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E D I T O R I A L

# Unwarranted Removal of Dental Amalgams

The amalgam toxicity issue remains a hot one in North America as well as around the world. The infamous "60 Minutes" television program by CBS this past December provided a biased view of the topic; it was biased in favor of those who preach that all dental amalgam is bad, and not only should we not be using it, but all amalgams might be better off removed. For sometime we have had in this country a small but ever-increasing number of dentists and other professionals, a vocal and persistent minority, who promote such a concept. Some dentists advertise that they practice dentistry without the hazards of mercury. The newsletters they send to patients and prospective patients in their area may not come right out and *say* that mercury causes a host of diseases such as multiple sclerosis, depression, neurologic and other terrible diseases, but they imply that this is so. These newsletters then say that if patients are concerned, they should see the dentist about the possibility of having such restorations removed.

Patients with serious diseases, whether imagined or real, will go to any length to seek help in the form of some miraculous cure. Because of antiamalgam promotion, patients are frequently duped into having a large amount of unnecessary dental treatment. One question we cannot answer is how many dentists are a part of this movement because of their erroneous conviction that amalgam is a health hazard, or because they are simply promoting this for their own pocketbook.

This unwarranted removal of serviceable dental amalgam generated by scare-tactics of the antiamalgamists is highly unethical and causes near financial ruin for some patients. It is my understanding that several older patients who have sought out these charlatans for full-mouth replacement of serviceable amalgam restorations have been forced to mortgage their homes to pay their dental bills. Talk about unethical and immoral standards! Something must be done!

The State of Washington Department of Health Dental Disciplinary Board is taking steps to protect the public from such unwarranted treatment. The Board has developed a rule that would classify the unwarranted removal of serviceable amalgams solely to substitute a material that does not contain mercury as unprofessional conduct. The new rule also states that "Any dentist who advertises that dental amalgams are toxic, unsafe or cause health problems or whose advertising promotes the replacement of amalgam restorations solely to subsititute a material that does not contain mercury shall be considered to have engaged in advertising which is false, fraudulent, or misleading in violation of RCW 18.130.180(3)."

It took a great deal of courage for this particular board to propose such a rule. They are to be congratulated!

The first public hearing was held in the Seattle, Washington, area on 12 October. At this hearing there were more than 130 individuals, most of whom were opposed to the adoption of this rule. The state had two experts testify for 30 minutes each. The people in the audience were each granted three minutes. Those opposed to the rule demanded that the State bring in two outside consultants for the antiamalgam group and that a new hearing be held for them. The State is proceeding with this, and a new hearing is being held on 9 November. After this hearing, I would anticipate that the board will follow sound scientific advice and adopt the new rule.

Let us hope that other states take a serious look at this emotionally charged issue and address it in a similar manner. It is time that we stop pussyfooting around this issue and deal with the offenders who indiscriminately remove serviceable restorations.

DAVID J BALES
Editor

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ORIGINAL ARTICLES

# Long-term Deterioration of Composite Resin and Amalgam Restorations

ROGER J SMALES


## Summary

**Previous long-term longitudinal studies of two different methods of placing an auto-cured conventional anterior composite resin, and of a low- and a high-copper amalgam alloy, had shown similar restoration survivals despite the different resin treatment methods used or the types of amalgam alloy placed. Therefore, the aim of the present study was to assess several clinical factors or characteristics of these restorations that were believed to affect the survival of the restorative materials. The 950 composite resin and the 1042 amalgam restorations examined were placed by many operators in numerous patients attending a dental hospital. The composite resin restorations were placed using unetched- and etched-enamel-bonding treatment methods, and the amalgam restorations were polished after insertion. Clinical ratings supplemented by color transparencies were used for the assessment of four factors for the resin, and four factors for the amalgam restorations. Significant deterioration differences were found for several of the clinical factors assessed for both the two different composite resin treatment methods, and for the two different amalgam alloys, which were not directly related to the restoration survivals.**


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## Introduction

In one longitudinal study over periods of up to 16 years of an anterior composite resin placed with or without enamel acid-etching and a bonding resin (Smales, 1991a), and in another separate study of up to 10 years of a low- and a high-copper amalgam alloy (Smales, 1991b), no statistically significant

differences in restoration longevity were found between either the two methods of resin placement or between the two different amalgam alloys.

Therefore it was decided to evaluate the restorations from each study for any differences that may have been present in the deterioration of several clinical factors or characteristics that were believed to affect the survival of the restorative materials.

## Materials and Methods

The materials assessed are listed in Table 1. Concise, an auto-cured conventional composite resin, was placed with and without enamel acid-etching and a low-viscosity Enamel-Bond (EB) resin. Details of the materials, their methods of placement and assessment have all been described previously (Smales, 1975, 1977a, 1977b, 1983a,b). The 471 Concise restorations were placed from 1971 and the 479 Concise EB restorations from 1974 by many dentists and supervised students in the anterior permanent teeth of eligible patients attending a dental hospital. The 774 New True Dentalloy low-copper amalgam restorations and the 268 Indiloy high-copper amalgam restorations were placed from 1967 and 1977 respectively by similar operators in the posterior permanent teeth of the same patient population as were the composite resins. All the amalgam restorations were polished after insertion.

Patients were examined at varying intervals over the two study periods, and the resin and amalgam restorations were assessed by "double blind" technique for several clinical factors using direct clinical observations supplemented by the examination later of Ektachrome 35 mm color transparencies (Eastman Kodak Co, Rochester, NY 14650) taken at 1:1 magnification. For the composite resin restorations, the clinical factors assessed were surface staining, marginal staining, marginal fracture, and color mismatch. Each factor was rated using an ordinal scale of 0 to 3, 0 representing no detection of any deterioration (Good), 1 slight changes and 2 obvious changes from ideal (Adequate), and 3 indicating that the changes were severe and required some form of treatment (Unsatisfactory). For the amalgam alloy restorations, the clinical factors assessed were surface roughness, surface tarnishing, marginal staining, and marginal fracture. Each factor was rated using an ordinal scale of 0 to 2, 0 representing no detection of any deterioration (Good), 1 slight or obvious changes from ideal (Adequate), and 2 indicating severe changes (Unsatisfactory). The assessment method has been described previously (Smales, 1983b).

As the rating assessments were based on ordinal variables, these were transposed before statistical analysis. The method involved ordering all the original ratings or scores by rank, calculating the empirical probability of each score from the data, and then selecting the Z-value from the normal Gaussian distribution curve with the same probability. All tied scores were treated by averaging their normal values. The transposed data for each clinical factor were then analyzed separately using a mixed-model analysis of variance. This model included a random effects term for patients, a co-variate effect for restoration age, and a fixed effect for each composite resin treatment method or amalgam alloy whose significance was determined by a likelihood ratio test. Statistical analysis was undertaken with BMDP program 3V (Dixon, 1990) on a minicomputer. Significant differences between the scored means over the studies were assumed to exist when there was an alpha probability value of 0.01 or less.

*Table 1. Materials Assessed*

| Material | Manufacturer | Composition |
|---|---|---|
| Concise/ Enamel Bond | 3M Dental Products Co, St Paul, MN 55144 | BIS-GMA resin, 70% Borosilicate |
| New True Dentalloy | S S White Ltd, Harrow, UK | Fine lathe cut, 2% Cu, 1% Zn |
| Indiloy | Shofu Dental Co, Kyoto, Japan | Spheroidal, 13% Cu, 4% In |

## Results

There were 732 Concise, 558 Concise EB, 1176 New True Dentalloy, and 425 Indiloy restoration observations made over the two study periods.

Comparisons of the percentages of observations rated as Good, Adequate, or Unsatisfactory over the studies for the two composite resin treatment methods and for the two amalgam alloys are shown in Tables 2 and 3. Apart from the surface tarnishing of Indiloy, there were relatively few Unsatisfactory ratings recorded for any of the clinical factors assessed. The percentage rating score changes over the two studies are illustrated for composite resin color mismatch in Figures 1 and 2, and for amalgam alloy surface tarnishing in Figures 3 and 4. All clinical factors deteriorated significantly with aging of the resin and amalgam restorations, $P < 0.001$.

For the two composite resin treatment methods, the Z-values for the four clinical factors and related asymptotic *t*-test results are shown in Tables 4 and 5. Apart from color mismatch and marginal staining, where the Concise EB treatment gave the better restoration results, there were no significant differences between the two methods for surface staining, or marginal fracture.

*Table 2. Percentages of Rating Scores of the Two Composite Resin Treatment Methods over the Study Period*

| | | Percentages of observations rated as: | | |
|---|---|---|---|---|
| **Clinical Factor** | **Treatment Method** | **Good (0)** | **Adequate (1,2)** | **Unsatisfactory (3)** |
| Clinical staining | Concise | 82.2 | 16.6 | 1.2 |
| | Concise EB | 80.6 | 17.6 | 1.8 |
| Marginal staining | Concise | 64.5 | 31.8 | 3.7 |
| | Concise EB | 74.7 | 22.6 | 2.7 |
| Marginal fracture | Concise | 62.0 | 36.1 | 1.9 |
| | Concise EB | 70.5 | 27.9 | 1.6 |
| Color mismatch | Concise | 26.5 | 63.5 | 10.0 |
| | Concise EB | 52.0 | 45.1 | 2.9 |

*Table 3. Percentages of Rating Scores for the Two Amalgam Alloys over the Study Period*

| | | Percentages of observations rated as: | | |
|---|---|---|---|---|
| **Clinical Factor** | **Treatment Method** | **Good (0)** | **Adequate (1,2)** | **Unsatisfactory (3)** |
| Surface roughness | NTD | 55.4 | 38.9 | 5.7 |
| | Indiloy | 72.2 | 26.2 | 1.6 |
| Surface tarnishing | NTD | 44.9 | 46.1 | 9.0 |
| | Indiloy | 29.9 | 48.9 | 21.2 |
| Marginal staining | NTD | 82.7 | 14.6 | 2.7 |
| | Indiloy | 81.6 | 15.1 | 3.3 |
| Marginal fracture | NTD | 48.8 | 43.5 | 7.7 |
| | Indiloy | 72.9 | 24.7 | 2.4 |

*Table 4. Z-Values over the Study Period for the Two Resin Treatment Methods*

| **Treatment Method** | **Surface Staining** | **Marginal Staining** | **Marginal Fracture** | **Color Mismatch** |
|---|---|---|---|---|
| Concise | - 0.01 | 0.08 | 0.06 | 0.23 |
| Concise EB | 0.01 | - 0.10 | - 0.08 | - 0.29 |
| *P* (from likelihood ratio test) | 0.37 | < 0.001* | 0.02 | < 0.001* |

*Significant difference present at the 1% level of probability

*Table 5. Asymptotic t-tests for the Two Resin Treatments for Predicted Cell Means*

| **Surface Staining** | **Marginal Staining** | **Marginal Fracture** | **Color Mismatch** |
|---|---|---|---|
| 0.89 | 4.06* | 2.35 | 8.08* |

*Values of 2.6 (1% probability level) or greater considered to be of statistical significance

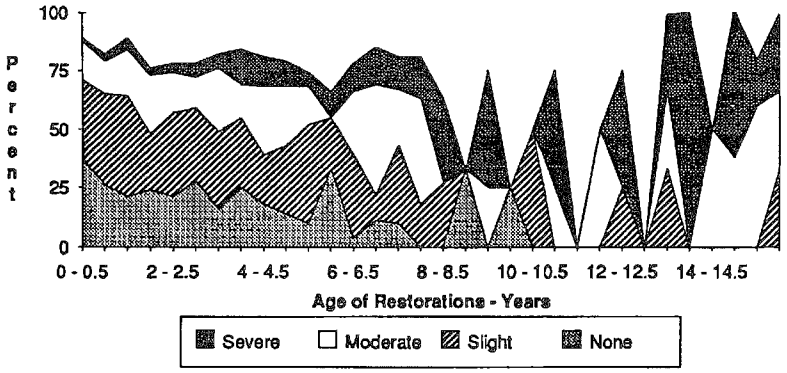


FIG 1. *Percentage rating scores for Concise color mismatch*

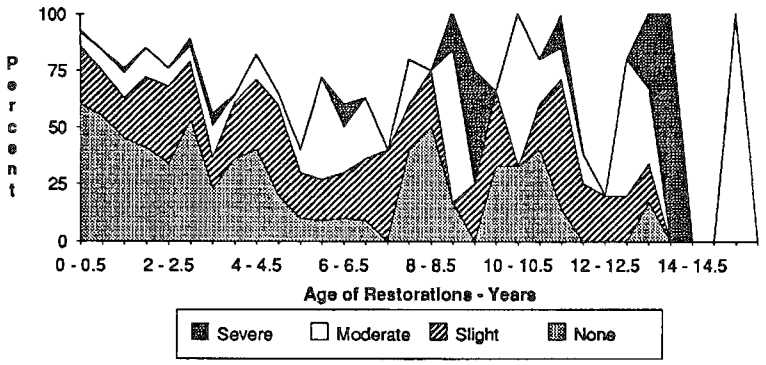


FIG 2. *Percentage rating scores for Concise EB color mismatch*

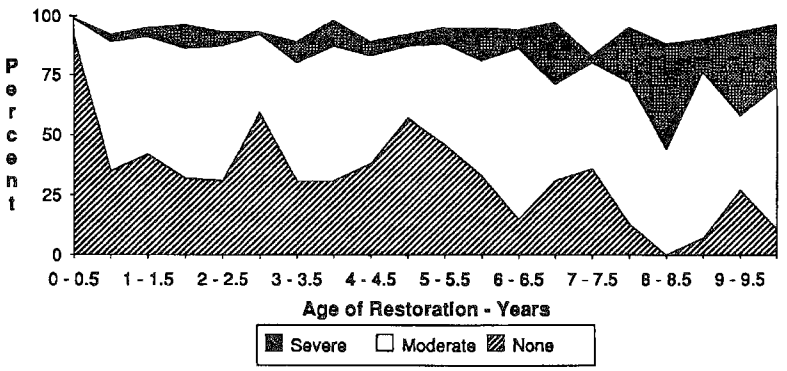


FIG 3. *Percentage rating scores for New True Dentalloy surface tarnishing*

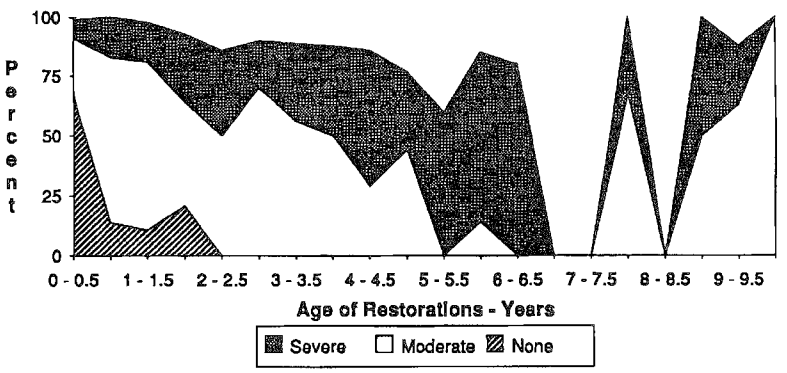


FIG 4. *Percentage rating scores for Indiloy surface tarnishing*

For the two amalgam alloys, the Z-values for the four clinical factors and related asymptotic *t*-test results are shown in Tables 6 and 7. Indiloy restorations showed significantly more surface tarnishing and New True Dentalloy significantly more surface roughness and marginal fracture, but there were no significant differences present between the two alloys for marginal staining.

*Table 6. Z-Values over the Study Period for the Two Amalgam Alloys*

| Material | Surface Toughness | Surface Tarnishing | Marginal Staining | Marginal Fracture |
|---|---|---|---|---|
| New True Dentalloy | 0.08 | - 0.09 | - 0.01 | 0.11 |
| Indiloy | - 0.22 | 0.24 | 0.01 | - 0.29 |
| *P* (from likelihood ratio test) | < 0.001* | < 0.001* | 0.03 | < 0.001* |

*Significant difference present at the 1% level of probability

*Table 7. Asymptotic t-tests for the Two Amalgam Alloys for Predicted Cell Means*

| Surface Roughness | Surface Tarnishing | Marginal Staining | Marginal Fracture |
|---|---|---|---|
| 3.81 | 7.05* | 2.21 | 4.20* |

*Values of 2.6 (1% probability level) or greater considered to be of statistical significance

## Discussion

In vitro studies of marginal leakage and bonding strengths have reported variable results with the use of low-viscosity enamel bonding resins, when compared to the direct application of the composite resin to the etched enamel (van Dijken & Hörstedt, 1987), but the relevance of such studies to the long-term survivals of different composite resin restorations has not been determined by controlled clinical trials.

Conventional auto-cured composite resin restorations placed with acid-etching of the enamel, and with bonding resin, have shown improvements in their marginal adaptation and discoloration when compared to restorations placed without enamel etching in several two-year studies (Bozell & Charbeneau, 1979; Oram & Lyders, 1981). However, the use of enamel acid-etching alone did not eliminate longer-term marginal discrepancies (Christensen & Christensen, 1982; van Dijken, Hörstedt & Meurman, 1985; van Dijken, 1986: Crumpler & others, 1988), marginal discoloration (van Dijken, 1986; Crumpler & others, 1988), caries and the loss of restoration retention, even with the later repeated use of low-viscosity intermediate bonding resins (van Dijken, 1986). In the present study, the Concise EB treatments showed significantly less marginal staining than did Concise placed in unetched preparations, $P < 0.001$, but arguably significantly less marginal fracture, $P = 0.02$.

The Concise EB treatments also showed significantly better color matchings than did the use of Concise placed in unetched preparations, $P < 0.001$. However, the etch-bonding treatments used in the study did not begin until 1974, and the Concise resin material was modified by the manufacturer on several occasions following its initial placement from 1971. Despite the significant color matching and marginal staining differences found between the two methods of resin placement, their restoration survivals were very similar: $P = 0.75$ (Smales, 1991a).

During the study period, there were significant increases in the rating scores for all clinical factors, and this deterioration trend was similar to that reported in earlier studies of the same populations (Smales, 1975, 1977a, 1983a). However, in one other study of a small retrieved population of Adaptic restorations placed without acid-etching or bevels, there were no significant changes found over 12 years for marginal adaptation and color match, and significant changes only occurred in the first three years for cavosurface discoloration (Osborne, Norman & Gale, 1990). Despite the findings of these studies, it would be unwise to discontinue enamel and/or dentin bonding

until reports from controlled studies are available.

In the present study, Indiloy amalgam restorations showed significantly more surface tarnishing, and New True Dentalloy restorations significantly more surface roughness and marginal fracture, $P < 0.001$, although their restoration survivals were again very similar, $P > 0.55$ (Smales, 1991b). All of the four assessed clinical factors deteriorated over the study in a manner similar to that described for marginal fracture of the two materials in earlier studies (Jordan, Suzuki & Mills, 1978; Letzel & Vrijhoef, 1984; Smales & Gerke, 1984; Smales & Rupinskas, 1985).

Surface tarnishing has been shown to be an unsatisfactory clinical characteristic of several high-copper amalgam alloys (Smales & Gerke, 1984, 1986; Smales & Rupinskas, 1989). However, as found in the present study, the tarnishing may not have a significant effect on the survival of the restorations, as relatively few were usually severely affected and could be easily repolished. Although surface tarnishing or discoloration is expected to be the forerunner of surface roughness caused by corrosion (Letzel & others, 1978), the present study did not support this supposition.

The relationship of marginal fracture to amalgam restoration survival remains controversial. While several studies have found an association between early high marginal fracture rates and later high restoration failures (Letzel & others, 1989; Osborne & others, 1989a; Osborne & Norman, 1990), other studies have not (Hamilton & others, 1983; Moffa, 1989; Osborne & others, 1989b). In the present study, New True Dentalloy showed significantly more marginal fracture, but arguably, significantly less marginal staining than did Indiloy, $P = 0.03$. The marginal fracture results may have been related more to the surface roughness of the two alloys, and the surface tarnishing results to the marginal staining of the two alloys, but these associations were not tested.

The findings of the present study would suggest that inappropriate clinical factors or characteristics for material deterioration may have been selected in this and many other studies to indicate future replacements or repairs for the two resin restorative treatments and two amalgams, because examination of the actual reasons for replacements showed that many failed restorations had been fractured or lost, or had marginal caries (Smales, 1991a,b). Such reasons for replacement cannot be confidently related to the clinical deterioration of the composite resin material, and only bulk fracture may be relevant to the deterioration of the amalgam materials (Lemmens & others, 1987). As has been noted previously (Dahl & Eriksen, 1978), the improved clinical appearance obtained by physical improvements to amalgam alloys may be of marginal importance for the durability of the restorations, and further long-term studies are required that correlate quality factors and the reasons for restoration replacement.

## Conclusions

From the findings of two large long-term longitudinal clinical studies of the deterioration of an auto-cured conventional composite resin placed using unetched- and etched-enamel-bonding treatment methods, and of a low- and a high-copper amalgam alloy, it was found that:

1. There were significant differences found in the deterioration of the restorations for several of the clinical factors assessed for both the two different treatment methods used with the composite resin and for the two different amalgam alloys;
2. Despite these differences in restoration deterioration, the survivals for the two different composite resin treatment methods were very similar, as were the survivals for the two different amalgam alloys;
3. Apart from the more severe surface tarnishing found for one amalgam alloy, there were relatively few Unsatisfactory ratings given for any of the clinical factors assessed; and
4. All the clinical factors deteriorated with aging of the resin and amalgam restorations.

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# Physical Properties of Proprietary Light-cured Lining Materials

L E TAM • D McCOMB • F PULVER


## Summary

Physical parameters of four photopolymerized lining materials were evaluated for comparative purposes and to contribute to the understanding of these novel materials. Properties investigated included compressive, diametral and flexural strength, modulus of elasticity, fluoride and calcium release for four weeks, and pH values for 24 hours.

In general Cavalite and TimeLine were significantly stronger than Vitrabond and XR-Ionomer and were essentially neutral in pH. All the materials exhibited a yield upon compressive strength testing. The fluoride release was intermediate for TimeLine and negligible with Cavalite. Vitrabond and XR-Ionomer released significant amounts of fluoride at all time periods. XR-Ionomer was the only material investigated to have significant calcium release, and this may have been related to its visible dissolution in water. This material also showed a low initial pH of 2.2. It, along with Vitrabond, demonstrated a gradual increase in pH over time. The behavior of TimeLine and Cavalite is therefore more consistent with that of a modified composite resin, whereas Vitrabond and XR-Ionomer are similar in nature to glass-ionomer liners.



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## INTRODUCTION

Restorations often require an intermediary base of lining material to provide patient comfort and protection of the pulp. It is necessary that such materials provide appropriate physical and biological properties for their clinical usage. Conventional liners and bases have certain known advantages and limitations and their use is tailored to the clinical situation. Recently new proprietary lining and base materials have been introduced in an effort to

improve upon materials currently available. Some are photopolymerized for control over the speed of set. Commercial claims of these photopolymerized materials indicate enhanced mechanical properties, bond strength, and therapeutic properties. However, independent studies are required to evaluate their relative properties and clinical performance. In this study, physical parameters of clinical relevance were evaluated in vitro for four new photopolymerized lining materials to contribute to the understanding of these novel materials. The properties tested were strength, including modulus of elasticity, fluoride and calcium release, and pH profile.

Strength and rigidity are necessary where occlusal stresses are placed on a restoration. Farah, Hood and Craig (1975) have shown that the fracture resistance of amalgam restorations decreases with lower modulus of elasticity of the base material. The strength parameters of proprietary forms of single classes of lining/base materials have been shown to vary widely. Twenty-four-hour compressive strengths for conventional glass-ionomer base and lining materials have been reported to vary from 46 to 127 MPa, and for calcium hydroxides, from 6 to 38 MPa (Tam & others, 1989). Knowledge of the strength factors for a particular material ensures appropriate clinical use.

Fluoride release has become a possible therapeutic property in recent years with the development of glass-ionomer liners and bases. Conventional glass-ionomer cements have been shown to release significant amounts of fluoride over sustained periods (Fukazawa, Matsuya & Yamane, 1987; Hattab, 1987; Muzynski & others, 1988). This has been attributed to glass-ionomer dissolution, the diffusion of $H^+$ and $F^-$ for exchange in the cement matrix, and the slow formation of complexes as the glass ionomer matures (Fukazawa & others, 1987). Resins containing fluoride, however, rely only on outward diffusion of fluoride (Arends & Ruben, 1988), and thus are limited with respect to amount and rate of fluoride release (Swift, 1989). Fluoride contributes to anticariogenicity by increasing the potential for enamel re-mineralization and by its antimicrobial effect (Hicks, Flaitz & Silverstone, 1986; Marquis, 1989; Reintsema, Lodding & Arends, 1986; Temin, Csuros & Mellberg, 1989). Tveit and others (1987) have, however, questioned the value of fluoride release at the tooth/liner interface in the prevention of secondary decay. Nevertheless, it has been suggested that the release of fluoride from a base/liner may prevent the formation of wall lesions and prevent caries at the margins of restorations (McCourt, Cooley & Huddleston, 1990). The use of a fluoride-releasing liner was shown to reduce the acid solubility of dentin (Söremark, Hedin & Röjmyr, 1969).

The compositional nature of the photopolymerized liners evaluated in this study varies between that of a resin and a glass ionomer. Two products, Cavalite and TimeLine, are essentially composite resin with novel inclusions. The manufacturers state that the formulation of TimeLine is based on a urethane dimethacrylate resin with glass and sodium fluoride inorganic phases. Cavalite contains both hydroxyapatite and glass-ionomer fluoroaluminum-phosphosilicate powder in polymerizable monomers. The other two products, Vitrabond and XR-Ionomer, are thought to be essentially glass ionomers, the liquid component in each case being an innovative polyacrylic acid with the addition of differing amounts of photopolymerizable pendant monomers. The addition of organic polymers to glass ionomers has been shown to increase toughness, decrease brittleness, increase shrinkage and decrease solubility in a glass ionomer (Mathis & Ferracane, 1989). This latter effect, however, may result in decreased fluoride release.

It has been suggested that calcium may activate adenosine triphosphatase activity, thus accelerating mineralization of hard tissue (Abiko, 1977), and combine with proteins to form calcium precipitates, which will act as a crystalline barrier and a matrix for odontoblastic alignment (Gordon, Ranly & Boyan, 1985). Thus calcium release would be an advantageous property in lining materials, particularly in a deep carious lesion. It is important, however, that neither fluoride nor calcium release be associated with rapid dissolution in the oral environment.

The pH profile of a material, from initial application to completion of the setting process, is an important physical property which relates to pulpal response and other biological properties. Acidity of an intermediary base material may contribute solely or synergistically with

other factors to cause an adverse pulpal response (Brännström, 1984; Gordon & others, 1985). An alkaline pH, such as that which occurs with calcium hydroxide cements (Tam & others, 1989; Abbasi & Barkhordar, 1987) appears to create an optimum environment for remineralization and against organism survival (Fisher & McCabe, 1978; Gordon & others, 1985). Knowledge of the pH profile for new materials provides valuable information for the clinician.

## MATERIALS AND METHODS

The lining materials studied were: Cavalite (Sybron/Kerr, Romulus, MI 48174), XR-Ionomer (Sybron/Kerr), TimeLine (L D Caulk Co, a Division of Dentsply International, Milford, DE 19963), and Vitrabond (3M Dental Products, St Paul, MN 55144). The two-component materials were dispensed by weight and mixed as per instructions prior to polymerization. One-paste systems were dispensed directly from the container. Exposure to visible light (Prismetics Lite, Model EU001, L D Caulk Co) consisted of at least 20 seconds for every 0.5 mm increment to ensure complete set.

### Strength Tests

Cylindrical specimens approximately 3 mm in diameter and 6 mm in height were prepared in split stainless steel molds for the compressive and diametral tensile strength testing. Polyethylene molds were used for fabrication of rectangular specimens (2 mm x 2 mm x 20 mm) for the flexural strength and modulus of elasticity tests. All specimens were maintained in distilled water at 37 °C until just prior to being tested on the Instron Universal Testing Machine (Model TTCM, Instron Corp, Canton, MA 02021) with the crosshead set at 0.05 cm/min. The force measured at ultimate fracture of the sample was recorded to calculate diametral tensile strength and flexural strength. However, during compressive loading, deformation of specimens occurred prior to ultimate fracture. Thus, compressive strength determinations reported here were based upon the force measured at the first point of this sample deformation, i e, yield compressive strength. The strength studies were conducted 24 hours after the start of sample preparation. At least six specimens were fabricated for each material for each strength test.

### pH Test

Each material was spread uniformly on a clean glass slide to a thickness of approximately 1 mm and photopolymerized for 40 seconds. Measurements were begun immediately at the end of mixing or dispensing (0 seconds) and subsequently were obtained at 1 minute, 5 minutes, 15 minutes, 30 minutes, 1 hour, 2 hours, 4 hours, 6 hours, and 24 hours. A combination electrode (flat surface polymer body combination electrode, Fisher, catalog #13-639-83, Pittsburgh, PA 15238) was used with an Accumet 620 pH/mV meter (Fisher, catalog #13-627-620) for pH measurement. The electrode was placed in intimate contact with the test material, with an intermediary film of distilled water for set material. Samples were stored in a humidity chamber (37 °C, > 80% RH) between testing intervals. The electrode was cleaned and stored in a phosphate buffer solution (pH 4) between measurements. A minimum of three trials was used for determination of the average pH change.

### Fluoride Release Test

The method for fluoride release measurements followed LaRochelle (1989). Disc-shaped specimens 10 mm in diameter and 2 mm thick were fabricated in silicone molds (Express Type I, 3M Dental Products). Four samples were prepared for each material and were weighed in order to verify standardization within each material test group. Immediately after polymerization, the discs were immersed into individual sealable glass bottles containing 15 mLs of deionized water. These containers were then left undisturbed in an incubator set at 37 °C. Four control samples consisting of bottles containing 15 mLs of deionized water without a disc were also prepared for testing. After 24 hours, the bottles were removed from incubation and the liquid contents gently stirred. Three mLs of solution were extracted from each bottle using a micropipette and placed into individual vials for further testing. The specimens were then returned to the incubator. Similar extractions were performed for each sample for each

material at 7 days, 14 days, and 28 days.

One ml of total ionic strength adjustment buffer was then added to one ml of the previous extracted solution and magnetically stirred. Fluoride release was determined by means of a specific fluoride ion electrode (Model 94, Fisher, catalog #940900) immersed into the solution and coupled to a Digital IonAnalyzer (Model 701A, Orion Research, Inc, Cambridge, MA 02129). The electrode had been previously calibrated with a series of standard solutions of sodium fluoride (Orion Research, Inc) whose molarity spanned the actual concentrations of fluoride to be measured.

### Calcium Release Test

0.5 ml of the previous 3 mL extracted was combined with 4.5 mL of 0.1% lanthanum oxide using a micropipette to avoid phosphate interference. The concentration of calcium ions in the solution was then determined by an atomic absorption spectrophotometer (2380, Perkin-Elmer Corp, Norwalk, CT 06859).

## RESULTS

The means and standard deviations for the strength testing are shown in Figures 1 and 2. Results for all strength tests were analyzed using the one-way analysis of variance and the Fisher's Least Significant Difference Test ($P < 0.05$). In general, Vitrabond and XR-Ionomer were significantly weaker than TimeLine and Cavalite. All the light-cured liners exhibited yield behavior during compressive loading. The moduli of elasticity for these materials ranged from 1.1 to 2.6 GPa, with XR-Ionomer and Cavalite significantly the more rigid materials.

Figures 3 and 4 illustrate respectively the calcium and fluoride release at the times

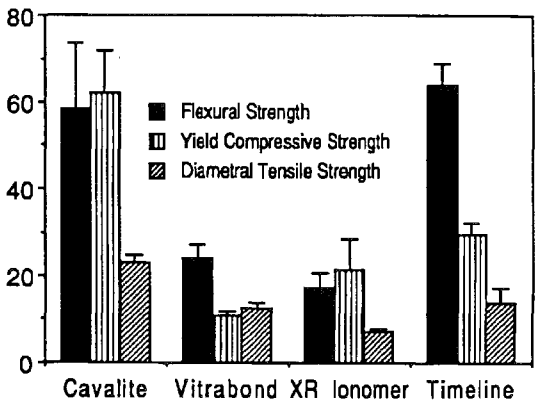


FIG 1. *Strength values (MPa)*

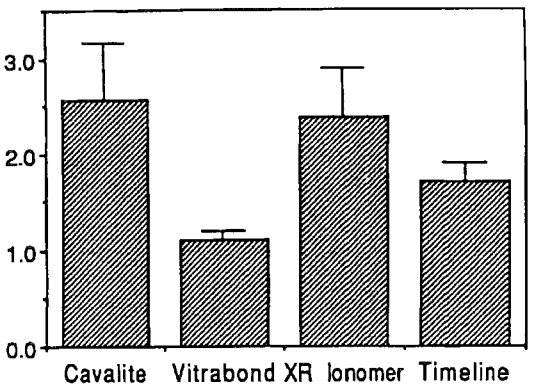


FIG 2. *Modulus of elasticity (GPa)*

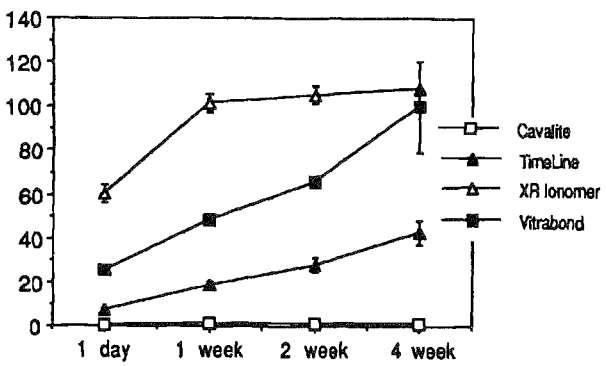


FIG 3. *Fluoride release (ppm)*

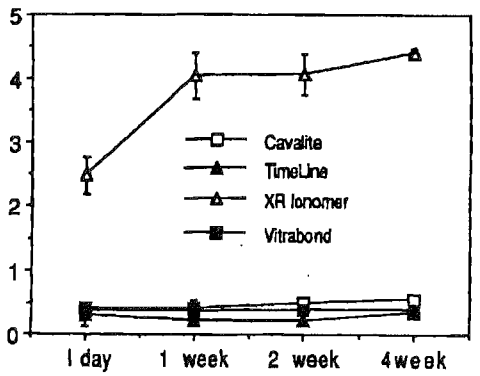


FIG 4. *Calcium release (μg/ml)*

tested. The two-way analysis of variance with one repeated variable test and the Fisher's Least Significant Difference Test were used for statistical analysis of these results ($P < 0.05$). At all time periods, XR-Ionomer had significantly greater levels of calcium release. TimeLine and Vitrabond had essentially no calcium release over time compared to the control samples. Cumulative fluoride release measurements from the light-cured liners was such that XR-Ionomer > Vitrabond >> TimeLine >> Cavalite. XR-Ionomer seemed to plateau in its fluoride release in the fourth week of testing at 108 ppm, whereas it appeared to continue rising for Vitrabond at 100 ppm.

The pH profiles are depicted in Figure 5. The pH range for XR-Ionomer over 24 hours was 2.2 to 5.5, the lowest of all the materials tested. It, along with Vitrabond, demonstrated a gradual increase in pH over time. Cavalite and TimeLine, however, showed a drop in pH immediately after curing, followed by a gentle rise.

## DISCUSSION

The light-cured base and lining materials demonstrated a yield upon compressive strength testing. This yield may have been related to incremental insertion or incomplete polymerization of the material during specimen fabrication. However, when the effect of prolonged curing and bulk insertion during sample preparation was subsequently tested, the yield was not eliminated. The yield compressive strength values were lower than those that would be based upon the ultimate fracture of the specimen, but were thought to be a more appropriate measure of response to loading.

Conventional lining and base cements do not undergo a compressive yield. It has, however, been reported to occur with a light-cured calcium hydroxide cement (Tam & others, 1989). The effect of this yield has yet to be assessed clinically. The standard strength test method employed can only provide relative results between the materials sampled and does not accurately mimic the clinical situation. Lloyd, Jeffrey and Mannion (1982) suggested that additional support may be given to the lining material by the rigid cavity walls as well as the overlying base in a clinical setting. In addition, the effect of this support would be increased as Poisson's ratio of the material is decreased. This may imply a greater relative increase in strength of the more fluid liners over rigid lining materials in a clinical environment. Farah and others (1975), however, reported that the modulus of elasticity should be as high as possible for a base material under a restoration supporting direct occlusal forces. Until the effect of the visco-elastic deformation of these materials can be established in a clinical setting, the selection of liners and bases with high values for all strength tests would be prudent under amalgam restorations and/or if used in greater thicknesses

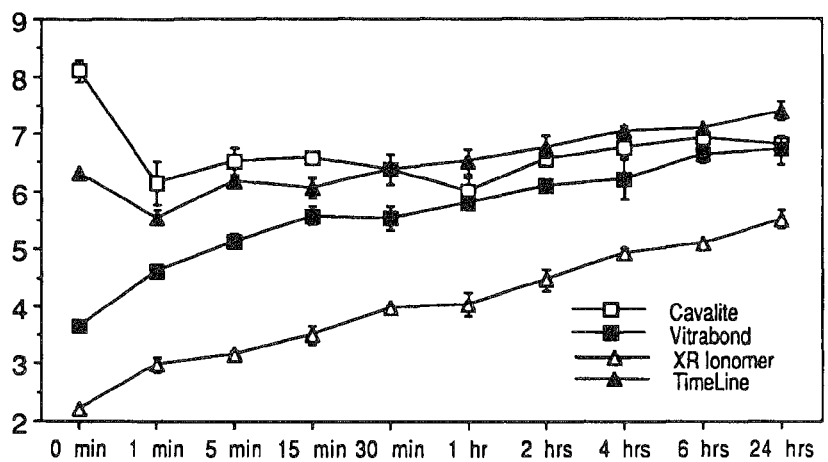


FIG 5. *pH of light-cured liners*

as a base. This would be of less significance in nonocclusally stressed restorations.

The fluoride release measurements revealed similar traits to those reported by McCourt and others (1990). The data presented here essentially divides the materials tested into two groups, one behaving as glass ionomers, the other as composite resin. Cavalite and TimeLine are essentially composite resins and thus have less fluoride release than glass ionomers. There was no significant difference for fluoride release measurements between Cavalite and control groups. This has also been cited in studies by DeSchepper, White and von der Lehr (1989) and LaRochelle (1989). The ability of TimeLine to release fluoride is therefore of interest. Fluoride release measurements for XR-Ionomer and Vitrabond were comparable to that of a conventional glass ionomer (Swift, 1989; LaRochelle, 1989).

The minimum amount of fluoride needed for anticariogenicity cannot be predicted at this time. Artificial recurrent caries under amalgam was reduced with the use of a conventional glass ionomer (García-Godoy & Jensen, 1990), Vitrabond and XR-Ionomer, but not with TimeLine (Jensen, García-Godoy & Wefel, 1990). DeSchepper and others (1989) proposed the minimum value of fluoride concentration necessary to inhibit *Streptococcus mutans* to be in the range of 20-300 ppm. TimeLine, XR-Ionomer, and Vitrabond all attained this value at one month. Studies support continuous release of small amounts of fluoride release for long-term anticariogenic effect (Swift, 1989). Conventional glass ionomers release the greatest amount of fluoride in the first few days (Tsanidis, Koulourides & Retief, 1990). McCourt and others (1990) indicated that all the lining materials tested exhibited sustained fluoride release over a 17-week period, except Cavalite.

XR-Ionomer was the only material tested with significant calcium release at all time periods. It was noted, however, that releasing particulate matter into solution during testing may have played a role in its high calcium release measurement. None of the materials studied was recommended for use as a pulp capping medicament, and certainly the initial pH of the XR-Ionomer would contraindicate such usage.

Vitrabond and XR-Ionomer had a pH spectrum ranging from 2.2 to 7.4 over 24 hours. These results are comparable to or slightly higher than pH values previously reported for conventional glass-ionomer lining and base materials (Tam & others, 1989; Smith & Ruse, 1986). The prolonged phasic setting reaction of typical glass ionomers are similarly reflected in the initial rapid pH rise followed by a slower increase over the course of 24 hours, despite the fact that the materials reported here are photopolymerized. The effect of acidity may vary, depending upon its duration, the quantity of available acid, the presence or absence of bacterial contamination, the degree of dentin permeability, and the area in contact with dentin. Maintenance of a pH of 2 for five minutes or more has been implicated as a critical situation for a setting cement for a damaging pulp response (Smith & Ruse, 1986). Immediate pH readings of Vitrabond and XR-Ionomer were 3.6 and 2.2 respectively. Acid release may be even greater if a thinner consistency of material were to be mixed (Woolford, 1989). In deep preparations, an intermediary layer of calcium hydroxide should be placed prior to the application of these materials. pH values for set TimeLine and Cavalite were between 5.5 and 7.4 over 24 hours. The uncured materials (0 minutes) had a slightly higher initial pH. These materials were essentially of neutral pH profile.

The overlying restorative material and its clinical environment will ultimately dictate the material choice for a lining and/or base. Light-cured lining materials provide certain practical advantages, but other factors should be considered: 1) short clinical history thus far, 2) unknown long-term pulpal effects, 3) limited light-curing potential with greater thicknesses, 4) polymerization shrinkage thus possible contributing to microleakage (Chan & Swift, 1989), 5) transient temperature increases with photopolymerization (Adamson, Lloyd & Watts, 1988), and 6) questionable antimicrobial effects (Scherer & others, 1990).

Increased knowledge of the physical attributes of these materials will provide more appropriate material selection for clinical usage.

## CONCLUSIONS

1. The light-cured lining materials tested all exhibited yield during compressive strength

testing. The effect of this yield has yet to be assessed in a clinical setting.

2. In general, Cavalite and TimeLine appeared to be stronger; Vitrabond and XR-Ionomer were significantly weaker. Vitrabond had the lowest modulus of elasticity.

3. All the materials except Cavalite demonstrated a significant fluoride release with time.

4. Only XR-Ionomer had significant calcium release at all time periods. This may have been related to its noted dissolution in water.

5. Cavalite and TimeLine had a neutral pH profile over 24 hours. In comparison, Vitrabond and XR-Ionomer had a lower pH range, which was similar to that of conventional glass ionomers.

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# Effect of Dentin Age on Effectiveness of Dentin Bonding Agents

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## Summary

**The purpose of this research effort was to investigate the effect of age changes in dentin on the effectiveness of two dentin adhesives in minimizing microleakage at the tooth-restoration interface. Cavities were prepared in permanent teeth extracted from patients below 20 years or over 55 years of age. Wedge-shaped cervical preparations were made with the gingival cavosurface margin on dentin. Treatment groups were randomly restored with one of two composite restorative materials, together with the appropriate dentin adhesives. The control group specimens were restored with the respective composite restorative materials without the adhesives. All specimens were thermocycled, then placed in 0.5% basic fuchsin dye solution for 24 hours and subsequently sectioned longitudinally. Microleakage at the tooth-restoration interface was assessed by dye penetration. The results showed that the use of adhesives significantly reduced microleakage along the tooth-restoration interface. The adhesive formulated with glycidyl methacrylate was significantly more effective in reducing microleakage in dentin of the over-55 age group.**



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## Introduction

The acid-etch technique introduced by Buonocore (1955) is effective in providing retention and marginal seal to cavity margins in enamel but not in dentin. In an attempt to improve adhesion of composite restorative materials to dentin, research in chemical bonding has led to the development of dentin adhesives. Existing dentin adhesive systems can only reduce the extent of microleakage but not eliminate it completely (Council on Dental Materials, Instruments, and Equipment, 1987).

In vitro and in vivo studies have examined the effectiveness of various types of dentin

adhesives. Previous in vitro studies on the effectiveness of dentin adhesives used shear (Causton, 1984) and tensile (Asmussen & Bowen, 1986) bond strength as well as marginal contraction gaps (Hansen, 1986) as the means of evaluation. Measurement of microleakage at the tooth-restoration interface using dye penetration is also a recommended method for assessing dentin adhesives (Fuks, Hirschfeld & Grajower, 1985). Most of the studies found that pretreatment of dentin cavities with dentin adhesives generally improved adhesion of composite restorative materials to dentin. However, the use of dentin adhesives did not produce a complete seal of the cavity, and the performance of adhesives was also found to be highly variable. Generally, none of the systems in use are consistent in the results they produce. One possible reason for the highly variable results is the difference in the physical and chemical structure of individual teeth. Aging changes the physical and chemical structure of dentin (Shells, 1988).

A previous study showed that cavity site had no influence on the effectiveness of dentin adhesives (Soh, Henderson & Chantler, 1988). Other investigators found dentin adhesives to be less effective in dentin close to the dental pulp (Causton, 1984; Finger, 1988). While much effort has gone into investigating factors affecting adhesives, possible influences of age changes in dentin on the adhesion of restorative materials have not been evaluated. This study was conducted to test the effectiveness of two dentin adhesives in minimizing microleakage between the tooth-restoration interfaces of cavities prepared on permanent teeth of patients in the under-20 and over-55 age groups. The purpose of this research effort was to investigate the effect of age changes in dentin on the effectiveness of two dentin adhesives in minimizing microleakage at the tooth-restoration interface of cervical preparations.

## Materials and Methods

Eighty human permanent premolars free of carious lesions or defects extracted from patients aged below 20 years or above 55 years were cleaned with pumice and stored in distilled water at room temperature. Wedge-shaped cavities with a mesiodistal width of 3 mm, an occlusogingival length of 2 mm, and a depth of 2 mm, were prepared at the cementoenamel junctions on the facial surfaces. The class 5 cavities were prepared with tungsten carbide burs (Shofu Inc, Kyoto, Japan) under an air and water spray. The occlusal margins were invariably in enamel, while the cervical margins extended into cementum and dentin at an obtuse cavosurface angle.

The prepared teeth were first separated into the two main age groups of the patients from whom the teeth were extracted. Within each age category, the teeth were randomly assigned to four equal groups. The first group of cavities from each age category was restored with Prisma Universal Bond 2 dentin adhesive and APH composite restorative material (L D Caulk Co, Dentsply International, Milford, DE 19963), while the second group was restored with Tenure adhesive and Marathon V composite restorative material (Den-Mat Corp, Santa Maria, CA 93456). The third and fourth groups of cavities in both age categories were each restored with one of the two composite restorative materials without the use of dentin adhesives. All materials were handled according to the manufacturers' instructions.

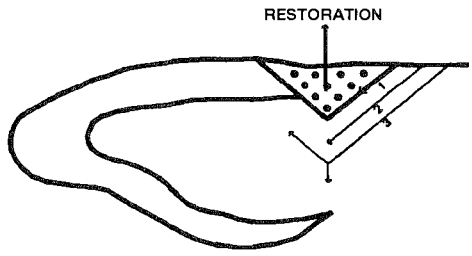
The enamel margin of each cavity was etched for 15 seconds with a 37% unbuffered phosphoric acid and washed with a water spray for 30 seconds before drying. The composite restorative materials were inserted in 1-mm increments and polymerized using a visible-light curing unit (Elipar Visio-Light System, ESPE Dental, Seefeld/Oberbay, Germany). The final layer of material was held under firm pressure with a Mylar celluloid strip while polymerizing. The excess resin was removed and the teeth were then stored in distilled water at 37 °C for 24 hours before the restorations were finished to the cavosurface margins, using a series of coarse- to fine-grit discs (Shofu Super-Snap Rainbow kit, Shofu Inc, Kyoto, Japan).

The restored teeth were thermocycled in baths of temperatures between 5 °C and 55 °C for 250 cycles with a dwell-time of 15 seconds in each bath. After thermocycling, the root apices of the restored teeth were sealed with utility wax and each tooth surface was covered with two coats of nail varnish

except for the restorations and an area 2 mm beyond, thus exposing them to the dye. The teeth were then incubated in 0.5% basic fuchsin solution maintained at 37 °C for 24 hours. In preparation for evaluation of microleakage, the nail varnish was removed and the teeth were sectioned longitudinally through the restorations. The degree of dye penetration around the gingival margins was evaluated under an Olympus zoom stereomicroscope, model SZ30 (Olympus Optical Co, Ltd, Tokyo, Japan), at X40 magnification by an independent examiner. The scoring criteria used to assess the extent of dye penetration at the tooth-restoration interface (see figure) were as follows:

0 = No dye penetration,
1 = Dye penetration to one-half the maximum depth of the cavity,
2 = Dye penetration to the full depth of the cavity, and
3 = Dye penetration beyond the full depth of the cavity.

The frequencies of the scores were tabulated for each of the treatment and control groups in each age category. A two-sample test of the median scores was carried out to test between-group differences using chi-square analysis for Fisher's Exact test when conditions for the former were not fulfilled. Comparisons were made between median scores for each treatment and its control group, and between the two treatment groups for each of the two age categories.




*Diagrammatic representation of scale used to measure the degree of dye penetration at the tooth-restoration interface*

## Results

There was no evidence of dye penetration at the occlusal margin of any of the specimens and materials tested. Hence, the scoring was limited to the gingival cavosurface margin, which is the main area of concern with dentin adhesives. The frequencies of the scores and their median values by type of treatment and age category are presented in the table. Generally, teeth restored with either of the two dentin adhesives presented with significantly less dye penetration at the tooth-restoration interface when compared to its control group for each of the two age categories ($P < 0.05$). Teeth of the under-20 age group that were treated with the Prisma Universal Bond 2/APH

*Microleakage of Dentin of Two Age Groups Restored with Two Dentin Adhesive Systems*

| | < 20 years Frequency of scores | | | | | | | | | > 55 years Frequency of scores | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | | 1 | | 2 | | 3 | | | 0 | | 1 | | 2 | | 3 | | | P** |
| Treatment & Control Groups | N | % | N | % | N | % | N | % | Median | N | % | N | % | N | % | N | % | Median | Value |
| Prisma Universal Bond 2/APH | 9 | 90 | 1 | 10 | 0 | 0 | 0 | 0 | 0 | 8 | 80 | 2 | 20 | 0 | 0 | 0 | 0 | 0 | NS |
| APH (Control)* | 1 | 10 | 2 | 20 | 0 | 0 | 7 | 70 | 3 | 2 | 20 | 0 | 0 | 0 | 0 | 8 | 80 | 3 | NS |
| Tenure/Marathon V | 3 | 30 | 6 | 60 | 1 | 10 | 0 | 0 | 1 | 8 | 80 | 2 | 20 | 0 | 0 | 0 | 0 | 0 | 0.03 |
| Marathon V (Control)* | 2 | 20 | 3 | 30 | 2 | 20 | 3 | 30 | 1.5 | 5 | 50 | 3 | 30 | 0 | 0 | 2 | 20 | 0.5 | 0.03 |

*Two-sample median test of median scores between each treatment and its control group for each age category was statistically significant at $P < 0.05$.

**Significance values of two-sample median tests of median scores between the two age groups for each of the treatment or control groups

system registered marginally less microleakage than those of the over-55 age group, but the difference in the extent of leakage recorded in the two age groups was not statistically significant. The reverse was observed on teeth restored with the Tenure/Marathon system, which seemed to be more effective in preventing leakage in teeth belonging to patients aged over 55 years. Teeth belonging to the over-55 age group when restored with the Tenure/Marathon system recorded significantly less microleakage than similarly restored teeth in the under-20 age group ($P < 0.03$).

## Discussion

A multiplicity of factors can influence the effectiveness of dentin adhesives other than those attributed to experimental variables. Two dentin formulations commonly used in dentin adhesives are either halophosphorus esters of bisphenyl A glycidyl methacrylate (BIS-GMA), which bonds chemically with the calcium in dentin, or aqueous solutions of glutaraldehyde and hydroxyl-ethyl-methacrylate (HEMA), which bonds with the collagen in dentin (Robinson & Moore, 1988). The organic and inorganic composition of dentin varies according to locations on the tooth (Scott & Symons, 1974). Similarly, fiber dimension and orientation differ across various regions of the tooth. Age changes in the form of physiological calcification will increase the inorganic composition of dentin and decrease the collagen content in the aging process. Such changes in the structure of dentin may influence the efficacy of dentin adhesives. Therefore, the extent of age changes on the efficacy of adhesives may be determined by the basic formulations of the adhesives.

In this study, the Tenure/Marathon system was found to be more effective in minimizing leakage in restored teeth of the older age group. The basic formulation of the Tenure adhesive contains N (p-toly) glycine glycidyl methacrylate (NTG-GMA), which bonds chemically with calcium. The higher content of mineralized tissue in aged dentin explains the improved adhesion in restored teeth belonging to the older age group. Structural changes manifest as a sizeable reduction (or obliteration) of dentinal tubules with increasing age. In theory such changes could alter not only the chemical structure but reduce the surface area of tissues that would bond effectively with restorative materials. It is postulated that adhesives relying on mineral bonding should be effective on older teeth, due to the increased availability of calcium. In contrast, those adhesives that bond to the organic constituents would be less effective because of the decrease in the availability of collagen arising from mineralization and tubule obliteration. Mixed-system adhesives should be similarly affected by the process of dentin aging, albeit to a lesser extent.

Only a marginal difference between the two age groups was evident in teeth restored with the Prisma Universal Bond 2/APH system. The Prisma Universal Bond 2 adhesive consists of a dentin primer that has a 1% weight/volume of glutaraldlehye with 30% weight/volume of HEMA that will bond with the collagen in dentin. It also contains a halophosphorous ester of bisphenyl A-glycidyl methacrylate that will bond to the inorganic component of dentin.

The clinical significance of this research is useful. The results obtained provide some guide as to the adhesive system to be used on teeth of different ages. This can improve the quality of the restorative service provided. It may be argued that results of in vitro studies may not be directly extrapolated to the clinical situation. However, in the absence of well-controlled clinical trials, results of laboratory studies are useful in providing some guidelines regarding the performance of restorative materials. It is hoped that this study will provide the impetus for further research, in particular, clinical studies of dentin adhesion.

## Conclusion

The use of dentin adhesives significantly reduced the extent of microleakage along the tooth-restoration interface of teeth restored with composite restorative materials. The adhesive with a formulation that contained glycidyl methacrylate was found to be significantly more effective in providing adhesion for teeth belonging to the over-55 age group. Formulation with a combination of glycidyl

methacrylate and glutaraldehyde with HEMA provided a more consistent sealing capability in both young and old teeth.

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# Comparison of Shear Bond Strengths of Some Third-Generation Dentin Bonding Agents

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## Summary

**Recently a series of new dentin bonding systems has been introduced to the dental profession. These agents are the third generation of systems that have been developed over the past two decades. Some dentin bonding agents are easily applied, clinically, while others are complicated, multi-step procedures. A comparison of the shear bond strengths of five of these systems was made at 15 minutes and after 24 hours stored in water at 37 °C.**

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## Introduction

Since Buonocore (1955) reported increased adhesion of acrylic filling materials to enamel following a phosphoric acid treatment, a significant effort has been made to establish a chemical bond between composite resin restorative materials and dentin.

Nakabayashi (1985a), Nolden (1985), and Bowen (1985) reviewed the status of dentin bonding agents in Japan, Germany, and the

United States respectively and reported that significant improvements had been made during the past 30 years. Bowen, Cobb and Rapson (1982), Bowen (1985), Blosser and Bowen (1988), Asmussen, Antonucci and Bowen (1988) and Retief and others (1988) all reported different and improved systems for attaching to dentin. Retief and others (1988) evaluated a dentin bonding agent and described a system for evaluating the shear bond strength. These authors also issued an appeal for standardization of test procedures for bond strengths.

The first-generation dentin bonding agents (Retief & Denys, 1989) included cyanoacrylates, polyurethanes, glycerophosphoric acid dimethacrylate, and the adduct of N-phenyl glycine and glycidyl methacrylate (NPG-GMA). Buonocore and others (1956) developed a glycerophosphoric acid dimethacrylate to bond to the calcium ions of dentin through the active phosphate group. Bowen (1965) developed the adduct of N-phenyl glycine and glycidyl methacrylate (NPG-GMA), and the system was marketed in Europe. Lee and others (1971) developed a polyurethane resin to be used as an adhesive for composite restorations.

A second generation of dentin bonding agents (Retief & Denys, 1989) included Scotchbond (3M Dental Products, St Paul, MN 55144), a halophosphorus ester of BIS-GMA. The smear layer was not removed in this technique. Gross, Retief and Bradley (1985) reported microleakage at the cervical margin of all restorations. Dentin Bonding Agent (Johnson & Johnson Dental Products, East Windsor, NJ 08520) is a halophosphorus ester of hydroxyethyl methacrylate. Creation Bonding Agent (Den-Mat Corp, Santa Maria, CA 93456) utilizes a phosphorus ester of BIS-GMA. Dentin Adhesit (Vivadent, Schaan, Liechtenstein) contains a prepolymer of toluene-diisocynate and trimethylol propane (urethane) in methylene chloride. Retief and others (1986) tested the tensile bond strengths of several dentin bonding agents and reported low tensile bond strengths and a number of spontaneous failures.

There are several dentin bonding systems that are now available to the dental profession that represent a third generation. Some of these are Mirage Bond (Chameleon Dental Products, Kansas City, MO 66101), XR-Bond (Kerr/Sybron, Romulus, MI 48174), ALL-BOND (BISCO, Inc, Downers Grove, IL 60515), ALL-BOND Kanca Technique or All-Etch (BISCO), and Prisma Universal Bond 2 (L D Caulk Co, Dentsply International, Milford, DE 19963).

Mirage Bond uses a nitric-NPG and PMDM combination. Conn, Duke and Barghi (1989) reported a shear bond strength of 5.8 ± 1.3 MPa.

XR-Bond is a system that consists of a primer that is an ethanol solution of a phosphonated dimethacrylate resin and a photo-initiator. The XR adhesive is a phosphonated BIS-GMA. Barkmeier and Cooley (1989) reported a mean shear bond strength of 15.6 ± 3.7 MPa.

Prisma Universal Bond 2 is comprised of a primer and an adhesive. The primer system is an ethanol solution of hydroxyethyl methacrylate (HEMA) and phosphonated penta-acrylic acid (PENTA). The adhesive is a phosphate ester of BIS-GMA, glutaraldehyde, and PENTA.

ALL-BOND was recently introduced to the profession. When the restoration will involve enamel and dentin, the ALL-BOND technique recommends etching the enamel with 32% phosphoric acid and treating the dentin with a conditioner.

ALL-BOND also has an alternate technique known as the "All-Etch or Kanca Technique." In this procedure the enamel and dentin are etched for 15 seconds with 10% phosphoric acid (All-Etch).

The purpose of this in vitro study was to compare the shear bond strengths of five third-generation dentin bonding agents, one of which utilizes phosphoric acid-etching of the dentin. This investigation utilized the test system advocated by Retief and others (1988). It also provides data that is directly comparable to other studies, thereby contributing to the standardization of shear bond strength testing.

## Materials and Methods

The bonding systems utilized in this project are listed in Table 1. These bonding systems were used in conjunction with Herculite XR (Kerr), a composite resin restorative material.

One hundred and fifty extracted, caries-free, permanent human molars were used in this study. Teeth were obtained from the Department of Oral and Maxillofacial Surgery, West Virginia University School of Dentistry. Immediately following extraction the teeth were cleaned with pumice and stored at room temperature in 70% ethanol. The roots of the teeth were removed with a carbide bur in an air tubine handpiece with copious water coolant. Each tooth was embedded into clear PVC tooth sleeves (Utilities Supply, Medford, MA 02174), using cold-cure acrylic resin (Formatray, Kerr). The teeth were randomly assigned to 10 groups of 15 each. Subsequently, two groups of 15 each were assigned to each material. One group of 15 was designated for testing at 15 minutes and the other at 24 hours. The teeth were mounted in such a manner that the occlusal surface projected above the PVC tooth sleeve. Just prior to testing, the enamel was removed by wet grinding with 240-grit silicon carbide paper, using an alignment block to obtain a flat surface. The dentin was exposed by wet grinding with 600-grit silicon carbide paper (ECOMET II Polisher/Grinder, Buehler Ltd, Evanston, IL 60204). Each group of 15 was treated as recommended by the manufacturer. One group for each material was bonded and allowed to remain in the bonding stand for 15 minutes before stressing to failure. One group for each material was bonded and allowed to stand for 15 minutes before being placed in distilled water at 37 °C for 24 hours before stressing to failure.

For each specimen the dentin surface was washed with water and air-dried. The manufacturers' recommendations for conditioning the dentin were followed for each product. Following treatment of the dentin, a layer of a low-viscosity adhesive was applied with a brush and thinned and cured for 30 seconds with visible light (Optilux 400, Demetron Research Corp, Danbury, CT 06810).

The mounted teeth that had just been treated with a dentin primer and adhesive were transferred to a special apparatus, as recommended by Retief and others (1988). Herculite XR restorative resin (Dark Gray Dentin) was then transferred to a split teflon mold in three increments. Each increment was cured with visible light for 30 seconds. The final increment was given an additional 30 seconds of visible-light cure. This resulted in a cylinder 3.5 mm in diameter and 5 mm in length that was bonded to the previously prepared and primed dentin. The bonded specimens were allowed to remain in the apparatus for 15 minutes without being disturbed before testing or storage.

The tooth sleeves were vertically mounted in a special device for testing. The mounted specimens were placed on the compression table of a

*Table 1. Dentin Bonding Systems and Composites Utilized*

| Material | Batch Number | Manufacturer |
|---|---|---|
| Prisma Universal Bond 2 | Dentin Primer #090189<br>Adhesive #0831891 | L D Caulk Co<br>Dentsply International<br>Milford, DE 19963 |
| Mirage Bond | Part #1 Conditioner #071089<br>Part #2 Hydrophilic Monomer #071089 | Chameleon Dental Products, Inc<br>Kansas City, MO 66101 |
| XR-Bonding System | XR Primer #93017<br>XR Bond #8407 | Kerr/Sybron<br>Romulus, MI 48174 |
| ALL-BOND | Dentin Conditioner #049180<br>Primer A #049180<br>Primer B #049180<br>Dentin/EnamelBonding Resin #118099 | BISCO, Inc<br>Downers Grove, IL 60515 |
| Uni-Etch | 32% Phosphoric acid #049090 | BISCO, Inc |
| All-Etch | 10% Phosphoric acid #049100 | BISCO, Inc |
| Herculite XR | Dentin Dark Gray | Kerr/Sybron |

*Table 2. Individual Specimen Values at 15 Minutes (MN/m²)*

| Sample # | Universal | Mirage | XR-Bond | ALL-BOND | All-Etch |
|---|---|---|---|---|---|
| 1 | 12.6 | 12.8 | 5.5 | 13.4 | 11.4 |
| 2 | 14.2 | 6.0 | 6.9 | 14.7 | 11.7 |
| 3 | 18.1 | 6.0 | 7.2 | 14.5 | 14.1 |
| 4 | 13.0 | 5.1 | 10.4 | 17.9 | 12.4 |
| 5 | 13.5 | 6.7 | 13.6 | 11.7 | 6.4 |
| 6 | 11.1 | 9.5 | 9.2 | 14.7 | 6.9 |
| 7 | 12.1 | 3.9 | 12.9 | 15.1 | 11.2 |
| 8 | 14.3 | 5.8 | 16.2 | 14.2 | 15.7 |
| 9 | 12.1 | 6.3 | 17.3 | 12.5 | 11.9 |
| 10 | 16.0 | 5.9 | 17.5 | 15.1 | 9.4 |
| 11 | 12.0 | 11.4 | 13.6 | 17.4 | 10.6 |
| 12 | 8.3 | 9.9 | 16.0 | 13.8 | 12.8 |
| 13 | 10.2 | 7.6 | 10.9 | 13.7 | 10.2 |
| 14 | 14.6 | 8.1 | 13.9 | 16.0 | 13.0 |
| 15 | 14.7 | 7.3 | 12.0 | 14.9 | 12.9 |

*Table 3. Individual Specimen Values at 24 Hours (MN/m²)*

| Sample # | Universal | Mirage | XR Bond | ALL-BOND | All-Etch |
|---|---|---|---|---|---|
| 1 | 12.0 | 4.2 | 16.5 | 9.6 | 9.4 |
| 2 | 9.9 | 6.6 | 14.8 | 16.2 | 11.9 |
| 3 | 19.8 | 7.0 | 21.6 | 15.5 | 9.3 |
| 4 | 6.5 | 6.5 | 16.0 | 19.1 | 19.1 |
| 5 | 12.3 | 4.7 | 17.6 | 16.2 | 20.8 |
| 6 | 14.9 | 5.6 | 21.4 | 18.5 | 20.1 |
| 7 | 16.1 | 4.9 | 23.9 | 19.7 | 15.4 |
| 8 | 15.8 | 6.5 | 20.0 | 15.2 | 19.3 |
| 9 | 18.6 | 9.5 | 21.4 | 10.8 | 10.8 |
| 10 | 15.3 | 8.8 | 17.2 | 14.3 | 10.4 |
| 11 | 17.7 | 10.4 | 16.6 | 10.2 | 10.0 |
| 12 | 12.9 | 9.5 | 16.2 | 14.5 | 9.3 |
| 13 | 14.7 | 13.4 | 17.1 | 14.0 | 15.9 |
| 14 | 16.7 | 5.1 | 21.4 | 10.0 | 16.6 |
| 15 | 12.5 | 5.5 | 21.8 | 11.1 | 12.7 |

Tinius-Olsen Testing Machine (Tinius-Olsen Testing Machine Co, Willow Grove, PA 19090), and a shear force was applied with a knife-edge device with a width of 0.5 mm at a crosshead speed of 0.02 inch/minute. The specimens were continuously loaded until fracture occurred. The shear bond strengths were then calculated and recorded in MN/m².

## Results

The individual shear bond strength values for the 15-minute specimens are reported in Table 2 and for the 24-hour specimens in Table 3. The means and standard deviations are illustrated in Table 4. ANOVA revealed statistically significant differences ($P < 0.05$) among the means at 15 minutes and at 24 hours (Tables 5 and 6). Tukey's multiple range test revealed statistically significant differences among the different dentin bonding systems at 15 minutes and at 24 hours (Tables 7 and 8). Figure 1 illustrates the rankings of the dentin bonding systems at 15 minutes. Systems that are not significantly different are connected by a horizontal line. Figure 2 illustrates the rankings of the dentin bonding systems at 24 hours. Systems that are not significantly different are connected by a horizontal line.

## Discussion

Further discussion is appropriate for the ALL-BOND and ALL-Etch techniques. When the restoration will involve enamel and dentin, the ALL-BOND technique recommends cleaning the tooth with pumice, preparing the cavity, placing a liner or base as required, etching the enamel with 32% phosphoric acid (Uni-Etch, BISCO) for 15 seconds with agitation, rinsing for 15 seconds, and drying with uncontaminated air. Dentin conditioner is applied to the dentin for 30 seconds with a brush and air-dried. One drop of Primer A is mixed with one drop of Primer B and then applied to

*Table 4. Means of Shear Bond Strengths at 15 Minutes and 24 Hours (MN/m²)*

| Material | Mean (SD) 15 Minutes | Mean (SD) 24 Hours |
|---|---|---|
| Universal Bond 2 | 13.12 (2.39) | 14.38 (3.46) |
| Mirage Bond | 7.49 (2.45) | 7.21 (2.58) |
| XR Bond | 12.21 (3.82) | 18.90 (2.82) |
| ALL-BOND | 14.64 (1.63) | 14.33 (3.37) |
| ALL-BOND/All-Etch | 11.37 (2.47) | 14.07 (4.31) |

*Table 5. Analysis of Variance (ANOVA): 15-Minute Samples*

| | SS | df | MS | F |
|---|---|---|---|---|
| Treatments | 431.1 | 4 | 107.83 | 15.39 |
| Error | 490.36 | 70 | 7.01 | |
| Total | 921.67 | 74 | | |

*Table 6. Analysis of Variance (ANOVA): 24-Hour Samples*

| | SS | df | MS | F |
|---|---|---|---|---|
| Treatments | 1051.15 | 4 | 262.79 | 23.12 |
| Error | 793.06 | 70 | 11.33 | |
| Total | 1844.21 | 74 | | |

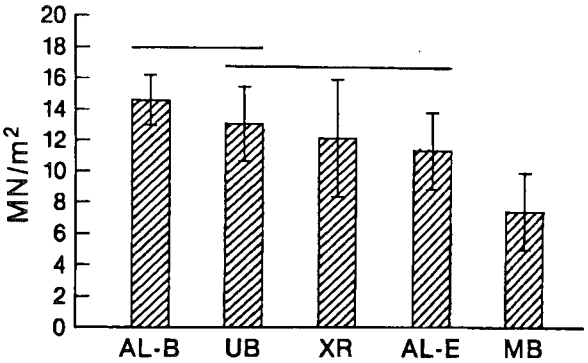


FIG 1. *Shear bond strengths, 15 minutes. Horizontal lines show no statistical difference.*

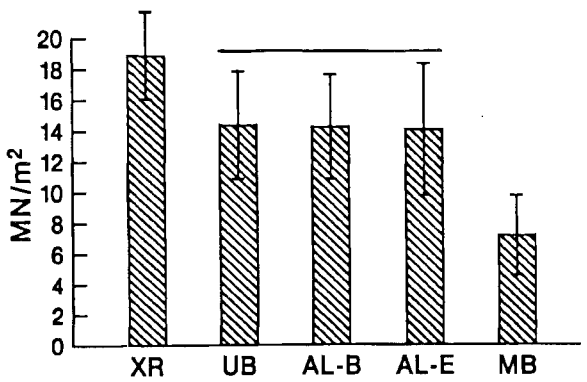


FIG 2. *Shear bond strengths, 24 hours. Horizontal line shows no statistical difference.*

*Table 7. Tukey's HSD Test: 15-Minute Samples*

| | UB | MB | XR | AL-B | AL-E |
|---|---|---|---|---|---|
| Universal Bond 2 (UB) = 13.12 | — | 5.63 | 0.92 | -1.51 | 1.75 |
| Mirage Bond (MB) = 7.49 | | — | -4.71 | -7.15 | -3.88 |
| XR Bond (XR) = 12.20 | | | — | -2.44 | 0.83 |
| ALL-BOND (AL-B) = 14.64 | | | | — | 3.27 |
| All-Etch (AL-E) = 11.37 | | | | | — |

a = 0.05, k = r, N = 75, n = 15, q = 3.97, HSD = 2.71

*Table 8. Tukey's HSD Test: 24-Hour Samples*

| | UB | MB | XR | AL-B | AL-E |
|---|---|---|---|---|---|
| Universal Bond 2 (UB) = 14.38 | — | 7.17 | -4.52 | 0.05 | 0.32 |
| Mirage Bond (MB) = 7.21 | | — | -11.69 | -7.12 | -6.85 |
| XR Bond (XR) = 18.90 | | | — | 4.57 | 4.84 |
| ALL-BOND (AL-B) = 14.33 | | | | — | 0.27 |
| All-Etch (AL-E) = 14.06 | | | | | — |

a = 0.05, k = 5, N = 75, n = 15, q = 3.97, HSD = 3.45

both the enamel and the dentin. Three to five coats of the mixed primer are applied and air-dried. Extra coats of primer are suggested, and it is recommended that all the mixed primer be used. The surface is then air-dried. Dentin/Enamel Bonding Resin (BISCO) is brushed thinly over the enamel and then the dentin and light-cured for 20 seconds. The composite is then placed in increments. It was our experience that it took more than five coats to utilize all the mixture of Primer A and B. In most cases it took eight to 10 coats to totally utilize this amount of primer mixture. This extended the time necessary to apply the primer. This could be a function of the drop size used.

In ALL-BOND's alternate technique, known as the "All-Etch or Kanca Technique," the enamel and dentin are etched for 15 seconds with 10% phosphoric acid, rinsed with water for 15 seconds, and then dried with compressed air. The primer is mixed and applied as previously described, followed by the Dentin/Enamel Bonding Resin and the composite resin restorative material.

The application of phosphoric acid to dentin has been and continues to be controversial. Retief (1973), Stanley, Going and Chauncey (1975), Macko, Rutberg and Langeland (1978), and Stanford (1985) reported on the effects of acid-etching the dentin and recommended that phosphoric acid should not be applied. Gwinnett (1977) reported that pretreatment of dentin with phosphoric acid enlarged the dentinal tubules. The depth of resin penetration using a normal clinical protocol was 0.2 mm. When the dentin was dried for 24 hours, the resin penetrated 2.00 mm. Brännström and Gaberoglio (1972) established the presence of odontoblast extensions at no more than 0.7 mm beyond the dentin-pulp interface. This lends credence to the recommendations that a liner should be applied when the remaining dentin is less than 1.0 mm.

Winson (1965) noted that $^{32}P$ did not penetrate the dentin in vivo even after two to six hours of contact. Johnson and others (1970) observed the same severity of inflammatory changes under distilled water as under phosphoric acid. Jennings and Ranly (1972) reported that dentin was penetrated by $^{32}P$, but the amount was so small that it was not detectable by the pulp. Lee and others (1973) applied 50% phosphoric acid or 50% citric acid to 1.0-mm disks of dentin and found that the acid etchants only affected the treated surface. Fusayama (1987) concluded that the most fundamental factor of pulp irritation is the separation of resin from dentin. Without this separation neither bacteria nor chemicals can irritate the pulp. Cox and others (1987) observed that chemical toxic factors such as acid and components of the restorative materials per se were less significant in causing pulpal injury than bacterial leakage around the restoration margins. Cox and Snuggs (1990) indicated that acidic components of silicate or zinc phosphate did not cause pulp inflammation or necrosis when surface-sealed to exclude bacterial infection. Kanca (1990) reported a clinical trial of 140 class 2 restorations at one year with no postoperative sensitivity, and all teeth pulp tested vital at one year.

The newer third-generation dentin bonding systems give excellent bonds to dentin, but they demand detailed attention (Erickson, 1989). Historically dentists have relied on dental materials being "forgiving" when they are utilized in the oral cavity.

It is important that dental practitioners comprehend what a dentinal bond formation entails, in order for them to appreciate the necessity of following specific procedural demands.

In order for dentin to bond to a polymeric resin, it is essential that the dentin be wetted by the material (Beech, 1982). A bond may be accomplished that is chemical or mechanical. Wetting promotes intimate contact to be formed, allowing bond formation to occur.

The role of the smear layer in dentin bond formation is arguable. One theory is that the smear layer should be removed because it is weakly attached and will adversely affect the formation of strong bonds to the underlying dentin. Another concept is to modify the smear layer, as opposed to removal (Bowen & others, 1984). An acidic pretreatment of the dentin may not be strictly a cleansing treatment, but may involve subsurface demineralization.

Nakabayashi (1985a & b) stressed that demineralization of the dentin must occur without denaturing the collagen.

Of the five systems evaluated, three utilize a

cleansing pretreatment of the dentin (Mirage Bond, ALL-BOND, ALL-Etch). Universal Bond and XR-Bond do not pretreat the dentin.

Each of the systems examined has a dentin primer as a component of the system. This primer aids wetting of the dentin and strengthens bond formation. This wetting is essential because dentin contains 13% water, and the resin materials are hydrophobic. Therefore the primers promote bond formation between a hydrophobic adhesive and a water-containing substrate, dentin.

After the dentin is primed and dried of water, a layer remains that can be resolubilized. This layer is very vulnerable in bond formation. It must not be rinsed or allowed to become contaminated.

An adhesive bonding resin is applied to the primed layer and thinned with air. The resin can resolubilize the primed layer, and the mixture of the adhesive and the primer is able to infiltrate into the dentin. Curing of this mixture creates a resin-reinforced layer of dentin that is capable of bonding to a restorative material.

## Conclusions

This in vitro study showed that ALL-BOND had the highest shear bond strength at 15 minutes, while XR-Bond had the highest values at 24 hours. The values for ALL-BOND showed little change from 15 minutes (14.64 ± 1.63 $MN/m^2$). The XR-Bond significantly increased from 12.21 ± 3.82 $MN/m^2$ at 15 minutes to 18.90 ± 2.82 $MN/m^2$ at 24 hours. All-Etch also showed an increase from 11.37 ± 2.47 $MN/m^2$ at 15 minutes to 14.07 ± 4.31 $MN/m^2$ at 24 hours. The reason for this increase would seem to indicate that the bonds were continuing to mature. ALL-BOND and Mirage Bond showed slight decreases in shear bond strength from 15 minutes to 24 hours; however, these changes were very slight (Table 4).

XR-Bond at 24 hours produced shear bond strength that was significantly higher than the other systems. There was no significant difference in the mean shear bond strength of Universal Bond, ALL-BOND, or All-Etch ($P < 0.05$).

All the systems tested produced bond strengths that were higher than previous first- and second-generation dentin bonding systems.

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# Direct Surface pH Determinations of Setting Cements

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## Summary

**Surface pH measurements were made over a 24-hour period for several luting cements and glass-ionomer lining and restorative materials. Of the luting cements, the water-mixed glass-ionomer cement, Ketac-Cem, had the lowest initial pH and the polycarboxylate, Durelon, had the highest. Three glass-ionomer lining materials showed differences in pH that increased over the entire test period. Two of the three visible-light-cured glass-ionomer liners evaluated showed similar pH values, while the third exhibited a considerably lower pH. The lower pH values found with the water-mixed glass-ionomer luting agent suggest that acidity may contribute to the posttreatment sensitivity associated with this material.**

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## Introduction

Glass-ionomer cements were developed in the late 1960s by Wilson and Kent and first described in 1972 (Wilson & Kent, 1972). These materials were produced by combining the powder component of the silicate cements (aluminosilicate glasses) with the liquid component of the polycarboxylate cements (polyacrylic acid). The production of the glass-ionomer materials in this manner stemmed from a desire to combine the beneficial aspects of the silicate cements with those of the polycarboxylate cements. It was hoped that this new cement would possess both the strength and translucency of the silicate

cements and the adhesiveness and biocompatability of the polycarboxylate cements (Smith & Williams, 1982).

Early clinical and laboratory testing seemed to confirm the presence of adhesiveness to tooth structure and relatively high compressive strength values. Cell culture and pulpal response studies provided additional positive information that affirmed that the glass-ionomer cements appeared to possess good biocompatability with pulpal and gingival tissues (Dahl & Tronstad, 1976; Kawahara, Imanishi & Oshima, 1979; Valcke & others, 1982; Nordenvall, Brännström & Torstensson, 1979).

The dental profession's response to the new restorative and luting agents was positive, and in the years following the introduction of the first glass-ionomer materials in the mid-1970s, dentists employed the glass-ionomer cements as restorative materials, luting agents, liners and bases, and pit and fissure sealants.

In 1984, however, reports began to surface of posttreatment sensitivity when certain forms of the glass-ionomer cements were used for luting (Council on Dental Materials, Instruments, and Equipment, 1984). The sensitivity, described as delayed in onset and progressive in development (Simmons, 1986), was most frequently seen with use of the "water-mixed" glass-ionomer cements, in which the polyacids were dehydrated and incorporated into the powder.

Several possible factors have been proposed as either sole or additive etiologic agents for the pain. Included among these are microleakage resulting from early moisture contamination (McLean, Wilson & Prosser, 1984) and low initial pH (Smith & Ruse, 1986).

Acidity of dental cements has long been held to be a major factor in causing pulpal irritation (Tompkins, 1922; Harvey, LeBrocq & Rakowski, 1944). Glass-ionomer materials, first believed to be nonirritating to the pulp because of certain physical characteristics of the polyacrylic acid component, have recently been shown to possess a low initial pH that remains low for an extended period of time (Smith & Ruse, 1986).

Because glass-ionomer cements have been shown to possess a low initial pH, which may play a role in causing posttreatment sensitivity, a study was conducted to more precisely determine the length of time during which these materials exhibit low pH values and to determine if differences exist between their different forms. Surface pH measurements were made of several glass-ionomer materials at selected time intervals for purposes of comparison to those of a zinc polycarboxylate cement and a zinc phosphate cement. In addition to pH values measured for mixes prepared at the manufacturer's recommended powder/liquid ratio, surface pH values were also measured for selected cements prepared with 25% more and 25% less powder.

## Materials and Methods

Three glass-ionomer liners, three glass-ionomer restorative materials, two glass-ionomer cements, three visible-light-cured glass-ionomer liners, one polycarboxylate cement, and one zinc phosphate cement were measured for surface pH (Table 1). The materials were mixed according to the manufacturers' instructions at a room temperature of 23 ± 2 °C and a relative humidity of 60 ± 5%. Two 4 mm high and 3 mm in diameter specimens were prepared for each cement, using a split silicone mold that was placed on the ground facial surface of a bovine incisor tooth. The mold consisted of a 4 mm high and 12 mm in diameter cylinder through which two 3 mm in diameter cavities had been prepared. Fifteen minutes after mixing, the silicone mold was removed from the specimens.

The light-activated glass-ionomer liners were mixed according to the manufacturers' instructions, placed into the silicone molds, and light-activated for the prescribed time. The silicone molds were then removed and the specimens were exposed circumferentially to the curing light to ensure that the entire depth of the material was fully exposed to the light. Immediately after preparation and between measurements, the specimens were stored at 37 °C and 100% humidity. Surface pH measurements were made using a standard pH electrode (model MI-410, Microelectrodes, Inc, Londonderry, NH 03053) and ion analyzer (model 501, Orion Research, Cambridge, MA 02129). Prior to measurement the ion analyzer was calibrated, using a buffered solution of pH 4.0. Immediately before each measurement

*Table 1. List of Cements*

| Material | Manufacturer | Type of Cement | Intended Use | Manufacturer's Powder/Liquid Ratio |
|---|---|---|---|---|
| Ketac-Bond | ESPE/Premier Norristown, PA 19404 | glass ionomer | liner | 3.4 g / 1.0 g |
| G-C Dentin Cement | G-C International Corp Scottsdale, AZ 85260 | glass ionomer | liner | 2.2 g / 1.0 g |
| G-C Lining Cement | G-C International Corp | glass ionomer | liner | 1.2 g / 1.0 g |
| Vitrabond | 3M Dental Products St Paul, MN 55144 | glass ionomer | liner | 1.4 g / 1.0 g |
| XR Ionomer | Sybron/Kerr Romulus, MI 48174 | glass ionomer | liner | 1.9 g / 1.0 g |
| Zionomer | Den-Mat Corp Santa Maria, CA 93456 | glass ionomer | liner | 2.5 g / 1.0 g |
| Ketac-Cem | ESPE/Premier | glass ionomer | cement | 3.4 g / 1.0 g |
| Durelon | ESPE/Premier | polycarboxylate | cement | 1.5 g / 1.0 g |
| Fuji Ionomer Type I | G-C International Corp | glass ionomer | cement | 1.4 g / 1.0 g |
| Fleck's | Mizzy, Inc Cherry Hill, NJ 08002 | zinc phosphate | cement | 2.4 g / 1.0 g |
| Fuji Ionomer Type II | G-C International Corp | glass ionomer | restorative | 2.4 g / 1.0 g |
| Ketac-Fil | ESPE/Premier | glass ionomer | restorative | precapsulated |
| Chemfil II | L D Caulk Co Dentsply International Milford, DE 19963 | glass ionomer | restorative | 6.8 g / 1.0 g |

approximately 0.01 cc of glass-distilled water was placed on the surface of each specimen. Testing was done at 2, 5, 10, 15, 20, 30, 45, 60, 90, 120 minutes and 24 hours from the start of mixing. Three separate trials were conducted for each cement and the results averaged.

Surface pH measurements of four luting cements were made after varying their powder/liquid ratios by 25% (Table 2). Three separate mixes were prepared: one at the manufacturer's recommended powder/liquid ratio, one with 25% more powder, and one with 25% less powder. A total of six specimens were made for each cement mix, and

*Table 2. Cement Powder/Liquid Ratios*

| Material | Manufacturer's Powder/Liquid Ratio | +25% Powder | -25% Powder |
|---|---|---|---|
| Ketac-Cem | 3.4 g / 1.0 g | 4.25 g / 1.0 g | 2.55 g / 1.0 g |
| Durelon | 1.5 g / 1.0 g | 1.88 g / 1.0 g | 1.13 g / 1.0 g |
| Fuji Ionomer Type I | 1.4 g / 1.0 g | 1.75 g / 1.0 g | 1.05 g / 1.0 g |
| Fleck's | 2.4 g / 1.0 g | 3.00 g / 1.0 g | 1.80 g / 1.0 g |

the results were averaged. The testing schedule and conditions were the same as previously described.

Mean pH values for the cements were compared at selected time periods by using one-way analysis of variance at the 0.05 probability level to test for statistically significant differences. When significant differences were found, Fisher's Least Significant Differences method was used to compare the means.

## Results

The results presented for each cement are mean values for the six specimens measured. With few exceptions, standard deviations were less than 0.15 pH units.

The results obtained for the four luting cements are presented in Figure 1. Initial pH values at two minutes ranged from 1.69 for the Ketac-Cem glass-ionomer cement to 3.4 for the Durelon zinc polycarboxylate cement. For the first five minutes after mixing, the water-mixed glass ionomer (Ketac-Cem) had pH values that were significantly lower than those of the non-water-mixed glass ionomer (Fuji I) and the zinc phosphate cement (Fleck's). The initial pH for Durelon was at least one pH unit higher than the others, and its pH remained higher for the first 60 minutes, until approached by Ketac-Cem as the rate of Durelon's increase slowed. The pH curve of the Fleck's cement remained the lowest for the majority of the test period, and final measurements revealed a pH considerably lower than the others. Ketac-Cem showed a rapid rate of increase in the first 15 minutes. By 60 minutes it had approached the levels of Durelon and at 24 hours was slightly higher. Final pH values at 24 hours ranged from 4.89 for Fleck's to 6.2 for Ketac-Cem. Although the final pH values for all of the cements were significantly different, Durelon, Ketac-Cem, and Fuji I were grouped together between 5.5 and 6.5, while Fleck's pH was under 5.

The initial pH values for the glass-ionomer cement liners (Fig 2) ranged from a low of 2.01 for G-C Dentin Cement to 2.90 for Ketac-Bond. For the entire test period, G-C Dentin Cement had pH values significantly lower than those of G-C Lining Cement and Ketac-Bond. The general pattern of pH change for the three lining materials was similar, with a rapid increase during the first 20 minutes, then it slowed to a more moderate rate after 45 minutes. Differences between the materials began to manifest themselves at 60 minutes, with a marked separation in pH measurements seen at 120 minutes. Twenty-four-hour values reflected the same relationship as the two-hour measurements, but differences between the materials were less. Final values were all significantly different and ranged from 5.87 for G-C Dentin Cement to 6.43 for G-C Lining Cement.

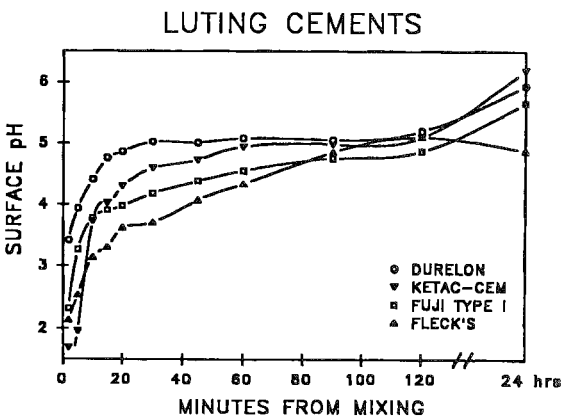


FIG 1. *Surface pH versus minutes from mixing for luting cements*

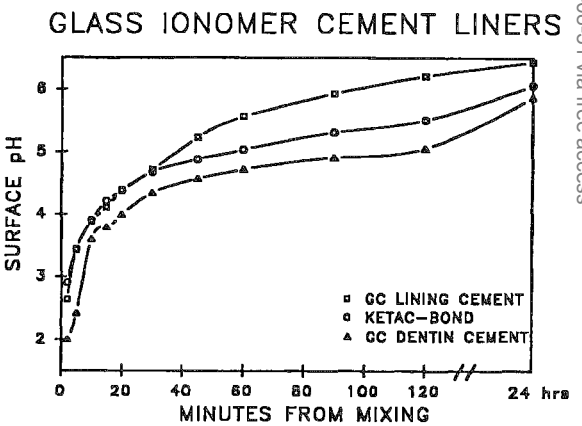


FIG 2. *Surface pH versus minutes from mixing for glass-ionomer cement liners*

The visible-light-cured glass-ionomer liners showed a distinct difference in values between two of the materials and the third, although patterns of pH increase were similar for all three (Fig 3). Initial pH measurements ranged from 3.26 for XR Ionomer to 4.94 for Zionomer. The pH values for Vitrabond and Zionomer were similar for the entire test period, particularly after the first 60 minutes. The XR Ionomer's values were significantly lower than those of the other liners for the entire test period. Twenty-four-hour measurements were 4.83 for XR Ionomer, 5.92 for Vitrabond, and 6.31 for Zionomer.

The glass-ionomer restorative materials, Chemfil II, Ketac-Fil, and Fuji Ionomer Type II, all showed similar patterns of pH increase (Fig 4). Initial pH values ranged from 2.27 for Fuji Type II to 2.98 for Chemfil II. At two minutes after mixing, Fuji Type II had a pH value significantly lower than those of the other two restorative materials. Chemfil II and Ketac-Fil had similar pH values, especially from the 45-minute point on. For the majority of the test, Fuji Type II exhibited pH values approximately 0.5 units lower than those of the other two cements. Final pH values ranged from 5.85 for Fuji to 6.28 for Chemfil II. At 24 hours, Ketac-Fil and Fuji Type II had pH values significantly lower than that of Chemfil II.

Fleck's zinc phosphate cement demonstrated distinct differences in pH values when mixed at powder/liquid ratios that varied from the one recommended by the manufacturer (Fig 5). The pH values for the mix prepared at the -25% powder/liquid ratio were the lowest at initial testing and remained the lowest for the entire testing period. Two minutes after mixing, the -25% mix had a pH value significantly lower than those of the other two mixes. With the exception of the 10-minute test value, measurements for the +25% mix were higher than the others and the values for the recommended powder/liquid ratio mix were approximately halfway between the other two. This pattern of differences is reflected in the 24-hour values of 4.25, 4.89, and 6.06 for the -25%, recommended, and +25% powder/liquid ratios respectively. All three values were significantly different from each other.

Fuji Ionomer Type I demonstrated a similar, but less pronounced, pattern of differences

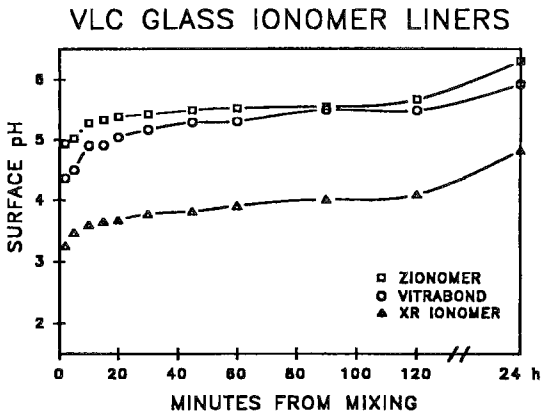


FIG 3. *Surface pH versus minutes from mixing for visible-light-cured glass-ionomer liners*

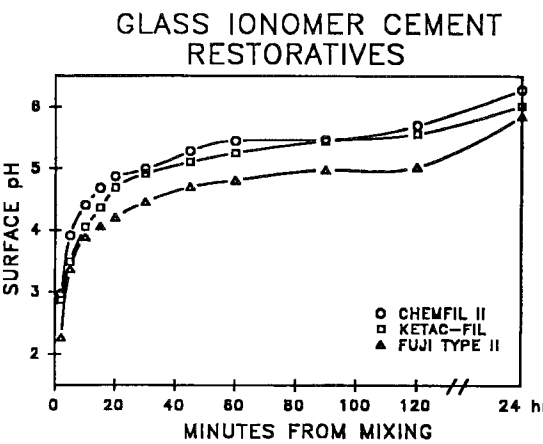


FIG 4. *Surface pH versus minutes from mixing for glass-ionomer cement restoratives*

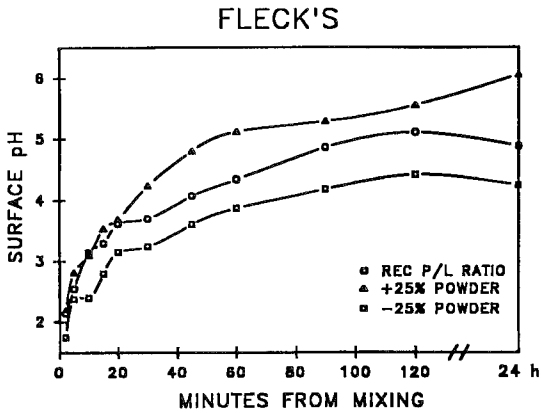


FIG 5. *Surface pH versus minutes from mixing for Fleck's*

between the various mixes (Fig 6). All three mixes had initial pH values of aproximately 2.3, but by 20 minutes they had begun to display the same order of differences as the zinc phosphate cement. These differences remained largely intact for the remainder of the 24-hour test period. Final pH measurements ranged from 5.41 for the -25% mix to approximately 5.70 for the recommended and the +25% mixes. The -25% mix had a 24-hour pH that was significantly lower than those of the other two mixes.

Durelon showed little variation in pH values for the three different mixes until the final measurement (Fig 7). Specimens showed an initial pH range of from 3.42 to 3.80 and a pattern of slow increase from 45 minutes to 120 minutes. The final pH values ranged from 4.83 for the +25% mix to 5.94 for the recommended powder/liqid ratio mix. All final values were significantly different.

Ketac-Cem similarly showed little variation between measurements for the different powder/liquid ratio mixes (Fig 8). All three mixes had an initial pH of approximately 1.7, and each demonstrated the same rapid rate of increase in pH during the first 15 minutes. At 24 hours, the recommended mix had a significantly higher pH than the other two mixes.

## Discussion

The general pattern of differences in pH seen for the four luting cements is consistent with the results obtained by Ban and others (1985), who found that a polycarboxylate cement had higher pH values over a 24-hour testing period than did a zinc phosphate cement and a glass-ionomer cement. They also found that Fuji Ionomer Type I had a higher initial pH than zinc phosphate cement, but had lower values beginning at 18 hours after mixing. A similar pattern is seen by comparison of the pH curves for the Fuji Type I and Fleck's cements (Fig 1), although the point at which Fuji began to register lower pH was considerably earlier in the testing period than that found by Ban and co-workers. This pattern of lower pH values for Fuji Type I compared to Fleck's had reversed itself by the final 24-hour point. The finding of higher pH measurements for Durelon than for the other three luting

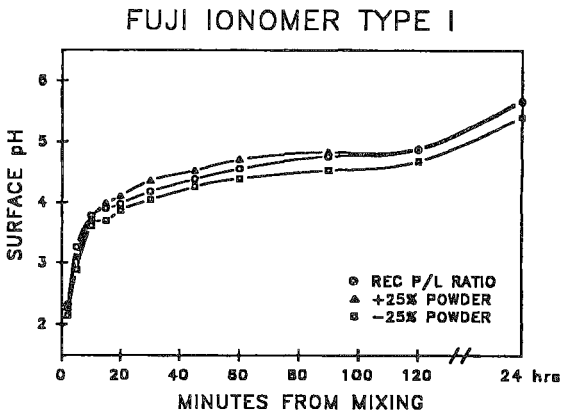


FIG 6. *Surface pH versus minutes from mixing for Fuji Ionomer Type I*

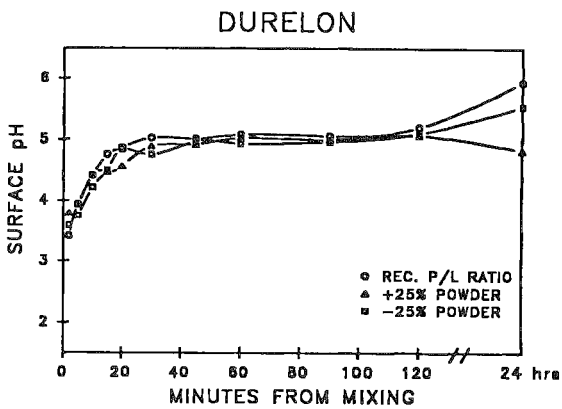


FIG 7. *Surface pH versus minutes from mixing for Durelon*

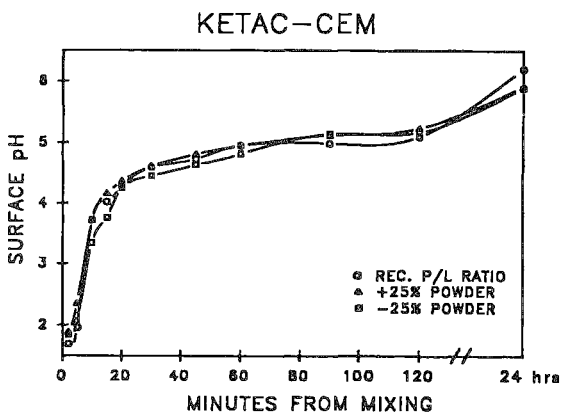


FIG 8. *Surface pH versus minutes from mixing for Ketac-Cem*

agents is consistent with the work of Brune and Evje (1984), who found that of a zinc polycarboxylate cement, a silicate cement, and a zinc phosphate cement, the zinc polycarboxylate cement consistently provided higher pH values.

The initial pH measurements for the glass-ionomer cement liner G-C Lining Cement are higher than those obtained by Smith, Ruse and Zuccolin (1988), who found that early pH values were below 2.0 and remained there for the first two minutes. The pH values obtained in this study show a higher initial pH (over 2.6) by the first two minutes. By the end of the test, the G-C Lining Cement's near neutral pH of 6.5 more closely agreed with Smith's result of 7.0.

The visible-light-cured glass-ionomer liners Vitrabond and Zionomer demonstrated pH values considerably higher than those of XR Ionomer. The differences in acidity between Vitrabond and XR Ionomer may in part be related to their compositional differences. Vitrabond, for example, contains 2-hydroxyethyl methacrylate (HEMA) in a 25% concentration, while XR Ionomer contains none of this monomer. The comparatively flat pH curves of all three liners may at least partially be due to the fact that they are light-activated materials. Light-activation produces rapid covalent crosslinking of the polymer chains, which may slow the free diffusion of hydrogen ions involved in the glass-ionomer reaction. This, in turn, would explain the relatively slow changes in pH that characterize these materials.

The lower pH values recorded for Fleck's zinc phosphate cement when mixed using a -25% powder/liquid ratio are consistent with the observations of past researchers who found that mixes produced from lower powder/liqid ratios had lower pH values (Norman & others, 1966).

Measurements of pH for Ketac-Cem made in this study were consistent with those of Smith and Ruse (1986). It had a longer period with pH below 3 than did the other glass-ionomer cement or the zinc polycarboxylate cement. Values for Ketac-Cem in both studies remained below 2 pH units for the first five minutes and then showed a marked increase by 10 minutes. Measurements correlated well and showed an average difference of 0.35 units. At 24 hours the final pH in both studies was the same (6.2).

Low pH values present during the early stages of setting for Ketac-Cem lend credence to the belief that high levels of acidity are in some measure related to the postcementation sensitivity experienced by patients. Researchers have expressed the opinion that in many cases pulpal inflammation is the result of cement acidity (Souder & Paffenbarger, 1942). The fact that initial pH values are lower for Ketac-Cem than for Fuji Type I is also in keeping with the observation that sensitivity most commonly occurs after the use of the water-mixed forms of the glass-ionomer cements.

The change in pH exhibited by other cements when their powder/liquid ratios are altered was not seen with Ketac-Cem. This suggests that the postcementation sensitivity problem, if indeed caused by low pH, would be affected very little by alterations in powder/liquid ratio.

One factor acting in association with lowered pH that has been identified as being a potential cause of sensitivity is the removal of the smear layer. The smear layer is routinely removed prior to the use of the glass-ionomer cements in an attempt to increase bond strength. Disruption of the smear layer reduces its ability to protect the pulp from toxic cement constituents (Meryon & Johnson, 1988). Additionally, the loss of the smear layer increases the surface area available for diffusion of bacteria and cement molecules (Pashley, Michelich & Kehl, 1981).

The difficulty that has been encountered in identifying the factor responsible for the post-treatment sensitivity associated with glass-ionomer cements suggests a multifactorial etiology. As a result, clinicians should actively attempt to reduce the impact that all of the factors may have in producing sensitivity. The smear layer, for example, should not be removed prior to using the luting forms of the glass-ionomer cements. In addition, protective bases should be placed in deep areas of the preparation where remaining dentin is believed to be minimal. In cases where a history of hypersensitivity exists, a less irritating cement such as polycarboxylate cement should be used.

## Conclusions

Surface pH measurements were made of several luting cements and glass-ionomer lining and restorative materials over a 24-hour period. For four luting cements, measurements were made after altering the powder/liquid ratio. Of the luting cements evaluated at the recommended powder/liquid ratio, Ketac-Cem had the lowest initial pH and Durelon had the highest. For two of the four materials, decreasing the powder/liquid ratio by 25% resulted in decreases in pH and increasing the powder/liquid ratio by 25% resulted in increases in pH. The three glass-ionomer cement liners showed differences in pH that increased with time until the final test measurement. Two of the three visible-light-cured glass-ionomer liners evaluated showed similar pH values, but the third reflected considerably lower pH over the entire test period.

Initial pH values of a water-mixed glass-ionomer cement were lower than those of a zinc phosphate cement. These data suggest that acidity may contribute to the posttreatment sensitivity associated with this form of the glass-ionomer cements. To prevent posttreatment sensitivity, it is recommended that the smear layer be left intact and that protective bases be placed when remaining dentin is thin.

The opinions expressed herein are those of the authors and do not necessarily reflect the opinions of the Department of Defense or the United States Air Force.

*(Received 31 October 1990)*

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# Clinician of the Year Award

Again this year I have the honor to be in a position to present the Clinician of the Year Award to a most worthy recipient. As you will recall, this is an award not given to one of our legendary members but is designed to recognize the contributions of the somewhat younger contingent of our Academy. The plaque has again been donated by the Ivoclar/Williams Company.

Since we created a possible medical emergency last year by not informing the recipient in advance, we thought it best to let this year's recipient in on the secret early in the game. When Dick Tucker was informed of the distinction, he immediately called our president and humbly suggested he must have received his father's mail. This was not the case--Richard D Tucker stands very comfortably on his own two feet.

We have all been impressed by Dick's clinical expertise and his virtually flawless class 5 foil preparation slides, blown up for all the world to see. Most of us would intentionally keep those of our own work slightly out of focus, but Dick doesn't find that necessary.

He has been busy outside of the Academy as well, serving as president of several groups, including his local dental society. He is active in at least four dental study clubs, has operated with excellence at many of our annual meetings, presented a number of table clinics and various essays. He has earned the respect of a good portion of our profession--the others don't know him.

His hobbies include fly fishing for steelhead, and sailing, having twice skippered a 33-foot sailboat in the race from Victoria to Maui, Hawaii. He has also been known to fire a mean shotgun, so it wouldn't pay to get on his bad side.

*Richard D Tucker*

Besides all these accomplishments, Dick Tucker is really a nice guy. It has been embarrassing to operate at the same Academy meeting with him, but it's an incentive to work fast and get the first audience. He is a perfectionist.

I am very pleased to be able to present this award to a most deserving individual. Congratulations!

RONALD D HARRIS, DDS, MSD

# Press Digest

**Retention and effectiveness of dental sealant after 15 years. *Simonsen, R J (1991) *Journal of the American Dental Association* 122 (11) 34-42.**

(*3M Company, Dental Products Division, Bldg 275-2SE-03, St Paul, MN 55144-1000)

This article reports on the longevity and effectiveness of a single application of pit and fissure sealant to all first molars after 15 years. The DFS data are compared to an age-, sex-, and geographically identical group in the same fluoridated community. The sealed group showed 74% sound first molar surfaces, while the control group showed 83% of the same surfaces either decayed or restored. For every two surfaces sealed, one surface was saved from caries or restoration. These data show that sealants are safe and effective in a single application. Maintained sealants in a private practice setting should show near 100% elimination of pit and fissure caries.

**Dentin bonding systems: a review of current products and techniques. *Johnson, G H, Powell, L V & Gordon G E (1991) *Journal of the American Dental Association* 122 (8) 34-41.**

(*University of Washington, School of Dentistry, Department of Restorative Dentistry, Seattle, WA 98195)

This article reviews most of the currently available dentin bonding agents. The article provides a concise tabular listing of components of each system, precautions for use and storage, and the retail cost of each system. A glossary defines the chemical terms used in the product components table. An additional table gives a well-organized overview of the time required for use of each product in a normal application. Times ranged from a low of 30 seconds to a high of 150 seconds.

The article reviews the indications for use of dentin bonding agents and briefly discusses the ADA's certification program for both posterior composites and dentin bonding agents. In addition the micromechanical nature of adhesion is discussed. Clinical studies do not show an advantage for placement of a cervical retentive groove in dentin nor does it appear necessary to bevel contiguous enamel. Material selections for classes of restorations are made along with a discussion of safety issues.

**Using antimicrobial agents to manage periodontal diseases. *Genco, R J (1991) *Journal of the American Dental Association* 122 (10) 31-38.**

(*State University of New York at Buffalo, School of Dental Medicine, Buffalo, NY 14214)

This article stresses that periodontal disease is a bacterial infection characterized by inflammation and destruction of the attachment apparatus. Since it is a bacterial infection, the author reviews the microbes associated with the disease, the limitations of mechanical periodontal therapy, and the treatment use of antibiotics and antimicrobial agents in systemic and local delivery forms. The article contains a number of tables that can be used as the basis for treatment of periodontitis with specific antibiotics for specific diseases and references for further reading. The toxicity of each periodontal antibiotic is described. A rationale of periodontal treatment purveying a medical model of care is presented.

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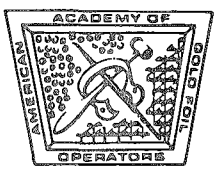


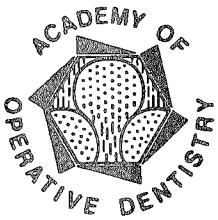


## volume 16
## 1991

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