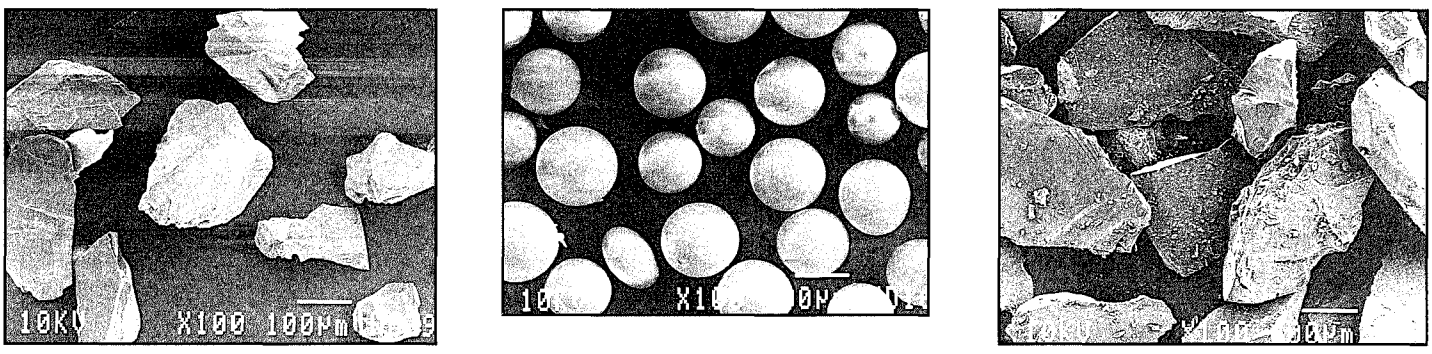
The purpose of this study was to determine an acceptable air-abrasion method that would enable clinicians to remove carious tissue, without damaging intact enamel and dentin.

## METHODS AND MATERIALS

### Materials Used and Sample Preparation

Four kinds of air-abrasive particles, alumina powders, glass beads, crushed glass powders (CGP), and crushed powders of polycarbonate resin (PR), were selected for this study (Table 1) (Figures 1 and 2).

Figure 1. *SEM photographs of abrasive particles; magnification X100*

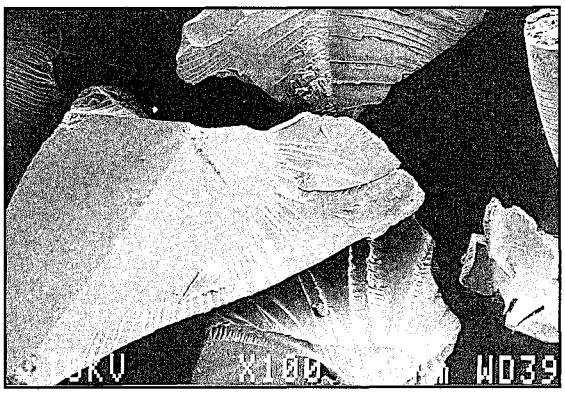



**A.** *Alumina powders #100*

**B.** *Glass beads #100*

**C.** *Crushed glass powders*

Figure 2. *SEM photographs of abrasive particles; magnification X100*



A. *Polycarbonate resin #80*

B. *Polycarbonate resin #60*

Alumina powders are the most commonly used abrasives in air-abrasive techniques. These particles were selected as the control.

Extracted human molars stored frozen at -20 °C were used within 30 days after extraction. The teeth were placed in acrylic tubes and embedded in dental stone (GC New Fujirock, GC Corp, Tokyo, Japan). For the enamel and dentin samples, the buccal surfaces of the teeth were reduced until the approximal part of enamel was exposed, by means of a model trimer (Y230, Yoshida Dental Mfg Co, Ltd, Tokyo, Japan), under running water. The surface was then finished with #600 SiC paper under running water (Figure 3).

For the caries-model dentin, teeth were demineralized with the Plank-Rychlo's decalcifying solution (Plank & Rychlo, 1952) for 1 week, neutralized with 5% sodium sulfate for 6 hours, and washed under running tap water for 6 hours. The demineralized teeth were placed in acrylic tubes and embedded in dental stone. The buccal surfaces were exposed, and finished as described above.

The samples were placed in a specially designed device, in the chamber of the air-abrasive machine (CL-FSG3, Heraeus Kulzer GmbH, Hanau, Germany) (Figure 4). A nozzle tip was fixed to the air-abrasive machine, perpendicular to the sample surface, and with a nozzle-sample distance of 2.0 mm.

## Effect of Air-Abrasive Particles on Abrasion Depth

Specimens were subjected to air abrasion using the different particles listed in Table 1. Since the air pressure applied to a dental unit is approximately 4.5 atm (atmospheres), air pressure of the abrasive machine was fixed to 4.5 atm. Nozzle diameter was 0.8 mm except when polycarbonate resin #60 particles were used. These resin #60 particles were

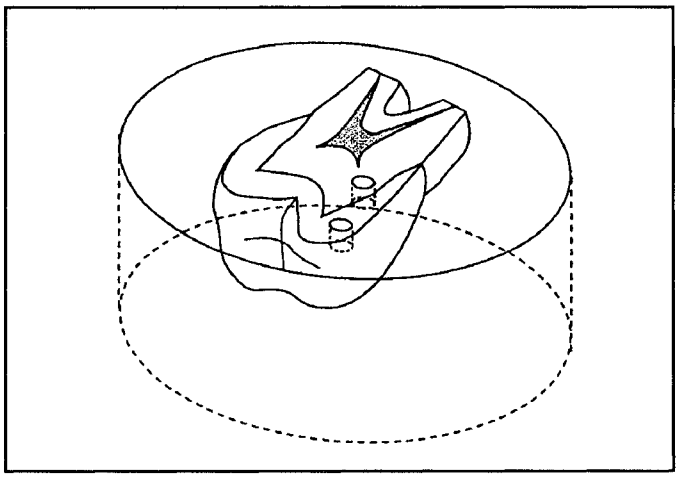


Figure 3. *Human molar sample; section surface of human molar embedded in dental stone was used to determine abraded depth of enamel and dentin.*

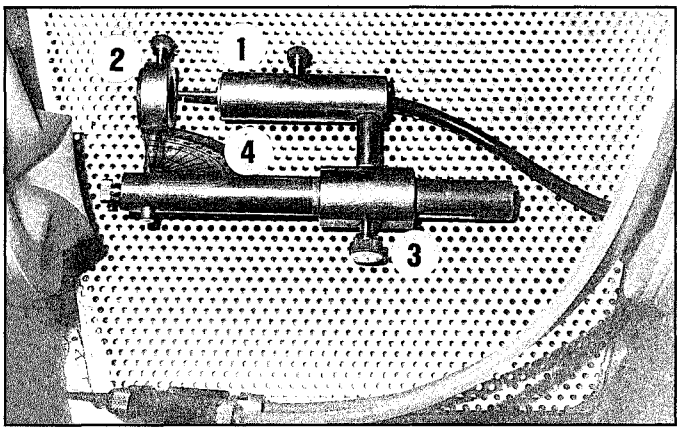



Figure 4. *Nozzle and samples were fixed with specially designed device in the chamber of the air-abrasive machine. 1 = nozzle holder; 2 = sample holder; 3 = nozzle-sample distance adjuster; 4 = burst angle adjuster.*

*Table 2. Correlation Coefficient*

| Parameters | Enamel | Dentin | Caries Model |
|---|---|---|---|
| abraded depth--average particle diameter (alumina powders) | 0.934 ($P < 0.05$) | 0.941 ($P < 0.05$) | 0.978 ($P < 0.05$) |
| abraded depth--average particle diameter (glass beads) | 0.862 ($P < 0.05$) | 0.406 (ns) | 0.178 (ns) |
| abraded depth--air pressure (alumina powders) | 0.941 ($P < 0.05$) | 0.909 ($P < 0.05$) | 0.958 ($P < 0.05$) |
| abraded depth--air pressure (glass beads) | 0.960 ($P < 0.05$) | 0.486 (ns) | 0.958 ($P < 0.05$) |

The level of significance is in parentheses; ns = no signficant correlation (r = 0).

large enough to obstruct the 0.8 mm nozzle, so a 1.2 mm-in-diameter nozzle was used only for this type of particle. The air-abrasion time was 3 seconds, and eight samples for all particles and substrates were produced. The center profile of the abraded cavity was traced with a surface recorder (SEF-30C, Kosaka Laboratory Ltd, Tokyo, Japan), and the abraded depth was represented by the distance from the uncut surface to the deepest point of the abraded cavity.

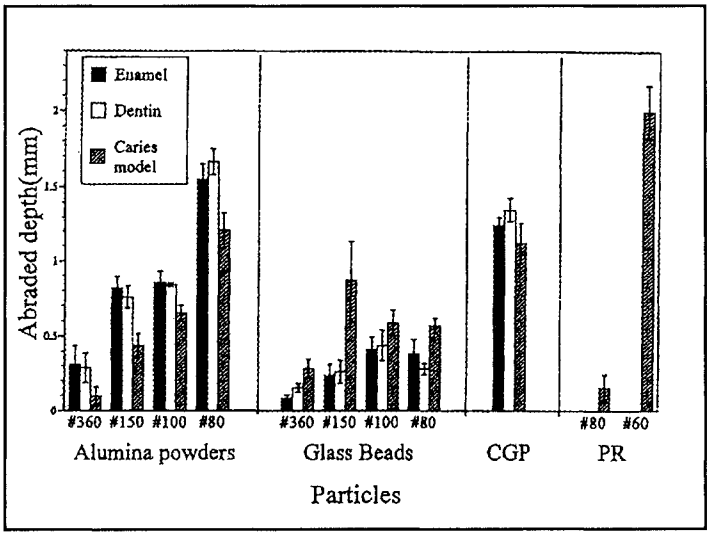



Figure 5. *The effect of air-abrasive particles on abraded depth. CGP = crushed glass powders; PR = polycarbonate resin. CGP (angular shape) showed deeper abrading depth than glass beads #80 (spherical shape). With alumina powders and glass beads, abraded depth increased as the particle size increased. PR #80 and #60 did not reduce enamel and dentin, and abraded only caries-model dentin. Abraded depth of caries-model dentin with PR #60 was the greatest of all.*

## Effect of Air Pressures on Abrasion Depth

To determine the effect of the air pressure on abrasion depth of the three substrates, the cutting effect was evaluated using alumina powder #100 and glass beads #100, changing the air pressure from 2.0 to 4.5 atm at 0.5 atm intervals. The other experimental conditions were identical to those described previously.

## Statistical Analysis

The correlation between abraded depth, particle size and abraded depth, and air pressures was analyzed by correlation coefficient. The statistically significant differences among abraded depth of enamel, dentin, and caries-model dentin were determined with a two-way analysis of variance (ANOVA).

## SEM Observation

The enamel, dentin, and caries-model dentin abraded with polycarbonate resin #60 were fixed overnight in 10% neutral buffered formalin at pH 7.4 and dehydrated in ascending grades of ethanol followed by freeze drying to critical point (EID-2, Elionix, Tokyo, Japan). They were sputter-coated with gold and observed under an SEM (JXA-840, JEOL Ltd, Tokyo, Japan).

## Removal of Human Carious Tissue

To visualize the effect of selective caries removal, dentin disks (thickness of 2.0 mm) with normal and carious enamel and dentin were cut from freshly extracted human teeth, with moderate occlusal caries, using a diamond microtome (Leitz 1600 Saw Microtome, Ernst Leitz GmbH, Wetzlar, Germany).

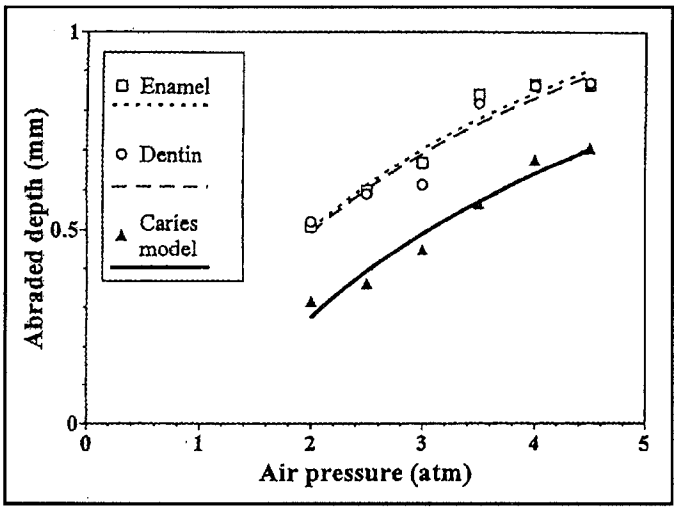



Figure 6. *Correlation between air pressures and abraded depths using alumina powders #100*

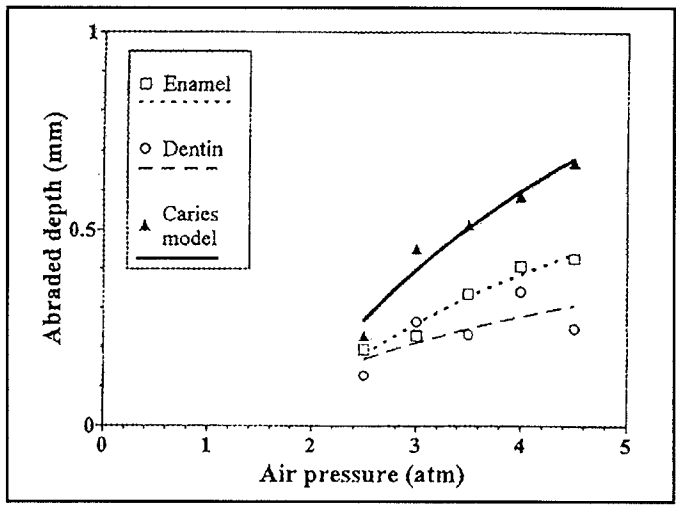



Figure 7. *Correlation between air pressures and abraded depths using glass beads #100. Since abraded depth at 2.0 atm was too low to determine, the data were excluded.*

Outer carious dentin was stained with a caries-disclosing dye solution (Caries Detector, Kuraray Co, Ltd, Osaka, Japan) (Fusayama, 1980) and photographed. The disks were then abraded with polycarbonate resin #60 until carious dentin was removed, then the disk was stained with dye solution, and again photographed.

## RESULTS

The effect of air-abrasive particles on abraded depths is graphically presented in Figure 5. With alumina powders, the abraded depth of enamel ($r = 0.93$; $P < 0.05$), dentin ($r = 0.94$; $P < 0.05$), and caries-model dentin ($r = 0.98$; $P < 0.05$) increased significantly as the particle size increased (Table 2). There was no significant difference in abraded depth between enamel and dentin. The caries-model dentin showed significantly less depth than either enamel or dentin (two-way ANOVA, $P < 0.05$).

Using glass beads, the abraded depth of enamel ($r = 0.86$; $P < 0.05$) increased significantly as the particle size increased. However, this was not observed for dentin ($r = 0.41$) and the caries-model dentin ($r = 0.18$)(Table 2)(Figure 5). There was no significant difference in depth of abrasion between enamel and dentin when using similar abrasive particle sizes, but the caries-model dentin showed significantly greater depth than enamel and dentin (two-way ANOVA; $P < 0.05$).

The average abraded depth of enamel (1.24 mm) and dentin (1.35 mm) with crushed glass powder (angular shape) was approximately three times greater than with glass beads #80 (enamel 0.38 mm, dentin 0.28 mm) (spherical shape), although both particles were of similar size. The abraded depth of the caries-model dentin (1.12 mm) was similar to that of enamel (1.24 mm) and dentin (1.35 mm) (Figure 5).

Crushed powders of polycarbonate resin #80 and #60 hardly reduced enamel and dentin, and abraded only the caries-model dentin. Abraded depth of the caries-model dentin with #60 was the greatest of all, and was significantly deeper than with #80 (Figure 5).

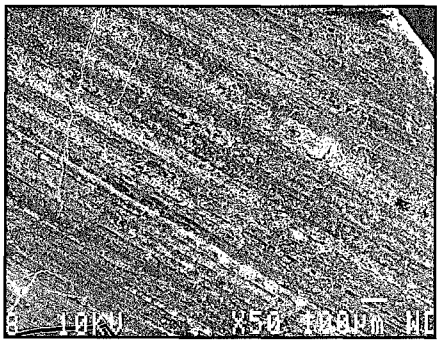
The effect of air pressure on the abraded depth with alumina powder #100 is shown in Figure 6. The abraded depth of enamel ($r = 0.94$; $P < 0.05$), dentin ($r = 0.91$; $P < 0.05$), and caries-model dentin ($r = 0.96$; $P < 0.05$) increased significantly as the air pressure increased (Table 2). The abraded depth of the caries-model dentin was significantly less than that of enamel and dentin (two-way ANOVA; $P < 0.05$).

When the glass beads were used, the abraded depth of enamel ($r = 0.96$; $P < 0.05$) and caries-model dentin ($r = 0.96$; $P < 0.05$) increased as the air pressure increased (Table 2) (Figure 7). The caries-model dentin showed significantly greater abraded depth than enamel and dentin (two-way ANOVA; $P < 0.05$).

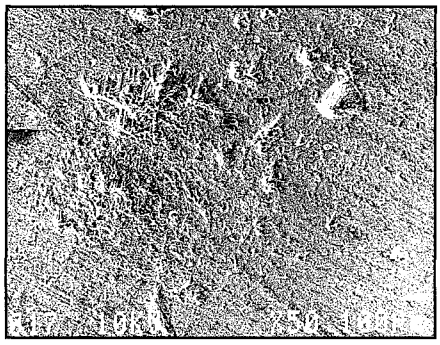
Figure 8 shows SEM pictures of the surfaces abraded with polycarbonate resin #60 particles. Only slight cutting effects were observed on enamel and dentin. However, the caries-model dentin was definitely removed, leaving a hollow cavity.

The effect of air abrasion using polycarbonate resin #60 is presented in Figure 9. The stainable layer (outer carious dentin) was completely removed by air abrasion, and transparent dentin still remained in the cavity floor. The entire procedure took about 30 seconds. Even with additional abrasive blasts, no additional abrasion was noted.

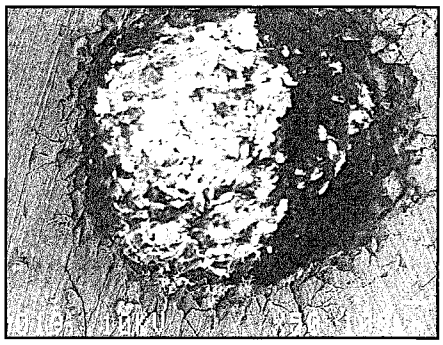
Figure 8. *SEM photographs of abraded surfaces with polycarbonate resin #60; magnification X50*



**A.** *Enamel surface: picture showed no abraded scar*



**B.** *Dentin surface: a few abraded marks were shown*



**C.** *Caries-model dentin surface: specimen was abraded, leaving hollow cavity.*

## DISCUSSION

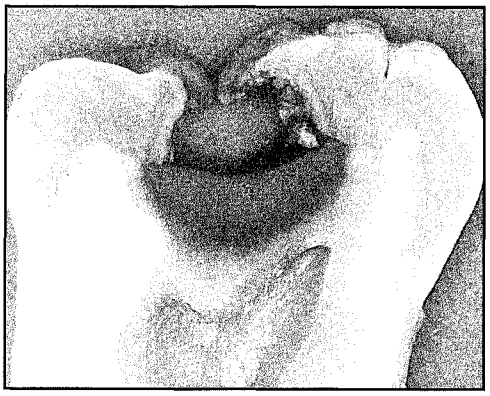
Air abrasion is essentially a cutting action by numerous abrasive particles. To obtain maximum cutting efficiency, the particle should be hard enough to indent the substrate it abrades, and irregular in shape with a sharp cutting edge. Round and smooth particles possess poor abrasive properties (Phillips, 1991). In the present study, these basic principles in abrasion of tooth substance were reconfirmed.

Comparing glass beads #80 (spherical shape) to crushed glass powders (angular shape), which had similar composition and particle size, the angular-shaped particles reduced intact enamel and dentin three times more than using spherical particles. Comparing alumina powders #80 to crushed glass powders with similar particle size and shape, the harder and heavier alumina powders reduced intact enamel and dentin more than the glass powders.

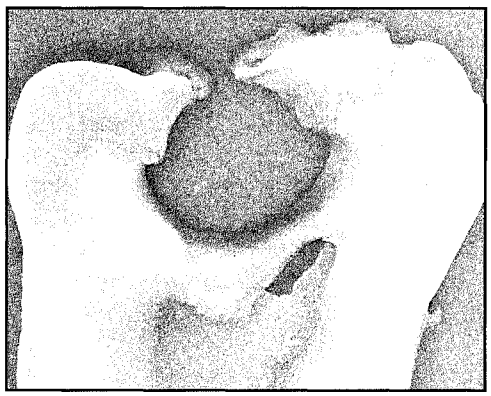
Particle size and air pressure were found to have a significant effect on abrasion power. Abraded depth of intact enamel and dentin with both alumina powders and glass beads increased as the particle size and air pressure increased. Increased air pressure provided an increased number and velocity of the particles. Abrading power must be proportional to kinetic energy of the particles, which is the function of mass and velocity of the particle.

Although microhardness of intact enamel and dentin was reported to be 300 and 70 Hv respectively (Ryge, Foley & Fairhurst, 1961), air abrasion was not sensitive to this substrate difference. However, caries-model dentin behaved differently from intact enamel and dentin using the air-abrasion technique, when using alumina powders and glass beads. Angular alumina powders and crushed glass powders

Figure 9. *Carious tooth before and after abrasion with polycarbonate resin #60*



**A.** *Before abrasion—outer carious dentin was stained with the caries detector.*



**B.** *After abrasion—outer carious dentin and carious enamel were completely removed; however, inner carious dentin (transparent dentin) remained. Pulp tissue was partially destroyed during sample preparation.*

reduced caries-model dentin less than intact enamel and dentin, whereas spherical glass beads abraded the caries-model dentin significantly more than intact enamel and dentin. Caries-model dentin used in the present study was completely demineralized human dentin, a soft tissue, in contrast to the brittle enamel and dentin. Its microhardness was too low to be determined with a microhardness tester. Kinetic energy of the particle must be the source of cutting by air abrasion. In addition, angular particles, which hit brittle enamel and dentin surface, cut into them and chip them. In contrast to this, the angular particles might penetrate the soft tissue of the caries-model dentin, losing some of their kinetic energy by the cushioning effect between the particles and the tissue. This would result in less destruction of soft-caries model dentin than that of brittle intact enamel and dentin. On the other hand, spherical particles with dull and smooth surfaces cannot penetrate deep into soft tissue. Their kinetic energy might be mainly lost through deformation and destruction of the soft tissue surface without losing their energy by penetration into the tissue.

Although it was interesting to note that the spherical particles of glass beads abraded carious dentin more than intact enamel and dentin, unlike angular alumina powders and crushed glass powders, these particles abraded intact enamel and dentin with all particle sizes and air-pressure levels tested in the present study.

For selective removal of carious dentin using air abrasion, abrasive particles are needed that can reduce only carious dentin without damaging intact enamel and dentin. In the present study, crushed powder of polycarbonate resin was the only particle that abraded caries-model dentin without reducing intact enamel and dentin. Polycarbonate resin (Hv = 40-50) has a similar hardness to human dentin (Hv = 70), and is much harder than the caries-model dentin. This meant that the particles were too soft for cutting intact enamel and dentin, but hard enough to cut caries-model dentin. Two particle sizes #60 (mean diameter = 338 μm) and #80 (mean diameter = 215 μm) were tested. Although both of them only reduced the caries-model dentin, the #60 particles showed significantly greater abraded depth than the #80 particles. SEM observation of both particles revealed that the #60 particles were much more angular and had greater sharpened edges than the #80 particles, which displayed crushed irregular surfaces with less sharp cutting edges (Figure 2). Abrasion power of polycarbonate resin also seemed to be particle size- and shape-dependent.

Selective removal of carious dentin was confirmed using extracted human teeth with moderate dentinal caries. The natural dentinal caries was comprised of outer and inner carious dentin (Fusayama, 1980). The outer layer, being demineralized and infected, was selectively stained by the caries detector. The inner layer was intermediately demineralized and uninfected. The middle and bottom parts of this layer were transparent dentin (Ogawa & others, 1983). Polycarbonate resin #60 could remove only the outer carious dentin with minimal effect on the inner dentin. This technique exhibited the potential for simplifying caries removal, thereby shortening the patient treatment time.

The present study found one method for selective caries removal by using angular particles with similar hardness to intact dentin. Further studies are required to determine the optimum particle size, shape, and optimum pressure level for performing the caries-removal operation. Any material having similar hardness to dentin might be an acceptable candidate for use as an abrasive particle for selective caries removal using the air-abrasive technique.

## CONCLUSION

Conditions of selective caries removal using the air-abrasive technique were studied by changing the size and shape of various particles, as well as varying the air pressures, with the following results:

1. The cutting efficiency of air abrasion depended on the size, shape, hardness, and density of the abrasive particles and air pressure;
2. For alumina powders and glass beads, which are harder than intact tooth substance, it was difficult to achieve selective caries removal even by changing particle size, shape, and air pressure;
3. Selective caries removal was found to be most effective by using particles with similar hardness to intact dentin, such as crushed powders of polycarbonate resin.

### Acknowledgments

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# Evaluation of Acidic Primers in Microleakage of Class 5 Composite Resin Restorations

V V GORDAN • M A VARGAS
D S COBB • G E DENEHY

**Clinical Relevance**
The results of this in vitro study indicate that both Clearfil Liner Bond 2 and Denthesive II may be used with or without etching on both enamel and dentin to restore class 5 cavity preparations.

## SUMMARY

**The purpose of this in vitro study was to evaluate the marginal seal of two adhesive systems on class 5 composite resin restorations. Two adhesive systems that use acidic primers were used in this study: Clearfil Liner Bond 2 and Denthesive II. Scotchbond Multi-Purpose was used as a control. Class 5 cavity preparations were made at the CEJ (half of the preparation was in enamel and half in dentin/cementum) of 60 extracted human molars. Both systems were used with and without etching. The control group was used with etching only. Composite resin restorations were placed, light cured for 40 seconds, and polished. All specimens were thermocycled 500 times. The specimens were immersed in erythrosin B, tetra-iodo-fluorescent 2% solution, then sectioned to obtain 600 mm-thick sections. These were examined under X13.2 magnification, and the degree of dye penetration was measured in microns. A chi-square test demonstrated no significant differences in microleakage among groups for either dentin ($P = 0.54$) or enamel ($P = 0.35$).**

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## INTRODUCTION

Microleakage, the marginal permeability to bacterial, chemical, and molecular invasion at the interface between the teeth and the restorative materials, remains a problem with composite resin restorations. Microleakage at the enamel/restoration interface has been practically eliminated since the introduction of acid etching by Buonocore in 1955 (Prati & others, 1990; Tani & Buonocore, 1969; Retief, 1989), but sealing of dentin margins still remains a challenge, (Retief & Denys, 1989; Rigsby & others, 1992).

Microleakage is the result of breakdown of the tooth/restoration interface, which may cause discoloration and recurrent caries, which in turn could lead to possible pulpal pathology (Brännström, 1984). Marginal breakdown has been attributed to many factors, including differences in the coefficients

of thermal expansion between the tooth structure and the restorative material, polymerization shrinkage of the resin materials, and inadequate adhesion to dentin (Anusavice, 1989; Feilzer, DeGee & Davidson, 1988; Philips, 1965; Davidson, DeGee & Feilzer, 1984).

Conditioners used with the current generation of bonding agents are generally designed to remove the smear layer and demineralize the tooth surface. When enamel is conditioned, the inorganic component is removed, creating microporosities (Buonocore, 1955). When the dentin is conditioned, the superficial dentin is decalcified, leaving a rich organic area, primarily collagen. Primer materials used after, or in conjunction with, acid conditioners are essential for achieving good wetting and penetration through the demineralized area (Nakabayashi, 1991).

The primer contains a hydrophilic part that has affinity for both the organic and inorganic component of dentin, and a hydrophobic part that has affinity for the adhesive resin. It also has good wetting characteristics that allow it to penetrate through the collagen (Nakabayashi, 1991). According to Nakabayashi (1991), the hydrophilic primer diffuses into the moist demineralized dentin surface to combine with the collagen and the hydroxyapatite at the front of the demineralization. Subsequently, an adhesive resin is applied that fills the spaces of the demineralized surface and forms a new material that is part tooth and part resin, known as the hybrid layer (Nakabayashi, Kojima & Masubara, 1982).

A modern approach to dentin adhesion is the use of acidic primers, also called self-etching primers. These primers simplify clinical adhesive procedures by combining the acid conditioning of dentin with the priming step. Thus, with self-etching dentin primers, the acid and the primer are combined in one solution to form an acidic monomer (Chigira & others, 1989).

Theoretically, in these systems, the acidic part of the primer dissolves and incorporates the smear layer into the mixture, as it demineralizes the dentin and encapsulates the collagen fibers and hydroxyapatite crystals (Nishida & others, 1993). Besides simplification, the rationale behind these systems is to superficially demineralize dentin and simultaneously penetrate it with monomers, which can be polymerized in situ (Sano & others, 1994).

The purpose of this in vitro study was to evaluate the marginal seal of two adhesive systems that contain acidic primers, with and without etching, on class 5 composite resin restorations.

## METHODS AND MATERIALS

Sixty extracted human molars free of visible caries were selected for this study. The ages of the patients were not identified, and the reasons for extraction ranged from periodontally compromised teeth to impacted teeth. The teeth were cleaned of gross debris and stored in 0.5% chloramine solution, except for 24 hours before beginning the experiment, when they were kept in distilled water.

A class 5 cavity preparation was cut on each buccal surface, centered at the cementoenamel junction using a # 57 carbide bur at high speed with adequate water cooling. Each bur was replaced every five cavity preparations. Cavity dimensions were 3 mm wide, 2 mm high, and 1.5 mm deep. The preparations were located half in enamel and half in dentin/cementum so that the occlusal margin was in enamel and the gingival was in dentin/cementum. The occlusal wall on enamel was beveled (45°, 0.5 mm) with a # 7901 finishing bur. After preparation, the teeth were randomly assigned to five groups of 12 specimens each. The bonding systems used were: Clearfil Liner Bond 2 (J Morita USA Inc, Tustin, CA 92680), Denthesive II (Heraeus Kulzer, Irvine, CA 92718), and as a control, Scotchbond Multi-Purpose (3M Dental Products, St Paul, MN 55144). All adhesive systems were used according to the manufacturers' recommendations, and in addition, Clearfil Liner Bond 2 and Denthesive II groups were used with Ultraetch (35% phosphoric acid, Ultradent Products, Inc, South Jordan, UT 84095) for 15 seconds prior to the primer application.

The preparations were restored with a microfill composite resin, Silux Plus XL (3M Dental Products), in two increments with the occlusal increment placed first. Each composite increment was cured for 40 seconds with an Optilux 401 visible-light-curing unit (Demetron Research Corp, Danbury, CT 06810). The output of the light unit was measured with a curing radiometer (Demetron Research Corp) every five restorations to ensure a constant value of at least 380 $mW/cm^2$. The restorations were finished with a football finishing bur and Sof-Lex disks (3M Dental Products), and polished with Vivadent Politip-P cups (Ivoclar North America, Amherst, NY 14228). To prevent any possible leakage, the apices of the teeth were sealed with cold-cure acrylic resin (Shure-Tray, Heraeus Kulzer).

The samples were coated with an acid-resistant varnish except for the restorations and a 1-mm rim of tooth structure around the restorations. The specimens were placed in distilled water at 37 °C for 48 hours and then thermocycled 500 times in a thermocycling machine (Type E52 #72179, Haake, Lichterfelde, Berlin, Germany). The cycles consisted of 26 seconds in baths of 5° ± 5 °C and 55° ± 5 °C with an exchange time of 13 seconds between baths.

After 48 hours, the specimens were immersed in erythrosin B, tetra-iodofluorescent 2% solution (Harleco, Philadelphia, PA 19143) for 24 hours. Following removal from the solution, the specimens were rinsed in tap water for 5 minutes to remove excess dye.

The samples were sectioned buccolingually with a Silverstone-Taylor hard tissue microtome (Sci-Fab, Littleton, CO 80026) to obtain four sections of approximately 600 microns. All sections were examined under a light microscope at X13.2. The section with the deepest dye penetration was considered representative of the sample and photographed. The photographs were later projected onto a digitizing table, and dye penetration was measured from the cavosurface margin into the resin/dentin or resin/enamel interface with values converted to microns.

A chi-square test was used to detect differences in microleakage among groups.

## RESULTS

A chi-square test for enamel microleakage ($P = 0.35$) revealed no differences among groups. Leakage between the enamel and the composite resin was found in only one specimen (Clearfil Liner Bond 2/ without etching; 200 mm leakage).

Similarly, the chi-square test (Table 1) for dentin microleakage ($P = 0.54$) demonstrated no significant difference among groups. Mean microleakage for dentin groups is presented in Table 2. The mean microleakage was highest for Denthesive II/without etching (67.08mm), decreasing among groups in the following order: Clearfil Liner Bond 2/with etching, followed by Denthesive II/with etching, followed by Scotchbond Multi-Purpose/with etching, and lowest in Clearfil Liner Bond 2/without etching (2.33mm).

*Table 1. Incidence of Microleakage--Dentin*

| Bonding Agents | n | No Leakage | Leakage |
|---|---|---|---|
| Denthesive II/No Etch | 12 | 8 | 4 |
| Clearfil Liner Bond 2/Etch | 12 | 8 | 4 |
| Denthesive II/Etch | 11 | 9 | 2 |
| Scotchbond MP | 12 | 10 | 2 |
| Clearfil Liner Bond 2/No Etch | 11 | 10 | 1 |

Chi-square value = 3.110; $P = 0.54$.

## DISCUSSION

Conventional adhesive systems use three different steps involving three different agents (a conditioning agent, a primer solution, and an adhesive resin) to accomplish the goal of bonding resin materials to dentin. A unique characteristic of the Clearfil Liner Bond 2 system is that it combines the conditioner and priming agent into a single acidic primer solution for simultaneous use on both enamel and dentin. Combining conditioning and priming into a single treatment step results in improvement in both time and cost-effectiveness.

*Table 2. Mean Microleakage (μm)--Dentin*

| Bonding Agents | n | Mean | SD | Max | Min |
|---|---|---|---|---|---|
| Denthesive II/No Etch | 12 | 67.08 | 122.89 | 375.0 | 0 |
| Clearfil Liner Bond 2/Etch | 11 | 48.18 | 135.41 | 450.0 | 0 |
| Denthesive II/Etch | 12 | 16.25 | 25.14 | 70.0 | 0 |
| Scotchbond MP | 11 | 9.09 | 30.15 | 100.0 | 0 |
| Clearfil Liner Bond 2/No Etch | 12 | 2.33 | 6.02 | 20.0 | 0 |

The conventional three-step bonding systems work by removing the smear layer, followed by application of primer and adhesive. Some questions have been raised about the possibility of the primer not completely reaching the demineralized zone, resulting in gaps (Van Meerbeek & others, 1992). The idea of the acidic primer is attractive because in theory this system decalcifies the inorganic component and infiltrates the collagen fibers at the same time, minimizing the potential for voids or discrepancy between the demineralized surface and the primer penetration.

According to Nishida and others (1993), acidic primers work by dissolving the smear layer and demineralizing the inorganic component of the dentin. The pH of the primer may affect its penetration and dissolution into the dentin. In the Clearfil Liner Bond 2 bonding system, after the LB primer A and LB primer B are mixed, the pH of the resulting solution is 1.4 (Barkmeier, Los & Triolo, 1995). Denthesive II has a pH between 2.5 and 3.3 after being mixed (personal communication with the company), and Scotchbond Multi-Purpose has a pH between 2.9 and 4 (personal communcation with the company).

The goal of adhesion with acidic primers is to obtain thorough infiltration of resin monomer through the smear layer while simultaneously demineralizing and infiltrating the dentin to form the hybrid layer (Nakabayashi, Nakamura & Yasuda, 1991). There are two ways to promote and enhance this adhesion to dentin. The first is to improve the impregnation of monomer into the substrate, and the second is to increase the diffusivity or the ability to penetrate into the dentinal substrate. HEMA used in combination with dentin adhesives was

found to improve wettability and hydrophilicity and to increase the bond strength of adhesive resin to teeth (Pashley, Horner & Brewer, 1992). All the dentin bonding systems used in the present study contain a wetting agent, hydroxyethylmethacrylate (HEMA), in either the primer composition (Denthesive II) or in both primer and adhesive composition (Clearfil Liner Bond 2 and Scotchbond Multi-Purpose), which acts as a bifunctional resin (Table 3). The hydroxyethyl portion of the molecule is hydrophilic and aids in establishing a dentin bond; the methacrylate component is hydrophobic and co-polymerizes with the BIS-GMA adhesive resin (Nakabayashi, 1991).

According to Yoshiyama and others (1996), in an SEM observation of Clearfil Liner Bond 2, the use of 20% Phenyl-P in HEMA seems to limit the depth of demineralization to slightly more than 1 μm in normal dentin and even less in sclerotic dentin. As the self-etching primer demineralizes the dentinal surface, the concentration of calcium and phosphates increases, thus neutralizing the primer and limiting further dissolution of apatite (Wang & Hume, 1988).

The presence of acetone or alcohol in the primer solution increases the diffusivity into dentin. The action of acetone or alcohol is to seek out the moisture in the dentinal tubules. This moisture, in effect, pulls the acetone into and between the tubules, taking with it the resin (Gwinnett & Kanca, 1992; Miller, Mabrito & Castellanos, 1993). The acetone- or alcohol-based primer, upon contacting the moisture in dentin, raises the boiling point of the acetone and lowers the boiling point of the water. This behavior is known as azeotrophism. The alcohol and the moisture then vaporize from the substrate, leaving the resin behind. Both Clearfil Liner 2 and Denthesive II have alcohol as a solvent in their composition.

In an in vitro study by Barkmeier and others (1995) no microleakage was observed in either enamel or dentin when the Clearfil Liner Bond 2 adhesive system was used. Similarly, under the conditions of this study, no difference in enamel microleakage among products nor among groups was demonstrated. For dentin, the highest mean microleakage was found using Denthesive II without etching (Table 2), although differences were not significant. In a previous study, Gordan and others (1997) found that Denthesive II presented inconsistent dentin shear bond strength, which was reflected in low bond strength and high standard deviation values when this product was used without etching. The high standard deviation obtained in the current study is due to the fact that few samples leaked (Table 1).

The critical area of adhesive restoration is the interface between composite resin and dentin or cementum. While bond strength of composite resin to enamel has achieved predictably high levels (O'Brien, Watts & Read, 1991), bonding to dentin has been inconsistent (Nakabayashi & others, 1991; Fortin & others, 1994). Enamel is primarily inorganic in nature, about 95% (Sturdevant & Pashley, 1989), as opposed to dentin, which is only 75% inorganic (Scott & Symons, 1974). The difference in composition between the enamel and the dentin may have accounted for the fact that the dentin side showed more leakage than the enamel side.

*Table 3. Composition of the Bonding Systems*

| Bonding System | Conditioner | Primer A | Primer B | Adhesive |
|---|---|---|---|---|
| Clearfil Liner Bond 2 (J Morita) | Not recommended | Phenyl-P, 5-NMSA, N,N-diethanol, p-toluidine, dl-camphoro-quinone, ethyl alcohol | HEMA, hydrophobic dimethacrylate, water | MDP, BIS-GMA, HEMA, silanated colloidal silica, dl-camphoroquinone, N,N-diethanol p-toluidine, hydrophobic dimethacrylate |
| Denthesive II (Heraeus Kulzer) | Not recommended on dentin | Maleic acid 2.0% | HEMA, hydroxypropyl-methacrylate, maleic acid-mono-2-methacryl-oyloxyethylester | TEG-DMA, BIS-GMA, maleic acid-mono-2-methacryloyloxy-ethylester |
| Scotchbond Multi-Purpose (3M Dental Products) | 35% $H_3PO_4$ | HEMA, light-cured polymer, water | | BIS-GMA, HEMA, photoinitiator |

Higher microleakage values were found in in vitro studies as opposed to in vivo studies (Ferrari & others, 1993, 1994). A study by Barnes and others (1993) also demonstrated that laboratory microleakage tests of thermocycled resin restorations produced more leakage than that found in clinical restorations.

A study by Kanca (1989) showed that microfills tend to exhibit less leakage than hybrids. The shrinkage stress associated with a microfilled composite resin is less than that associated with a high elastic modulus hybrid composite resin. A highly filled system would stress the bonding resin to a higher degree during polymerization, thereby increasing the chance of increased dentin leakage.

It has been suggested that the time immediately following placement of a restoration is critical, since enamel and dentin bonding must counteract composite shrinkage (Davidson & DeGee, 1984). The clinical failure of a restoration, marginal discoloration, bacterial penetration through the dentin tubules, and pulp damage may occur during the first minutes after placement of composite (Prati & others, 1990). In the current study, the thermocycling time, in addition to the 48 hours' delay before specimen testing, controlled these factors. Although thermocycling time varies among studies, research by Burger, Cooley, and García-Godoy (1992) and Crim & García-Godoy (1987) has demonstrated that thermocycling time is not a significant variable.

## CONCLUSIONS

The purpose of this in vitro study was to evaluate the marginal seal of two adhesive systems that contain acidic primers, used with and without etching, on class 5 composite resin restorations. The following conclusions were determined:

1. Both Clearfil Liner Bond 2 and Denthesive II used without etching produced marginal seals comparable to Scotchbond Multi-Purpose in both enamel and dentin.
2. Both Clearfil Liner Bond 2 and Denthesive II used with etching produced marginal seals comparable to Clearfil Liner Bond 2 and Denthesive II used without etching in both enamel and dentin.

Future research involving longer-term clinical analysis of acidic primers, SEM examination, shear bond strength tests, and clinical trials are important to substantiate the clinical relevance of the results of the current study.

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# Effect of Etchant, Etching Period, and Silane Priming on Bond Strength to Porcelain of Composite Resin

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**Clinical Relevance**
Etching feldspathic porcelain with hydrofluoric acid for more than 30 seconds followed by silane priming considerably enhanced bond strength of composite resin to the porcelain.


## SUMMARY

**The purpose of this study was to evaluate the effect of etching and silane priming on bond strength to a feldspathic porcelain (VMK 68) of a composite resin (Clearfil APX). Two hydrofluoric acid etchants (2.5% and 5%) and seven different etching times (0, 30, 60, 90, 120, 150, and 180 seconds) were used to etch the porcelain specimens respectively. A self-curing bonding agent containing a silane coupler (Clearfil Porcelain Bond) was used on both etched and unetched porcelain surfaces. Etched relief patterns were observed by means of a scanning electron microscope, and the bond strengths between the photocured composite resin and the porcelain were determined. Scanning electron micrographs revealed complicated etching patterns with increased etching time periods. Shear testing results showed that the bond strength to the unetched porcelain of the composite resin was very low, and that etching periods for more than 30 seconds effectively enhanced the bond strength. Of the two etching agents applied to the unsilanated porcelain, the buffered 2.5% etchant produced higher bond strengths than the 5% etchant for all etching time periods except for 180 seconds. Silane priming was effective and critical for improving bond strength to the porcelain. Application of the silane bonding agent to the porcelain after hydrofluoric acid etching appeared to be suitable for achieving consistent bonding between the composite resin and the porcelain.**



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## INTRODUCTION

The technique of bonding composite resin or resin cement to porcelain restorations has made it possible to repair intraoral fractures of metal-ceramic restorations that occasionally occur in practice (Eames &

others, 1977). This technique also allows seating of all-ceramic restorations such as inlays, onlays, and laminate veneers onto tooth structure (Rochette, 1975; Horn, 1983; Calamia, 1985; Jensen, 1988).

Horn (1983) and Calamia (1985) established that although other acids can be used as feldspathic porcelain etchants, the best results were obtained with the shortest etch time when using hydrofluoric acid. Grinding and air abrasion (Bertolotti, Lacy & Watanabe, 1989; Appeldoorn, Wilwerding & Barkmeier, 1993; Suliman, Swift & Perdigao, 1993; Kupiec & others, 1996), whether or not combined with acid etching, have subsequently been used on the bonding surface of ceramic restorations to create a mechanically retentive surface. The etchant concentrations and times used for etching ceramic materials are important factors for obtaining sufficient bond strength.

Although extensive research on porcelain bonding has been reported, limited information is available concerning the contribution, if any, of combinations of etchant, etching period, and silane priming on the repair bond strength of composite resin to porcelain. The purpose of this study was, therefore, to evaluate the effect of different etching agents, etching times, and the combined use of etching and silane priming on the bond strength of a composite resin to a feldspathic porcelain.

## METHODS AND MATERIALS

### Shear Testing

A feldspathic porcelain designed for metal-ceramic restorations (VMK 68, 542 dentin A3, Vita Zahnfabrik GmbH, Bad Säckingen D-7889, Germany) was selected for the substrate material. Two hundred and eight porcelain disks (10 mm in diameter by 2.5 mm thick) were vacuum-fired in cylindrical refractory investment molds by means of a computerized furnace (Commodore 75 VPF, Jelenko/Morita, Suita Osaka 564, Japan) and the disks were finished with 600-grit wet abrasive paper.

Figure 1 is a diagram illustrating specimen preparation. The porcelain disks were divided into three groups. One was an unetched control group of 16

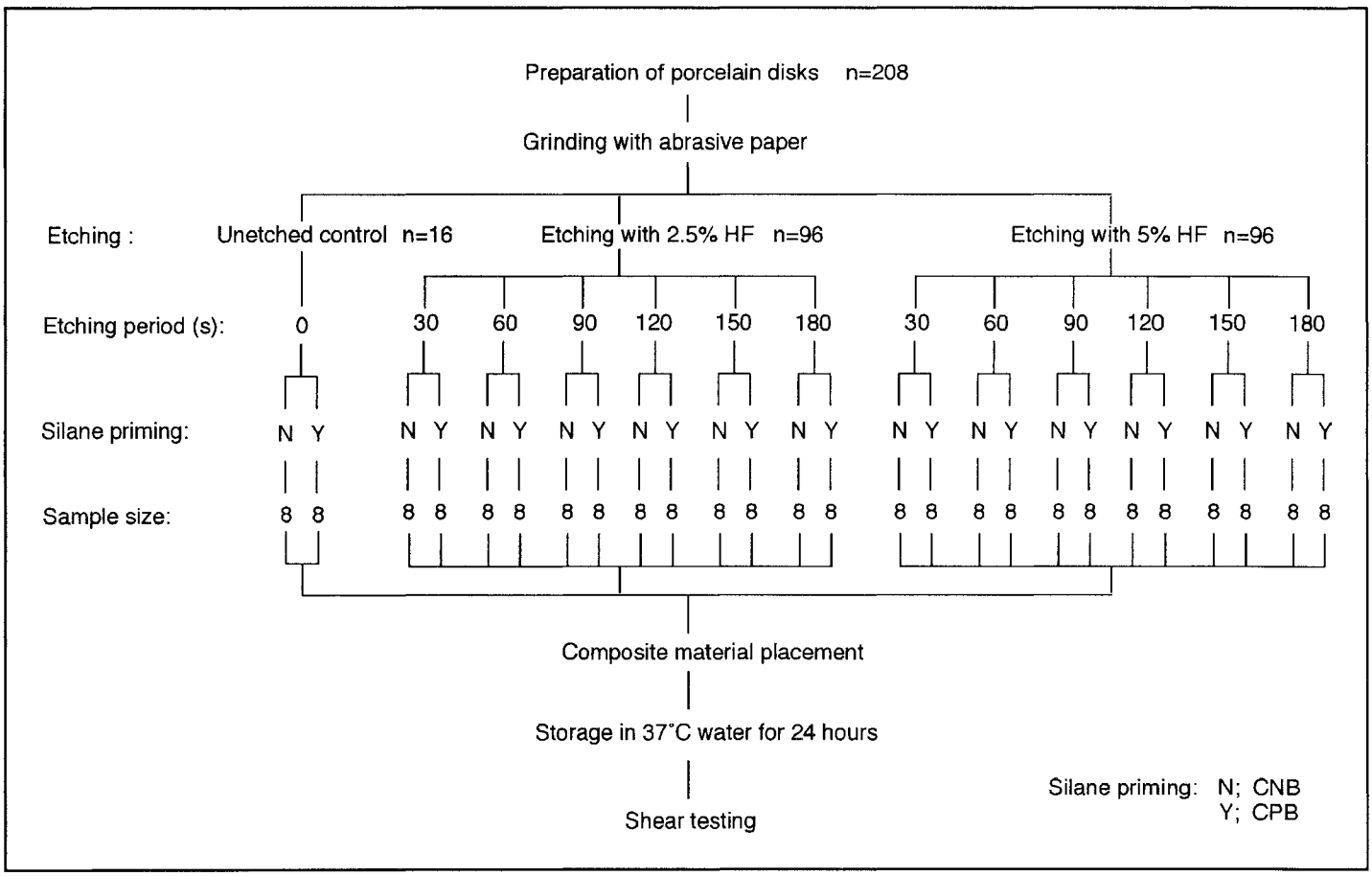



Figure 1. *Diagram illustrating specimen preparation*

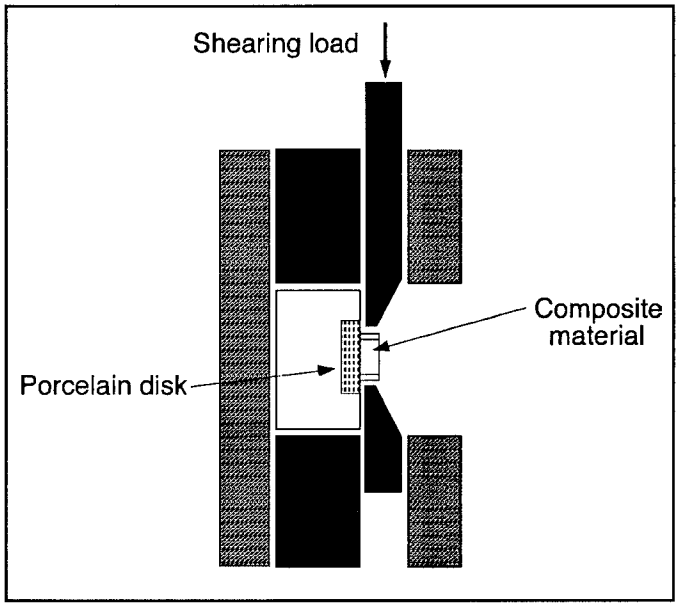



Figure 2. *Assembly used for determination of shear bond strength*

specimens, while the other two were porcelain etchants with different hydrofluoric acid (HF) concentrations (buffered 2.5% HF, PorceLock Porcelain Etching Solution, Den-Mat Corp, Santa Maria, CA 93456; and 5% HF, GC Hydrofluoric Acid Gel, GC Corp, Tokyo 113, Japan). The 96 disks in each of the two experimental groups corresponding to the two etchants were further divided into subgroups according to etching time (30, 60, 90, 120, 150, and 180 seconds). Each of the 12 subgroups therefore consisted of two sets of eight disks. The 16 specimens in each subgroup were etched for the same time periods. After etching, specimens were washed with tap water, ultrasonically cleaned in a water bath, and air-dried. A piece of double-coated tape with a circular hole 5 mm in diameter was positioned on each porcelain disk to define the area of bond.

Half of the unetched control disks (eight disks) and half of the etched disks (12 sets of eight disks) were treated with a two-liquid self-curing bonding agent that did not contain silane (Clearfil New Bond, J Morita USA Inc, Tustin, CA 92680), whereas the remaining disks were treated with a three-liquid silane bonding agent (Clearfil Porcelain Bond, J Morita USA Inc). After application of both bonding agents, the surface of each specimen was lightly dried with compressed air, and a brass ring (6 mm in inside diameter by 2 mm long and 1 mm-thick wall) was placed on the tape to surround the 5-mm-in-diameter area of primed surface. The ring was filled with a photo-activated composite restorative material (Clearfil APX, shade A3, J Morita USA Inc) and exposed to visible radiation for 40 seconds by a high-intensity photo-curing unit (Quick Light, J Morita Corp, Suita Osaka 564, Japan).

After 30 minutes of preparation, specimens were stored in 37 °C water for 24 hours. Each specimen was then embedded in an acrylic mold with a self-curing acrylic resin and mounted in an ISO/TR 11405 shear testing jig (Figure 2). Shear bond strength was determined by means of a universal testing device (DCS-500, Shimadzu Corp, Kyoto 603, Japan) at a cross-head speed of 0.5 mm/min following the procedure described by Matsumura and others (1989). For each condition, the shear bond strength mean and standard deviation of eight tests were calculated.

The values of each group were compared by three-way analysis of variance (ANOVA). The three factors analyzed were etchant, etching period, and bonding agent. After the three-way ANOVA, further ANOVA and Duncan new multiple range intervals were performed ($P < 0.05$).

*Table 1. Shear Bond Strength (MPa ± SD); 2.5% HF Etchant*

| Etching Periods | Bonding Agents | | | |
|---|---|---|---|---|
| | CNB | | CPB | |
| 0 (control) | 3.0 ± 0.4 | | 25.2 ± 4.2 | a |
| 30 | 30.2 ± 4.1 | b,c | 36.0 ± 4.9 | e |
| 60 | 28.1 ± 4.0 | a,b | 36.4 ± 4.6 | e |
| 90 | 30.5 ± 4.4 | b,c,d | 35.0 ± 5.1 | d,e |
| 120 | 25.3 ± 3.7 | a | 35.0 ± 5.1 | d,e |
| 150 | 35.4 ± 4.4 | e | 37.4 ± 6.1 | e |
| 180 | 26.5 ± 3.4 | a,b | 33.4 ± 4.2 | c,d,e |

n = 8; CNB = Clearfil New Bond; CPB = Clearfil Porcelain Bond (contains a silane coupler). Letters indicate values that are not significantly different ($P > 0.05$).

## Microscopic Observation

Porcelain disks were prepared and etched according to each of the procedures described above for the different shear testing groups. Disks were desiccated, sputter-coated with gold, then observed with a scanning electron microscope (SEM, S-520, Hitachi Corp, Chiyoda, Tokyo 101, Japan) operated at 20 kV.

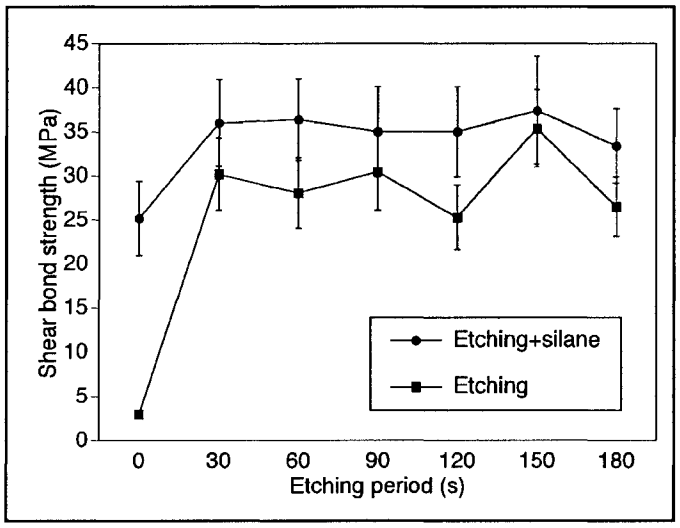



Figure 3. *Shear bond strengths of 2.5% HF etchant subgroups*

*Table 2. Shear Bond Strength (MPa ± SD); 5% HF Etchant*

| Etching Periods | Bonding Agents CNB | CPB |
|---|---|---|
| 0 (control) | 3.0 ± 0.4 | 25.2 ± 4.2 c |
| 30 | 18.8 ± 4.0 a | 32.9 ± 5.5 d,e |
| 60 | 19.7 ± 1.6 a,b | 31.3 ± 4.4 d,e |
| 90 | 21.9 ± 2.3 a,b,c | 31.5 ± 3.2 d,e |
| 120 | 19.9 ± 2.8 a,b | 32.2 ± 4.9 d,e |
| 150 | 30.0 ± 2.3 d | 30.5 ± 3.2 d,e |
| 180 | 23.4 ± 4.5 b,c | 34.3 ± 4.6 e |

n = 8; CNB = Clearfil New Bond; CPB = Clearfil Porcelain Bond (contains a silane coupler). Letters indicate values that are not significantly different ($P > 0.05$).

## RESULTS

Three-way ANOVA performed on the shear testing results revealed that bond strengths were influenced by bonding agent ($F = 298.6$; $P = 0.0001$), etchant ($F = 77.6$; $P = 0.0001$), and etching period ($F = 6.2$; $P = 0.0001$). Bond strengths were also affected by the combinations of: 1) etchant and etching period; 2) etchant and bonding agent; and 3) etching period and bonding agent ($P < 0.05$), whereas they were not affected by the combination of etchant, etching period, and bonding agent ($F = 1.2$; $P = 0.3000$).

Shear testing results were then analyzed by four sets of two-way ANOVAs. For the first step, a two-way ANOVA was run on the results generated with the groups etched with 2.5% HF. Since the two-way ANOVA indicated that bond strengths were influenced by the bonding agent ($F = 105.7$; $P = 0.0001$), etching period ($F = 44.2$; $P = 0.0001$), and their combination ($F = 9.0$; $P = 0.0001$), a one-way ANOVA and the Duncan new multiple range intervals were performed. Table 1 and Figure 3 show the shear testing results generated with the groups in which specimens were etched with the 2.5% HF etchant. The results indicated that the etching procedure significantly enhanced the bond strength of unsilanated groups, and that a maximal bond strength (35.4 MPa) was obtained by 150-second etching. The results from unetched control specimens (etching period 0) showed that silane priming was also effective in elevating bond strength. Silane priming following etching enhanced bond strength except for the 90-second and 150-second etching groups. Bond strengths generated with the etched and silane-primed groups ranged from 33.4 MPa to 37.4 MPa. These values, however, were not statistically different, and extension of the etching time period did not significantly increase the bond strength.

The two-way ANOVA of the 5% HF etchant groups indicated that bond strengths were influenced by the bonding agent ($F = 277.1$; $P = 0.0001$), etching period ($F = 32.3$; $P = 0.0001$), and their combination ($F = 12.1$; $P = 0.0001$). A one-way ANOVA and the Duncan new multiple range intervals were then performed. Table 2 and Figure 4 show the shear bond strengths of the composite resin bonded to the porcelain etched with the 5% HF etchant. Although the results demonstrated similarity to Table 1, the difference in bond strength between the etched-alone groups and the etching-and-silane-treated groups was

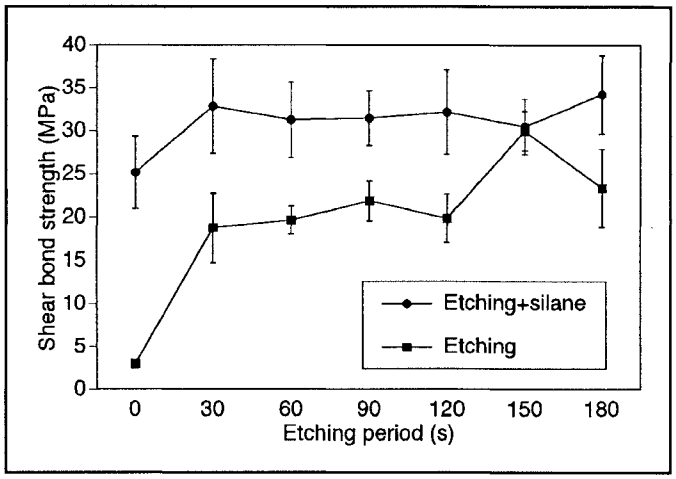



Figure 4. *Shear bond strengths of 5% HF etchant subgroups*

*Table 3. Shear Bond Strength (MPa ± SD); CNB Bonding Agent (without Silane)*

| Etching Periods | Etchant | | | |
|---|---|---|---|---|
| | 2.5% HF | | 5% HF | |
| 0 (control) | 3.0 ± 0.4 | | 3.0 ± 0.4 | |
| 30 | 30.2 ± 4.1 | g | 18.8 ± 4.0 | a |
| 60 | 28.1 ± 4.0 | e,f,g | 19.7 ± 1.6 | a,b |
| 90 | 30.5 ± 4.4 | g | 21.9 ± 2.3 | a,b,c |
| 120 | 25.3 ± 3.7 | c,d,e | 19.9 ± 2.8 | a,b |
| 150 | 35.4 ± 4.4 | h | 30.0 ± 2.3 | f,g |
| 180 | 26.5 ± 3.4 | d,e,f | 23.4 ± 4.5 | b,c,d |

n = 8; CNB = Clearfil New Bond. Letters indicate values that are not significantly different ($P > 0.05$).

*Table 4. Shear Bond Strength (MPa ± SD); CPB Bonding Agent (Contains Silane)*

| Etching Periods | Etchant | | | |
|---|---|---|---|---|
| | 2.5% HF | | 5% HF | |
| 0 (control) | 25.2 ± 4.2 | | 25.2 ± 4.2 | |
| 30 | 36.0 ± 4.9 | a,C | 32.9 ± 5.5 | b,C |
| 60 | 36.4 ± 4.6 | a | 31.3 ± 4.4 | b |
| 90 | 35.0 ± 5.1 | a,D | 31.5 ± 3.2 | b,D |
| 120 | 35.0 ± 5.1 | a,E | 32.2 ± 4.9 | b,E |
| 150 | 37.4 ± 6.1 | a | 30.5 ± 3.2 | b |
| 180 | 33.4 ± 4.2 | a,F | 34.3 ± 4.6 | b,F |

n = 8; CPB = Clearfil Porcelain Bond (contains a silane coupler). Identical lower case letters indicate that the values within each etchant group are not significantly different ($P > 0.05$). Identical capital letters indicate that the two values of an identical etching period are not significantly different ($P > 0.05$).

greater than that observed with the 2.5% etchant. The highest range of bond strengths (30.5 to 34.3 MPa) was again recorded for the etched-and-silane-treated groups.

Table 3 summarizes the results of the groups that did not use the silane bonding agent. Although the difference in bond strength between the groups etched with the 2.5% HF etchant and the groups etched with the 5% HF etchant was not significant for the 180-second etching group, the former exhibited greater bond strength than the latter for the 30-, 60-, 90-, 120-, and 150-second etching groups.

Table 4 shows the shear testing results for the silane-treated groups. The results indicated that bond strengths were affected by the etchant ($F = 12.6$; $P = 0.0006$), but not by etching period ($F = 0.1$; $P = 0.9896$), or by combination of etchant and etching period ($F = 1.3$; $P = 0.2909$). The difference in bond strengths between the two etching agents was significant for the two etching period groups: 60-second etching and 150-second etching.

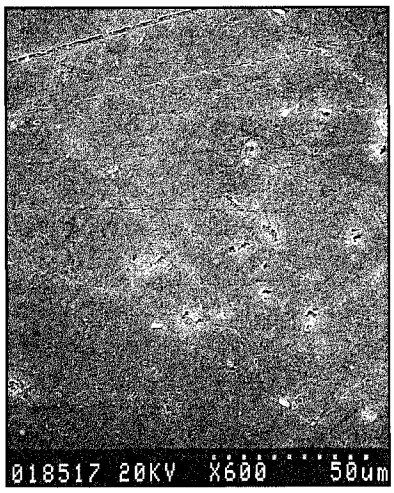



Figure 5. *Scanning electron micrograph of the porcelain before etching*

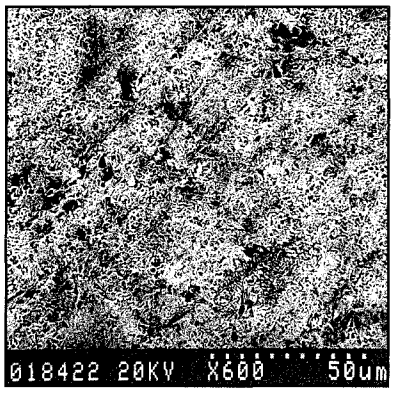
Failure modes varied with the individual groups. Both the unetched control group and the unsilanated group consistently failed at the interface as revealed by optical microscopy. Other groups overwhelmingly failed as the porcelain broke down.

Figures 5 through 7 show porcelain surfaces before and after etching. For both etchants, the surfaces etched for more than 60 seconds were rougher than those etched for 30 seconds. Contrary to the shear bond testing results, the porcelain surface etched with the 5% HF etchant appeared to be more retentive due to a more complicated relief pattern than that produced with the 2.5% HF etchant.

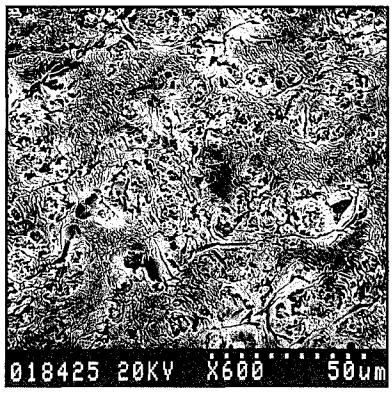
## DISCUSSION

The present experiment assumed that bonding composite resin to porcelain is necessary in cases that require intraoral porcelain repair. Although HF is considered a dangerous, harmful, and irritating compound that is categorized as a poisonous reagent, both laboratory evaluations (Llobell & others, 1992;

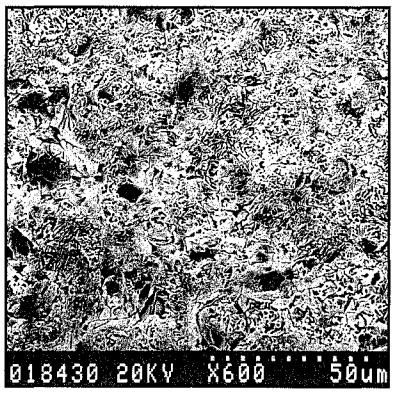
Figure 6. *Scanning electron micrographs of the porcelain etched with the 2.5% etchant for*



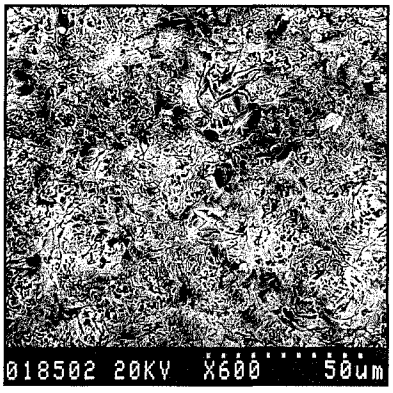

**A**. *30 seconds*




**B**. *60 seconds*

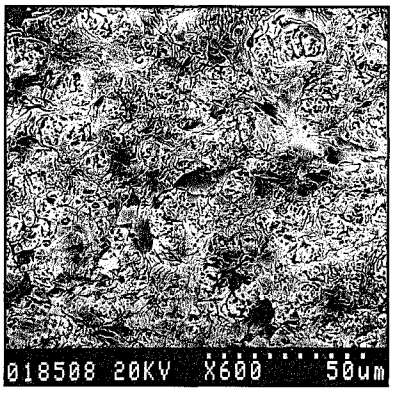



**C**. *90 seconds*




**D**. *120 seconds*


**E**. *150 seconds*



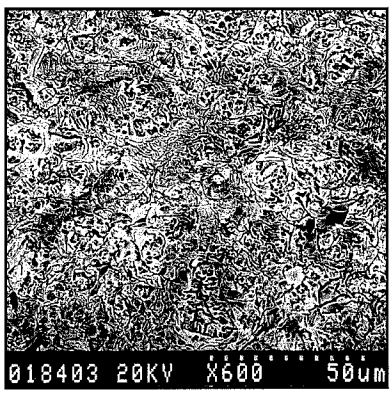

**F**. *180 seconds*

Wolf, Powers & O'Keefe, 1992) and clinical procedures (Rada, 1991; Berksun, Kedici & Saglam, 1993) concerning the use of HF for intraoral porcelain bonding have been reported. Moreover, an etchant containing HF designed for intraoral application is on the market (Oral Ceram Etch, Llobell & others, 1992). These reports suggest that HF may be used in clinical procedures if sufficient care is taken to avoid iatrogenic side effects. Two products with different concentrations of HF were therefore assessed as potential intraoral porcelain etchants.

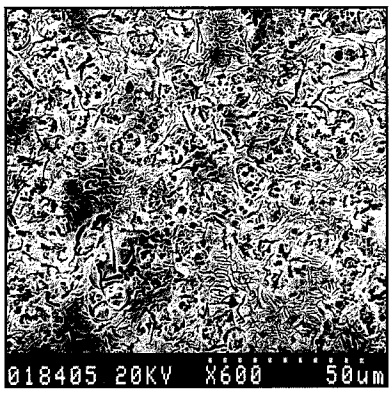
Shear bond testing results without silane priming demonstrated that the relationship between etching period and bond strength was not particularly distinct, especially for the 2.5% etchants. On the basis of the present results, 30-second etching with the 2.5% HF etchant may be clinically acceptable for achieving sufficient bond strength. As revealed by the shear testing, extension of the etching period did not proportionally increase the bond strength. This may be related to the surface structure change of the porcelain. Specifically, Figures 6 and 7 do not exhibit remarkable changes in the etched pattern with extension of etching period, and the slight change of bond strength with extension of etching may be attributed to the slight change of the etched relief pattern.

The shear testing results indicated that the porcelain etched with the 2.5% HF etchant generated a greater bond strength than that etched with the 5% HF etchant when the etching period was 150 seconds or less. These results may be due to the difference in the resistance of the adhesive interface to fracture between resin-porcelain junctions generated with the use of different etching agents. If the porcelain surface were over-etched, bond strength would be adversely affected. Possible problems are: 1) difficulty in removing etchant and solvent water from the etched surface using chair-side equipment, 2) wettability of intermediate resins, and 3) postcuring stress concentration due to complicated adhesive interface structure. The resistance to fracture of the adhesive interface depends partly on mechanical interlocking between the bonding agent and etched porcelain. It is probable that the stronger the resistance to fracture of a thin layer of etched porcelain surface, the greater the recorded bond strength, because the adhesive interface consists of a roughened porcelain surface and penetrated bonding resin. The authors speculate that the fracture resistance of a thin layer of etched porcelain

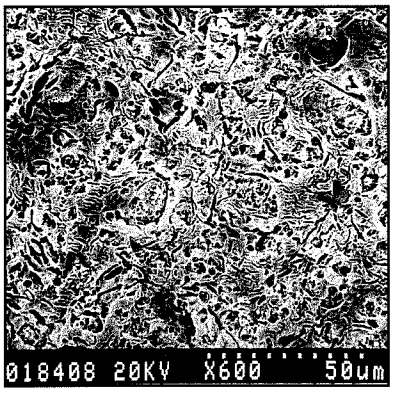
Figure 7. *Scanning electron micrographs of the porcelain etched with the 5% etchant for*



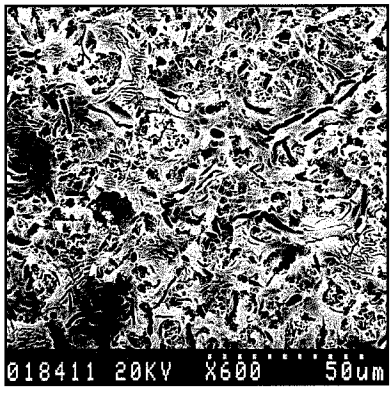

**A.** *30 seconds*



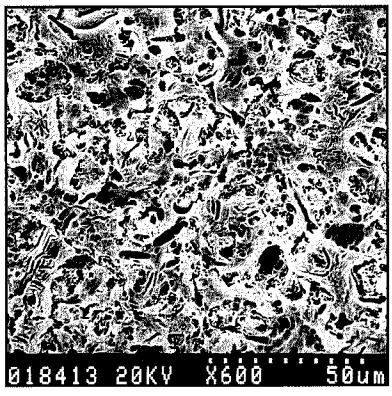

**B.** *60 seconds*



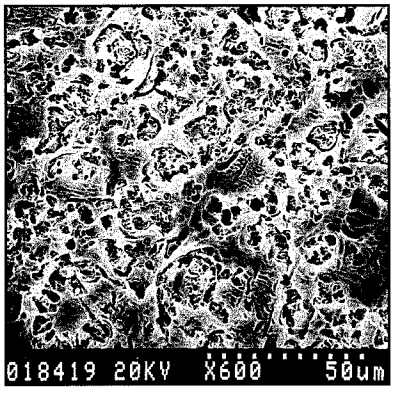

**C.** *90 seconds*




**D.** *120 seconds*




**E.** *150 seconds*




**F.** *180 seconds*

is stronger when etched with the 2.5% HF etchant than when etched with the 5% HF etchant, hence greater strength was recorded with the use of the 2.5% etchant.

The manufacturer of the 2.5% etchant stated that the agent was buffered. One of the purposes of buffering appears to be the reduction of side effects such as chemical burn when the reagent accidentally contacts the mucous membrane. Whether buffering affects bond strength cannot be determined from the present study, but further investigation should be conducted using different concentrations of the buffered etchants.

Silane priming also considerably improved bond strength to the porcelain of the composite material. The effectiveness of the Clearfil Porcelain Bond silane bonding agent has been reported by several researchers (Llobell & others, 1992; Lu & others, 1992; Wolf & others, 1992; Appeldoorn & others, 1993; Suliman & others, 1993; Kato & others, 1996), and the results of the present study correlated well with these reports. The effectiveness of the Clearfil Porcelain Bond material may be attributed to: a) the fact that the material contained an initiator for enhancing conversion of the monomer component at the adhesive interface, and b) the fact that the material also contained a polymerizable hydrophobic acid catalyst (Kato & others, 1996). Since the Clearfil New Bond and Clearfil Porcelain Bond bonding agents consist of similar ingredients made by an identical manufacturer, the viscosities of the two agents were similar. The difference between the two bonding agents was that the Clearfil Porcelain Bond agent contained a silane coupler. According to a report on bonding porcelain teeth to denture base acrylic resin, the mechanism of the bond between porcelain and resin is condensation between the trimethoxysilyl groups in the silane coupler and the hydroxy groups of the porcelain surface (Paffenbarger, Sweeney & Bowen, 1967). Since the silane monomer and the porcelain structure in the present study were almost identical to those in their report, the bond mechanisms would be nearly the same. The only difference between the two systems was the presence of a catalyst for condensation. Instead of using a hydrophilic acid catalyst or heating, some intraoral porcelain repair systems employ hydrophobic acid functional monomers with nonaqueous solvent as a catalyst. This concept was reported by Matsumura and others in 1989, and materials that employ this type of catalyst

system include Clearfil Porcelain Bond, Porcelain Liner M (Etch-Free Primer), and Tokuso Ceramics Primer (Kato & others, 1996).

Although the present study demonstrated the effectiveness of the combined application of hydrofluoric acid etching and a self-curing silane bonding agent, future evaluation of bond durability is necessary to predict the service period of the repair systems. Further study is also needed to determine whether similar results would actually be found in an intraoral environment.

## CONCLUSIONS

Repair bond strength to a feldspathic porcelain of a photoactivated composite material was evaluated in vitro using two hydrofluoric acid etchants, seven etching duration periods, and a self-curable three-component silane bonding agent. Under the present laboratory experimental conditions, the following conclusions can be made:

1. The shear bond strengths of the composite material to the porcelain without silane priming improved considerably with the use of either etchant. The porcelain etched with the 2.5% HF etchant exhibited a greater bond strength than that etched with the 5% HF etchant for the 30-, 60-, 90-, 120-, and 150-second etching periods.
2. Without silane priming, shear bond strength was not improved when the etching period was extended from 30 seconds to 120 seconds.
3. The use of the silane bonding agent dramatically enhanced bond strength to the unetched porcelain of the composite material.
4. Combined application of the 2.5% HF etchant for 30 seconds or longer and use of the silane bonding agent appeared to be the best system. The bond strengths of the etched and silane-treated systems were not affected by the type of etching agent or the etching duration period, except for 60 and 150 seconds.

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# Influence of Air-Abrasion Treatment on the Interfacial Bond between Composite and Dentin

M HANNIG • T FEMERLING

**Clinical Relevance**
Adaptation between composite resin and cavity walls located in dentin is significantly enhanced by combining air-abrasion treatment with the application of a dentin adhesive; however, the obtained interfacial bond cannot prevent gap formation between composite and dentin due to thermal load.


## SUMMARY

**The purpose of this in vitro study was to investigate the effect of air abrasion on the interfacial bond between composite and dentin. Dentin surfaces surrounded by enamel were produced by removing the incisal part of the crown from extracted human anterior teeth. The dentinal area of the specimen surfaces was conditioned either by air-abrasion treatment (27 μm aluminum oxide particles, 120 psi), application of dentin adhesives, and combination of air abrasion and dentin adhesives, or left nontreated (controls). The enamel was conditioned by acid etching, by air abrasion, or by air abrasion and subsequent acid etching. A 2 mm layer of six different composite resins (Brilliant, Charisma, Helioprogress, Herculite, Pekafill, Z-100) was applied on the specimen surfaces and light cured. The composite dentinal interface was evaluated in cross-cut sections of 324 specimens by SEM analysis, and the mean gap width (MGW) between composite and dentin was calculated. SEM analysis revealed gap formation ranging from 1.1-3.6 μm in control specimens. Gap width was reduced significantly due to air-abrasion treatment of the dentinal surface (MGW: 0.3-1.4 μm) or by application of dentin adhesives (MGW: 0.06-0.6 μm). Combined air-abrasion treatment and application of dentin adhesives resulted in a gap-free adaptation between composite and dentin in the majority of specimens. However, thermocycling of the specimens (5 °C/55 °C; 1000 cycles) caused a significant increase in gap formation (MGW: 0.03-2.9 μm) at the composite-dentin interface. It is concluded that air-abrasion treatment and subsequent application of a dentin adhesive is an effective procedure to resist the contraction stress at the dentinal surface during polymerization of the composite resin and to improve the internal bond of composite resin restorations.**


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## INTRODUCTION

The clinical failure of composite resin restorations is often associated with the polymerization shrinkage of the resin. The contraction can manifest itself in gap formation between the restoration and the cavity margins, in deformation of the tooth, and in residual stress within the restoration. Interfacial gap formation between the composite resin restoration and the dentinal surface promotes dentinal fluid percolation. This phenomenon may cause pulpal sensitivity during the functional load of the restoration. In addition, internal gaps enhance microleakage, bacterial invasion, and subsequent development of secondary caries whenever the marginal integrity of the restoration fails. Therefore, both a permanently perfect marginal adaptation and a gap-free internal bond between composite and dentin are important for the long-term clinical success of composite resin restorations.

The creation of a microretentive surface pattern by phosphoric acid etching of the enamel has proven to be a clinically reliable method for obtaining a tight marginal seal of composite resin restorations. Another method for producing a microretentive surface structure is the so-called kinetic cavity preparation or air-abrasion technique (Goldstein & Parkins, 1994). Kinetic energy generated by a high-velocity stream of aluminum oxide particles can effectively increase the surface roughness of dentin and enamel (Laurell, Lord & Beck, 1993; Doty & others, 1994; Keen, von Fraunhofer & Parkins, 1994; Moritz & others, 1996). Previous studies suggest that the air-abrasive technology has the potential to prepare enamel bonding surfaces comparable to those attained by acid etching (Laurell & others, 1993; Doty & others, 1994; Keen & others, 1994; Moritz & others, 1996). In addition, air abrasion may have the potential to enhance the internal adaptation between composite resin and cavity walls located in dentin.

The purpose of this in vitro study was to evaluate the influence of air-abrasion treatment on the interfacial seal between composite and dentin in restorations placed on flat dentin surfaces surrounded by enamel. The composite resin was applied both with and without dentin bonding agents.

## METHODS AND MATERIALS

Dentin surfaces surrounded by enamel were prepared in 324 extracted anterior human teeth by cutting off the incisal part of the crown with a high-speed bur. The resulting enamel-dentin surfaces were flattened and polished by wet-grinding with 1200-grit abrasive paper. After such preparation, the 324 specimens were randomly divided into nine groups with 36 specimens each, and restored with six different composite resins (Table 1). In each individual group (Groups 1-9), six randomly selected specimens were

*Table 1. Composite Resins and Dentin Adhesives Used*

| Composite Resins (Manufacturer) | Type of Composite | Dentin Adhesives (Manufacturer) | Components and Application of Dentin Adhesives |
|---|---|---|---|
| Brilliant (Coltène, Konstanz, Germany) | fine-hybrid | ART Bond (Coltène) | Primers A and B: mixing, 60 seconds scrubbing into dentinal surface, air drying<br>ART Bond: 20 seconds application, 20 seconds light curing |
| Charisma (Heraeus-Kulzer, Wehrheim, Germany) | fine-hybrid | Denthesive II (Heraeus-Kulzer) | Denthesive II A and B: mixing, 30 seconds scrubbing into dentinal surface, air drying<br>Adhesive Bond II: 15 seconds scrubbing into surface, 20 seconds light curing |
| Helioprogress (Ivoclar, Schaan, Liechtenstein) | inhomogeneous microfilled | Syntac (Ivoclar) | Syntac-Primer: 15 seconds application, air drying<br>Syntac-Adhesive: 15 seconds application, air drying<br>Heliobond: 20 seconds light curing |
| Herculite XRV (Kerr, Karlsruhe, Germany) | fine-hybrid | OptiBond (Kerr) | OptiBond Prime: 30 seconds scrubbing into dentinal surface, air drying, 20 seconds light curing<br>OptiBond Adhesive: 20 seconds light curing |
| Pekafill (Bayer Dental, Leverkusen, Germany) | fine-hybrid | Gluma CPS (Bayer Dental) | Gluma Cleanser: 30 seconds application, rinsing, air drying<br>Gluma Primer: 30 seconds application, air drying<br>Gluma Sealer: 10 seconds application, 20 seconds light curing |
| Z-100 (3M Dental Products, St Paul, MN 55144) | fine-hybrid | Scotchbond MP (3M Dental Products) | Etchant: 15 seconds application, rinsing, air drying<br>Primer: 15 seconds application, air drying<br>Adhesive: 20 seconds light curing |

*Table 2. Experimental Conditions in Group 1-9 (Each Group: n = 36)*

| | First Experimental Series (without use of dentin adhesives) | | | Second Experimental Series (application of dentin adhesives) | | | Third Experimental Series (application of dentin adhesives, thermocycling of specimens) | | |
|---|---|---|---|---|---|---|---|---|---|
| | Groups | | | Groups | | | Groups | | |
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| **Treatment of Enamel** (ee = enamel etching; KCP = air abrasion) | ee | KCP | KCP ee | ee | KCP | KCP ee | ee | KCP | KCP ee |
| **Treatment of Dentin** (KCP = air abrasion; DA = dentin adhesive) | -- | KCP | KCP | -- DA | KCP DA | KCP DA | -- DA | KCP DA | KCP DA |
| Thermocycling (TC) | -- | -- | -- | -- | -- | -- | TC | TC | TC |

restored with each of the six different composite resins.

Restoration of specimens took place in three different experimental series (Table 2). In the first experimental series the composite materials were applied to the dentin-enamel surfaces without the use of dentin adhesives (Groups 1, 2, 3). In the second series of experiments the resin composites were placed after application of the system-equivalent dentin bonding agents (Groups 4, 5, 6). In the third series of experiments specimens were restored with composite resins using dentin adhesives, and were subjected to subsequent thermocycling (Groups 7, 8, 9). In each of the three experimental series, application of the composite resins took place (Table 2) after selective etching of the enamel without air abrasion (Groups 1, 4, 7); after conditioning both dentin and enamel by air abrasion without any etching (Groups 2, 5, 8); and after air-abrasion treatment of enamel and dentin followed by selective etching of the air-abraded enamel surface (Groups 3, 6, 9).

Air-abrasion conditioning of both the dentin and enamel surfaces was accomplished with the KCP 1000 unit (American Dental Technologies, Troy, MI 48084) using a 27 μm aluminum oxide particle stream at 120 psi pressure. The KCP treatment took place at a distance of approximately 1 mm from the dentin or enamel surface. Enamel etching was performed for 30 seconds with 37% phosphoric acid gel. Subsequently, the enamel was rinsed for 30 seconds with water and carefully air dried. Application of the various dentin adhesives was made in strict accordance with the manufacturers' directions (Table 1). No wet-bonding technique was used. The composite resins were applied to the surface of the specimens in a 2 mm-thick bulk layer and light cured for 40 seconds from the incisal direction (Translux, Heraeus-Kulzer, Wehrheim, Germany). Thermal cycling (Groups 7, 8, 9) included 1000 cycles within a temperature range of 50 K for the duration of 60 seconds at a maximum temperature of 55 °C and a minimum temperature of 5 °C. Table 2 presents a general overview of the experimental conditions in Groups 1 to 9.

The internal bond of the restorations was analyzed on cross-cut sections at the composite-dentin interface. All teeth had been transversely sectioned mesiodistally in two halves with a diamond saw and polished with 1200-grit abrasive paper. Impressions (President light body; Coltène, Konstanz, Germany) of the polished surfaces were taken immediately after polishing for replication purposes. SEM evaluation was performed on epoxy resin replicas (Stycast; Grace, Westerlo, Belgium) of the polished sections. The entire length of the composite-dentin bond was analyzed on each section at a X1250 magnification, and the gap width between dentin and composite was measured at three defined points (Figure 1). The mean gap width of the six specimens of each composite material was calculated in every group. The results of the SEM analysis were subjected to a nonparametric statistical test procedure (Bonferrioni-adjusted

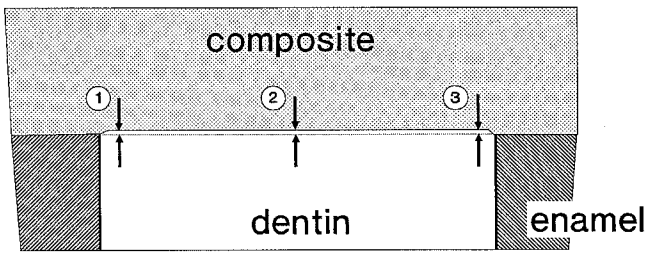


Figure 1. *Schematic diagram of a cross-cut sectioned specimen, indicating the points (1, 2, 3) defined for the quantitative SEM analysis of the composite-dentin interface. The distance between the dentinoenamel junction and measurement points 1 and 3 was approximately 150 μm.*

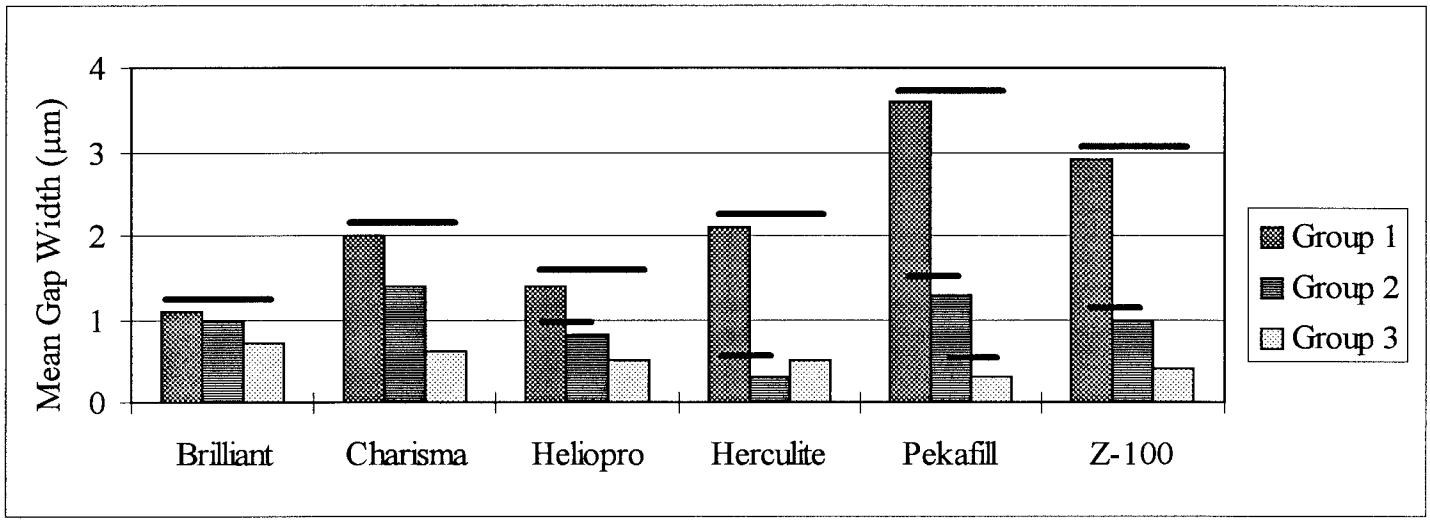


Figure 2. *Results of SEM analysis: mean gap width at the composite-dentin interface in Groups 1, 2, 3 (application of composite resin without dentin adhesive). Brackets indicate significant differences (P < 0.05) between groups. Comparison of gap width values of the various materials revealed significant differences in Group 1 between Brilliant and Pekafill, Brilliant and Z-100, and Helioprogress and Pekafill, in Group 2 between Charisma and Herculite, and in Group 3 between Brilliant and Pekafill. Group 1 = enamel etch; Group 2 = KCP; Group 3 = KCP + enamel etch).*

multiple Mann-Whitney tests, $P < 0.05$).

SEM analysis of the composite-enamel seal was not quantitative, but qualitative, determining whether or not gaps between the composite resin and enamel could be detected on the cross-cut sections of the specimens.

## RESULTS

The results of the quantitative SEM analysis are summarized in Figures 2 to 4 and in Tables 3 to 5.

The SEM analysis of the composite-dentin interface without the use of dentin adhesives and without air-abrasion conditioning (Group 1) revealed gap formation between composite and dentin on the full length of the interface (Figure 2). The mean gap width varied from 1.1 to 3.6 µm, depending on the specific composite resin applied. Air-abrasion treatment of the dentinal surface (Groups 2, 3) as well as application of the system equivalent dentin adhesives (Groups 4, 5, 6) significantly reduced interfacial gap formation between composite and dentin (Figures 2, 3; Tables 3-5). Best results were obtained in Groups 5 and 6, where the dentin surfaces had been conditioned by air abrasion before the application of dentin adhesives. With most materials (Charisma,

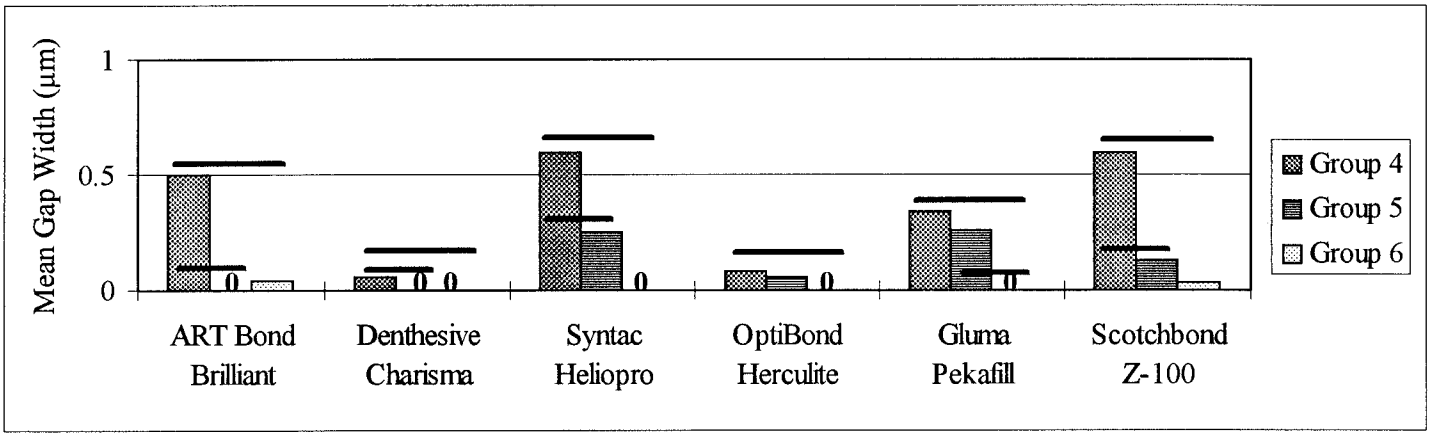


Figure 3. *Results of SEM analysis: mean gap width at the composite-dentin interface in Groups 4, 5, 6 (application of composite resin with dentin adhesives). Horizontal lines (brackets) indicate significant differences (P < 0.05) between groups. Statistical analysis did not reveal any significant differences of mean gap width with values between the various materials in Groups 4, 5, 6 respectively. Group 4 = enamel etch; Group 5 = KCP; Group 6 = KCP + enamel etch.*

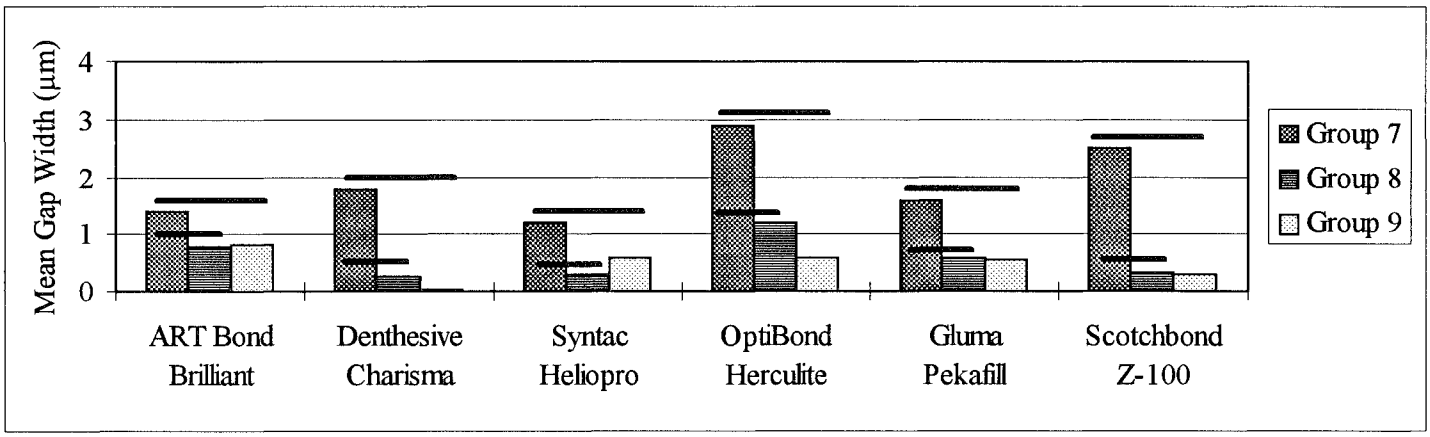


Figure 4. *Results of SEM analysis: mean gap width at the composite-dentin interface in Groups 7, 8, and 9 (application of composite resin with dentin adhesives followed by thermocycling of the specimens). Brackets indicate significant differences (P < 0.05) between groups. Values of mean gap width differ statistically significantly in Group 9 between Denthesive/Charisma and ART Bond/Brilliant. Group 7 = enamel etch; Group 8 = KCP; Group 9 = KCP + enamel etch.*

Helioprogress, Herculite, Pekafill), a full-length, gap-free bond between the composite and dentin was observed in Group 6. No significant differences were detected between the specimens conditioned by air abrasion only (Group 5) and those conditioned by air abrasion and subsequent enamel etching (Group 6), with the exception of the specimens restored with Pekafill (Figure 3).

In the vast majority of the analyzed specimens, thermocycling caused a significant increase in interfacial gap formation (Figure 4, Tables 3-5). The mean gap width at the composite-dentin interface varied between 1.2 and 2.9 µm in Group 7 (without air-abrasion conditioning) and 0.03 to 1.2 µm in Groups 8 and 9 (with air-abrasion conditioning).

The SEM analysis of the composite-enamel interface revealed an insufficient adaptation in some specimens belonging exclusively to Groups 2 and 8 (air abrasion without acid etching of the enamel) (Table 6). The specimens of all other groups manifested a full-length, gap-free adaptation at the composite-enamel interface.

## DISCUSSION

Composite resin materials shrink during polymerization, generating contraction stress within the resin up to 7 MPa (Hegdahl & Gjerdet, 1977; Bowen, Nemoto & Rapson, 1983; Davidson, DeGee & Feilzer, 1984; Feilzer, DeGee & Davidson, 1987). The magnitude of this stress varies depending on the cavity configuration. In a three-dimensional cavity preparation, only one

*Table 3. Results of the Statistical Analysis; Comparison of Groups 1 (Nontreated Dentin) and 4 (Dentin Adhesives), and Groups 4 and 7 (Dentin Adhesives; Thermocycling)*

| Composite Resin | Group 1 vs Group 4 | Group 4 vs Group 7 |
|---|---|---|
| Brilliant | sig dif* | sig dif |
| Charisma | sig dif | sig dif |
| Helioprogress | sig dif | sig dif |
| Herculite XRV | sig dif | sig dif |
| Pekafill | sig dif | sig dif |
| Z-100 | sig dif | sig dif |

*sig dif = significant difference, $P < 0.05$.

*Table 4. Results of the Statistical Analysis; Comparison of Groups 2 (Air-Abrasion Treatment) and 5 (Air-Abrasion Treatment, Dentin Adhesives), and Groups 5 and 8 (Air-Abrasion Treatment, Dentin Adhesives; Thermocycling)*

| Composite Resin | Group 2 vs Group 5 | Group 5 vs Group 8 |
|---|---|---|
| Brilliant | sig dif* | sig dif |
| Charisma | sig dif | sig dif |
| Helioprogress | sig dif | n s |
| Herculite XRV | n s | sig dif |
| Pekafill | sig dif | n s |
| Z-100 | sig dif | ns |

*sig dif = significant difference, P < 0.05; n s = no signficant difference.

*Table 5. Results of the Statistical Analysis; Comparison of Groups 3 (Air-Abrasion Treatment, Etching) and 6 (Air-Abrasion Treatment, Etching, Dentin Adhesives), and Groups 6 and 9 (Air-Abrasion Treatment, Etching, Dentin Adhesives; Thermocycling)*

| Composite Resin | Group 3 vs Group 6 | Group 6 vs Group 9 |
|---|---|---|
| Brilliant | sig dif* | sig dif |
| Charisma | sig dif | n s |
| Helioprogress | sig dif | sig dif |
| Herculite XRV | sig dif | sig dif |
| Pekafill | sig dif | sig dif |
| Z-100 | sig dif | n s |

*sig dif = significant difference, $P < 0.05$; n s = no significant difference.

*Table 6. SEM Evaluation of the Enamel Composite Bond, Indicating the Number of Samples (out of n = 6) with Gaps between Composite and Enamel in Groups 2 and 8 (Air-Abrasion Treatment without Enamel Etching)**

| Composite Resin | Group 2 | Group 8 |
|---|---|---|
| Brilliant | 2 | 2 |
| Charisma | 2 | -- |
| Helioprogress | 1 | 2 |
| Herculite XRV | -- | 2 |
| Pekafill | 1 | 2 |
| Z-100 | 2 | 3 |

*In all other groups the enamel composite interface showed a full-length gap-free adaptation.

surface of the composite resin is unbonded. This strongly limits the flow of composite material and produces stress within the restoration or at the interface between the restorative material and the cavity walls (Davidson & others, 1984; Feilzer & others, 1987). In the present study a very simple cavity design was chosen to reduce the number of cavity-related factors that could influence the composite-dentin bond. When the composite resin is bonded to a single flat surface, as designed for the present study, flow relaxation can occur within the composite during setting, reducing some of the contraction forces. However, as clearly shown in the experimental series without the use of dentin adhesives (Group 1), these flow phenomena could not prevent the formation of interfacial gaps between dentin and composite resin. Polymerization shrinkage caused the composite resin to recede from the dentin, whereas the microretentive surface of the adjacent acid-etched enamel counteracted the shrinkage forces during polymerization and secured a tight marginal seal. The forces producing gaps at the composite-dentin interface are essential considerations for designing appropriate experimental conditions to determine whether air abrasion and dentin bonding have an influence on the internal seal of composite resin restorations. The gap width found in Group 1 was considered as control standard for the SEM evaluation of the other groups. It was expected that the extent of gap formation between composite resin and dentin would increase from the well-bonded enamel-composite interface to the central area of the specimen's dentinal surface. Therefore, two measurements were made at about 150 μm from the dentinoenamel junction, and one measurement was performed at the center of the composite-dentin interface. However, SEM investigations revealed a similar magnitude of gap width on almost the entire length of the composite-dentin interface, and statistical analysis of data failed to show any significant differences between the values of gap width near to the dentinoenamel junction and at the center of the specimens. These findings justified calculation of mean gap width from values measured at the three defined points.

SEM analysis indicated that the mean gap width between composite and dentin was significantly reduced by using air abrasion to roughen the surface before placing the resin composites in Groups 2 and 3, as opposed to Group 1. These results confirmed the observation that KCP treatment increased the shear bond strength of composite resin to dentin (Laurell & others, 1993). In contrast, other authors could not detect an improvement in composite-dentin shear bond strength due to microabrasion pretreatment (Rainey & Barghi, 1995).

A comparison of the Groups 1, 2, 3 (first series, without use of dentin bonding agents) and Groups 4, 5, 6 (second series, use of dentin bonding agents) indicated that air-abrasion treatment did not eliminate the need for dentin adhesives to bond the composite materials to the dentinal surface. Similar findings were established by shear bond strength measurements (Roeder & others, 1995), showing that air-abrasion treatment of the dentin alone resulted in lower bond strengths than in dentin additionally conditioned with adhesives. Dentin surfaces that were air abraded, primed, and adhesively bonded yielded bond

strengths in excess of 20 MPa (Roeder & others, 1995). These shear bond strength values fell within the same range as those achieved between composite resin and enamel prisms cut transversely by phosphoric acid etching (Gwinnett & Kanca, 1992). Under the conditions of this in vitro study, air abrasion of the dentinal surface seemed to improve the effectiveness of dentin bonding agents in counteracting the polymerization-induced shrinkage stress at the composite-dentin interface (Figure 3). Enlargement of the dentinal surface area as well as an increase in surface roughness due to air-abrasion treatment may be relevant factors, which could influence the effect and reaction mechanisms of dentin bonding agents. On the other hand, it has been demonstrated that the smear layer created during tooth preparation could adversely affect the effectiveness of dentin bonding agents (Prati & others, 1990; Yu & others, 1991; Gwinnett & Kanca, 1992; Nakabayashi & Saimi, 1996). Resin infiltration into intertubular dentin and formation of the hybrid layer is more easily accomplished in fractured dentin than in smear layer-covered dentin (Pashley & others, 1993; Nakabayashi & Saimi, 1996). Air-abraded dentinal surfaces are covered at the most by smear layer residues, and therefore may provide more favorable conditions for dentin bonding as compared to the smear layer-covered dentinal surface in nonair-abraded specimens. Further investigations are in progress now to clarify the mode of action of dentin adhesives on air-abraded dentinal surfaces.

In the present study air-abrasion treatment of dentin and enamel surfaces was performed with the KCP 1000 unit using a 27 μm instead of the 50 μm particle stream recommended by the manufacturer for conditioning purposes of the tooth hard substances. Roeder and others (1995) reported that aluminum oxide particle size (27 μm or 50 μm) had no influence on the bond strength of composite resin to dentin. In addition, we conducted some preliminary experiments, which also revealed no statistically significant differences in the interfacial bond and adaptation between composite and dentin after air-abrasion conditioning with the 27 μm or 50 μm particles respectively. Therefore, in this study the gentle, less abrasive pretreatment of the dentin with a medium pressure (120 psi) 27 μm aluminum oxide particle stream was used.

In the vast majority of experiments, no statistically significant differences could be detected between specimens conditioned by air abrasion alone and those conditioned by air abrasion with subsequent acid etching of the enamel, except for the Pekafill/Gluma specimens (Figures 2, 3). However, the present investigation focused primarily on the internal seal of the composite resin restorations. Concerning the marginal seal, in vitro studies indicate that conditioning the enamel cavity margins by air abrasion without subsequent acid etching results in a marginal adaptation with significantly more microleakage (Eakle, Wong & Huang, 1995) and lower shear bond strengths than in enamel margins etched in addition to air-abrasive pretreatment (Roeder & others, 1995; Horgesheimer & others, 1995). These findings suggest the need for etching the enamel surfaces after air-abrasion conditioning prior to applying the composite resin. The present study confirmed this recommendation by the SEM detection of marginal gaps at the composite-enamel interface in some specimens whose enamel margins had been conditioned by air abrasion only (Table 6).

The results of the experimental series exposing the specimens to thermocycling revealed that thermal stress strongly influenced the internal seal of a composite resin restoration, even in specimens treated by a combination of air abrasion and dentin adhesives (Groups 8, 9). The loss of interfacial adaptation due to thermocycling may be explained by differences in coefficient of thermal expansion of the tooth surface and the adjacent composite resin restoration. The gradual increase in microleakage with thermal stress is commonly observed in the marginal adaptation of composite resin restorations (Momoi & others, 1990). Moreover, results of this study show that the internal seal at the dentin-composite interface in composite resin restorations was especially affected by thermal load, even when the marginal adaptation of the restoration at the composite-etched enamel interfaces remained unaltered and stable during thermocycling. These findings offer possible evidence for a thermally related stress relaxation within the composite resin, which subsequently led to a deformation of the restoration, resulting in additional stress on the composite-dentin bond. This hypothesis needs further corroboration, especially in complex class 2 composite resin restorations. From a clinical point of view, the results after thermocycling of the specimens indicated that the long-term stability of the internal seal between composite and dentin in resin composite restorations was questionable, even when dentin surfaces were conditioned by combined air-abrasion treatment and application of dentin bonding agents.

Six different composite resins and bonding agents were included in the experiments. The purpose of this study was not to grade the different materials, but to assess the effect of air-abrasion treatment on a broad range of commonly used dentin adhesives in composite resin systems. Values of mean gap width showed material-dependent differences in the experimental groups (Figures 2-4). However, statistical comparison of the various materials within each individual group revealed that these differences were significant only in a few cases (see legends of Figures 2-4).

## CONCLUSIONS

Under the conditions of this in vitro study the following conclusions were reached:

1. The combined air abrasion/dentin bonding treatment represented an effective method to reduce gap formation at the composite resin-dentin interface; however,
2. Thermocycling caused a significant deterioration of the interfacial bond between composite and dentin in resin composite restorations.

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# Color Attributes and Accuracy of Vita-based Manufacturers' Shade Guides

A U J YAP

**Clinical Relevance**
Manufacturers' shade guides are not similar to the Vita Lumin shade guide to which they are supposedly keyed.


## SUMMARY

**The shades of several tooth-colored restoratives are now keyed to the Vita Lumin shade guide. The purpose of this study was to determine whether manufacturers' shade guides had color attributes that were similar to the Vita Lumin guide to which they are supposedly keyed. The overall color accuracy between the different guides was also compared via colorimetry. Results showed that none of the manufacturers' shade tabs evaluated had all L*, a*, and b* values that were identical to their respective Vita shade tabs. For the shades evaluated, manufacturers' tabs were generally darker than their corresponding Vita shade tabs. When shade guides were compared, the color accuracy of Z100 and Fuji II LC shade guides was significantly better than that of Durafill VS and TPH. Dyract's shade guide was the most accurate and was significantly better than the shade guides of Fuji II LC, Durafill VS, and TPH. The accuracy of manufacturers' shade guides was not consistently product dependent but was shade dependent.**



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## INTRODUCTION

Tooth-colored restoratives are the most heavily researched material in dentistry today. New restoratives are launched almost daily and are modified or removed from the market with equal rapidity. The majority of these new tooth-colored restoratives are now keyed to the Vita Lumin shade guide (Vita Zahnfabrik, Bad Säckingen, Germany), which is widely used for porcelain shade selection. The Vita shade range is divided into four groups designated by letters A, B, C, or D. These shades have brown, yellow, gray, and red characters respectively. Shade tabs of a specific letter group have similar hue (Miller, 1988), and each hue group includes several tabs of increasing chroma and decreasing value designated in numeric order (e g, A1, A2, A3, A3.5, A4) (Schwabacher & Goodkind, 1990).

As more manufacturers adopt this practice, the Vita shade guide has become the de facto standard in clinical dentistry. The objective of this practice was to reduce the need for different shade guides, with

colors that were arbitrary, subjective, and varied (Wozniak & others, 1985), and to improve interpractitioner communication. Clinical procedures involving different materials would then be theoretically simplified as colors are identical. Previous research (Yap, Bhole & Tan, 1995), however, does not support the claim by manufacturers that their products correspond with the colors of the Vita shade guide. In addition, color match of restoratives to the Vita Lumin shade guide was found not to be material dependent but tended to differ among the value shades evaluated. The manufacturers' customized shade guides (Wieder, 1990) made from the actual restorative material were thus recommended.

Color has three main attributes that allow it to be described. These attributes are dependent on the color system used. The most commonly used color systems are the Munsell color system and the CIE color system. The Munsell system, despite its disadvantages, is the most popular method for color description and is widely used in the dental literature (Sproull, 1973). In it the three main attributes are called Hue, Chroma, and Value, where Hue is defined as the particular variety of a color, chroma the intensity of a Hue, and Value the relative darkness and lightness of a color.

The CIE color system (CIELAB) was determined by the Commission Internationale de l'Eclairage (International Commission on Illumination) in 1978. This method of color evaluation is related to human color perception by three attributes (figure). Equal distances in the color space represent approximately equally perceived gradations. The CIE system thus has an advantage over the Munsell system, in which the gradations are more arbitrary (Rosenstiel, Land & Fukimoto, 1988). Because of this, the CIE system is becoming more widely used in dental research (O'Brien, Boenke & Groh, 1991; Swift, Hammel & Lund, 1994). The three attributes of color in this system are L*, a*, and b*, where L* is a lightness variable proportional to Value in the Munsell system and a* and b* are chromacity coordinates. The a* and b* coordinates designate positions on red/green and yellow/blue axes respectively (+a = red, -a = green; +b = yellow, -b =blue) and do not correspond directly with Munsell's Hue and Chroma.

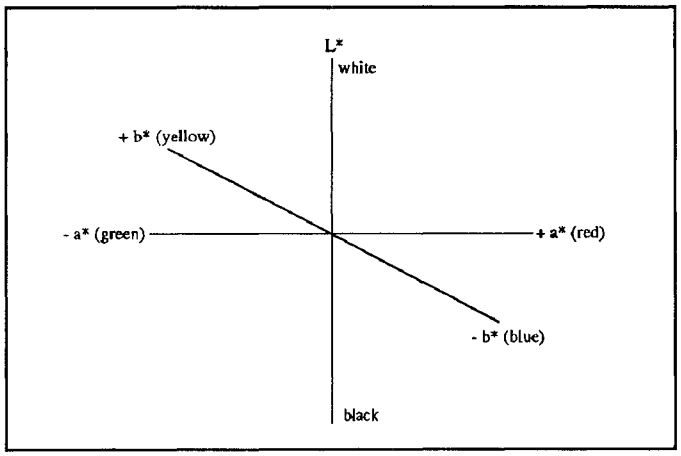


*Diagram of the CIELAB color space*

The purpose of this study was to determine whether manufacturers' shade guides had color attributes that were similar to the Vita Lumin guide to which they are keyed. The overall color accuracy between the different guides was also compared.

## METHODS AND MATERIALS

Shade guides from five Vita-based restorative products were used in this study: Z100 (3M Dental Products, St Paul, MN 55144), Dyract (De Trey/Dentsply, Surrey, England), Fuji II LC (GC, Tokyo, Japan), Durafill VS (Kulzer, Friedrichsdorf, Germany), and TPH (L D Caulk/Dentsply, Milford, DE 19963). All common shades (Vita shades A2, A3, B3, C2, and C4) from these five products were selected for evaluation. A small-area (3 mm-in-diameter measuring area) colorimeter (Dental Colorimeter, Minolta Camera Co Ltd, Tokyo, Japan) was used to determine CIELAB coordinates of each specimen. Illumination corresponding to "average" daylight (CIE illuminant D65) from a pulsed xenon light source was used. The colorimeter was calibrated before each measurement period using the white calibration tile (calibration cap) supplied by the manufacturer. Measurements were taken from five tabs for each product and shade combination. Three readings were taken at the body region for each tooth-shaped tab, via means of a transparent silicone adaptor, and at the center of each flat tab. The L*a*b* values were averaged to obtain a single set of values for each product. The means and standard deviations for the L*a*b* coordinates for each specimen were calculated using SPSS for Windows version 6.0.

Significant differences between mean L*, a*, and b* values were determined by Kruskal-Wallis one-way ANOVA, with shade and products as the main factors. Mann-Whitney *U* and Wilcoxon's Rank Sum W tests were then used to detect significant differences in color attributes between the manufacturers' and Vita Lumin shade tabs. The color difference (ΔE), the algebraic distances between two points in the color space, between the manufacturers' and Vita Lumin tabs were calculated using the equation:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

The ΔE values of the different shades were subsequently used to compare the color accuracy between the different manufacturers' shade guides. Significant

*Table 1. Mean L*a*b* Values and Standard Deviations (SD) of the Manufacturers' Shade Tabs Evaluated.*

| SHADE | PRODUCT | L* (SD) | a* (SD) | b* (SD) | ΔE |
|---|---|---|---|---|---|
| A2 | Vita Lumin | 72.57 (0.06) | -0.33 (0.21) | 19.70 (0.10) | ----- |
| | Z100 | 70.47 (0.15) # | -1.20 (0.36) # | 19.07 (0.15) # | 2.36 |
| | Dyract | 69.93 (0.11) # | 0.43 (0.25) # | 17.40 (0.35) # | 2.30 |
| | Fuji II LC | 70.20 (0.26) # | -1.90 (0.45) # | 20.73 (0.73) # | 3.02 |
| | Durafill VS | 72.63 (0.37) | -1.37 (0.49) # | 25.53 (2.23) # | 5.92 |
| | TPH | 67.17 (0.15) # | -2.00 (0.10) # | 24.23 (0.51) # | 7.24 |
| A3 | Vita Lumin | 70.30 (0.10) | -0.07 (0.15) | 22.53 (0.06) | ----- |
| | Z100 | 68.37 (0.15) # | 0.43 (0.42) | 22.33 (0.21) | 1.97 |
| | Dyract | 69.23 (0.25) # | 1.10 (0.20) # | 20.07 (0.32) # | 2.87 |
| | Fuji II LC | 67.93 (0.15) # | -1.57 (0.32) # | 25.17 (0.40) # | 3.85 |
| | Durafill VS | 68.37 (0.89) # | -0.60 (0.62) | 29.60 (2.12) # | 7.35 |
| | TPH | 64.10 (0.20) # | -1.63 (0.15) # | 21.87 (0.41) # | 6.43 |
| B3 | Vita Lumin | 70.87 (0.15) | -0.63 (0.29) | 27.13 (0.31) | ----- |
| | Z100 | 68.77 (0.06) # | 0.40 (0.26) # | 29.73 (0.32) # | 3.35 |
| | Dyract | 68.03 (0.31) # | 1.70 (0.26) # | 27.60 (0.72) | 3.07 |
| | Fuji II LC | 68.83 (0.20) # | -1.97 (0.15) # | 28.70 (0.20) # | 2.90 |
| | Durafill VS | 69.63 (0.15) # | -2.50 (0.62) # | 32.17 (0.28) # | 5.52 |
| | TPH | 64.77 (0.20) # | -0.93 (0.35) | 22.83 (0.75) # | 7.47 |
| C2 | Vita Lumin | 67.30 (0.17) | -0.90 (0.35) | 21.40 (0.20) | ----- |
| | Z100 | 65.70 (0.10) # | -0.43 (0.15) | 24.60 (0.26) # | 3.61 |
| | Dyract | 66.33 (0.32) # | 0.73 (0.31) # | 18.77 (0.49) # | 2.81 |
| | Fuji II LC | 65.03 (0.11) # | -1.97 (0.20) # | 24.07 (0.40) # | 3.66 |
| | Durafill VS | 65.10 (0.32) # | -4.63 (0.60) # | 22.13 (0.85) | 4.39 |
| | TPH | 69.80 (0.30) # | -3.40 (0.40) # | 18.60 (0.36) # | 4.51 |
| C4 | Vita Lumin | 60.13 (0.15) | 1.13 (0.15) | 23.77 (0.42) | ----- |
| | Z100 | 56.70 (0.10) # | 0.57 (0.55) # | 21.17 (0.11) # | 4.34 |
| | Dyract | 58.83 (0.21) # | 0.37 (0.23) # | 23.83 (0.38) | 1.51 |
| | Fuji II LC | 62.57 (0.20) # | -0.63 (0.20) # | 28.37 (0.81) # | 5.23 |
| | Durafill VS | 60.20 (0.62) | 0.70 (0.34) # | 29.47 (0.11) # | 5.72 |
| | TPH | 58.47 (0.15) # | 2.10 (0.50) # | 27.00 (1.40) # | 3.76 |

# denotes statistically significant differences in L*a*b* values between the Vita Lumin and manufacturers' shade tabs (Results of Mann-Whitney *U* and Wilcoxon's Rank Sum W tests at significance level 0.05).

differences in ΔE were determined by Kruskal-Wallis one-way ANOVA and Mann-Whitney *U* Wilcoxon's Rank Sum W tests.

## RESULTS

Mean L*a*b* values and standard deviations for each product are listed by shade in Table 1. One-way ANOVA revealed that differences between mean L*, a*, and b* values were significant at $P < 0.05$ for all shades. When the mean L* values of manufacturers' guides were compared with the Vita Lumin shade guide, almost all product and shade combinations, with the exception of Durafill VS shade A2 and C4, exhibited significant differences. For the shades evaluated, manufacturers' shade tabs were generally darker than their corresponding Vita tabs. Only the C2 shade of TPH and C4 of Fuji II LC were lighter than their respective Vita Lumin tabs. When a* and b* values were compared, no obvious trends in difference of chromacity coordinates were observed. Only 16% of the a* and b* values of the different manufacturers' shade tabs were statistically insignificant when compared to the a* and b* values of their corresponding Vita tabs. None of the manufacturers' shade tabs evaluated had all L*, a*, and b* values identical to their respective Vita Lumin shade tabs.

The mean ΔE values and standard deviation of the different manufacturers' shade guides are reflected in Table 2. The statistical comparison of overall color accuracy (ΔE values) between manufacturers' shade guides are shown in Table 3. Ranking in the order of best to worst color accuracy (smallest to largest ΔE values) was: Dyract < Z100 < Fuji II LC < Durafill VS

*Table 2. Mean ΔE Values and Standard Deviations for the Different Manufacturers' Shade Guides*

| Products | ΔE Values | Standard Deviation |
|---|---|---|
| Z100 | 3.13 | 0.95 |
| Dyract | 2.51 | 0.62 |
| Fuji II LC | 3.73 | 0.93 |
| Durafill VS | 5.78 | 1.05 |
| TPH | 5.88 | 1.66 |

< TPH. The color accuracy of Z100 and Fuji II LC shade guides were significantly better than that of Durafill VS and TPH. The Dyract shade guide was the most accurate and was significantly better than the shade guides of Fuji II LC, Durafill VS, and TPH.

## DISCUSSION

Small-area colorimeters use photodetectors to measure reflectance of a light source from a sample. The reflectance data obtained are subsequently transformed via a microprocessor into the color dimensions of the object. The technical problem of comparing flat with irregular, curved shade tabs may have limited the accuracy of color measurements in this study. In addition, some shade tabs actually represent a gradation of different shades from cervical to incisal aspect. Emphasis was thus placed on measuring only the body shade. Although errors may occur in absolute color measurement, accurate quantitative color measurements can still be made with these devices (Seghi, Johnston & O'Brien, 1989b; Seghi, 1990). Photodetectors can, however, measure much smaller color changes than those detectable by the human eye, and the clinical relevance of color differences recorded by colorimetry must therefore be assessed. Ruyter, Nilner, and Moller (1987) showed that under clinical settings the human eye can sense ΔE values of 3.3 or greater. Under controlled environments, other investigators found that the human eye could perceive changes of color between ΔE 1 and 2 (Seghi, Johnston & O'Brien, 1986; Seghi, Hewlett & Kim, 1989a). Based on a ΔE value of 3.3 as the lower limit of perceptibility for color changes, 68% of the shade tabs evaluated were expected to have clinically detectable color differences from their corresponding Vita tabs. Only one product (Z100) had a mean ΔE value that was less than 3.3 for all shades. The results of this study show that the color of manufacturers' shade guides was not similar to the Vita Lumin shade guide to which they were supposedly keyed. If manufacturers' shade tabs are true reflections of material shade, the restorative materials will also have a clinically perceptible color difference when compared with Vita shade tabs. The Vita Lumin shade guide should, therefore, not be used in place of manufacturers' shade guides for the products evaluated. A study is currently being undertaken to determine the color accuracy of the restorative materials compared to manufacturers' and Vita Lumin shade tabs.

ΔE values indicate the overall color difference, taking into account differences in L*a*b* coordinates. As in natural teeth (Preston & Bergen, 1980), the chroma of shades was relatively low, so differences in L* attributes may have greater influence on the overall color differences (Swift & others, 1994). In general, the different manufacturers' shade tabs were darker than their respective Vita tabs. Assuming that manufacturers' guides are a true reflection of restorative color, clinical shade selection using the Vita Lumin guide (in place of manufacturers' guides) will result in restorations that are darker than the tooth tissue to which they are matched. Difference in L* values between Vita and manufacturers' shade tabs ranged from 0.06 for Durafill VS shade A2 to -6.2 for TPH shade A3. TPH had the greatest difference in L* values for four of the five shades evaluated. L* values, however,

*Table 3. Statistical Comparison of Overall Color Accuracy (ΔE Values) between Manufacturers' Shade Guides.*

| PRODUCTS | Z100 | Dyract | Fuji II LC | Durafill VS | TPH |
|---|---|---|---|---|---|
| **Z100** | ----- | NS | NS | S | S |
| **Dyract** | NS | ----- | S | S | S |
| **Fuji II LC** | NS | S | ----- | S | S |
| **Durafill VS** | S | S | S | ----- | NS |
| **TPH** | S | S | S | NS | ----- |

S denotes statistical significance and NS denotes no statistical significance (Results of Mann-Whitney *U* and Wilcoxon's Rank Sum W tests at significance level 0.05).

did not always have the greater influence on overall color. In 52% (13 out of the 25) of the product and shade combinations, it was found that the b* value had greater influence on overall color difference.

Differences in a* and b* attributes indicated differences along the red/green and yellow/blue color axes respectively. For a designated Vita shade, a tab with a higher b* value would be more yellow. One with a higher a* value would be more red. The difference in a* values between manufacturers' and the Vita shade tabs was relatively smaller than the difference in L* values. The range was only from -0.3 for TPH shade B3 to -3.73 for Durafill VS shade C2. Difference in b* values was, however, much larger and ranged from 0.06 for Dyract shade C4 to 7.07 for Durafill VS shade A3. Sixty percent (15 of the 25) of the shade tabs evaluated were more yellow than their corresponding Vita shade tabs. Clinically, shade selection with the Vita Lumin guide in place of manufacturers' guides may result in restorations that are more yellow than the tooth tissues that they are to replace. The assumption here is that the b* values of the manufacturers' guide are identical to their product.

When shade guides were compared, the color accuracy of Z100 and Fuji II LC shade guides compared to the Vita Lumin shade guide was significantly better than that of Durafill VS and TPH. The Dyract shade guide was the most accurate and was significantly better than the shade guides of Fuji II LC, Durafill VS, and TPH. The results were not expected, especially since Dyract shade tabs were made of the actual restorative material instead of plastic like Z100, Fuji II LC, and TPH. The shade tabs of Durafill VS were also fabricated from the actual restorative materials. The accuracy of manufacturers' shade guides was not consistently product dependent but was shade dependent.

## CONCLUSIONS

Results of this study showed that

1. None of the manufacturers' shade tabs evaluated had color attributes that were identical to their respective Vita shade tabs.
2. For the shades evaluated, manufacturers' tabs were generally darker than their corresponding Vita shade tabs.
3. The difference in a* values was relatively smaller than the difference in L* values.
4. When shade guides were compared, the color accuracy of Z100 and Fuji II LC shade guides was significantly better than that of Durafill VS and TPH.
5. The Dyract shade guide was the most accurate and was significantly better than the Fuji II LC, Durafill VS, and TPH guides.
6. The accuracy of manufacturers' shade guides was not consistently product dependent but was shade dependent.
7. The Vita Lumin shade guide should not be used in place of manufacturers' shade guides in clinical shade selection.

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# Change in Size of Replaced Amalgam Restorations: a Methodological Study

I A MJÖR • R L REEP
P S KUBILIS • B E MONDRAGÓN

**Clinical Relevance**
If marginal discrepancies or recurrent caries were present, replacement restorations showed a statistically significant increase in size compared with the original restorations.

## SUMMARY

**The purpose of this study was to evaluate the pre- and postoperative size of amalgam restorations that were scheduled for replacement. A video imaging system in combination with specially designed devices to align the stone casts prepared from impressions of the teeth gave reproducible results for both an in vitro and in vivo series. The in vivo series showed that removal of the occlusal part of amalgam restorations could be done without significantly increasing the size of the cavity in the tooth, provided the restorations did not have marginal discrepancies. If marginal discrepancies or recurrent caries were present, the replacement restorations showed a statistically significant increase in size compared with the original restorations.**

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## INTRODUCTION

The preservation of tooth structure during cavity preparation, both in the treatment of primary caries and when replacing failed restorations, is of paramount importance to maintain the strength of restored teeth (Hood, 1991). It is generally accepted that repeated replacements result in larger restorations, but limited data are available to show the detailed extent of the increased size following replacement in clinical studies. Elderton (1977) made direct measurements on epoxy models prepared from impressions of occlusal amalgam restorations that had failed due to marginal degradation and the subsequent cavity preparation. He found that the average cavity margin was cut back 0.34 mm. If recurrent caries was diagnosed, the cavity margin was cut back on the average 0.52 mm, compared with

0.25 mm when no caries was present. Brantley and others (1995) recorded additional unrestored surfaces that were recommended to be included when restoring teeth, but no detailed measurements were made.

Replacement of failed restorations constitutes about 65-80% of all operative work on adult patients (Mjör, 1981; Nuttall, 1984; Klausner & Charbeneau, 1985; Maryniuk & Kaplan, 1986; Boyd, 1989). The purpose of the present study was to assess the increase in size of replaced amalgam restorations compared with that of failed restorations with emphasis on the methodology involved in the assessment. A number of techniques were considered and evaluated before selection was made of a video imaging system to measure the size pre- and postoperatively of restorations in conjunction with special alignment devices.

### Direct Measurements

Attempts to make measurements directly on stone casts of the teeth preoperatively and after completion of the cavity preparation did not provide reproducible results on repeated measurements. Measurements at low magnification in a dissecting microscope were unreliable because of difficulties in aligning the occlusal surfaces on the two stone models and in selecting identical measuring points. Some of the same difficulties were experienced if photographs were taken of the occlusal surfaces and the measurements made on enlarged black-and-white prints. Attempts to orient the two specimens by providing them with a base using the preoperative impression for the orientation of both stone casts (Figure 1) also proved to be of limited value in the orientation of the specimens. Finally, a special alignment device was constructed that allowed three-dimensional orientation of the specimen (Figure 2). This device used in combination with a video imaging system similar to that employed by Whitehead and Wilson (1988) proved suitable to quantify the occlusal surface area. The present report will describe this technique and the results obtained from an in vitro series and from two in vivo series, involving replacement of amalgam restorations with and without occlusal defects.

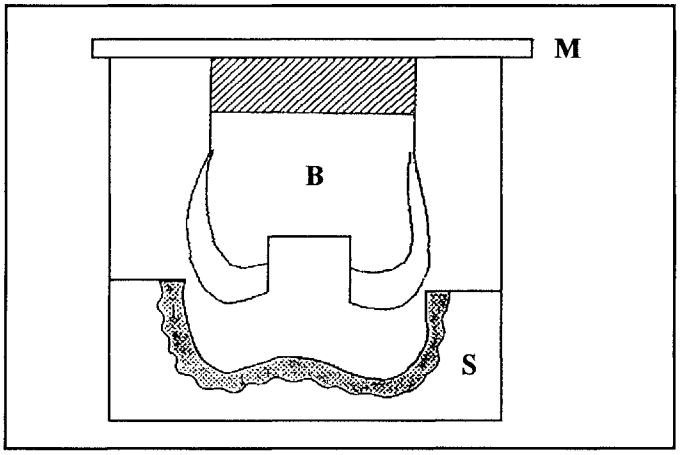


Figure 1. *Diagram of device used to align the bases of the preoperative and postoperative stone casts. The impression of the tooth taken preoperatively is used to prepare a stone cast of the tooth (not shown), which is carefully removed. The impression is then placed in dental stone (S) in a sturdy plastic cylinder. The stone cast of the tooth prepared from a postoperative impression was then positioned into the preoperative impression. The hatched area of the postoperative stone cast (B) is filled with dental stone after the cast has been positioned in the impression. Then the preoperative stone cast was repositioned in the impression and its base completed (the hatched area) against the microscope slide (M) on which the postoperative stone cast was already glued.*

## METHODS AND MATERIALS

Two series of measurements using a video imaging system (Kontron IBAS-AT, Kontron Elektronik GmbH, Munich, Germany) were performed (1) on amalgam restorations in plastic teeth and of cavity preparations after the removal of the restorations and (2) on stone casts prepared from impressions of amalgam restorations and cavity preparations prepared in vivo by students in the clinic of the Department of Operative Dentistry, University of Florida. The students were not informed about the purpose of the study, and several projects involving the same clinical recordings were ongoing at that time.

Bases for the stone casts in the in vivo series were prepared using the device shown in Figure 1. This device was used to obtain similar orientation of the pre- and postoperative casts. The specially designed stand (Figure 2) allowed final alignment of the occlusal surfaces of the two stone casts of the same tooth by controlled x-y translational movement,

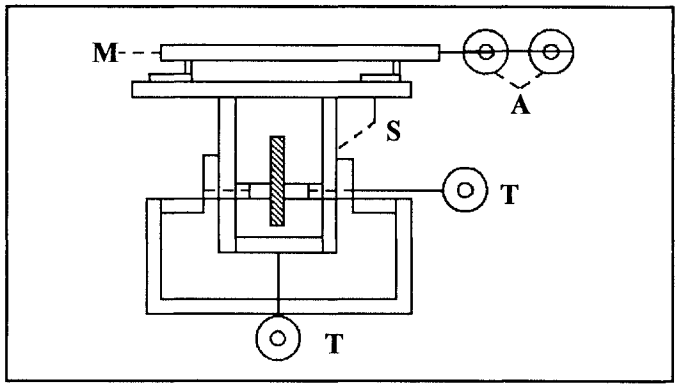


Figure 2. *Diagram of device used for detailed alignment of preoperative and postoperative specimens. M = microscope stand with two adjustable screws (A) for x-y movement; S = laboratory surveyor that could be rotated and tilted in two directions (T).*

rotation, and tilt. The essential parts of this device were a microscope table that allowed movements in two dimensions and a laboratory surveyor that allowed controlled rotation and tilting. Thus, the two stone casts of the same tooth could be three-dimensionally aligned using anatomical landmarks such as fissures, grooves, and ridges for guidance.

The recordings were limited to the size of the occlusal surfaces. For class 2 restorations, cut-off lines were selected based on anatomical landmarks to exclude interproximal areas or extensions from the occlusal surface (Figures 3A&B). Lighting proved to be a critical factor in video imaging, and optimal contrast was obtained using diffused light generated by two copy stand lamps positioned on either side of the specimen, elevated approximately 30 cm above the plane of the specimen, and tilted at about 45° toward the specimen.

Images were digitized using a Dage CCD camera (Dage MTI, Inc, Michigan City, IN 46360) equipped with a macro lens, at a fixed magnification of X10-15 for each pair of specimens, and displayed on the image display monitor. The same magnification for each pair of specimens was used, but since teeth vary in size, magnification was varied slightly to make measurements on images that were as large as possible. Pixel resolution of the cursor used to outline perimeters of areas of interest was 1 pixel. Although this would translate to slightly better absolute resolution at X15 compared to X10, the large perimeters outlined (i e, large relative to cursor pixel resolution), together with the high reliability of the replicate measurements, make it very unlikely that variations of X10-15 in pair magnifications influenced the results. The edge of the original cavity preparation was then traced using the graphics cursor, and its occlusal area was computed in pixels. Upon completion of area measurements for the first specimen of a pair, the video input was switched to on-line and the second specimen of the pair brought into view and aligned. Landmark features were marked with the graphics cursor for use in alignment of the second specimen of the pair. Usually, x-y translation and slight rotation, with little or no tilt, were sufficient to produce good alignment of landmarks with the graphic overlay. This image was then digitized and the area measurement made. Black-and-white photographs were made of selected specimens for illustration purposes using a matrix film recorder (Figures 3A&B). However, these photographs did not do justification to the resolution observed on the display monitor.

### In Vitro Study

Class 1 and class 2 cavity preparations were prepared in 20 Dentoform (Kilgore International, Inc, Coldwater, MI 49036) molar teeth and restored with amalgam by one of the investigators (IAM). The teeth were fixed on a microscope slide with glue and the occlusal outline of the restorations recorded. An occlusal cut-off line based on identifiable anatomical landmarks was selected in the isthmus area of class 2 restorations. The restorations were

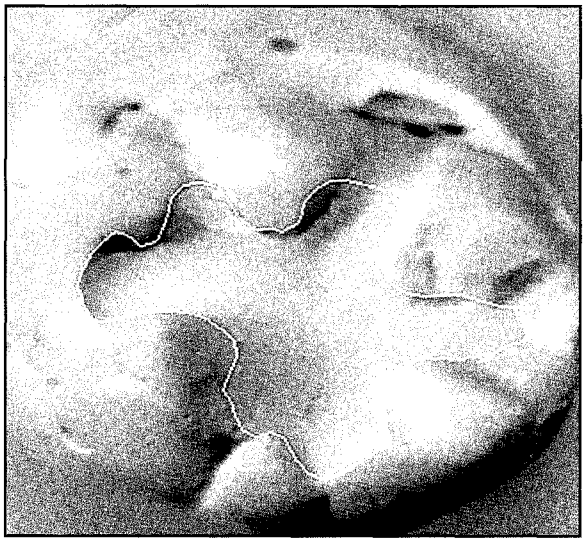

Figure 3A

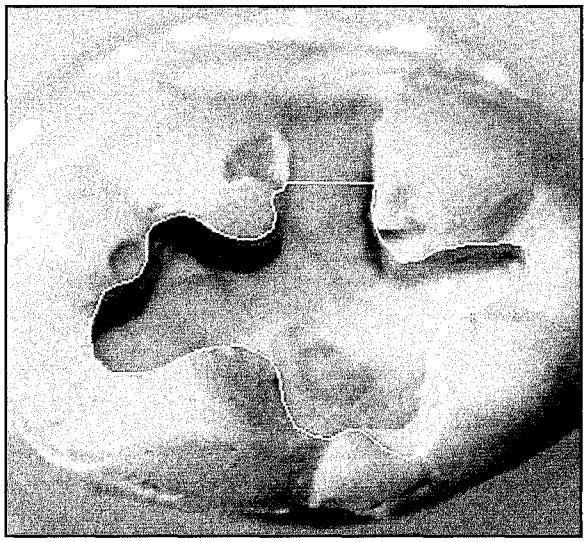

Figure 3B

Figures 3A & B. *Photographs directly off the video screen of stone cast preoperatively (A) and postoperatively (B) of a specimen with an OL restoration, which was replaced due to leakage. Note tracing outlining the periphery of the restoration and of the cavity preparation. A cut-off line excluded the buccal extension of the cavity from the area measurement. The preoperative measurement was 17,360 pixels and postoperative measurement was 17,668 pixels.*

then removed by intentionally extending the size of the preparation in 11 teeth and by attempting to remove the restorations with minimal increase in cavity size in nine teeth. Measurements of the size of the cavity preparations were then performed and again the occlusal cut-off line at the isthmus area of the class 2 preparations using a photograph of the restored tooth for identification was used as the identifiable landmark. Special landmarks could also be placed on the plastic teeth, but since these teeth were on the original base as prepared by the manufacturer throughout the procedure, no alignment difficulties were experienced.

Duplicate measurements were performed immediately following the initial measurements and also after several days to assess the reproducibility of the measuring technique. One investigator performed all the tracings of the restoration and cavosurface margins (RLR). Positioning and shadowing of the specimen were done by two of the investigators (IAM and BEM). Duplicate measurements performed after several days were blinded, i e, the measurer did not know the result of the previous measurements.

### In Vivo Study

Impressions of 20 restorations that had been scheduled for replacement following routine treatment planning in the student clinic at the University of Florida were included in the study. These were divided into two groups. Group 1 comprised 10 restorations with occlusal marginal defects: seven with the clinical diagnosis of ditching/poor margins and three with the diagnosis of occlusal secondary caries. Group 2 comprised 10 restorations with clinically intact occlusal surfaces that needed replacement for defects at some other part of the restorations. Impressions were made prior to removal of the restorations and of the prepared cavities immediately prior to restoring the teeth. Stone casts were prepared for each impression (Silky-Rock Die Material, Whip Mix Corp, Louisville, KY 40217) and the size of the occlusal area measured using the video imaging system. Cut-off lines were selected as appropriate (Figure 3). The size of the occlusal restorations to be replaced and of the final cavity preparation were compared. Two to four readings were made in three pairs of specimens to test the reproducibility of the measuring technique.

### Statistical Analysis

The reliability of replicate measurements for the in vitro study was assessed using the intraclass correlation coefficient (ICR). The ICR can be interpreted as the proportion of variance that can be attributed to error-free variability among measurements, i e, variability not introduced by the measurer. For specimens with variation in the number of replicate measurements, the harmonic means of replicates per tooth for each type and status were used to compute the ICR and its lower 95% confidence limit.

For the in vivo study the average change between the size of the final cavity preparation and the original restoration was estimated along with corresponding 95% confidence intervals. The paired *t*-test was used to determine if the observed mean change differed significantly from 0. The independent sample *t*-test was used to determine if the observed mean change differed significantly between Groups 1 and 2. The reliability of replicate measurements of the size of the final cavity preparation and the restoration to be replaced and the corresponding within-replicate differences were assessed using the ICR. Since the number of replicate measurements varied, the harmonic means of replicates per tooth were used to compute the ICR and its lower 95% confidence limit. All statistical analyses were done by one of the investigators (PSK).

## RESULTS

### In Vitro Samples

The reproducibility of measurements was very good, with ICRs ranging from 0.947 for the occlusal part of class 2 specimens to 0.987 for class 1 specimens. Thus, the percentage of variability in measurements that can be attributed to error-free variability among teeth ranged from 94.7% to 98.7%. Lower 95% confidence limit for the ICR ranged from 0.907 to 0.950 for class 1 measurements and from 0.832 to 0.836 for the class 2 measurements.

### In Vivo Samples

The data for Group 1 and Group 2 measurements are illustrated in Figures 4 and 5. The area values expressed in video scale units for the restoration (preop) and after completed cavity preparation (postop) in the same tooth are connected in the center part of the illustrations. The mean preop and postop measurements are indicated by short, thick, horizontal lines.

The mean within-tooth change in the size of the final cavity preparation relative to the original restoration differed significantly from 0 in Group 1 ($P = 0.007$), but not in Group 2 ($P = 0.101$). The reliability of within-replicate measurements in Group 1 (96.4% with a lower limit of 91.2%) is comparable to the reliability of preop and postop measurements (98.0% and 96.2% with corresponding lower limits of 90.7% and 91.2%). The reliability of the preop and postop measurements in Group 2 was

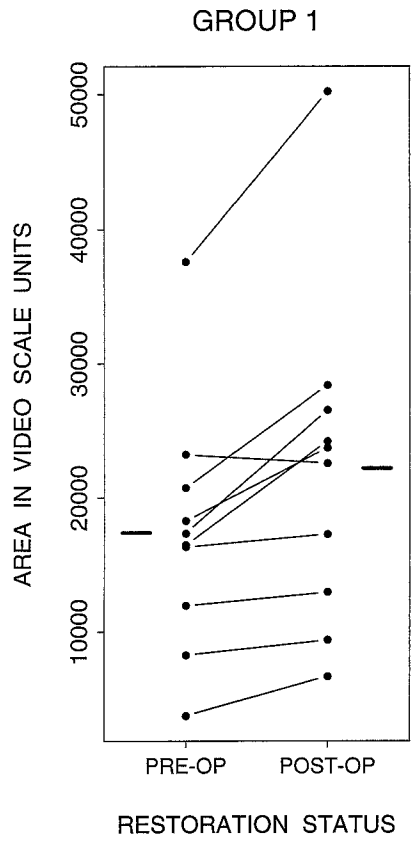


Figure 4. *Results of occlusal area measurements of restorations (preop) and completed cavity preparation (postop) from Group 1, where the replacement of the restoration was due to occlusal marginal defects, including ditching and recurrent caries. Preoperative and postoperative values for the same tooth are connected in the center part of the illustration. The degree of inclination of these lines is an expression of the increase in size from the original to the replaced restoration. Preoperative and postoperative means are indicated by the short, thick, horizontal bars.*

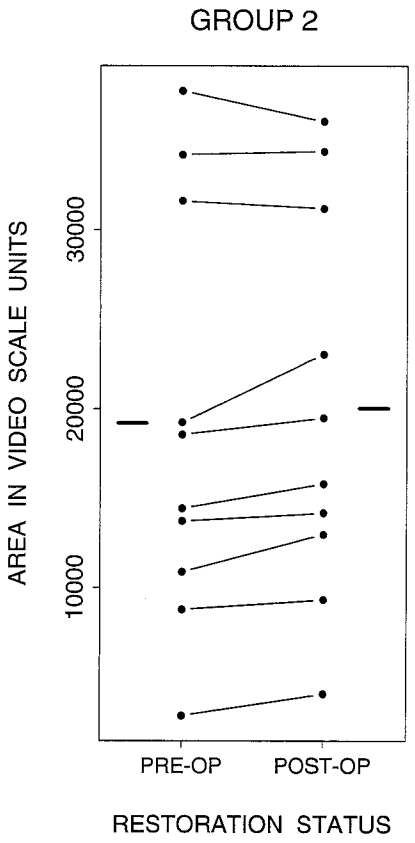


Figure 5. *Results of occlusal area measurements of restorations (preop) and after completed cavity preparation (postop) from Group 2, where the replacement of the restoration was due to defects at locations other than at the occlusal surface. Preoperative and postoperative values for the same tooth are connected in the center part of the illustration. The degree of inclination of these lines is an expression of the increase in size from the original to the replaced restoration. Preoperative and postoperative means are indicated by short, thick, horizontal lines.*

also excellent (99.8% and 99.7% within corresponding lower limits of 99.1% and 99.7%), but was effectively 0 for within-replicate area differences.

## DISCUSSION

The in vitro part of this study showed that the imaging technique employed was suitable for recording the periphery and, therefore, the area of restorations and of cavity preparations. Thus, suitability of the technique used by Whitehead and Wilson (1988) was confirmed. The reproducibility was excellent, although better for class 1 samples than for class 2. The lower magnitude of confidence limits for class 2 measurements may reflect variations in the occlusal cut-off lines, but it may also be attributable to a smaller number of replicates per tooth available for class 2 than for class 1 analysis.

The in vivo part of the study showed that the imaging technique was suitable for recording detailed changes in the size of restorations and cavity preparations performed under clinical conditions. Duplicate readings confirmed that the technique also was reproducible when applied on stone casts of teeth taken pre- and postoperatively. The specially designed devices (Figures 1 and 2) allowed for controlled three-dimensional, optimal orientation of the pre- and postoperative specimens. In two instances, one in each group, where a larger value for the area of the final cavity preparation was recorded as smaller than the original restoration, it turned out that the amalgam restoration had extended beyond the original cavity preparation.

The difference between pre- and postoperative

dimensions in Group 1 where marginal discrepancies or recurrent caries were clinically diagnosed confirmed results by Elderton (1977), who showed that more tooth tissue was lost in teeth with the clinical diagnosis of recurrent caries than in teeth with no caries. However, the present results indicated that on an average no significant loss of tooth structure occurred when no marginal discrepancies were diagnosed (Figure 4), although some variations were noted that may be due to operator technique (Hunter, Treasure & Hunter, 1995).

Elderton (1977) showed that all amalgam restorations increased in size following replacement irrespective of the reason for replacement. This finding was not confirmed in the present study. Thus, the present in vivo results indicate that no significant amount of tooth structure should be lost if no marginal discrepancies are present, which agrees with in vitro data published by Whitehead and Wilson (1988) and the in vitro results from the present study. Other in vitro data have indicated marked operator variation and also variations depending on the material to be replaced (Hunter & others, 1995). In these studies significantly more tooth structure was lost when replacing composite than amalgam restorations, probably because the tooth-colored material was hard to differentiate from tooth structure. In addition, resin-impregnated acid-etched enamel must be removed prior to inserting a new bonded tooth-colored restoration. The results from the composite part of that report need to be confirmed in clinical studies, and the imaging technique employed in this study is suitable for such investigations.

## CONCLUSIONS

The following conclusions were reached based upon the data obtained by this study:

1. Occlusal amalgam restorations can be removed without increasing their size provided no marginal discrepancies exist.
2. A significant increase in size can be expected for replacement restorations where marginal discrepancies and/or recurrent caries are present.
3. The use of a video imaging system combined with specially designed devices provided reproducible in vitro and in vivo results.

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# STUDENT AWARDS

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## OPERATIVE PEARLS

Please submit your own wonderful, yet secret, tips for practicing at a higher level and/or comments regarding this section via FAX (206) 543-7783 or via e-mail to rmccoy@u.washington.edu.

### REMOVAL OF TISSUE-RETRACTION MATERIAL
**Contributed by:**
**Dr Warren Johnson, Seattle, WA**

Wash the preparation and the tissue-retracting material to remove any excess hemostatic agent, saliva, or blood coagulants. Keep area dry and retracted with cotton rolls, dri-angles, cotton roll holders, or any other successful means. Air is gently blown into the sulcus by the assistant as the retraction material is slowly being withdrawn by the operator. The tissue will remain retracted while the preparation is being inspected for adequate retraction and the sulcus is free of blood or saliva. At this time the impression material may be mixed and inserted for an excellent, one-time impression where the margins are all clearly visible.

### TISSUE-RETRACTING MATERIAL
**Contributed by:**
**Dr Warren Johnson, Seattle, WA**

Three- or four-strand white cotton yarn can be purchased at a fabric or craft store. The yarn should be soft and expandable. It can be precut into lengths of ½-inch or 1 cm, 1 inch or 2.5 cm, and 1½-inch or 3.5 cm. The shortest length can be used to retract interproximal tissue or add to already retracted tissue where loose tissue needs more lateral retraction. The middle length can be used to retract partial-coverage preparations or full-coverage preparations of all teeth except molars. The longest length can be used to retract tissue for full-coverage preparations. Different lengths, number of strands, and numbers of cords may be used to mix and match for achieving adequate tissue retraction. Cotton pellets used as is or teased into strands can also be used to achieve extra retraction, if needed. The yarn lengths, cotton pellets, or cotton strands can be wetted first into the hemostatic agent of choice prior to placement for tissue retraction.

## ANNOUNCEMENTS

### 28th ANNUAL MEETING of the ACADEMY OF OPERATIVE DENTISTRY

**17-19 February 1999**
**FAIRMONT HOTEL**
**CHICAGO, ILLINOIS**

For meeting information please contact Dr Gregory Smith, P O Box 14996, Gainesville, FL 32604-2996; FAX (352) 371-4882.

**NOTE THE CHANGE OF MEETING LOCATION FROM THE WESTIN TO THE FAIRMONT HOTEL.**

### AMERICAN ACADEMY OF GOLD FOIL OPERATORS MEETING

**7-10 October 1998**
**University of Minnesota**
**School of Dentistry**
**Minneapolis, Minnesota**

For details contact the AAGFO Secretary-Treasurer, Dr Ronald Harris at 17922 Tallgrass Court, Noblesville, IN 46060; (317) 867-0414; FAX (317) 867-3011.

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