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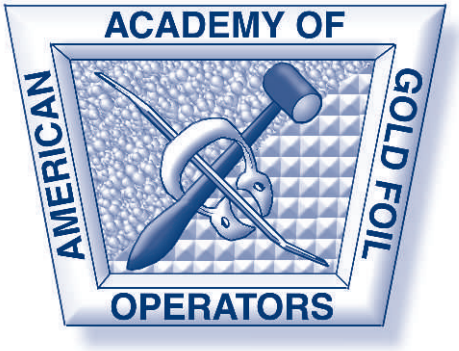

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Root Caries

INTRODUCTION

The Academy of Operative Dentistry has developed the following recommendations on diagnosis, treatment and prevention of root caries. Patients afflicted with this disease should be educated regarding etiologies, prevention methods, treatment alternatives and expected outcomes. Failure to appropriately prevent or treat root caries can result in the loss of teeth or tooth structure, weakening of teeth, need for endodontic therapy or increased risk of developing additional caries lesions.

Clinical Assessment and Diagnosis

A. Definition

A *root caries* lesion is "a soft, irregularly shaped lesion either (1) totally confined to the root surface or (2) involving the undermining of enamel at the cementoenamel junction but clinically indicating that the lesion initiated on the root surface" (Katz, 1995). Root caries occurs only if the root surface is exposed to the oral environment (Brown, Brunell & Kingman, 1996; Marcus & others, 1996; Winn & others, 1996).

B. Clinical Features and Methods of Assessment

Root caries lesions appear as a softening or cavitation in the root surface with no initial involvement of the adjacent enamel. These lesions generally begin at or slightly occlusal to the free gingival margin but can extend into the gingival sulcus or undermine the coronal enamel as the lesion progresses. Lesions also begin at the margins of restorations that terminate on root structure. Two reports (Mjör, 1981, 1985) indicate that secondary caries occurs more often at cervical margins of restorations, because they frequently end on root surfaces where access and isolation are most difficult. An active root caries lesion usually spreads laterally and eventually can encircle the tooth if left untreated.

Early lesions can be difficult to detect by appearance, as color changes are frequently not obvious until some progression of the caries activity has occurred. New lesions may appear as small, well-defined areas of a yellow to light brown color. On probing, the dentin in an active lesion is softer than adjacent, unaffected cementum or dentin. As it progresses, the surface of the lesion frequently has a "leathery" consistency that can easily be peeled away with a sharp excavator. Advanced lesions appear darker brown to black and may be as hard or harder than the normal root surface. Some researchers have attempted to categorize lesions based on color (lighter lesions more active, darker lesions inactive) and/or texture (the harder the lesion, the more inactive) (Beighton, Lynch & Heath, 1993; Billings, Brown & Kaster, 1985; Fejerskov & others, 1991; Syed & others, 1975). While some relationships between color, texture and dominant organism have been shown, the data have been conflicting and the links remain tenuous at best (Lynch, 1996).

Lynch (1996) found texture to be the best predictor of microbiological activity in root caries lesions. Careful tactile exploration using only moderate pressure should be performed, because the root surface is inherently softer than enamel. The gradient in tactile sensation is much less between sound and carious cementum/dentin than that between sound and carious enamel (Katz, 1995). Active lesions may or may not display obvious cavitation. They are generally described as "tacky" or "leathery" to probing, while offering some resistance to removal of the explorer tip. One study demonstrated that an explorer tip, bent at a 30° angle near its tip, increased the ability of the operator to detect root caries lesions (Newitter, Katz & Clive, 1985).

Caries activity may occur on any exposed root surface, but initial lesions on the buccal and approximal surfaces are most common (Katz & others, 1982). Some studies have suggested that 50 to 75% of root caries lesions begin interproximally (Hals & Selvig, 1977; Wagg, 1984). Lingual and palatal locations are seen much less frequently as isolated lesions. In the mandible, molars appear to be the most susceptible to root caries, followed by premolars, canines and

incisors, while in the maxilla, the order is reversed (Katz & others, 1982; Lawrence, Hunt & Beck, 1995; Leske & Ripa, 1989).

Although clinicians detect root caries lesions by judging changes in color (yellow, brown, black), texture (soft, hard) and surface contour (regular, irregular), examination strategies should focus on patients at risk for root caries. Therefore, the first step in diagnosing root caries is early identification of contributory factors and oral hygiene practices. Commonly, these lesions are obscured by plaque, food debris and calculus, so that accurate diagnosis is best accomplished following debridement and prophylaxis. Gentle tissue displacement with an air syringe and retraction with hand instruments can offer a better view of subgingival and interproximal areas. Use of transillumination and/or lighted mirrors and intra-oral cameras also enhances visibility and improves diagnostic capability.

Radiographs can be useful in identifying early interproximal root lesions but are occasionally prone to misinterpretation because of cervical "burnout" artifacts. Vertical bitewing radiographs permit better evaluation of the interproximal root surfaces in persons with significant loss of attachment (Jones, 1995).

Diagnostic tools and techniques, such as dye-enhanced laser fluorescence (DELF) and quantitative laser fluorescence (QLF), have shown promise *in vitro* (Ando & others, 1995; Ferreira, Analoui & Ando, 1995) and *in vivo* (Al-Khateeb & others, 1998) studies in enamel. This type of diagnostic aid should eventually be helpful in differentiating between active and inactive lesions by correlating the lesion severity and degree of mineral loss.

C. Etiology

The caries process that occurs on root surfaces is similar to that of coronal caries. Bacteria, which are capable of metabolizing dietary carbohydrates into acids, are present and produce a drop in pH to initiate decalcification of the tooth structure. Root surfaces are more vulnerable to chemical dissolution than enamel surfaces (Nyvad & Fejerskov, 1982). The pH decrease necessary for demineralization in cementum and dentin (pH 6.2-6.7) is less acidic than that required for enamel (pH 5.4-5.5) (Atkinson & Wu, 1994; Hunt, Eldredge & Beck, 1989). Initiation and progression of caries lesions occur more rapidly in dentin than in enamel. In addition, acid challenges can occur more readily and may continue for an extended period of time in dentin (Erickson, 1994).

Ramsay and Ripa (1969) report that the cementum and enamel are not confluent in as many as 30% of teeth. Additionally, the cementum on accessible root surfaces is often partially removed during scaling and root-planing procedures. Therefore, root caries commonly begins in dentin. Surface dissolution continues, followed by further demineralization and destruction of the collagen matrix (Johansen, 1965). Early microcavitation enlarges and produces the characteristic circumferential spreading seen with these lesions.

Currently, no specific microorganisms have been conclusively proven to cause root-surface caries. It is likely that root caries is a continuous, destructive process involving a succession of bacterial populations that vary depending on the condition of the substrate and the depth of the lesion (Schupbach, Osterwalder & Guggenheim, 1996; van Strijp, van Steenbergen & ten Cate, 1997).

D. Prevalence and Incidence

Numerous studies have reported the prevalence of root caries and its relationship with increasing age (Banting, Ellen & Fillery, 1980, 1985; Beck, 1993; Katz & others, 1982; Lohse, Carter & Brunelle, 1977), while international surveys have estimated the disease affects 60% to 90% of adults (Fejerskov & others, 1991; Hellyer & others, 1990; Katz, 1985; Locker, Slade & Leake, 1989; Luan & others, 1989; Miller & others, 1987). In addition, it has been suggested that approximately one in nine root surfaces at risk becomes carious. Studies (Fure & Zickert, 1990; Gustavson, Clive & Tveit, 1988; Hellyer & others, 1990; Kalsbeek & others, 1991; Katz & others, 1982; Whelton, Holland & O'Mullane, 1993) suggest that approximately 15% to 20% of all teeth with gingival recession are affected by root caries, and the mean number of teeth attacked per person is about 2.8 (Katz, 1985; Nyvad & Fejerskov, 1982).

There is general agreement that the prevalence of root caries will certainly increase in the dentate older population. The prevalence of untreated caries generally has been found to be constant with age (NIDR, 1987; Todd & Lader, 1991). Statistically, however, as the number of teeth decreases with age, the ratio of caries per tooth at risk increases, and root caries is a component of this. Thus, the ongoing loss of teeth with age is likely to produce an underestimation of the prevalence of root caries. It has been predicted that, in the United States, the percentage of dentate patients over age 65 in the total population will reach 85% or higher by 2020 (Beck, 1993).

E. Risk Factors/Assessment

Exposure of the root surface to the oral environment is a prerequisite for root-surface caries, so that patients with attachment loss, gingival recession or periodontal pocketing are at risk (El-Hadary & others, 1975). Patients with interproximal and cervical restorations ending on cementum should not be overlooked. Even though the root surface may not

be readily visible, the need for and placement of these restorations has satisfied the primary risk criteria.

All normal risk factors for caries development are applicable to root caries, including inadequate oral hygiene, cariogenic diet and poor utilization of routine dental services. Past caries and restorative experiences also have a strong correlation and generally indicate the presence of conditions or behaviors that support caries activity (Beck, Kohout & Hunt, 1988; Joshi, Papas & Giunta, 1993; Lawrence & others, 1995; Locker, 1996; MacEntee, Clark & Glick, 1993; Powell, Mancl & Senft, 1991; Scheinin & others, 1992).

Salivary flow rate is considered the most important of the non-microbial salivary parameters (Clarkson, 1995). The cariostatic activity and efficacy of other salivary parameters are dependent on the flow rate (Tenovuo, 1997). Xerostomia or "dry mouth" has been shown to have a positive correlation to a number of adverse oral conditions, including rapidly progressive dental caries and periodontal disease (Haveman & Redding, 1998; Ravald & Hamp, 1981).

Xerostomia can be caused by a variety of factors including radiation therapy of the head and neck, immunosuppressive therapy, radioactive iodine therapy, autoimmune diseases, HIV infection and a myriad of commonly prescribed medications (see Table 1) (Astroth, 1996; Atkinson & Wu, 1994; Haveman & Redding, 1998). Basic management of xerostomic patients involves finding ways to reduce their oral dryness. If functioning salivary gland tissue is present, natural flow stimulation is preferable to saliva substitutes. Pilocarpine can be an extremely effective salivary gland stimulant (Ferguson, 1993). However, pilocarpine has numerous side effects, contraindications and drug interactions, making consultation with the patient's primary care physician mandatory before recommending its use. Oral moisturizers are sometimes the only option for relieving the symptoms of xerostomia. These saliva substitutes can be used on a regular basis, but some commercial products have been found to have a pH below the demineralization point of enamel and should be avoided (Haveman & Redding, 1998).

Because they contribute to the retention of food debris and gingival recession, removable partial dentures have also been identified as a risk factor in this disease (Shay, 1994). Other factors that contribute to the potential for root caries include previous caries/restorative experience. Studies have indicated that individuals who have coronal caries are 2-3.5 times more likely to develop root caries (Hand, Hunt & Beck, 1988a,b; Pappas, Koski & Giunta, 1992). Root caries is generally more prevalent and severe among males than females (NIDR, 1987). Smoking has also been implicated as a risk factor in both periodontal disease (Tonetti, 1998) and the root caries process (Ravald & Birkhed, 1992).

Table 1: *Medications That Induce Xerostomic Changes*

Anorexiants	Cold Medications
Antiasthmatics	Decongestants
Anticholinergics	Diuretics
Anticonvulsants	Expectorants
Antidepressants	Muscle Relaxants
Antiemetics	Neuroleptics
Antihistamines	Psychotropic Drugs:
Antihypertensives	CNS Depressants
Antiinflammatories	Dibenzoxazepine Derivatives
Antinauseants	Phenothiazine Derivatives
Antiparkinsonians	MAO Inhibitors
Antipruritics	Tranquilizers
Antispasmodics	Sedatives
Appetite Suppressants	Sympathomimetics

Therapeutic Goals and Considerations

A. Preventive and Chemotherapeutic Strategies

Clinical observations suggest that root caries lesions can be arrested, obviating the need for restorative therapy (De Paola, 1993; Emilson, Ravald, & Birkhed, 1993). The majority of evidence relating to de- and remineralization of root caries lesions comes from *in vitro* research. However, *in vivo* studies (Nyvad & Fejerskov, 1986; Schupbach & others, 1996) and *in situ* (Nyvad, ten Cate, & Fejerskov, 1989; Nyvad & Larsen, 1992; Nyvad, ten Cate, & Fejerskov, 1997) have demonstrated success in preventing or arresting root caries through plaque removal, diet modification and topical fluoride application.

Plaque removal alone has been shown to play an important role in arresting active root caries lesions (Emilson & others, 1993). *In situ* studies have confirmed that when both plaque thickness and acidogenic response to sucrose exposure are significantly reduced, lesions become inactive (Nyvad & Larsen, 1992). A 0.12% chlorhexidine rinse (Peridex, Perioguard) can also be used to treat root caries. While chlorhexidine has been used primarily as an antimicrobial treatment for gingivitis and periodontal disease, it is very effective in eliminating cariogenic bacteria.

Topical fluoride is accepted as an appropriate chemotherapeutic agent in the management of root caries. Prevention and arrest of root-surface lesions have been demonstrated in both *in situ* and clinical

studies using fluoridated water (Brustman, 1986; Hunt & others, 1989; Stamm, Banting & Imrey, 1990), fluoride gels (Billings & others, 1985), fluoride mouthrinses (Teranaka & Koulourides, 1987), fluoride dentifrices (Jensen & Kohout, 1988), fluoride varnishes (Emilson & others, 1993), fluoride chewing gum (De Los Santos & others, 1994) and intra-oral fluoride releasing devices (De Los Santos & others, 1994; Mirth & others, 1982).

Studies have shown the benefits of substituting dietary polyols for sucrose in chewable dietary items. Xylitol, a five-carbon sugar alcohol, is not metabolized by *S mutans* and has been shown to have an anticariogenic effect (Nuuja, Meurman & Torkko, 1993; Trahan, 1995), decrease plaque formation (Isotupa & others, 1995), increase plaque pH (Aguirre-Zero, Zero & Proskin, 1993) and enhance remineralization (Wennerholm & others, 1994). A 5-10 gram daily consumption of xylitol in the form of gum can result in a 30% to 85% reduction in dental caries (Mäkinen & others, 1995, 1998).

The goal of the dental practitioner should be to initiate preventive and remineralization therapies that will inhibit or eliminate the disease process before the destruction of dental tissues occurs. The excavation of actively carious tooth structure and placement of restorative materials is, at best, a repair of the damages inflicted by the disease process and does not address the control of the disease itself.

B. Restorative Treatment

Clearly, many root-caries lesions do not require restorative treatment. Accessible, shallow lesions can be made caries-free and cleansable through debridement using hand instruments, finishing burs or polishing disks (Billings & others, 1985; Wallace, Retief & Bradley, 1993). Arrested lesions with a hard to leathery surface often are amenable to treatment with topical fluorides and chlorhexidine rinse (Beighton & others, 1993).

When root caries has progressed to the point where restoration of lost structure is necessary, the dentist faces difficulties that differ considerably from those posed by most coronal lesions. The challenges to the restorative dentist include impaired visibility, difficult access, moisture control, pulpal proximity and the nature of the dentin substrate itself. All of these factors tend to compromise the ideal restoration, which should conserve remaining tooth structure and provide long-term integrity of marginal seal. There is general agreement today that, when possible, adhesive fluoride-releasing materials are preferred (Burgess, 1995).

Isolation is the key to long-term success when restoring root surfaces. Inability to obtain a dry operating field and reasonably unobstructed access and visibility frequently results in a badly compromised restoration. The use of rubber dam and retractors, retraction cord or surgical exposure in extreme cases will usually satisfy the necessary criteria. At times, the isolation may take more time than the actual preparation and restoration but, in such cases, a satisfactory result could not be obtained in any other way.

A brief discussion of restorative materials and their application to treat root caries lesions follows:

1. Amalgam—Amalgam has the longest clinical history of the direct restorative materials with the exception of direct filling golds. It has excellent wear characteristics, increasing marginal seal over time and some limited bacteriostatic properties. Amalgam is relatively easy to place and is less sensitive to variations in handling than many other restorative materials. However, it must be mechanically retained and requires adequate bulk of material to resist fracture. It does not offer any chemotherapeutic benefit. The use of amalgam in cervical lesions has declined, but it still might be the material of choice when isolation is a problem.

2. Glass Ionomer Cement/Resin-Modified Glass Ionomer Cement—Glass ionomers might be the materials of choice for restorative treatment of the majority of root caries lesions. These materials offer adhesive bonding, long-term fluoride release and the ability to "recharge" or uptake fluoride when exposed to an external source (topical application, mouthrinse). Clinical studies have demonstrated successful longevity for 10 years (Matis, Cochran & Carlson, 1996) and reasonable success in xerostomic patients (Haveman & others, 2003).

3. Resin Composite—With the advent of relatively reliable dentin bonding agents, resin composite materials, including compomers (polyacid modified resins) and flowable composites, have become extremely popular. Unfortunately, these materials undergo polymerization shrinkage that can severely stress the adhesive interface, resulting in a loss of marginal seal and microleakage. Fluoride release from compomers is less than from glass ionomers, and fluoride re-charging is less likely. Fluoride release from fluoride-containing conventional composites occurs at a very low and likely non-therapeutic level. Composites are primarily indicated in root caries situations where esthetics is of major importance. Microfilled resin composites appear to offer some advantages over compomers and flowable composites.

4. Indirect Restorations—Indirect restorations generally are not indicated for treatment of root caries lesions unless it is determined that the affected tooth requires a crown of some type and the area of the lesion can be included in the tooth preparation without compromising an excessive amount of tooth structure or providing additional compromise to the periodontal situation.

Outcomes Assessment

The primary desirable outcome is the prevention of root caries lesions through good home care, hygiene procedures and chemotherapeutic regimens. If a lesion has already been initiated, a satisfactory result of nonrestorative therapy is remineralization of the area, absence of progression of the lesion, absence of symptoms caused by the lesion and absence of occurrence of any new lesions. The satisfactory outcome for a restoration is the absence of recurrent lesions, the long-term retention of the restoration without loss of contour and absence of symptoms associated with the restored tooth.

Adapted from Cochran MA & Matis BA: Chapter 12, Diagnosis and Treatment of Root Caries, in Fundamentals of Operative Dentistry, A Contemporary Approach*, edited by Summitt JB, Robbins JW & Schwartz RS, Chicago: Quintessence Publishing Co Inc, 2001, for the Academy of Operative Dentistry Special Project Committee (Triolo, PT—chair) by Berry TG, Summitt JB & Swift EJ Jr.*

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Clinical Research

A Retrospective Observational Study of the Effect of Surface Treatments and Cementing Media on the Durability of Gold Palatal Veneers

RG Chadwick • KI Linklater

Clinical Relevance

The provision of gold palatal veneers provides a conservative method of restoring palatal tooth surface loss. Success can be optimized by alumina blasting the fit surface of the restoration and acid etching the tooth prior to cementation with Panavia 21. Further potential for improved retention is offered by oxidation of the fit surface of the restoration prior to cementation.

SUMMARY

This paper reports on the results of a retrospective observational study that sought to determine both the longevity and effects of surface treatments of gold palatal veneers used to restore tooth surface loss. Details of all gold palatal veneers fabricated from Mattident 60 were sourced from hospital records spanning 11 years and 9 months. The case notes of each individual were accessed and, for each restoration, a note was made of the date and method of cementation, together with the period of patient follow-up. When patients continued their routine check-ups, the records were scrutinized closely for evidence of restoration failure. This yielded a data set of 151 palatal gold veneer cementations for which the surface treatments and/or cementing media were known. Survival analysis by the Kaplan-Meier method of alumina blasted veneers revealed median survival times of 4,663 days when cemented with Panavia 21 and 687 days if cemented with Aquacem. Veneers that were alumina blasted, oxidized and cemented with Panavia 21 had a survival probability of 1.0. A Logrank test revealed highly statistically significant differences between the survival curves ($p<0.0001$). It was concluded that: (1) Alumina blasting the fit surface of a gold veneer prior to cementation with Panavia 21 resulted in a significantly more durable restoration compared to alumina blasting and cementation with Aquacem and no etching of tooth substance. (2) Due to low MST cementing, gold palatal veneers with a conventional glass polyalkenoate cement are not recommended. (3) Pre-treatment of gold palatal veneers by alumina blasting and oxidation prior to cementing with Panavia 21 appears to

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improve the chances of obtaining a dependable restoration. A greater number of restorations would be required to statistically test this trend.

INTRODUCTION

Recent surveys conducted within the United Kingdom have identified dental erosion as affecting just over 50% of children (O'Brian, 1994; National Diet and Nutrition Survey, 2000). Dental erosion has been described as the loss of dental hard tissue resulting from dissolution by acids that are not of bacterial origin (Pindborg, 1970). Such acids may be either intrinsic or extrinsic in origin (Jarvinen, Rytomaa & Heinonen, 1991). Extrinsic factors include acidic foods and drink as well as certain medicines and tonics (Smith & Knight, 1984; Meurman & Murtomaa, 1986). Intrinsic erosion results from regurgitation of gastric contents into the mouth, for example, from a hiatus hernia or psychological disorders that include bulimia and anorexia. Either source may result in severe tooth destruction, with the loss of maxillary anterior palatal tooth substance and possible attendant thermal sensitivity and chipping or occlusal tooth surface loss from molars and premolars with an associated reduction in occlusal vertical dimension being common sequelae that challenge the restorative dentist.

Some studies have advocated restoration of the maxillary incisor palatal tooth surface loss using porcelain (Milosevic, 1990) or gold (Hussey, Irwin & Kime, 1994; Darbar, 1994) palatal veneers. Since both may be bonded adhesively to the residual tooth structure, they are conservative restorations. Although stable in the oral environment, porcelain can fracture in use and needs to be of a reasonable thickness for strength and does not lend itself to intra-oral adjustment (McAndrew, 1995). On the other hand, palatal veneers made of gold alloys are stronger in thin section (Hussey & others, 1994), bring about only slight change in the labial appearance of thin teeth (Livaditis & Tate, 1988) and produce comparatively little wear of the opposing teeth (Craig, 1989; Hussey & others, 1994).

In recent years, gold's lack of adhesion to tooth substance has been overcome by a variety of surface treatments that include alumina blasting, tin plating and heat treatment (Tanaka & others, 1988; Eder & Wickens, 1996) prior to cementation with a chemically active resin. Laboratory investigation has demonstrated that blasting the fit surface with 50 μm alumina and air firing the restoration at 400°C for four minutes prior to cementation with a chemically active resin gives a superior bond to tooth substance as assessed by tensile loading to failure (Eder & Wickens, 1996). Despite these numerous advantages, only one report exists on the long-term clinical performance of this type of restoration (Nohl & others, 1997). This report studied nickel chromium palatal veneers placed at a teaching hospital for up to 4.7 years and reported an overall success rate of 89% with a 56 month survival probability of 0.74. These veneers were air abraded with 50 μm aluminum oxide prior to cementation with either the chemically active resin Panavia Ex (Kuraray, New York, NY, USA) or the glass polyalkenoate (Ionomer) Aquacem (Dentsply De Trey GmbH, D-78467, Konstanz, Germany). Although veneers constructed from yellow gold were included in this study, no survival curve was presented and, as such, no reports exist on the long-term clinical performance of gold palatal veneers.

This paper reports on the results of a retrospective observational study that sought to determine the longevity and effects of surface treatments of gold palatal veneers used to restore tooth surface loss.

METHODS AND MATERIALS

This retrospective study was conducted at the Dundee Dental Hospital and School (Scotland). Details of all gold palatal veneers fabricated from Mattident 60 (Cookson's Precious Metals Ltd, Birmingham, UK), whose composition is show in Table 1, were sourced from the restorative laboratory's production records spanning 11 years and 9 months. The production records enabled lists of patients to be compiled for whom restorations of this type had been fabricated. The case notes of each individual were accessed, and for each restoration, a note of the restored tooth, date and method of cementation and period of patient follow-up was made. In the case of patients continuing to attend for routine check-ups, the records were scrutinized closely for evidence of restoration failure. If failure had occurred, the date when it was first noted was considered the date of failure. This, along with all other details, was entered into a relational computer database (Paradox 3.5, Borland International, Scotts Valley, CA, USA) customized by the investigators. To conform to the Data Protection Act (1984), all computer records associated with this study were registered with the Data Protection Registrar through the NHS

Table 1: *The Composition of Mattident 60 Alloy*

	% (w/w)
Gold	60%
Copper	19%
Silver	15%
Palladium	4%
Zinc	1%
Platinum	0.5%

Compiled from manufacturer's data.

Tayside Research and Development Consortium. For each restoration, the investigation revealed:

- The method of surface treatment.
- The cementing medium.
- The period of follow-up.
- The length of time of known survival or failure.

For each restoration type, survival curves were generated by the Kaplan-Meier method and Prism (Version 4.0, GraphPad Software Inc, San Diego, CA, USA). This was carried out for all such restorations and according to the various surface treatments/cementing media identified by the retrospective search of patient records. Restorations that had survived were treated as censored data, whereas, failures were classified as uncensored.

RESULTS

Figure 1 shows a typical example of a gold palatal veneer.

Data for a total of 195 palatal gold veneer cementations were available for review. Of these, the surface treatments and/or cementing medium was known for 151. Where failure occurred, this was exclusively the result of the restoration debonding from the tooth. Two types of cement had been used to affix these restorations in the alumina blasted state with and without oxidation. They were self-curing resin cement Panavia 21 (Kuraray) and conventional glass polyalkenoate (ionomer) cement Aquacem (Dentsply De Trey GmbH). Where Panavia 21 had been used, the entire palatal surface of the tooth had been acid etched according to the manufacturer's instructions. No acid etching was undertaken for those veneers luted in place with Aquacem in accordance with the manufacturer's instructions. The choice of cement was based on personal operator preference.

Table 2 summarizes the period of follow-up for each surface treatment and cementing medium applied to the gold palatal veneers in the study. Table 3 summarizes the number of restorations in each subgroup, together with the number of failures, % failure rate and median survival time (MST). Since all the palatal veneers that were blasted, oxidized and cemented with Panavia survived, it was not possible to calculate a median survival time from the available data.

Figure 2 shows the survival curves for each method of gold palatal veneer surface treatment and cementation included in the study. A comparison of the survival curves by the Log Rank test revealed highly statistically significant differences between them ($p<0.0001$).

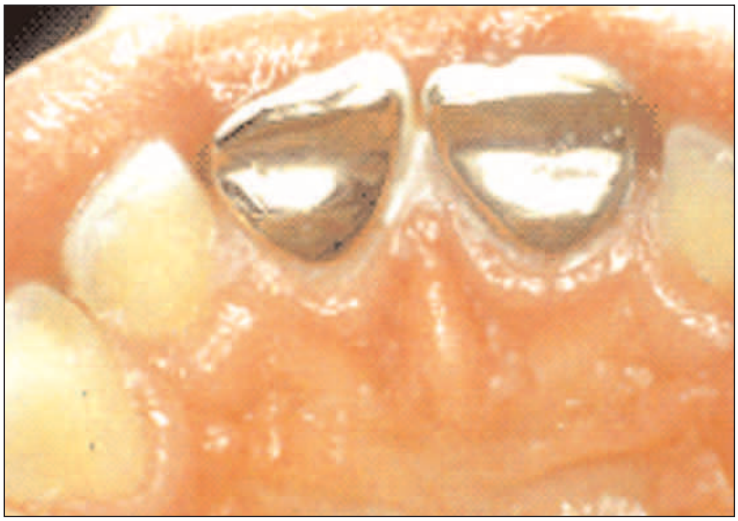

Figure 1. *Gold palatal veneers on the palatal aspects of permanent maxillary central incisors.*

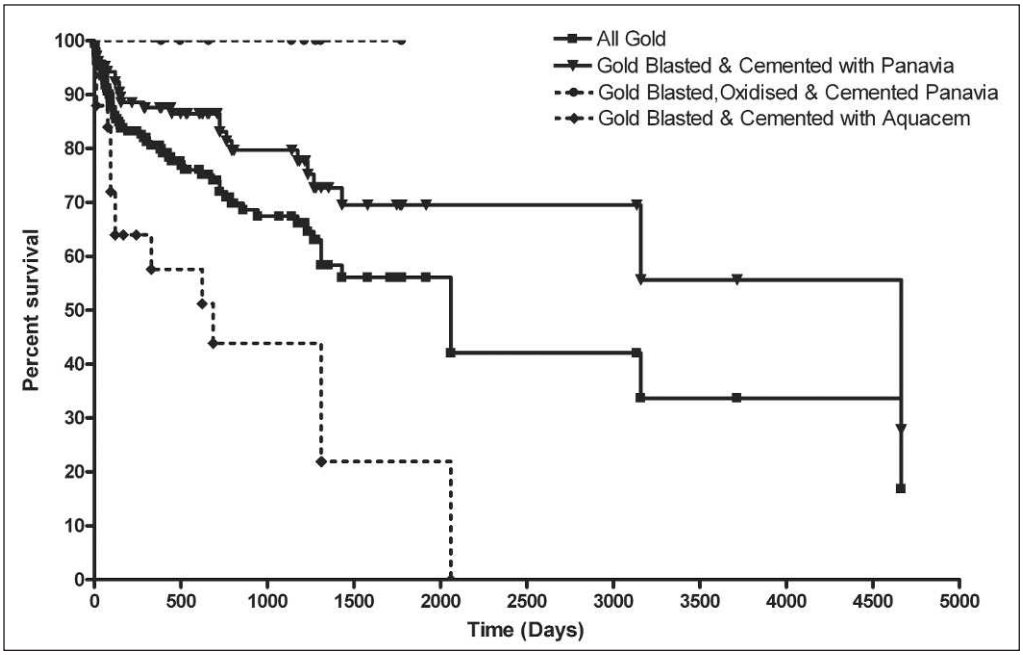

Figure 2. *A graph of the percent of restoration survival versus time to failure (days) for palatal gold veneers placed using various surface treatments and cementing media. The all-gold grouping is the combined survival analysis of all sub-groupings on this graph.*

Table 2: *Summary of Follow-up Period (days) for Gold Palatal Veneers According to Both Surface Treatment and Cementing Medium*

Follow-up Period/ Surface Treatment	Blasted[a] & Cemented with Panavia[b]	Blasted, Oxidized[c] & Cemented with Panavia	Blasted & Cemented with Aquacem[d]	Pooled Data for All Gold Veneers in Study[e]
Minimum	0	148	0	0
25% Percentile	252	388	94	123.5
Median	627	901.5	167	440
75% Percentile	1252	1310	998.5	1141
Maximum	4663	1777	2061	4663
Mean (SD)	874.2 (888.8)	896.7 (561.4)	516 (635.4)	675.3 (781.0)

Details of surface treatments and cementing media:

[a]Blasted with 50 µm alumina to even matt finish.

[b]Panavia 21 (Kuraray, New York, NY, USA).

[c]Placed in porcelain furnace at 400°C and held for four minutes before removal. Allowed to cool to room temperature under normal room ambient conditions.

[d]Aquacem (Dentsply De Trey GmbH, D-78467, Konstanz, Germany).

[e]Includes all veneers in preceding columns and also gold palatal veneers whose surface treatment and cementing medium are not identifiable from the patient notes.

To avoid duplication, superscripts are only placed within the table on the first occasion a particular treatment/cement is mentioned.

Table 3: *Gold Palatal Veneers—Summary According to Sub-grouping of Number of Restorations, Number of Failures, % Failure Rate and Median Survival Time (days)*

	Blasted[a] & Cemented with Panavia[b]	Blasted, Oxidized[c] & Cemented with Panavia	Blasted & Cemented with Aquacem[d]	Pooled Data for All Gold Veneers in Study[e]
Number of Restorations	105	21	25	195
Number of Failures	24	0	17	59
% Failure	22.9	0	68.0	30.3
Median Survival Time	4663	Not computed	687	2061

Details of surface treatments and cementing media as for Table 2.

DISCUSSION

This study is unique, for it has examined the longevity of gold palatal veneers that were never previously reported in the literature. The study only utilized survival data from patients who continued to attend the Dundee Dental Hospital and School for routine examination over the follow-up period. As a consequence, assessment of durability was based on clinical examinations—important criteria concerning the validity and quality of such a study (Downer & others, 1999).

In order to make survival predictions regarding the longevity of restorations, the data was both pooled and subdivided according to the nature of the surface treatment received by the veneers and the cement used. The study did not take into account the quality or quantity of residual enamel available for etching on the palatal aspect of the treated teeth. Such a mixture model has previously been applied successfully to assess the survival characteristics of named dental restorative materials (Smales, Webster & Leppard, 1991; Chadwick & others, 2001). The low Median Survival Time (MST), 687 days for blasted Aquacem cemented gold palatal veneers, agrees with the reported experiences of Nohl and others (1997), who also concluded that the adhesion afforded by such a glass ionomer cement was less effective than a chemically active resin, presumably because the surface of the tooth was not acid etched to afford micromechanical retention. This raises the question of why such a cement was used in this application. Although no longer used in the Dundee Dental Hospital and School for this purpose, as a consequence of poor retention of palatal veneers, it was perceived at the time that acid etching of dentin, as required for cementation by a chemically active resin, could cause moderate to severe pulpal reactions similar to when extrapolated from animal studies (Pashley, 1992). In contrast, glass polyalkenoate (ionomer) cement was known not to produce such a marked reaction (Gaintantzopoulou & others, 1994) and, as such, was considered relatively inert to the pulp. In fact, the pulpal irritation seen in such studies was due to the microleakage of bacteria and their products (Pashley, 1992), and with hindsight, such caution was probably not necessary.

Although not exactly comparable to this study, because it related to tin plated surface oxidized nickel chromium palatal veneers cemented with a chemically active resin (Panavia-Ex, Kuraray), it is worth comparing the results of Nohl and others (1997) to the results of this study. These workers reported a survival proba-

bility of 0.74 at 56 months (1,736 days). In the current study, gold palatal veneers blasted and cemented with Panavia 21 obtained a survival probability of 0.70 for this time period compared to only 0.23, when blasted gold veneers were cemented with Aquacem (Figure 2). These results are comparable to Nohl and others (1997) and further support their claim that cast metal veneers provide a useful method of restoring maxillary anterior teeth affected by acid erosion. Such survival rates can potentially be enhanced if oxidation of the veneers occurs prior to cementation with Panavia 21 (Figure 1—56 Month Survival Probability = 1.0). The relatively small number of restorations treated in this way (n=21) and the high survival rate does not permit an MST to be meaningfully calculated. However, this emerging trend supports laboratory studies which have demonstrated that such surface treatment enhances retention (Eder & Wickens, 1996). Also noteworthy is that concern has been expressed regarding the potential for allergic responses to nickel containing alloys (Pierce & Goodkind, 1989; Kansu & Aydin, 1996) and the use of gold alloy in this study avoids exposure to this potentially allergenic element. Given also that an alternative method of treating such cases would be to provide conventional crowns, destroying further tooth substance and risking future periradicular periodontitis (Saunders & Saunders, 1998), the provision of palatal veneers offers both a predictable and conservative alternative.

CONCLUSIONS

1. Alumina blasting the fit surface of a gold veneer prior to cementation with Panavia 21 results in a significantly more durable restoration compared to alumina blasting and cementation with Aquacem and no etching of tooth substance.
2. Due to a low MST, cementing gold palatal veneers with a conventional glass polyalkenoate cement is not recommended.
3. The pre-treatment of gold palatal veneers by alumina blasting and oxidation prior to cementing with Panavia 21 appears to improve the chances of obtaining a dependable restoration. A larger number of restorations would be required to significantly statistically test this trend.

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Natural History of Treatment Outcomes for Teeth with Large Amalgam and Crown Restorations

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Clinical Relevance

After five and 10 years, teeth with large amalgam and crown restorations received less subsequent treatment and less major treatment than teeth with just a large amalgam restoration. Treatment outcome trees can be a useful tool for clinicians as a way of portraying treatment outcomes.

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SUMMARY

The natural history of posterior teeth treated with a four or more surface amalgam restoration (LA) or a large amalgam restoration and a full-coverage crown (LAC) were compared over five- and 10-year periods. Subsequent treatment information was used to construct Treatment Outcome Trees (TOT), which described treatment that the teeth received after placement of LA and LAC restorations. Data were collected for all treatments provided to patients who received a four or five surface LA in 1987 or 1988 at the University of Iowa, College of Dentistry (UICD). The probability that these teeth would receive subsequent treatment and the type of subsequent treatment were placed into a TOT.

In general, a higher percent of teeth with an LA received subsequent treatment and were more likely to receive major treatment (root canals, extractions, crowns) five years post placement than teeth with an LAC. Between five and 10 years, this trend continued, with the percentage of teeth with an LA receiving subsequent treatment increasing more (48% to 64%) than teeth with an LAC (12% to 22%). Regardless of the initial restoration type (LA/LAC), women were less

likely to receive subsequent treatment and major treatment compared to men. The use of a TOT was found to be an effective observational approach for evaluating the natural history of teeth with alternative restorative treatment.

INTRODUCTION

To understand how to provide the best clinical care and make educated treatment decisions, information about the outcomes of different procedures, especially routine procedures, is necessary. Initially, this information should describe the outcomes of these procedures as they are routinely used in common practice. The lack of information regarding the outcome of alternative treatments and procedures often leads to significant variation in the way dentists plan for and implement treatment for patients, even for routine procedures (Elderton & Nuttall, 1983; Nuttall & Elderton, 1983; Hazelkorn, 1985; Shugars & Bader, 1992; Bader, Shugars & Rozier, 1993; Rytömaa, Järvinen & Järvinen, 1979; Bader & Shugars, 1993; Merrett & Elderton, 1984; Bailit & Clive, 1981; Bailit & others, 1979; Diehr & Grembowski, 1990; Grembowski, Milgrom & Fiset, 1990a,b; Bader & Shugars, 1995; Shugars & others, 1997; Davies, 1984). Traditionally, variation in dental treatment has been accepted, acknowledging differences in dentists' skills and experience and patient factors and interests. Variation in dental treatment is currently driven by the lack of an evidence base for which procedures produce the best outcomes in a given situation. This allows for a practitioners' personal preferences based upon their clinical experience to drive the treatment planning process (Shugars & Bader, 1992; Bader & Shugars, 1995). An Institute of Medicine report on quality in health care concluded that there is a "significant cultural shift... away from unexamined reliance on professional judgment toward more structured support and accountability for such judgment" (Institute of Medicine, 2001). This can only occur when there is an adequate, accurate statistical base to support clinical decision-making. Better information on expected outcomes would help providers increase their certainty in making treatment decisions and reduce variation (Brook & Lohr, 1985).

Understanding the natural life history of a tooth following different treatments adds to the evidence base in dentistry. There is currently little information regarding the outcome of different types of restorative care for individual patients or teeth over a long period (Clarkson, Worthington & Davies, 2000). Cross-sectional studies at one point in time have examined the reasons for placement and replacement of restorations; however, teeth are not followed beyond restoration failure and, therefore, the natural history of teeth cannot be determined (Rytömaa & others, 1984; Letzel & others, 1989; Wilson, Burke & Mjör, 1997; Cheetham, Makinson & Dawson, 1991; Qvist, Qvist & Mjör, 1990; Burke & others, 1999; Mjör, 1997). There have been a few longitudinal studies on dental care provided to patients. However, these studies follow the number and types of restorations patients have as a whole, and do not follow specific teeth subsequent to a certain restorative procedure (Elderton & others, 1985; Elderton, 1983).

Some information is available on the long-term outcome of amalgam restorations and crowns, yet most of these studies evaluate *restoration* longevity or failure and do not follow subsequent treatment, if any (Robinson, 1971; Allan, 1977; Crabb, 1981; Bentley & Drake, 1986; Letzel & others, 1989; Osborne & Norman, 1990; Robbins & Summitt, 1988; Smales, 1991; Plasmans, Creugers & Mulder, 1998; Walton, Gardner & Agar, 1986; Leempoel & others, 1985; Hawthorne & Smales, 1997; Smales & Hawthorne, 1997; Martin & Bader, 1997).

In this study, the natural history of posterior teeth treated with a four or more surface amalgam restoration (LA) and posterior teeth treated with an LA and full-coverage crown (LAC) were followed and compared over five and 10 years. The subsequent treatment information was then used to construct a Treatment Outcome Tree (TOT), which compared the treatment received by teeth with LA and LAC restorations. The authors developed the concept of the TOT as a way to graphically depict the natural history of a tooth subsequent to a definite procedure, such as placement of the LA or LAC. The TOT provides assistance for clinical decision making by evaluating the long-term outcomes following various treatment pathways.

METHODS AND MATERIALS

Data for all treatments provided to patients who received an LA in 1987 or 1988 at the UICD were collected from the College's administrative database. The data included all procedures provided at the College from 1985 to 2001. The data for procedures in 1985 and 1986 were used to exclude teeth with root canals prior to placement of the LA. Only patients who were continuous utilizers of care (visited UICD at least once every two years) for five and 10 years post-LA were included in this study. If a patient had more than one LA placed in 1987 or 1988, one tooth was randomly selected to avoid multiple and related observations. Third molars and teeth that subsequently served as a bridge abutment were excluded from this study. This resulted in following 756 patients for five years and 518 patients for 10 years.

Additional information regarding the patient (birthdate and gender) and dental visits (dates of service, ADA procedure codes, tooth number, experience level of

provider-undergraduate, graduate or faculty) was also obtained from the administrative data. If a tooth with an LA received a crown within 365 days of placement of the LA, the tooth was classified as a crown (LAC), indicating that it was likely to have been treatment planned for a crown at the time the LA was placed. All remaining teeth were placed in the LA category.

The date of service was used to determine the timing of subsequent treatment after the LA or LAC was placed. These services were then placed in chronological order of occurrence. Subsequent treatment codes were categorized into the following groups: extraction, root canal therapy, crown, large amalgam (four or more surfaces), major restoration (three or four surfaces—not including four or more surface amalgam restorations), minor restoration (one or two surfaces) and no subsequent treatment.

To evaluate the outcomes of teeth with LAs and LACs, TOTs were constructed. The TOTs provided an observational approach for evaluating the treatment provided to LA and LAC teeth over five to 10 years. The TOT has a structure similar to a decision tree. However, it has treatment nodes instead of decision and chance nodes. Thus, the probabilities associated with each treatment node represent the procedures that actually occurred and not, as in a decision tree, the change in health state of the tooth. Branches of different subsequent treatment nodes extend from each treatment node and the percent of teeth associated with each branch. Treatment nodes and branches are added until all subsequent treatment is accounted for. Similar to decision trees, the percentages of all branches from an individual node must add up to 100%.

In this study, DATA 3.5 for healthcare (TreeAge Software Inc, Williamstown, MA, USA) was used to create the TOTs. The first treatment node contained two branches, representing teeth with LAs and LACs. The second treatment nodes contained branches representing the first treatment (either extraction, root canal therapy, crown, large amalgam, major restoration, minor restoration or no subsequent treatment) that the tooth received subsequent to placement of the LA or LAC restoration. This process was continued until all subsequent treatments were displayed. The TOT was then simplified or "pruned," prioritizing the branches based on the severity of the subsequent treatment and frequency at which each subsequent treatment occurred. Extraction was considered the most severe treatment, while minor restoration was con-

Table 1: *Patient and Tooth Variables Distributed by Restoration Type for Individuals Who Were Continuous Users of Care for Five Years (n=756)*

Variable	Levels	LA	LAC	*p*-value
		N (%)	N (%)	
		398 (53)	358 (48)	
Patient Specific Variables				
Age	20-34	48 (12)	20 (6)	*Wp*=0.031
	35-44	68 (17)	54 (15)	
	45-54	79 (20)	62 (17)	
	55-64	86 (22)	122 (34)	
	65-74	90 (23)	86 (24)	
	75+	25 (6)	14 (4)	
Gender	Male	189 (48)	152 (43)	*Cp*=0.166
	Female	208 (52)	205 (57)	
Average number of visits per year	0-3	29 (7)	36 (10)	*Wp*<0.000
	>3-5	109 (27)	125 (35)	
	>5-7	108 (27)	105 (29)	
	>7-9	77 (19)	53 (15)	
	>9-11	49 (12)	27 (8)	
	>11	26 (7)	12 (3)	
Average number of recall visits per year	0-1	192 (48)	143 (40)	*Wp*<0.000
	>1-2	168 (42)	164 (46)	
	>2	38 (10)	51 (14)	
Average number of preventive visits per year	0-1	143 (36)	101 (28)	*Wp*=0.000
	>1-2	188 (47)	179 (50)	
	>2	67 (17)	78 (22)	
Tooth Specific Information				
Number of Pins	0	163 (41)	117 (33)	*Wp*=0.001
	1-2	181 (46)	169 (47)	
	>2	54 (14)	72 (20)	
Tooth Type	Premolar	104 (26)	129 (36)	*Cp*=0.003
	Molar	294 (74)	229 (64)	
Tooth Arch	Maxillary	209 (53)	171 (48)	*Cp*=0.192
	Mandibular	189 (48)	187 (52)	
Experience Level of Provider	Undergraduate	248 (62)	233 (65)	*Cp*=0.653
	Graduate	41 (10)	31 (9)	
	Faculty	109 (27)	94 (26)	

Cp=*Chi-square p-value*

Wp=*Wilcoxon p-value, reported using variable continuously, not categorical as displayed.*

sidered the least severe. For example, if a tooth received a minor restoration and was subsequently extracted, it was classified as having been extracted. The probabilities of each branch occurring and the median, maximum and minimum amount of time for that subsequent treatment were added to the TOT. For each branch, time was defined as the amount of time between placement of the LA or LAC and the time when the tooth received the subsequent treatment specified by the branch.

Table 2: *Patient and Tooth Variables Distributed by Restoration Type for Individuals Who Were Continuous Users of Care for 10-years (n=518)*

Variable	Levels	LA	LAC	*p*-value
		N (%)	N (%)	
		255 (49)	263 (51)	
Patient Specific Variables				
Age	20–34	20 (8)	13 (5)	*Wp*=0.143
	35-44	43 (17)	38 (15)	
	45-54	56 (22)	46 (18)	
	55-64	65 (26)	94 (36)	
	65-74	59 (23)	63 (24)	
	75+	11 (4)	9 (3)	
Gender	Male	109 (43)	112 (43)	*Cp*=0.970
	Female	145 (57)	150 (57)	
Average number of visits per year	0-3	8 (3)	17 (7)	*Wp*=0.001
	>3-5	67 (26)	86 (33)	
	>5-7	74 (29)	84 (32)	
	>7-9	56 (22)	43 (16)	
	>9-11	31 (12)	22 (8)	
	>11	19 (8)	11 (4)	
Average number of recall visits per year	0-1	90 (35)	86 (33)	*Wp*=0.036
	>1-2	127 (50)	128 (49)	
	>2	38 (15)	49 (17)	
Average number of preventive visits per year	0-1	64 (25)	53 (20)	*Wp*=0.068
	>1-2	127 (50)	143 (54)	
	>2	64 (25)	67 (26)	
Tooth Specific Information				
Number of Pins	0	105 (41)	89 (34)	*Wp*=0.043
	1-2	110 (43)	124 (47)	
	>2	40 (16)	50 (19)	
Tooth Type	Premolar	64 (25)	88 (34)	*Cp*=0.037
	Molar	191 (75)	175 (67)	
Tooth Arch	Maxillary	129 (51)	134 (51)	*Cp*=0.934
	Mandibular	126 (49)	129 (49)	
Experience Level of Provider	Undergraduate	155 (61)	168 (64)	*Cp*=0.686
	Graduate	25 (10)	21 (8)	
	Faculty	75 (29)	74 (28)	

Cp=*Chi-square p-value*

Wp=*Wilcoxon p-value, reported using variable continuously, not categorical as displayed.*

To evaluate the potential differences in the populations that received LA and LAC, chi-square tests were performed with the categorical variables (gender, arch type and experience level of provider), while the Wilcoxon Rank Sum test was performed with the continuous variables (age, average number of visits, recall visits and preventive visits per year and the number of pins). The SAS (Gary, NC, USA) statistical software version 8.2 was used to conduct data management and analysis.

RESULTS

Demographic information for patients with LA and LAC teeth who continuously visited UICD for five (n=756) and 10 years (n=518) are reported in Tables 1 and 2. There were similar distributions of patients in the LA and LAC populations at both five and 10 years. At baseline, LAC restorations were more common among the older age groups (55-74). Patients with LA restorations had more visits per year but fewer recall or preventive visits compared to those with LAC. More female patients received both LA and LAC restorations at both time intervals.

Regarding tooth specific characteristics, a greater proportion of teeth in the LAC group were premolars. LAC teeth were also more likely to have pins placed at the time of the initial large amalgam. Restorations were relatively equally distributed between the maxillary and mandibular arch. A majority of the treatment for both types of procedures was performed by undergraduate dental students (approximately 63%).

The hierarchical TOTs at five and 10 years are depicted in Figures 1 and 2. At five years, 52% of the LA teeth received no further treatment compared to 88% of the teeth with LAC. For LA teeth, 6% were extracted and 7% received root canal therapy, while for the LAC teeth, only 1% were extracted and 5% received root canal therapy. LAC teeth had a minimal number of replacement crowns, while LA teeth were replaced with crowns 18% of the time. Additionally, LA teeth had a higher percent of non-crown restorations (16%) com-

pared to LAC teeth (3%). After five years, LAC teeth were replaced with a crown nearly 4% of the time, yet the median time until the crown was placed was less than two months (0.1 year). Many of these crowns may have been improperly coded as complete at the initial seating appointment, but due to problems, the crown was remade, subsequently seated and coded again as complete.

Between five and 10 years, 238 individuals were lost to follow-up, resulting in 518 individuals in the study population after 10 years. The percent of teeth with no further treatment declined for both LA and LAC teeth. This decline was more dramatic for LA teeth, dropping from 52% to 36%, whereas for LAC teeth, the reduction was from 88% to 78%. LA teeth had a larger percentage increase in all levels of subsequent treatment received except for the major restoration/large amalgam category. The two greatest percent increases in subsequent treatment for LA teeth were the crown and extraction categories. For LAC teeth only the percent of non-crown restorations and extractions increased. Both LA and LAC teeth had an equal increase in extractions by approximately six percentage points.

The initial TOTs contained many branches and were too large and complex to be displayed. Among the information that was reduced to create the final TOT, at five years, both the LA and LAC teeth had received a maximum of four treatment procedures, with the LA teeth having a higher percent of treatment and a greater variety of treatment patterns. At 10 years, both the LA and LAC teeth had received up to six treatment procedures. Although the LA teeth still had a higher percent of treatment, the divide was smaller than at five years.

Additional details for the TOTs are shown in Figure 3 for the 20 teeth that received root canal therapy following the initial LA. This branch was a portion of the TOT at 10 years prior to pruning and is an example of the type of treatment branches pruned because the number of teeth affected was so small. After the LA teeth received root canal therapy, they received a wide variety of treatments. Yet, regardless of the different treatment sequences after root canal therapy, 75% of the teeth received a subsequent crown.

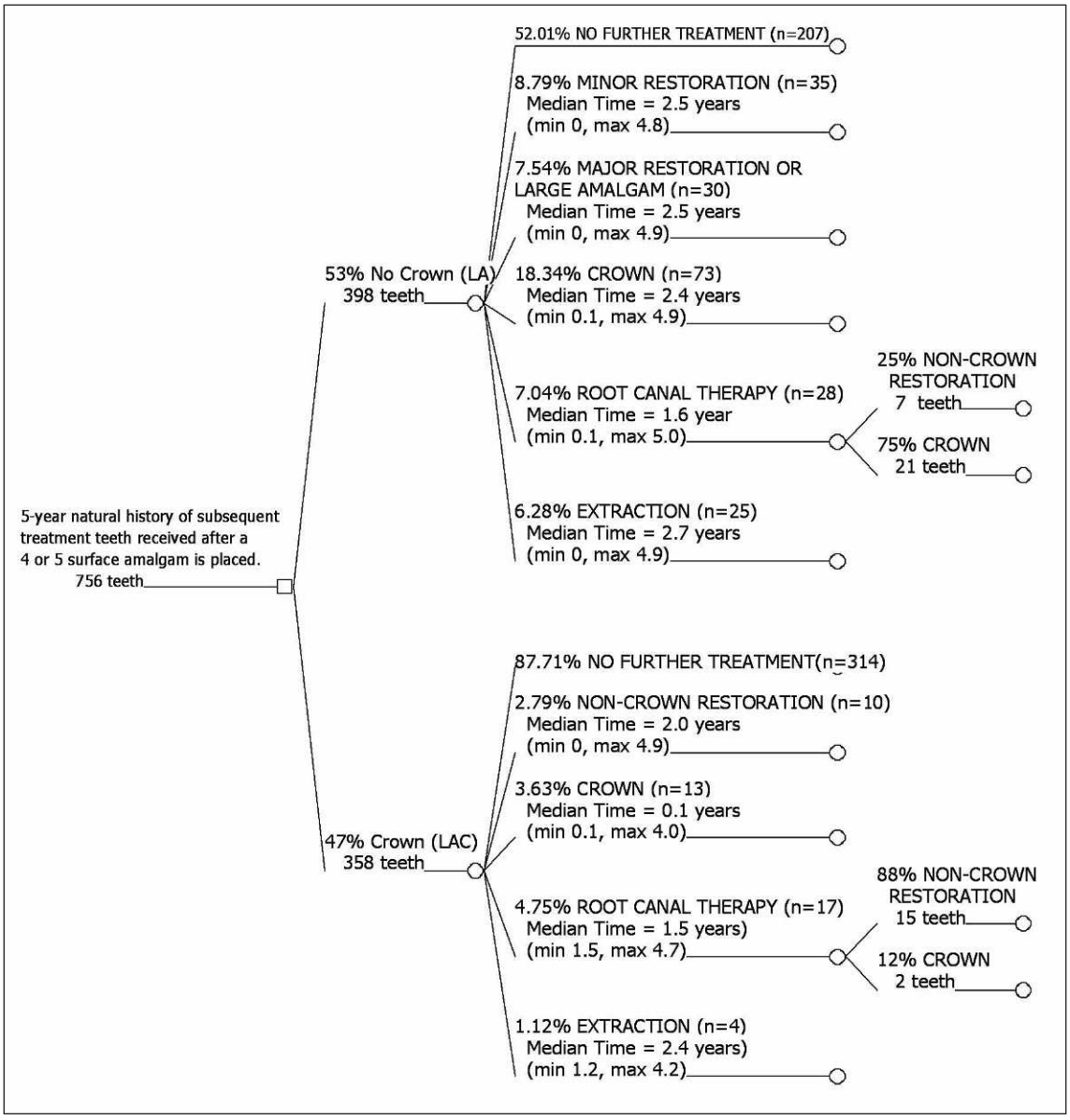

Figure 1. *Five-year hierarchical "Treatment Outcome Tree" for LAs and LACs (n=756).*

Figure 4 displays the subsequent treatment for men and women over 10 years. For the LA teeth, women had a higher percentage of receiving no further treatment, while men had a higher percentage of receiving a major restoration/large amalgam or root canal therapy. Nearly the same percentage of men and women had extractions. For LAC teeth, the patterns were similar, except that the men had a higher percentage of extractions.

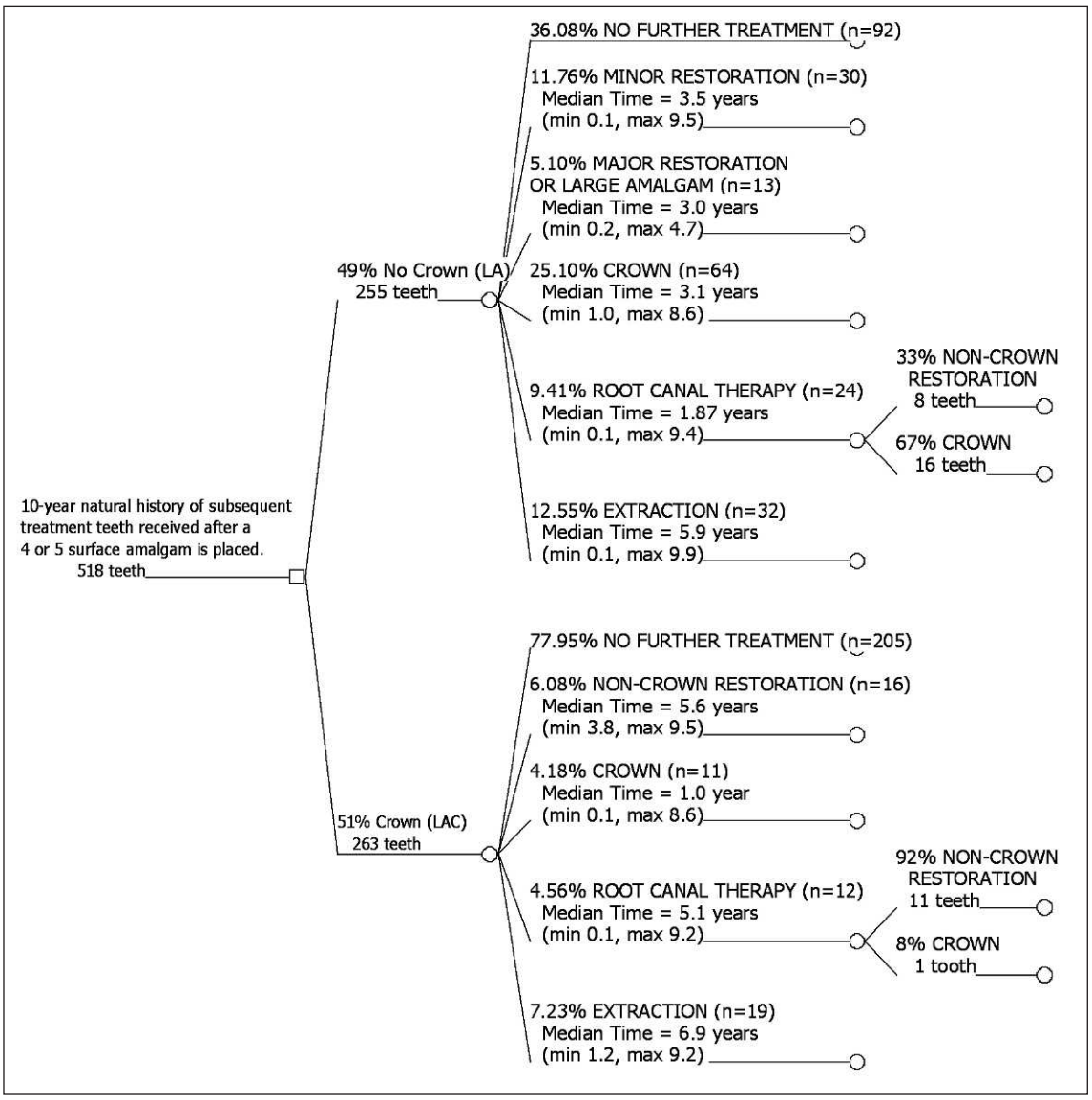

Figure 2. *Ten-year hierarchical "Treatment Outcome Tree" for LAs and LACs (n=518).*

DISCUSSION

In general, a higher percentage of LA teeth received subsequent treatment and the LA teeth were more likely to receive major procedures (root canals, extractions or crowns) at five years than the LAC teeth. Between five and 10 years, this trend continued, with the percentage of LA teeth receiving subsequent treatment increasing more (48% to 64%) than the LAC teeth (12% to 22%). Regardless of the initial restoration type (LA/LAC), women were less likely to require further treatment or major treatment compared to men.

There are several methodologic approaches used in this study that allow these results to add to the knowledge of the long-term outcomes of teeth restored with LA and LACs. The use of TOTs to follow the natural history of teeth with large restorative procedures is a unique approach for evaluating outcomes. Another different aspect of this study is that it evaluated the treatment provided to a tooth over a five- and 10-year period; it did not just follow the life of the restoration. Also, the definition of restoration outcome (receipt of subsequent treatment) is more comprehensive than the definition used in many studies (failure of the restoration).

Martin and Bader's study of the five-year outcomes of large amalgam and crown restorations found similar results (Martin & Bader, 1997). They reported a higher percentage of large amalgams as not having further treatment (62% for four-surface amalgams and 55% for five-surface amalgams) and a lower percentage of crowns not having further treatment (82% for porcelain crowns and 80% of gold crowns). Most of the extraction and endodontic therapy rates found in this research were comparable to Martin and Bader's, except that the rate of endodontic therapy for LAC teeth was much lower in this study (1%) than that found by Martin and Bader (6% for porcelain crowns and 7% for gold

crowns). Additionally, teeth with LA were much more likely to receive a subsequent crown in this study (18%) than in Martin and Bader's study (7% for four-surface amalgams and 10% for five-surface amalgams).

This study introduces the concept of a TOT as an observational method, comparing the outcomes and treatment states following alternative procedures. Although the method is similar to decision trees, the concept and method for creating a TOT is new. To describe the restorative history of teeth, the TOT provides both the percentage of subsequent treatment that occurred and the minimum, median and maximum time till the subsequent treatment occurred. It provides practitioners with a decision-making graph to evaluate alternative treatment methods.

Although teeth with crowns received less subsequent treatment compared to those with large amalgams, this does not mean that teeth with large amalgams received inappropriate care. For example, it may be that the teeth judged to be at highest risk for a poor outcome (little remaining tooth structure, possible pulpal involvement) at the time the LA was placed were less likely to receive a crown. This may be due to uncertainty of the long-term health of the tooth, potentially combined with financial considerations, making patients and/or clinicians less likely to choose the more expensive treatment alternative for teeth (LAC). Furthermore, it could be that patients who chose LAs over LACs had a greater susceptibility for future caries or other adverse oral health outcomes.

This study investigated post-restoration treatment in continuous patients who sought treatment in a dental school over a five- and 10-year period. Therefore, the results should be generalized carefully beyond this population. Dental school patients, as a group, may be fundamentally different from patients seeking care in a private practice setting. Dental school patients may be less likely to have insurance and therefore may seek lower fees. Additionally, most patients at a dental school receive treatment from many practitioners over a five- to 10-year period. This allows for significant variation in recommended and received treatment and may have an effect on outcome. Variation in treatment options may also be a result of treatment directed towards educational requirements. For example, there may be a substantial amount of time between appointments due to the relative inefficiency of students and their academic schedule, which may also have an effect on outcomes. Patients who sought care continuously (visited UICD at least once every two years) were selected to obtain a sample of patients who were likely to only receive treatment at UICD. This was done in an effort to capture all treatment provided for the targeted tooth. Information on non-continuous users of care is not available. Therefore, it is unknown whether outcomes for non-continuous users would differ from continuous users.

A future study needs to include information from non-continuous care seekers using an analytic approach called survival analysis. This will provide a stronger analytic model and glean information for patients who only sought care for a portion of the five- or 10-year period. More information about the cost implications of the two treatment options should also be factored into a future study. Although the long-term outcome may be better for a crowned tooth, the substantial cost difference may indicate that it is more cost effective to place

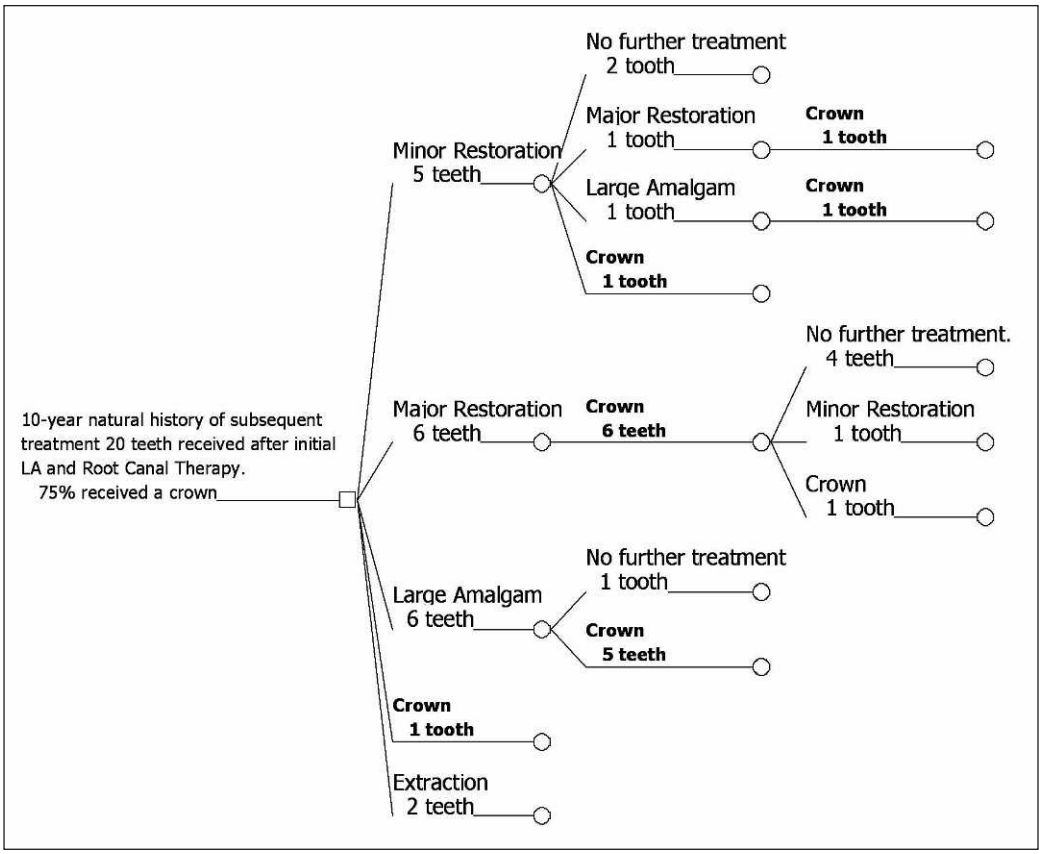

Figure 3. *Ten-year "Treatment Outcome Tree" for teeth that received a LA and subsequent root canal therapy (n=20).*

and replace a large amalgam over a long period of time.

CONCLUSIONS

Teeth that received an initial large amalgam restoration with a subsequent crown restoration had a reduced need for subsequent treatment, and the treatment that was provided was less severe than for teeth that received only a large amalgam restoration. The use of a TOTs was found to be an effective observational approach to evaluate the natural history of teeth with alternative restorative treatments.

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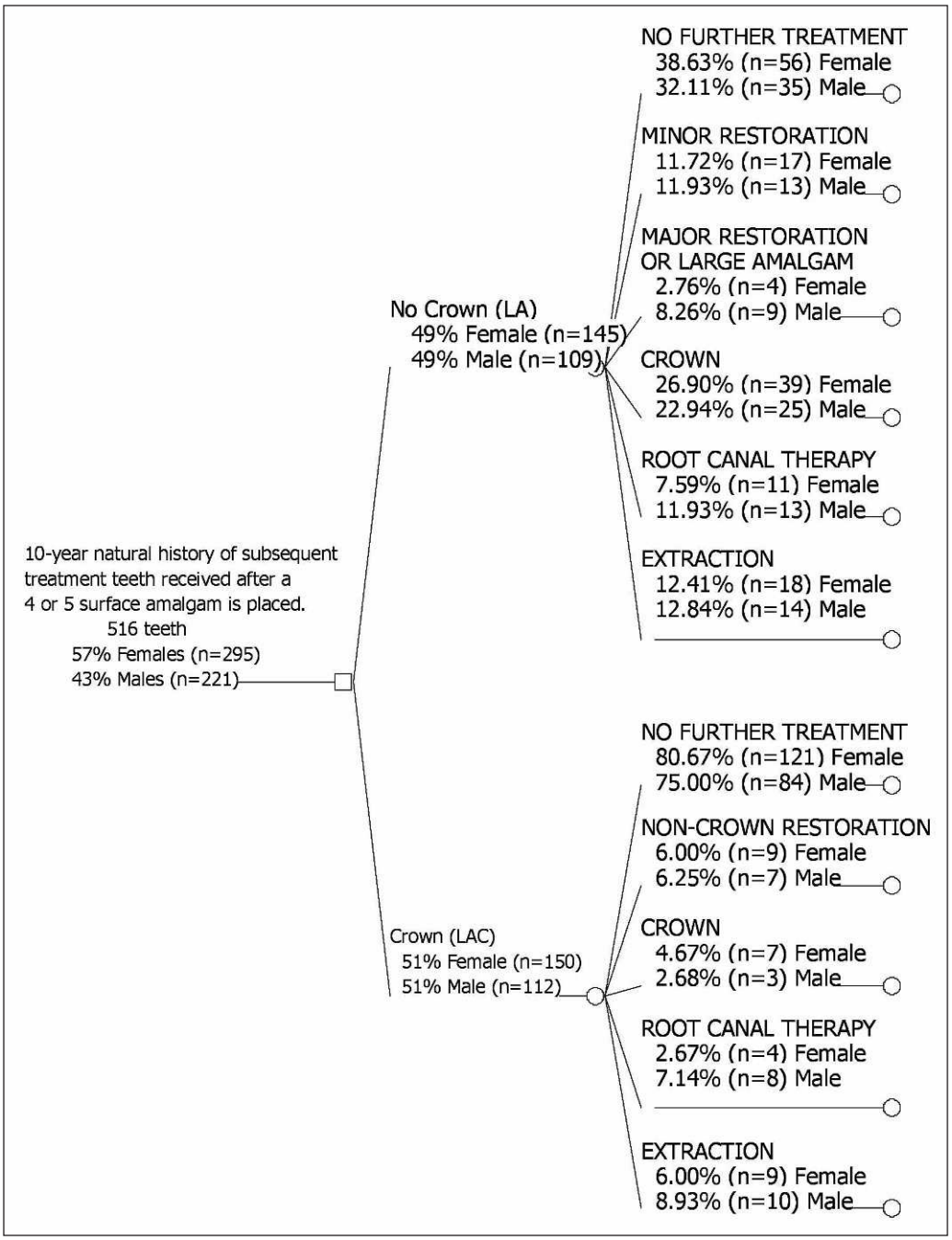

Figure 4. *Ten-year hierarchical "Treatment Outcome Tree" for LAs and LACs distributed by gender (n=516).*

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A Six-month Study of Two Self-applied Tooth Whitening Products Containing Carbamide Peroxide

PA Brunton • R Ellwood • R Davies

Clinical Relevance

Self-applied tooth whitening systems containing either 18% or 16.4% carbamide peroxide effectively whiten teeth when applied twice daily for two weeks, with the majority of the whitening benefit sustained for six months.

SUMMARY

Bleaching offers a non-interventive way of improving the appearance of sound, yet discolored anterior teeth. Until recently, the whitening agent was applied using a tray, but now other methods of delivering whitening agents, such as those using brush applicators, are available. This study investigated the tooth whitening efficacy of two novel, self-applied tooth whitening systems containing either 18% (Group 1) or 16.4% (Group 2) carbamide peroxide. Ninety-five subjects, ranging in age from 18 to 70 with anterior teeth A3 or darker, were recruited and randomly allocated to a group. The subjects were instructed to apply the formulation to all maxillary anterior teeth after brushing in the morning and evening. At baseline, two weeks and six months the upper six anterior teeth of the subjects were measured using the Vita shade guide tab system. In addition, the gingival health of the labial surfaces of the upper six anterior teeth was assessed using the Loee and Silness Gingival index (Loee & Silness, 1963) at baseline and at two weeks. The mean (SD) reduction in shade guide scores was 4.1 (2.4) shade guide tabs for subjects in Group 1, compared to 3.7 (2.6) shades for those in Group 2. This difference was not statistically significant (p=0.5). During the course of study, the gingivitis scores reduced from a mean (SD) of 0.91 (0.62) at baseline to 0.44 (0.55) at final examination (48% reduction). At the six-month recall, the mean (SD) reduction in shade guide scores was 2.3 (2.7) shade guide tabs for subjects in Group 1, compared to 2.5 (2.5) shades for those in Group 2. The different concentrations tested were found to be equally effective in improving the whiteness of upper anterior teeth by approximately four shades over a two-week period and the majority of the whitening benefit (c.60%) was sustained at six-month recall.

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INTRODUCTION

Prior to the availability of tray based vital bleaching, discolored teeth were often treated with porcelain laminate veneers or resin (dentin) bonded and porcelain fused to metal crowns. The introduction of tray-based vital bleaching, using various concentrations of carbamide peroxide, has proved to be a simple, non-invasive and effective treatment for sound, yet discolored anterior and posterior teeth (Haywood, 1997) which has proven to be very popular with practitioners (Haywood & Heymann, 1989; Christensen, 1997). Carbamide peroxide (10%) has been shown to produce tooth whitening and reports suggest that some changes may be stable for periods of up to seven years (Haywood & Leonard, 1998; Leonard, 1998). Despite some fears about the safety of this technique, no side effects other than transient sensitivity have been reported (Li, 1997). The availability of these materials to practitioners in the United Kingdom and across Europe has been sporadic, not for reasons of safety, but due to the legal classification of these products (BDA Guidelines, 1999). More recently, trayless, self-applied tooth whitening systems have been developed (Gerlach & Zhou, 2001; Nathoo & others, 2002).

This study investigated the tooth-whitening efficacy of two self-applied tooth whitening systems containing either 18% or 16.4% carbamide peroxide after two weeks treatment and six months after treatment. The study also investigated whether the whitening systems affected the gingival tissues.

METHODS AND MATERIALS

This study was a double blind, randomized, controlled, parallel group clinical trial. After receiving Ethical Committee (Institutional Review Board) approval, potential participants were screened. Those who satisfied the inclusion/exclusion criteria, had a minimum Vita shade of A3 on one or more upper incisors and signed a consent form were recruited into the study. The inclusion/exclusion criteria were as follows:

Inclusion Criteria

1. Male and female subjects ranging in age from 18 to 70 years inclusive.
2. Good general and oral health.
3. All maxillary anterior teeth present.
4. Availability for the six month duration of the study.
5. A minimum Vita Shade of A3 on one or more upper central incisors.

Exclusion Criteria

1. Presence of orthodontic appliances or any anterior tooth with a prosthetic crown or veneer.
2. Tumors or significant pathology of the soft or hard tissues of the oral cavity.
3. Moderate or advanced periodontal disease, rampant caries or any condition that the dental examiner considered exclusionary from the study.
4. Five or more carious lesions requiring immediate care.
5. Participation in any other study within 30 days preceding the clinical study.
6. Pregnant or lactating females.
7. A history of allergies to tooth whitening products, personal care consumer products or their ingredients.
8. Restorations on the teeth to be scored, which might interfere with scoring procedures.

At baseline and subsequent examinations, one operator (PB) recorded the shade value of the labial surfaces of the six upper anterior teeth using a Vita Shade Guide. The shade guide was arranged with the 16 shade tabs in order from B1 (1) to C4 (16). The subject's mean value score was computed by adding the six value scores together and dividing by the number of teeth examined.

The subjects were randomly assigned to one of two groups as follows:

1. Colgate Simply White (Colgate Palmolive, New York, USA) containing 18% carbamide peroxide.
2. Colgate Simply White (Colgate Palmolive) containing 16.4% carbamide peroxide.

The subjects had a soft tissue examination at both the baseline and subsequent examinations, and the gingival health of the labial surfaces of the upper six anterior teeth was assessed using the Loee and Silness Gingival index (Loee & Silness, 1963).

Subjects received a soft-bristled adult toothbrush for home use and two 50-ml tubes of Colgate Cavity Protection (Colgate Palmolive) non-whitening toothpaste. They were instructed to brush their teeth for one minute at least twice a day (morning and evening) and apply their whitening treatment as follows:

- Dry the front surface of their upper six anterior teeth with a tissue or cotton ball.
- Apply a layer of the whitening treatment over the front surface of the teeth, refilling the brush between applications.
- Remain with their mouth open for 30 seconds while the gel dried.
- Refrain from eating or drinking for 30 minutes.
- Apply the gel after brushing in the morning and before going to bed at night for the duration of the study (two weeks).

- Subjects were instructed to refrain from using any other oral hygiene products, but there were no restrictions regarding diet and smoking habits during the course of the study.

At the end of the study, subjects were asked whether they had experienced any sensitivity to their teeth or gingivae. Their responses were graded from 0 (none) to 5 (severe).

RESULTS

A total of 95 subjects were recruited into the study. Two subjects failed to complete the two-week study (one from each group) for reasons unrelated to the study.

Tooth Color

A total of 93 subjects, 46 in Group 1 (18% carbamide peroxide) and 47 in Group 2 (16.4% carbamide peroxide), completed the study. Those subjects in Group 1 had a mean reduction in shade guide scores of 4.1 (SD 2.4) compared to 3.7 (2.6) for Group 2 (Table 1). This difference was not statistically significant (p=0.5). The 44 subjects with a baseline Vita score of A3–A3.5 improved by four shades; the 49 subjects with baseline values of B4–C4 improved by 3.8 shades.

Gingivitis Scores

The mean gingivitis score for subjects at baseline was 0.91 (SD 0.62) and 0.44 (SD 0.55) after two weeks. This difference was statistically significant (p<0.001).

Tooth and Gingival Sensitivity

Of the 93 subjects completing the study, 79 (85%) reported little or no tooth sensitivity (scores 0 and 1), 12 (13%) reported moderate tooth sensitivity (scores 2 and 3) and 2 (12%) reported moderate-severe (score 4) sensitivity (Table 2). Seventy-nine (85%) subjects reported little or no gingival sensitivity and 14 (15%) moderate sensitivity (Table 3). Both gingival and tooth sensitivity were reported to be transient and caused none of the subjects to withdraw from the study.

Six-months Results

At six months, 51 subjects, 28 from Group 1 and 23 from Group 2, were available for examination. Of the 42 subjects not included in the six-month recall, 20 were involved in other non-related studies and were ineligible, 5 were on vacation, 14 did not respond or did not want to take part in the study and 3 failed to keep their appointments. The subjects in Group 1 had a sustained mean reduction in shade guide scores of 2.3 (SD 2.7) compared to 2.5 (2.5) for Group 2 (Table 4). This difference was not statistically significant (p=0.5).

Table 1: *Baseline, Two Weeks and Difference in Mean Value Scores for Subjects*

	Shade			
Group	**N**	**Baseline (SD)**	**Final (SD)**	**Diff (SD)**
1	46	12.3 (2.4)	8.2 (3.4)	4.1 (2.4)
2	47	12.6 (2.4)	8.9 (4.0)	3.7 (2.6)

Table 2: *Tooth Sensitivity Scores*

Group	Score (#, %)				
	0	1	2	3	4
1 n=46	32, 70	7, 15	6, 13	1, 2	0, 0
2 n=47	32, 68	8, 17	5, 11	0, 0	2, 4
Total	64, 69	15, 16	11, 12	1, 1	2, 2

Table 3: *Gingival Sensitivity Scores*

Group	Score (#, %)			
	0	1	2	3
1 n=46	32, 69	9, 20	4, 9	1, 2
2 n=47	31, 66	7, 15	7, 15	2, 4
Total	63, 68	16, 17	11, 12	3, 3

DISCUSSION

Both formulations tested in this study produced similar increases in whiteness of the upper anterior teeth. Although there was a reduced whitening effect for all subjects in Group 2, this was to be expected, given the reduced concentration of carbamide peroxide. However, the difference in whitening between the two groups was neither statistically nor clinically different. Treatments of the type described might be expected to have a reduced benefit in terms of the longevity of the whitening effect produced, but this did not appear to be the case.

Both formulations produced a four-shade improvement in whiteness on the Vita shade guide. Typically, a two-week treatment with a tray-based system using 10% carbamide peroxide will achieve about a seven-shade increase in value or whiteness. While there will still be a place for tray-based tooth whitening systems, a self-applied system of the type described could be used to top up tray-based or in-surgery treatments as an interim treatment or alternatively provide a low cost option for those who want a modest improvement in the whiteness of their teeth.

Table 4: *Baseline, Two Weeks, Six Months, Final and Difference in Mean Value Scores for Subjects Available for Examination After Six Months*

Group	Baseline	2 Weeks	6 Months	Baseline-2 Weeks	Baseline-6 Months	2 Weeks-6 Months
1 n=23	12.4 (2.4)	8.7 (3.5)	10.2 (2.6)	3.8 (2.4)	2.3 (2.7)	-1.5 (3.3)
2 n=28	12.4 (2.3)	8.2 (4.0)	9.9 (3.1)	4.2 (2.5)	2.5 (2.5)	-1.7 (2.7)

The whitening treatments described in this study are applied directly to the teeth, with gel being inadvertently applied to the gingival tissues. This did not appear to irritate the gingival tissues and, indeed, an improvement in gingival health was noted. This is likely to be due to the topical effects of the hydrogen peroxide produced by the whitening agent, coupled with improved oral hygiene and brushing during the course of the study. This is not surprising since hydrogen peroxide has been used extensively for the treatment of various oral inflammatory lesions with reduced inflammation noted (Marshall, Cancro & Fischman, 1995).

A significant complaint of patients who have tray-based whitening treatments is transient sensitivity, which can affect patient compliance with the treatment. Sensitivity rates reported for tray-based systems have been as high as 52% (Haywood & others, 1994). An important advantage of the topically self-applied whitening system described was the reduced levels of sensitivity (Tables 2 and 3). Therefore, practitioners should consider recommending a self-applied tooth whitening treatment when sensitivity with a tray-based system is problematic. This might be appropriate for patients with recession or excessive non-carious tooth tissue loss.

In this study, the agent was applied twice a day for a two-week period. It is suggested, given the improvement in whitening, that increased frequency of application and extended treatment times might produce greater improvement. However, further research is needed to investigate these variables.

CONCLUSIONS

Within the limitations of the study, the following conclusions can be drawn for both concentrations of the system tested:

- Both products effectively whitened teeth with a treatment time of two-weeks.
- The different concentrations tested were equally effective in improving whiteness.
- The whitening systems tested produced little tooth or gingival sensitivity.
- Some whitening benefit is sustained for at least six months after cessation of treatment.

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Laboratory Research

Curing Units' Ability to Cure Restorative Composites and Dual-cured Composite Cements Under Composite Overlay

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Clinical Relevance

When deep cavities need to be restored with a composite inlay, the composite inlay can be seated using restorative composites as luting materials if the depth of the deep cavity is reduced to 1.5 mm by placing base materials in the cavity. In this instance, the curing efficiency of the restorative composites is better than the dual-cured resin cements and the PAC system can most effectively cure both restoration types.

SUMMARY

This study compared the efficacy of using conventional low-power density QTH (LQTH) units, high-power density QTH (HQTH) units, argon (Ar) laser and Plasma arc curing (PAC) units for curing dual-cured resin cements and restorative resin composites under a pre-cured resin composite overlay.

The microhardness of the two types of restorative resins (Z100 and Tetric Ceram) and a dual-cured resin cement (Variolink II) were measured after they were light cured for 60 seconds in a 2 mm Teflon mold. The recorded microhardness was determined to be the optimum microhardness (OM).

Either one of the two types of restorative resins (Z100, Tetric Ceram) or the dual cured resin cement (Variolink II) were placed under a 1.5-mm thick and 8 mm diameter pre-cured Targis (Vivadent/Ivoclar AG, Schaan, Liechtenstein) overlay. The specimens that were prepared for each material were divided into four groups depending upon the curing units used (HQTH, PAC, Laser or LQTH) and were further subdivided into subgroups according to light curing time. The curing times used were 30, 60, 90 and 120 seconds for HQTH; 12, 24, 36 and 48 seconds for the PAC unit; 15, 30, 45 and 60 for the Laser and 60, 120 or 180 seconds for the LQTH unit. Fifteen specimens were assigned to each sub-

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group. The microhardness of the upper and and lower composite surfaces under the Targis overlay were measured using an Optidur Vickers hardness-measuring instrument (Göttfert Feinwerktechnik GmbH, Buchen, Germany). In each material, for each group, a three-way ANOVA with Tukey was used at the 0.05 level of significance to compare the microhardnesses of the upper and lower composite surfaces and the previously measured OM of the material. From the OM of each material, 80% OM was calculated and the time required for the microhardness of the upper and lower surface of the specimen to reach 100% and 80% of OM was determined.

In Z100 and Tetric Ceram, when the composites were light cured for 120 seconds using the HQTH lamp, microhardnesses of the upper and lower surfaces reached OM. When they were cured with the PAC unit, only 48 seconds was needed for the upper and lower surfaces to reach OM. When they were cured using the laser, the lower surface did not reach OM in any of the groups. When the specimens were cured using the LQTH lamp, 180 seconds of curing was needed for Z100 to reach OM, whereas Tetric Ceram did not reach OM. In Z100, 60, 12, 30 and 60 seconds were needed in HQTH, PAC, Laser and LQTH, respectively, for the specimens to reach 80% OM. Tetric Ceram was needed 60, 24, 45 and 180 seconds to reach 80% OM.

In the Variolink II specimen, microhardness of the upper and lower surfaces did not reach OM even though they were light cured with the HQTH lamp for 120 seconds. When they were cured with the PAC unit, 48 seconds was insufficient for them to reach OM. When they were cured with laser for 45 and 60 seconds, microhardness reached OM on the upper surface but not on the lower surface. However, when they were cured using the LQTH lamp, microhardness did not reach OM on the upper and lower surfaces even though the curing time was extended to three minutes. In Variolink II, 120, 36, 45 and >180 seconds were needed in HQTH, PAC, Laser and LQTH, respectively, for the specimens to reach 80% OM.

In conclusion, the PAC system is the most effective curing system to cure the restorative composite and dual cured resin cement under the 1.5 mm Targis overlay, followed by the laser, HQTH and LQTH units. In addition, the restorative composites cured more efficiently than the dual-cured resin cements.

INTRODUCTION

Resin cements have been used extensively in restorative dentistry for a variety of indirect applications. Dual cured-resin cements, where the peroxide/amine components are included in the light-cured system, are widely used. Theoretically, the benefit of such a product is that a portion of the cement that had initially received an insufficient intensity of light to initiate adequate polymerization would be polymerized after light exposure by a delayed chemical reaction where free radicals are formed. However, the chemical component of the cure has always been lower than when the specimen was exposed to any light condition, and there is no evidence that substantial chemically induced polymerization of a dual-cure resin cement occurring after the light exposure was completed (Breeding, Dixon & Caughman, 1991; Rueggeberg & Caughman, 1993).

Besek and others (1995) reported that the restorative composite could also be used as a luting material even though dual-cured resin cements have been widely used as a luting material for composite and ceramic inlays and onlays. When a porcelain inlay made of the Vita MK II system and machined using the Cerec system was cemented in the MOD cavity, the use of restorative resin as a luting cement showed better results than when the dual cured resin cement was used (Besek & others, 1995). This technique has been widely used in luting Cerec system.

The light from the curing units is attenuated through the ceramic or composite overlay. Therefore, a more powerful curing light may better cure dual-cure resin cement and restorative composites that are used as luting materials. This would be more important in composite overlays, because they have a lower translucency than ceramics. When the composites were light cured through the pre-cured composite overlay with a conventional quartz tungsten halogen (QTH) visible light curing unit and a power density of 400-500 mW/cm^2, they were not cured adequately due to the lower translucency of the pre-cured composite overlay (Rueggeberg & Craig, 1988).

Ar lasers have been used to polymerize the resin composite. It was reported that the composite polymerized for 10 seconds with an Ar laser exhibited superior physical properties compared to the composite polymerized for 40 seconds with a conventional lower power density QTH (LQTH) unit (Kelsey & others, 1989). The laser was recommended in polymerizing the composite luting cement under an indirect composite restoration (Michael, 1998).

Recently, a high power density QTH unit (HQTH) with power ranging from 600 to 900 mW/cm^2 has been introduced. In addition, a new type of light curing system, Plasma arc curing (PAC), has also been introduced. The PAC system uses a high-frequency electrical field to generate its plasma energy. A significant amount of energy released during this process is used to cure the photosensitive composites. Compared with the LQTH

unit, the PAC unit emits light with a higher power density (1370 mW/cm^2). The wavelength of the emitted light of the PAC is approximately 470nm, whereas, it is somewhere between 400-520nm in the QTH unit.

As the Laser, HQTH or PAC unit polymerizes the light curing composites more effectively than the LQTH system, they may have advantages over the LQTH system in being able to cure dual-cured resin cements or restorative composites under a composite overlay. This study compared the efficacy of the LQTH system, HQTH unit, Ar laser and the PAC unit in curing dual-cured resin cement and restorative resin composites under a pre-cured resin composite overlay.

METHODS AND MATERIALS

a) Determination of Optimum Microhardness (OM)

Two restorative composites and a dual-cured resin cement were used (Table 1). They were placed in a Teflon mold with a 2-mm thickness and an 8-mm diameter with a cover glass. The light curing tip of the QTH visible light curing unit (Optilux 400, Demetron/Kerr, CT, USA), with a power density of 450mW/cm^2, was positioned on the glass surface and the composites were light cured for 60 seconds. Fifteen specimens were created with each material. When the curing was finished, the specimen was removed from the mold and cover glass. The composite surface that was closer to the curing tip was marked. The specimens were then stored in dark, dry conditions at 37°C for seven days. The microhardness of the upper and lower composite surfaces were measured with an Optidur Vickers hardness measuring instrument (Göttfert Feinwerktechnik GmbH). The microhardness between the upper and lower surfaces was compared with a paired *t*-test at 0.05 level of significance.

The microhardness of the upper surface of each material obtained using this process was determined to be the optimum microhardness (OM).

b) Determination of Minimum Polymerization Time (MPT)

The minimum curing time to obtain the OM of 2-mm thick Tetric Ceram, Z100 and Vario-link II on both the upper and lower surfaces was defined as the minimum polymerization time (MPT) for each curing system.

To determine the MPT of PAC, HQTH and the laser system (Table 2), Tetric Ceram, Z100 and Variolink II were placed in a 2-mm thick, 8-mm diameter Teflon mold that was then covered with a cover glass. The power density of PAC and HQTH was measured as 1360 mW/cm^2 and 900mW/cm^2, respectively, by the

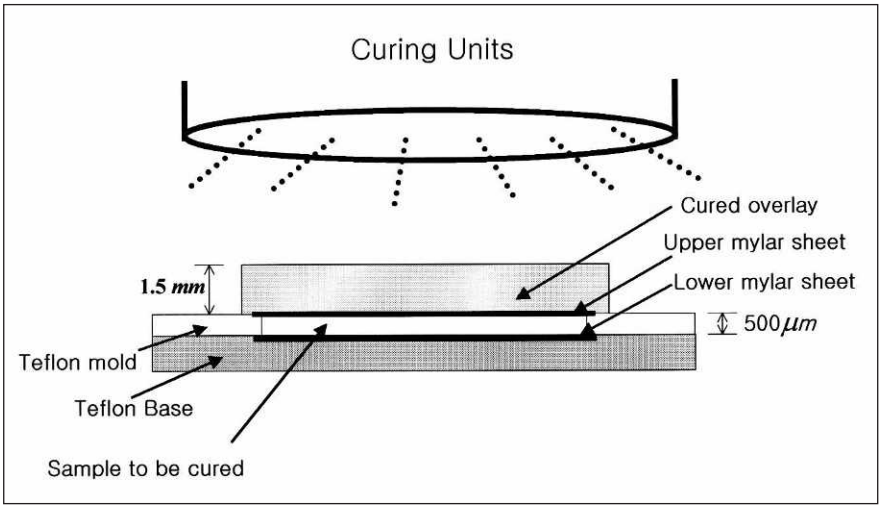


Figure 1. *Diagram of the specimen preparation.*

Table 1: *Materials Used in Study*

Materials	Manufacturer	Lot #
Z100	3M ESPE, St Paul, MN, USA A2 shade	20010310
Tetric Ceram	Vivadent, Schaan, Liechtenstein A2 shade	C46776
Variolink II	Vivadent, Schaan, Liechtenstein High viscosity	D14172, D205

Table 2: *Curing Units Which Were Used to Cure Dual-cure Resin Cement and Restorative Resin Composites Under the Pre-cured Composite Overlay*

System	Products	Manufacturer
HQTH	Coltolux 75	Coltene/Whaledent Inc, Mahwah, NJ, USA
LQTH	Optilux 400	Demetron/Kerr, Danbury, CT, USA
PAC	Apollo 95E	Dental/Medical Diagnostic System Inc, Westlake Village, CA, USA
Laser	HGM Blue/Green Dental 200 Laser system	HGM Inc, Salt Lake City, UT, USA

HQTH: High power density quartz tungsten halogen unit
LQTH: Low power density quartz tungsten halogen unit
PAC: Plasma arc curing unit

Coltolux light meter (Coltene/Whaledent Inc, Mahwah, NJ, USA). According to the manufacturer, the power output of the laser was 230 mW. The composites were light cured for 10, 20 or 30 seconds for HQTH, 3, 6, 12 or 15 seconds for PAC and 10, 15, 30 or 60 seconds for the Ar laser. The specimens were then stored in dark, dry conditions at 37°C for seven days. The microhardness of the upper and lower composite surfaces were measured with a Vickers hardness-measuring instrument (Göttfert Feinwerktechnik GmbH, Germany). The microhardness of the composites with different curing times were compared using a one-way ANOVA with Tukey. The microhardness of the upper and lower surface for each material was compared using a paired *t*-test at 0.05 level of significance.

c) Microhardness Measurement of Restorative Resins and Resin Cement Under Composite Overlay

The two types of restorative resin (Z100, Tetric Ceram) or dual cured resin cement (Variolink II) (Table 1) were placed into a 5-mm diameter, 0.5-mm thick Teflon mold lined with a mylar sheet and a Teflon base. A mylar strip was then placed on the upper composite surface. A 1.5-mm thick, 8-mm diameter Targis (Ivoclar/Vivadent AG) overlay, previously cured with the QTH unit (Optilux 500, Demetron/Kerr, Danbury, CT, USA) and Targis Power (Ivoclar/Vivadent AG, Schaan, Liechtenstein) was placed over the mylar sheet and Teflon mold. The tip of the light curing unit was placed 2-mm above the Targis overlay surface with special jig (Figure 1). The specimens were then divided into four groups (HQTH, PAC, Laser or LQTH) according to the curing units used for composite polymerization (Table 2). These groups were further subdivided into subgroups according to the light curing time. For HQTH, PAC and Laser, the curing time was MPT, 2xMPT, 3xMPT and 4xMPT. For the LQTH unit, the curing time was 60, 120 and 180 seconds. Fifteen specimens were assigned to each subgroup and 225 specimens were used for this study. The specimens were then light cured and removed from the Teflon mold and mylar strip. The upper surface of the specimen, which was closer to the curing tip, was marked. The specimens were then stored in dark, dry conditions at 37°C for seven days. The microhardness of the upper and lower composite surfaces were measured with an Optidur Vickers hardness-measuring instrument (Guttfert Feinwerktechnik GmbH). In each material, for each group, three-way ANOVA with Tukey was used at 0.05 level of significance to compare the microhardness of the upper and lower composite surfaces and previously measured OM of the material.

Table 3: *Microhardness Measurement of 2-mm Thick Composite Specimens on the Upper and Lower Surface to Determine Optimum Microhardness (OM). The Microhardness of the Upper Surface of Each Material Was Determined to be OM*

	Z100	Tetric Ceram	Variolink II
Upper surface	101.4 (1.8)	54.4 (2.1)	55.3 (2.1)
Lower surface	101.7 (1.8)	53.3 (1.3)	55.2 (1.4)

The figures in the parenthesis are the standard deviations. The vertical lines indicate the same microhardness at the p=0.05 level.

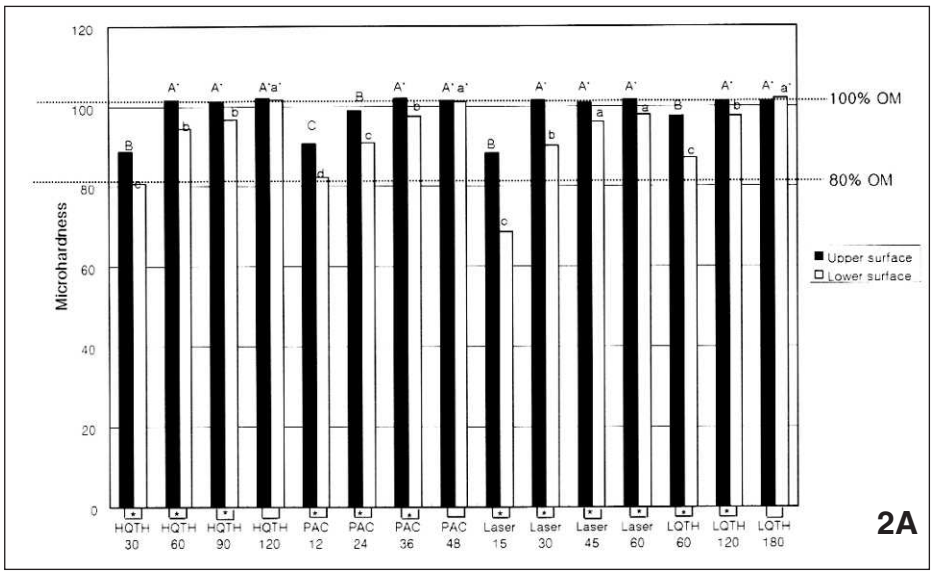
d) Determination of Curing Times Required for the Specimen Under the Pre-cured Composite Overlay to Reach 100% and 80% of OM

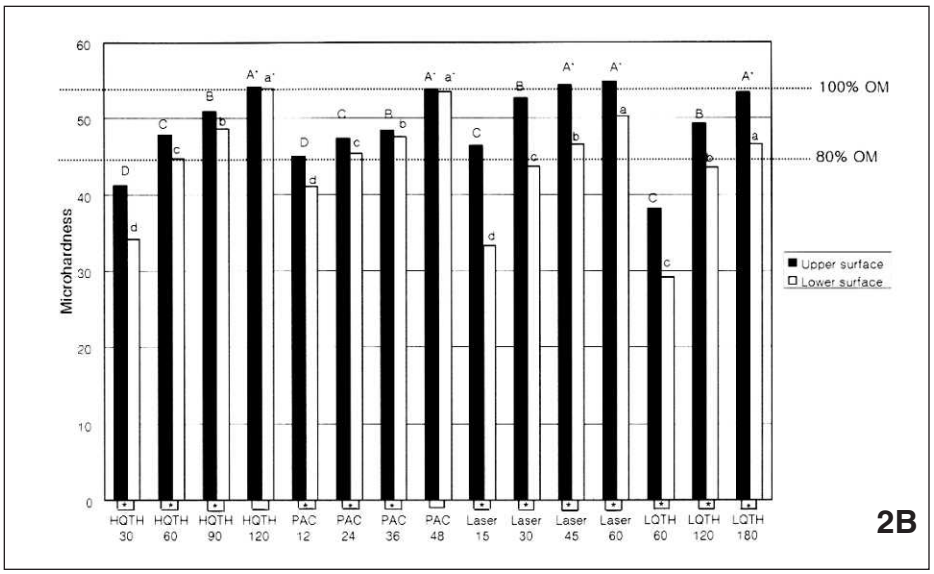
It has been reported that composite curing of a deep cavity layer is considered to be complete if the minimum hardness value is more than 80% of the maximum value measured on the specimen surface (Lutz, Krejci & Frischknecht, 1992; Breeding &

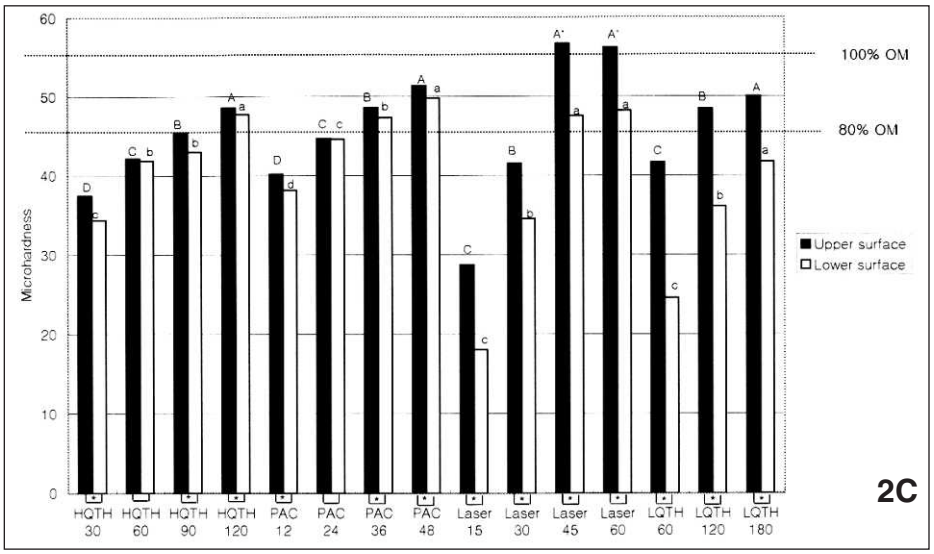
Table 4: *Microhardness Measurement of the 2-mm Thick Test Materials on the Upper (U) and Lower (L) Surface for Determination of Minimum Polymerization Time (MPT)*

		HQTH			PAC			Laser		
	Sec	**10**	**20**	**30***	**6**	**12***	**18**	**10**	**15***	**30**
Z100	U	76.1 (1.2)	81.5 (1.6)	100.9 (2.4)	96.8 (1.8)	100.4 (1.7)	101.4 (0.8)	99.8 (2.2)	100.2 (1.5)	101.3 (1.2)
	L	65.2 (3.0)	77.0 (2.4)	101.6 (1.7)	96.2 (1.4)	101.4 (1.7)	100.2 (1.3)	92.7 (2.0)	99.8 (1.8)	100.1 (1.2)
		HQTH			**PAC**			**Laser**		
	Sec	**10**	**20**	**30***	**6**	**12***	**18**	**10**	**15***	**30**
Tetric Ceram	U	45.1 (2.8)	51.5 (1.7)	53.8 (1.4)	52.1 (1.8)	54.0 (1.4)	53.8 (1.3)	51.0 (1.9)	53.5 (1.6)	52.9 (1.2)
	L	42.3 (3.0)	48.8 (2.4)	52.9 (1.7)	48.8 (1.4)	53.5 (1.7)	53.5 (1.3)	49.8 (2.0)	52.8 (1.8)	52.9 (1.2)
		HQTH			**PAC**			**Laser**		
	Sec	**10**	**20**	**30***	**6**	**12***	**18**	**10**	**15***	**30**
Vario-link II	U	52.0 (0.8)	52.5 (0.9)	55.3 (1.7)	51.2 (1.8)	53.9 (1.7)	53.9 (0.8)	51 (2.2)	54.0 (1.1)	53.9 (1.9)
	L	50.0 (3.0)	49.7 (2.4)	54.4 (1.7)	49.3 (1.7)	53.8 (1.5)	53.8 (1.9)	49 (2.0)	53.8 (1.7)	53.8 (1.0)

**indicates the MPT that was required to obtain the same microhardness as OM on both the upper and lower surface at 0.05 level of confidence. The figures in the parenthesis are the standard deviations.*







others, 1991). From the OM of each material, 80% OM was calculated. Depending on the microhardness data from c) Microhardness measurement of Restorative Resins and Resin Cement Under Composite Overlay, the time required for microhardness of the upper and lower surface of the specimen to reach 100% and 80% of OM was determined.

RESULTS

a) Determination of Optimum Microhardness (OM)

Table 3 shows the OM results. There was no statistically significant difference in microhardness between the upper and lower surfaces in all materials ($p>0.05$). According to the definition of OM, it was 101.4 for Z100, 54.4 for Tetric Ceram and 55.3 for Variolink II.

b) Determination of Minimum Polymerization Time (MPT)

The results are listed in Table 4. From this test, the MPT for HQTH, PAC and laser were found to be 30, 12 and 15 seconds, respectively.

c) Microhardness Measurement of Restorative Resins and Resin Cement Under Composite Overlay and Determination of Curing Time Required for the Specimen Under the Pre-cured Composite Overlay to Reach 100% and 80% of OM

In Z100 specimens, microhardness of the upper and lower surfaces reached OM when the composites were light cured for 120 seconds using the HQTH lamp. When cured

Figure 2. *Microhardness of Z100 (a), Tetric Ceram (b) and Variolink II (c) samples under a pre-cured 1.5-mm thick composite overlay.*
OM: Optimum Microhardness
The capital letters above the bar indicate a different microhardness on the upper composite surface in each group at the p*=0.05 level.*
The small letters above the bar show a different microhardness on the lower composite surface in each group at the p*=0.05 level.*
' was added to A or a if the microhardness was the same as the 100% OM at the p*=0.05 level.*
**shows a significant difference in microhardness between the upper and lower surface at the* p*=0.05 level.*

with the PAC unit, only 48 seconds was needed for the upper and lower surface to reach OM. When Z100 was cured with the laser for 30, 45 or 60 seconds, the microhardness of the upper surfaces reached OM, whereas microhardness of the lower surfaces did not. When cured with the LQTH lamp, 180 seconds was needed to reach OM (Figure 2a). The required curing time for the specimens to reach 80% OM was 60, 12, 30 and 60 seconds in HQTH, PAC, Laser and LQTH, respectively (Table 5, Figure 2a).

Table 5: *Needed Curing Time for the Upper and Lower Surface of the Specimen to Reach 100% or 80 % of OM*

	HQTH		PAC		Laser		LQTH	
	100%	80%	100%	80%	100%	80%	100%	80%
Z100	120	60	48	12	>60	30	180	60
Tetric Ceram	120	60	48	24	>60	45	>180	180
Variolink II	>120	120	>48	36	>60	45	>180	>180

In the Tetric Ceram specimens, the microhardness of the upper and lower surfaces reached OM when the composites were light cured for 120 seconds using the HQTH lamp. When they were cured with the PAC unit, only 48 seconds was required for the upper and lower surfaces to reach OM. When Tetric Ceram was cured with the laser, microhardness of the upper surfaces reached OM when they were cured for either 45 or 60 seconds. However, on the lower surface, they did not reach OM even when the light activation time was extended to 60 seconds. When Tetric Ceram was cured using the LQTH lamp, microhardness of the upper surface reached OM when cured for 180 seconds. However, it did not reach OM on the lower surface. (Figure 2b). The required curing time for the specimens to reach 80% OM was 60, 24, 45 and 180 seconds in HQTH, PAC, Laser and LQTH, respectively (Table 5, Figure 2b).

In the Variolink II specimens, microhardness of the upper and lower surfaces did not reach OM, even though they were light cured with the HQTH lamp for 120 seconds. When they were cured with the PAC unit, 48 seconds was insufficient for them to reach OM. When they were cured with the laser for 45 and 60 seconds, microhardness reached OM on the upper surface but not on the lower surface. When they were cured with the LQTH lamp, microhardness did not reach OM on both surfaces even though the curing time was extended to 180 seconds (Figure 2c). The required curing time for the specimens to reach 80% OM was 120, 36, 45 and >180 seconds in HQTH, PAC, Laser and LQTH, respectively (Table 5, Figure 2c).

DISCUSSION

In this study, compared to the LQTH unit, the PAC, Laser and HQTH units cured restorative composites and dual cured resin cement more effectively under the composite overlay. This appears to be related to the high power density of the curing units.

Ar laser light differs significantly from the QTH system. Differences include: 1) energy emission over a narrower band of wavelengths (about 40 nm vs 120 nm) centered around 470 nm, which is the optimal wavelength for activating the photoinitiator camphoroquinone and 2) collimation of the Ar laser light, resulting in more consistent power density over the distance. In contrast, the power density of QTH decreases with distances, due to greater light divergence from the source (Kelsey & others, 1989). In this study, the MPT was 15 seconds for laser and 30 seconds for HQTH, which is consistent with a previous study by Vargas, Cobb and Schmit (1998). They reported that the Ar laser adequately polymerized resin composite to a depth of 2 mm in half the time for the hybrid and two-thirds the time for the microfil composites compared to conventional light curing units. The materials used in this study can be categorized as small particle hybrid (Z100, Tetric Ceram) and hybrid (Variolink II).

In this study, the MPT for the PAC system was 12 seconds, which is consistent with a previous study by Park, Krajeci and Lutz (2002). They reported that the PAC system effectively cures the composite at a reduced activation time. They recommended 12 seconds of light curing for shallow cavities not exceeding 2 mm.

Also in this study, the restorative composites Z100 and Tetric Ceram cured more effectively than the dual cured resin cement Variolink II under the Targis overlay. The results of this study do not mean that the use of composites as luting material is highly recommended nor that resin composite cures better than dual-cured resin cements in all cases. The results of this study merely show the possibility of using restorative resin composites as luting materials under a 1.5-mm composite overlay. Of course, a longer curing time would be needed as the thickness of the composite overlay increases, and when it exceeds the limit where the curing light cannot pass through it, the restorative materials may not work as luting materials. For a thicker layer, more research will be needed that forms part of an ongoing study. Currently, when composite inlay restorations are planned using restorative composite materials as luting agents in deep cavities, reducing the depth of the cavity using base materials such as glass ionomer or compomer and keeping the depth of the inlay at 1.5 mm would be advisable.

When the laser was used to cure the composite under the composite overlay, only 30 seconds was needed for Z100 and 45 seconds for Tetric Ceram for the microhardness of the upper surfaces to reach OM. However, it did not reach OM on the lower surfaces of the Z100 and Tetric Ceram specimens even though the curing time was extended to 60 seconds. The laser appears to be scattered when it passes through the composite overlay, which means that insufficient light energy would be delivered to the restorative light-curing composites. In this study, the laser unit required a long intermission time to reactivate after one minute of activation. Therefore, the application of a laser for this purpose would be inconvenient in clinical situations that require a longer curing time due to the significant size of a restoration.

Z100 was cured more effectively under the composite overlay compared to the Tetric Ceram specimens. Park and others (2002) reported in their linometer study that Z100 cured much faster than Tetric Ceram. In addition, they reported that the curing rate in the first 10 seconds when Z100 was cured with the HQTH lamp was so fast that there was no difference compared to the PAC system. The wavelength of the emitted light of a PAC system is approximately 470 nm, whereas it is between 400-520 nm in a QTH lamp. Therefore, for composites containing both camphorquinone (CQ) and photo initiators absorbing at a shorter wavelength, the QTH unit may cure them more effectively than the PAC unit. Since the initiator system in Z100 is CQ only, another factor appears to contribute to the more efficient polymerization of Z100. One factor that should be considered is the new initiator-activator system in Z100. In Z100, a three-component initiator system (camphorquinone, tertiary amine and Idonium salt) was introduced (US Patent #5,545,676). According to Dr JD Oxman (3M Dental Products), iodonium salt plays an important role in increasing the curing efficiency. The new initiator-activator system in Z100 may be more efficient in composite polymerization than Tetric Ceram.

When using a high viscous restorative filling material as a luting agent, ultrasound can be used in a non-destructive manner to change the viscosity of the material. A thixotropic substance will change from a solid-like state to a fluid state when subjected to ultrasound. This phenomenon can be used to change the viscosity of the luting agent when seating the composite inlay (Noack, Roulet & Bergmann, 1991). With this method, the composite can be easily removed from the tooth surface, as the material returns to high viscosity when the vibrations are stopped. Siemens teaches this technique as a standard method in the Cerec course for cementing a Cerec inlay using Tetric Ceram as the luting material.

Dual-cured composite cements were reported to exhibit higher wear, because of the lower filler content and relatively inferior physical properties (Noack & Roulet, 1991; Van Meerbeek & others, 1992). Considering the high wear resistance of the contemporary restorative composite materials, the use of restorative composites as luting cements may contribute to the reduced wear of the cementing materials. This possibility requires further research.

In this study, when the Variolink II specimens were cured under a 1.5-mm thick composite overlay, the microhardness of the upper and lower surface did not reach OM in all groups. When the light curing efficiency of the restorative composites and dual cure resin cements was high, the restorative composites cured more effectively under a 1.5-mm thick composite overlay. A few factors may be related to this. In dual-cured resin cements, the initiation of chemically induced free radicals must be delayed so that an adequate working time can be provided during restoration manipulation and light curing (Rueggeberg & Caughman, 1993). To achieve this, a significant amount of inhibitors are required (Cook & Standish, 1983), which also interfere with the polymerization involving light-induced initiation (Cook & Thomasz, 1983). In addition, large amounts of chemically induced free radical generators cannot be placed in these materials, because they would reduce shelf life (Venz & Antonucci, 1988). El-Mowafy, Rubo and El-Badrawy (1999) reported that the microhardness of dual-cured resin cement that was not light cured under the ceramic overlay was lower than that of the dual-cured resin cement, which was light cured. In addition, microhardness of the dual cured resin cements that were under the >2 mm ceramic overlay was always lower than the resin cement that was light cured without the ceramic overlay. Therefore, it was concluded that the physical properties of the dual cured resin cement ultimately depended upon light energy from the curing unit.

In this study, the microhardness was measured seven days after irradiation. The results of the microhardness of the dual-cured resin cement indicated that the extent of curing was dependent on the length of light curing and an additional delayed chemical cure did not occur. The initial light exposure will cause a rapid increase in resin conversion, resulting in a very viscous gel (Rueggeberg & Caughman, 1993). This rapid increase in viscosity hinders the migration of active radical components that would be responsible for further chemical induced polymerization (Korolev & Berlin, 1963).

The curing times required for the upper and lower surface of the specimen to reach 80% OM are summarized in Table 5. As it may be applied to one surface of a restoration, additional time would be required for restorations that cover more than one tooth surface.

Considering the results of this study, 60 seconds (12 seconds x 5) to 180 seconds (36 seconds x 5) would be needed to cure luting materials with the PAC system in MOD composite inlay restoration of a premolar. As this may be a long time for some clinicians, the development of a more effective polymerization unit would be desirable.

It has been suggested that composite cured at a lower power density has a better marginal adaptation (Uno & Asmussen, 1991a). However, this procedure leads to inferior material properties (Uno & Asmussen, 1991a). Another way to minimize wall-to-wall contraction is to allow the flow of a resin composite during setting by means of controlled polymerization. This can be done by prepolymerizing at a low power density followed by a final cure at a high power density (Mehl, Hickel & Kunzelmann, 1997). The reduced rate of polymerization may allow for increased flow of the material, decreasing the polymerization shrinkage stress in a restoration (Uno & Asmussen, 1991b), which may be more favorable to the marginal integrity (Uno & Asmussen, 1991a; Feilzer & others, 1995; Mehl & others, 1997). The use of a PAC unit with a higher power density may cause a more rapid development of the polymerization contraction force. However, the marginal gap of the PAC and QTH units were similar (Peutzfeldt, Sahafi & Asmussen, 2000). Peutzfeldt and others (2000) indicated the possibility that a low degree of conversion was compensated for by the rapid cure that resulted in gaps similar to those obtained using the conventional QTH unit. In this study, the burst-curing mode was used in HQTH, PAC and laser. The effect of using PAC, HQTH or laser on the margin of the indirect composite restoration has not been fully understood and requires further research.

In this study, the thickness of a composite specimen was 500 μm. As the thickness of the luting materials for the indirect composite and ceramic restorations was approximately 40-240 μm (Krejci, Lutz & Gautschi, 1994), the specimen was too thick for use in clinical situations. Initially, the thickness of the specimen was designed to be 200 μm. However, in a pilot study, there were difficulties in measuring the hardness on both the upper and lower surfaces of the specimen with such a thin specimen. Therefore, the sample thickness was increased to 500 μm.

The specimens were stored under dry conditions. When stored in wet conditions, as in our pilot study, large standard deviations in microhardness were measured, particularly when the samples were not cured sufficiently. This is believed to relate to water sorption in the specimens.

CONCLUSIONS

When the restorative composites and dual-cure resin cements were light cured under a 1.5-mm Targis overlay, the PAC system was the most effective curing system followed by the laser, HQTH unit and LQTH unit. In addition, the restorative composites cured more efficiently than the dual-cured resin cements.

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Effect of Fluoride Varnishes on Color Stability of Esthetic Restorative Materials

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Clinical Relevance

Fluoride varnishes can be used without adversely affecting the color of esthetic restorative materials.

SUMMARY

Fluoride varnish applications were applied to two hybrid resin composite materials, Z-100 (3M Dental Products, St Paul, MN, USA) and Esthet-X (Dentsply Caulk, Milford, DE, USA), shades A1 and A2 and a glass ionomer, GC Fuji IX GP Fast (GC Corporation, Tokyo, Japan), shade A2, to evaluate color stability. Specimens (12.6-mm dia x 2.3 mm) were prepared using a polyethylene frame, light-cured and polished through a 1-µm alumina finish. After the initial baseline color measurements, the discs were suspended in Fusayama artificial saliva (FAS) solution at 37˚C for 48 hours. Post immersion, the specimens were divided into five groups (n=15 each). The following fluoride varnishes were applied to four groups of test specimens: Duraphat (Colgate Oral Pharmaceutical, Inc, Canton MA, USA), Cavity Shield (OMNII Oral Pharmaceuticals, West Palm Beach, FL, USA), Duraflor (Pharmascience Inc, Montreal, Canada) and Fluor Protector (Vivadent, Ivoclar North America, Amherst, NY, USA). The varnish was allowed to dry for five minutes before immersion. The control group was not coated with varnish, although the specimens were immersed in FAS. All specimens were incubated in newly prepared FAS at 37˚C for 24 hours, cleaned with an electric toothbrush and the process repeated using newly prepared FAS. CIE L*a*b* color measurements were recorded five times: at baseline, after 48 hours FAS immersion, after cleaning the first and second fluoride varnish applications and after the final brushing using a commercial toothpaste (Crest). A Minolta CR-300 tristimulus colorimeter with an 8-mm aperture (Ramsey, NJ, USA) was used to record color measurements with the daylight (D_{65}) setting. Calculations were performed for using CIE parameters ΔE*, ΔL*, Δa*, Δb*. Analysis of variance (ANOVA) and post-hoc test (Fisher's PLSD) were used for statistical analysis. After immersion in saliva, the tested glass ionomer (Fuji IX) produced the most significant color changes (ΔE*=1.19 and ΔL*=-1.03), indicating the effect of the color change was due to absorption. After fluoride varnish applications, Duraphat varnish produced significant changes in all tested materials and shades, resulting in color changes with ΔE greater than (>) 1 but less than (<) 3. These color changes are considered visually perceptible, yet have been reported in dental literature as clinically acceptable. Fluoride varnishes can be used without adversely affecting the color of restorative materials.

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INTRODUCTION

For more than 30 years, fluoride varnishes have been the practice standard for the professional application of topical fluoride in Europe (Beltrán-Aguilar, Goldstein & Lockwood, 2000). Their use as anti-cavity varnishes and in treatment for hypersensitive teeth is increasing among US dentists. The primary reason for the wide acceptance of fluoride varnishes is the easy, safe, convenient and well-accepted application procedure (Ogard, Seppa & Rolla, 1994). With fluoride varnishes, the amount of fluoride exposure for patients can be better controlled. Also, less chair time is required for the patient compared with the time required for the application and use of conventional foams and gels (Ogard & others, 1994). Fluoride varnish covers the teeth with an adherent film for a limited period of time, thereby, enhancing the uptake of fluoride ions into the tooth structure.

Currently, there are four fluoride varnishes marketed in the United States: Duraflor (Pharmascience Inc), Duraphat (Colgate Oral Pharmaceutical, Inc), Fluor Protector (Vivadent) and CavityShield (OMNII Oral Pharmaceuticals). Duraphat, Duraflor and CavityShield contain 5% sodium fluoride (2.26 wt% F) (Beltrán-Aguilar & others, 2000). Due to the natural resin carrier, Duraphat, Duraflor and CavityShield set with moisture, producing a light-amber colored film on the surface of the teeth. Fluor Protector contains 0.9 wt% of difluorosilane (0.1 wt% F) in a polyurethane based varnish and sets to a thin transparent film on the tooth surface (Beltrán-Aguilar & others, 2000).

While tooth-colored materials provide successful aesthetic dental restorations, unacceptable color match or staining are major reasons to replace anterior fillings (Kroeze & others, 1990). Intrinsic factors due to physico-chemical reactions in the deeper portions of the restoration or extrinsic factors, such as absorption or accumulation of stains, can cause discoloration (Horsted-Bindslev & Mjör, 1988). The development of colorimetry has facilitated study related to color stability, enabling one to identify particular color science parameters (Dietschi & others, 1994).

Color science phenomena are represented by the universally used CIE color parameters L*, a* and b*. L* refers to the lightness or whiteness coordinate, with a value range from 100 (perfect white) to zero (perfect black). The parameters a* and b* are chromaticity, or color coordinates on the red-green axis and the yellow-blue axis, respectively. Positive a* values signify red; negative values represent green. Similarly, positive b* values signify the yellow axis and negative b* values represent the blue axis. The difference in measurement values is referred to as the delta (Δ) and connotes a difference or a change in color. A negative ΔL* value signifies a decrease in whiteness, known as value, and results in a darker appearance. Each of the single parameters contributes equally to the CIE color difference formula $\Delta E = [(\Delta L^*)2 + (\Delta a^*)2, (\Delta b^*)2]^{1/2}$ (CIE Colorimetry Committee 1974. Consequently, a shift in the Δ value for a single parameter contributes to the overall effect of color difference (Anusavice, 1996).

Colorimeters are used to evaluate *in vitro* color changes, because the instrumental measurements eliminate subjective interpretation of visual-color comparison (Seghi, Hewlett & Kim, 1989; Okubo & others, 1998; Iazzetti & others, 2000). Studies in the dental literature have reported various aspects of color perceptibility and acceptability. While color changes are a major reason for replacing resin-based materials (Mjör, Moorhead & Dahl, 2000), not all perceptible differences necessitate replacement. Color differences between composites with a ΔE 3.3 or greater were found unacceptable in a study of 12 observers (Ruyter, Nilner & Moller, 1987). However, the acceptability limit for ceramics was reported as ΔE less than (<) 3.7 (Johnston & Kao, 1989) by two of three observers. Kuehni and Marcus (1979) reported that color differences between two objects (or treatments) yielding a ΔE higher than (>) 1 were visually perceptible to those with normal color vision. This implies that the ΔE values between 1 and 3 for the total color change are considered both visually detectable, but as reported in two studies, clinically acceptable.

Given its color and resinous nature, fluoride varnish coating might affect a temporary change in the color of restorative materials. However, data reporting the effects of fluoride varnishes on the color of esthetic restorative materials are lacking. This *in vitro* study determined the effect of two applications of four different fluoride varnishes on the hue and value of two hybrid resin composites and one glass ionomer.

METHODS AND MATERIALS

Two resin composite materials, Z-100 (3M Dental Products) and Esthet-X (Dentsply Caulk), shades A1 and A2, and a glass ionomer, GC Fuji IX GP Fast (GC Corporation), shade A2, were used to prepare experimental specimens. Individual specimens (12.6 mm diameter x 2.3 mm) of each material were formed using a polyethylene frame and light-cured (Demetron, Division of Kerr Corporation, Danbury, CT, USA) for 40 seconds on each side. Each specimen was individually wet-polished on both sides with 1200 and 2000 grit silicon carbide abrasive (Mark V Laboratory, East Granby, CT, USA) and finished with 1 μm aluminum oxide slurry (Fisher Scientific Company, Pittsburgh, PA, USA).

Each specimen was suspended in an individual polystyrene vial (Fisher Scientific Co) containing 20 ml FAS (Fusayama artificial saliva) solution (Fusayama,

Katayori & Nomoto, 1963) before the fluoride varnish applications. The vials were placed in an Eviron-Shaker (Lab-wine Instruments Inc, Melrose Park, IL, USA), agitated at 60 rpms and incubated at 37˚C for 48 hours.

The specimens were removed from the FAS, air dried and randomly divided into the five different treatment groups (n=15). A soft mini-brush was used to apply one of the following fluoride varnishes to the respective group; Duraphat (Colgate Oral Pharmaceutical Inc); Cavity Shield (OMNII Oral Pharmaceuticals); Duraflor (Pharmascience Inc) and Fluor Protector (Vivadent). Upon coating, each specimen was suspended in air to dry for approximately five minutes prior to immersion in FAS. All of the specimens were again suspended in newly prepared FAS (20 mL) and incubated at 37˚C for 24 hours; they were then individually cleaned for two minutes with an electric toothbrush (Sonicare, c/o Philips Oral Health Care, Snoqualmie, WA, USA). The fluoride varnish coating process was repeated for a second 24-hour period, after which the cleaning process was repeated as previously described. The controls were not coated with varnish, although they were immersed in FAS and incubated.

Tristimulus color measurements (L*, a*, b*) were taken using a Minolta CR-300 colorimeter with an 8-mm aperture (Ramsey, NJ, USA) and D_{65} (daylight) illuminant. The colorimeter was calibrated using its specified calibration plate before each series of measurements. For each parameter, three measurements were taken and an average was calculated for each specimen. The hue and value measurements were recorded at five intervals: baseline (dry), after 48 hours FAS immersion; after cleaning the first and second fluoride varnish applications and finally, after brushing with a commercial toothpaste (Crest, Procter & Gamble, Cincinnati, OH, USA). During color measuring, each specimen was placed on a neutral gray background referred to as Munsell N-7 for standardized, minimal background influence and color consistency. Calculations were performed for CIE parameters showing the total color difference, $\Delta E=[(\Delta L^*)^2 + (\Delta a^*)^2, (\Delta b^*)^2]^{1/2}$. Changes ($\Delta$) for L*, a* and b*, the individual value and hue parameters, were determined by subtracting the parameter variable for one experimental period from another. For the color change analysis, the following intervals were used: FAS–the difference between baseline (dry) and the initial 48 hours saliva immersion, F1–the difference between 48 hours initial saliva immersion and the first fluoride varnish applications, F2–the difference between 48 hours initial saliva immersion and second fluoride varnish applications and Brush–the difference between 48 hours initial saliva immersion and the final cleaning.

STATVIEW 5.0 software and analysis of variance (ANOVA) was used for statistical analysis of color changes. Data were analyzed from the color measurements recorded at each of the five intervals (before and after saliva immersion, after two separate fluoride applications and after cleaning with toothpaste). The analysis was done for total color change (ΔE) and for each parameter (ΔL^*, Δa^*,Δb^*). A post-hoc analysis (Fisher's PLSD) was used for pair-wise comparisons of different groups.

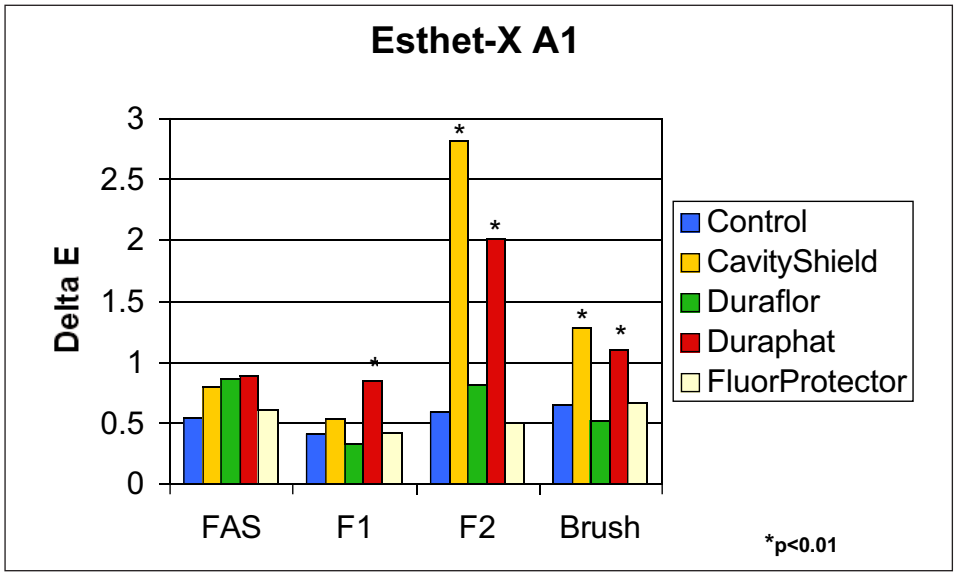

Figure 1.

Table 1: *Mean ΔE, Δa^*, Δb^*, and ΔL^* with Standard Deviations (SD) and Statistical Significance Levels of Composites when Compared to a Glass Ionomer (Fuji IX) after the Initial 48 Hours Saliva Immersion*

Material n=30	ΔE (SD)	Δa*	Δb*	ΔL*
Fuji IX A2	1.19 (0.39)	0.18 (0.06)	0.33 (0.29)	-1.03 (0.54)
Z-100 A1	0.71 (0.54)*	0.14 (0.14)	-0.49 (0.44)*	-0.24 (0.52)*
Z-100 A2	0.55 (0.20)*	0.28 (0.09)*	-0.26 (0.17)*	-0.21 (0.34)*
Esthet-X A1	0.74 (0.17)*	0.52 (0.13)*	-0.20 (0.15)*	-0.41 (0.23)*
Esthet-X A2	0.74 (0.50)*	0.38 (0.05)*	-0.07 (0.41)*	-0.49 (0.49)*

*Significance level: *p<0.0001*

Table 2a: Color Changes for Z-100 A1 (n=30)

Z-100 A1	**ΔE**	**Δa***	**Δb***	**ΔL***
Control	0.90 (0.52)	0.16 (0.11)	-0.04 (0.73)	-0.33 (0.73)
Duraflor	0.99 (0.16)	0.09 (0.03)	-0.24 (0.32)	-0.93 (0.11)*
Duraphat	1.54 (0.11)*	0.56 (0.06)*	0.05 (0.11)	-1.43 (0.13)*
Fluor Protector	1.10 (0.41)	0.29 (0.07)*	-0.23 (0.58)	0.87 (0.44)*
CavityShield	1.28 (0.25)	0.04 (0.06)*	0.13 (0.40)	-1.23 (0.18)*

Table 2b: Color Changes for Z-100 A2 (n=30)

Z-100 A2	**ΔE**	**Δa***	**Δb***	**ΔL***
Control	0.53 (0.16)	-0.05 (0.03)	-0.47 (0.25)	-0.12 (0.24)
Duraflor	1.10 (0.45)	-0.11 (0.09)	-0.24 (0.29)	-0.96 (0.56)
Duraphat	2.53 (0.93)*	0.21 (0.22)*	1.29 (0.91)*	-2.09 (0.63)*
Fluor Protector	1.15 (0.22)	-0.08 (0.03)	-0.84 (0.22)	-0.69 (0.37)
CavityShield	1.32 (1.30)	-0.07 (0.05)	-0.26 (0.26)	-0.85 (1.66)

Table 2c: Color Changes for Esthet-X A1 (n=30)

Esthet-X A1	**ΔE**	**Δa***	**Δb***	**ΔL***
Control	0.64 (0.15)	-0.05 (0.06)	-0.27 (0.13)	-0.56 (0.17)
Duraflor	0.52 (0.23)	-0.29 (0.04)*	-0.03 (0.11)	-0.40 (0.27)
Duraphat	1.10 (0.35)*	0.07 (0.15)*	0.58 (0.33)	-0.89 (0.24)
Fluor Protector	0.67 (0.22)	0.02 (0.02)	-0.47 (0.39)	-0.17 (0.38)*
CavityShield	1.28 (0.50)*	-0.40 (0.12)*	-0.07 (0.28)	-1.18 (0.48)*

Table 2d: Color Changes for Esthet-X A2 (n=30)

Esthet-X A2	**ΔE**	**Δa***	**Δb***	**ΔL***
Control	0.75 (0.18)	-0.07 (0.05)	-0.41 (0.18)	-0.59 (0.20)
Duraflor	0.57 (0.37)	-0.13 (0.06)	-0.05 (0.23)	-0.41 (0.50)
Duraphat	2.53 (2.87)*	0.42 (0.15)*	0.49 (0.80)*	-0.95 (0.42)
Fluor Protector	1.20 (0.65)	0.06 (0.07)*	-0.86 (0.41)	0.72 (0.68)*
CavityShield	0.63 (0.19)	-0.14 (0.02)	0.06 (0.25)	-0.57 (0.17)

Table 2e: Color Changes for Fuji IX (n=30)

Fuji IX A2	**ΔE**	**Δa***	**Δb***	**ΔL***
Control	0.53 (0.16)	-0.05 (0.03)	-0.47 (0.25)	-0.12 (0.24)
Duraflor	1.08 (0.45)	-0.11 (0.09)	-0.24 (0.29)	-0.96 (0.56)
Duraphat	2.53 (0.93)*	0.21 (0.22)*	1.29 (0.91)*	-2.09 (0.63)*
Fluor Protector	1.15 (0.22)	-0.08 (0.03)	-0.84 (0.22)	-0.69 (0.37)
CavityShield	1.32 (1.29)	-0.07 (0.05)	-0.26 (0.26)	-0.85 (1.66)

RESULTS

Table 1 shows the color changes after the initial 48 hours immersion in artificial saliva. A post-hoc analysis (Fisher's PLSD) showed that the glass ionomer tested in this study (Fuji IX) had the most significant color change (ΔE=1.19), when compared to other tested materials, ANOVA F (4,140)=10.191, $p<0.0001$.

Changes in the chroma (a* and b*) values were produced after the initial 48 hours saliva immersion, verifying the effect on chroma due to absorption of the aqueous solution. The most significant Δa* change was noted with Esthet-X, shade A1 (Δa*=0.52), ANOVA F (4,140)=68.351, $p<0.0001$. The most significant change in b* value was noted with Z-100, shade A1 (Δb*=-0.49), ANOVA F (4,140)=24.970, $p<0.0001$.

Similarly, the L* value was affected by the initial 48 hours of immersion in artificial saliva. All materials tested produced a negative ΔL*, indicating a decrease in value (darkening) due to absorption. However, a glass ionomer (Fuji IX) produced the most significant negative ΔL* value (-1.03), ANOVA F (4,140)=15.502, $p<0.0001$.

The total color changes (ΔE) for Esthet-X, shade A1 are shown in Figure 1. The greater proportion of overall color changes could be noted after the second fluoride varnish applications (F2) with CavityShield and Duraphat. The changes were still significant after final cleaning (Brush). This pattern could also be noted in other tested materials (data not shown).

The effects of the total color changes (ΔE) on the restorative materials after the fluoride varnish applications and the final brushing are shown in the Table 2 series (2a through 2e). These ΔE values were calculated using CIE L*, a*, b* measurements recorded between the initial 48 hours immersion in Fusayama artificial saliva (FAS) and the final brushing. A post-hoc analysis (Fisher's PLSD) showed that Duraphat varnish produced significant changes in all tested materials and shades when compared to their respective controls ($p<0.01$). Also, CavityShield varnish produced a significant change in Esthet-X material, shade A1 (ΔE=1.28, $p<0.01$) (Table 2d).

The values for each of the ΔL* Δa* Δb* parameters were analyzed separately. Composite Z-100, shade A1 was the only material for which a noteworthy change occurred in Δa* value; this change was observed only after Duraphat varnish applications (Δa* = 0.56, $p<0.01$) (Table 2a). Composite Z-100, shade A2 and the glass ionomer (Fuji IX) produced significant changes in Δb* value only after Duraphat varnish applications (Δb*=1.29, $p<0.01$) (Table 2b).

Duraphat had the most significant effect on ΔL* for all three materials (Z-100, Esthet-X, Fuji IX) and

shades. CavityShield produced a noticeable effect on the shade A1 for both Z-100 (ΔL*=-1.23) and Esthet-X (ΔL*=-1.18), indicating that the material was perceptibly darker after two applications and cleaning. Duraflor produced a negative ΔL* value (ΔL*=-0.93) for Z-100, shade A1, indicating that it does contribute to a decrease in value.

DISCUSSION

Color stability is critical to the long-term success of esthetic restorations. The use of fluoride varnish is increasing among US dentists. As some varnish applications create a superficial, yellowish film on teeth for a designated time, concern regarding the effect of varnishes on the color stability of materials is justified.

This study reported the ΔE values for the total color change between 1 and 3, which are considered both visually detectable and clinically acceptable. It is important to note that the three parameters L*, a* and b* each contribute equally to the total color change (ΔE). Most dental studies report only the ΔE values. One such study (Douglas & Brewer, 1998) reported chromatic acceptability limits for reds (a*) having a ΔE less than (<) 1.1. The ΔE limit for yellow (b*) in the same study was reported as 2.1. In our study, not all shades or materials demonstrated the same degree of color change. Shade A1, composite Z-100, exhibited the greatest change in a* (red/green axis) after the Duraphat treatment (Δa*=0.56), while b* (yellow/blue axis) was most effected in Z-100 A2 and Fuji IX (Δb*=1.29). These are noteworthy changes considering that the total difference (ΔE) is perceptible at a value of one. It is, however, important to note that in this study both the axes a* and b* exhibited differences within limits previously reported as clinically acceptable. Fluor Protector had virtually no effect on the chromatic parameters. This might be due to its transparent color.

Also, it was noted that color changes increased after the second fluoride varnish application, especially when Duraphat and CavityShield varnishes were applied. This is probably due to the light amber color of the varnish and its stickiness. After the final brushing with toothpaste, the color change was reduced significantly. Further experimentation might demonstrate that repeated cleanings after fluoride applications might continue to reduce the color change in restorative materials, which was not examined in this study.

In this study, it was visually and instrumentally observed that the glass ionomer specimens became darker than the other materials after the initial 48 hours of immersion in artificial saliva. Darkening involves a decrease in value (L*). In dentistry, value is often considered the most critical and easily recognized component in shade matching (Shillingburg & others, 1997). Douglas and Craig (1982) reported that hydrophilic materials stain more than hydrophopic materials. The glass ionomer material, most likely due to its hydrophilicity and greater surface degradation, exhibited greater color change than the composites used in this study. The Δa*=0.5 after only 48 hours immersion in FAS indicates that there is a definite chromatic effect from saturation in an aqueous solution. In this case, the red alone increased to half of the perceptible total ΔE value. While chromatic effects vary, such an effect should be considered at the time of shade selection for restorations. However, in this study water sorption itself did not seem to alter the perceived color of materials to an unacceptable extent.

In a study by Debner, Warren and Powers (2000), the color stability of a compomer, hybrid ionomer and composite was determined after staining with the fluoride varnishes Duraphat, Duraflor and Fluor Protector. In their study, the varnishes were applied only once and cleaning was performed with a toothbrush and toothpaste. After brushing, the only combination that exhibited a perceptible difference was TPH Spectrum (Dentsply/Caulk) composite with Duraphat varnish (ΔE=5.4). Although our experimental design was different, it supports the findings that Duraphat varnish can cause perceptible color changes.

Staining by absorption is one of the extrinsic factors that produces discoloration. The composition and size of the filler particles affect both surface smoothness and susceptibility for extrinsic staining (Peutzfeldt & Asmussen, 1990). The relative susceptibility of glass ionomer for staining could be attributed to the porosity of the glass particles. Z-100 is a hybrid composite with filler loading 66% by volume, with a particle size range of 3.5 to 0.01 microns (3M Dental Products). Esthet-X is a microhybrid with a glass particle size of 0.6-0.8 microns, with a particle size distribution of 0.02-2.5 microns. The total percentage by volume of fillers is 60 volume % (Dentsply Caulk). The relative susceptibility of Z-100 could be seen when parameters a* and b * were analyzed, with changes resulting in both the red-green and yellow-blue axis. This may be due to the larger particle size and possibly the rougher surface of Z-100. Rough surfaces mechanically retain surface stains more than smooth surfaces (Van Groeningen, Jongebloed & Arends, 1986). Surface roughness in glass ionomers seems to be more related to cracking and porosity of the material. Christensen (1993) reported that glass ionomers dehydrate after setting and drying. Mair (1991) noticed that the micro-cracks allow for stain penetration and discoloration. In this study, the porosity of glass ionomer discs was noticeable and the varnish was clinically retained in the cracks. These factors could also contribute to the variability in color measurements observed in this study.

Color measurements were recorded after immersion in artificial saliva to show the effect of absorption and

the subsequent color change simulating the clinically relevant oral environment. Composites allow water to penetrate the matrix or filler-matrix interface (Braden & Clarke, 1984; Fan & others, 1985). Consequently, immersion of the specimens in artificial saliva before staining may have reduced the overall stain absorption.

The small sample size in different groups and surface finishing might contribute to the high standard deviations. However, the temporary staining effect of Duraphat varnish on tested materials could be observed, as well as measured.

CONCLUSIONS

In this study, all the color changes in the tested materials after different and multiple fluoride varnish applications and subsequent cleanings were within the reported clinically acceptable range. It is important to educate patients about the temporary color change after fluoride varnish applications, especially when using Duraphat and CavityShield, which can last until the teeth are thoroughly cleaned multiple times with toothpaste. Further experimentation might demonstrate that repeated cleanings after fluoride application might continue to reduce the color change in restorative composite materials, which was not examined in this study. This study supports the conclusion that fluoride varnishes can be used on patients with esthetic restorations without adversely affecting the color of these materials.

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Hardness and Degree of Conversion of Posterior Packable Composites

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Clinical Relevance

Although some manufacturers of packable composite materials claim polymerization up to 5-mm increments, the degree of conversion and hardness was generally greater at the surface than at 4-mm depth.

SUMMARY

Knoop microhardness and the degree of conversion of three packable composites (SureFil, Alert and Solitaire), a microfil composite (Heliomolar), a microhybrid composite (Herculite) and an indirect laboratory-processed composite (Belleglass) were evaluated as a function of distance from the irradiated surface. Cylindrical specimens (5.0 mm [diameter] x 6.0 mm [length]) of each material were visible light cured for 60 seconds in black-backed Teflon molds and sectioned. Knoop microhardness values were then obtained at 0-, 2- and 4-mm using a 50-gram load and 20 second dwell time. Degree of conversion was determined using Fourier Transform Infrared Spectroscopy. ANOVA ($p<0.001$) and Tukey Multiple Comparison Test ($p<0.05$) showed the indirect laboratory-processed composite Belleglass exhibited the highest mean values for both hardness and degree of conversion. Alert exhibited significantly greater hardness than SureFil and Solitaire at 0-mm depth. SureFil had significantly greater hardness than Alert at 4-mm depth. The degree of conversion of SureFil packable composite at 4-mm depth was significantly higher than any of the other direct composites tested.

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INTRODUCTION

The technology of composite materials has changed over the past decade. New developments, along with an increase in patient esthetic awareness, have led many practitioners to use composites to restore posterior teeth. The clinical success of composite for posterior restorations has been documented; however, several questions still remain regarding the durability of composites in the oral environment (Wilson & others, 1988; Barnes & others, 1991; Turkun & Aktener, 2001;

Wassell, Walls & McCabe, 1995, 2000; Scheibenbogen-Fuchsbrummer & others, 1999; Manhart & others, 2000; Tyas, 1990).

In addition to reported clinical success, a number of problems have been documented for direct posterior composite restorations. The direct placement of large posterior composite restorations may result in microleakage, recurrent decay, incomplete cure, restoration fracture and poor marginal adaptation (Inoue & Hayashi, 1982; Caputo & Standlee, 1987; Ferracane, 1992; Ernst & others, 2001). Additional problems may include insufficient proximal contacts, wear, voids and postoperative sensitivity (Bryant, 1987, 1992; Robinson, Moore & Swartz, 1987). Laboratory-processed composite was introduced to address some of these deficiencies (Chalifoux, 1998). Laboratory processing offers more control of the depth of cure and porosity (Walton, 1992; Terry & Touati, 2001). More recently, direct packable composite has been introduced, claiming advantages such as improved marginal adaptation, decreased polymerization shrinkage, the potential to cure in greater increments and manipulation characteristics that are more similar to amalgam.

The long-term durability of composite restorations is difficult to predict intraorally. During polymerization of Bis-GMA based materials with visible light, a cross-linked matrix is formed. Subsequently, a significant number of the carbon-carbon double bonds remain unreacted when the resin is cured in the oral environment (Ferracane, 1985). Fourier Transform Infrared Spectroscopy has been used to evaluate the degree of conversion in Bis-GMA-based composite materials (Ferracane & Greener, 1984; Ferracane, 1985; Chung & Greener, 1990). The reported range for degree of conversion has been 43% to 78% (Ferracane & Greener, 1984; Ferracane, 1985; Chung & Greener, 1990). The mechanical properties of a material may be affected by the amount of residual monomer; therefore, the degree of conversion may provide valuable information regarding the durability of a material (Chung & Greener, 1990).

The degree of conversion of a resin material may affect physical properties, such as compressive strength, wear and hardness (Ferracane & Greener, 1986; Ruyter & Oysaed, 1982b). Highly polymerized composites characterized by increased cross-link density and low residual monomer have been shown to exhibit greater wear resistance, hardness and flexural strength (Wendt, 1987; McCabe & Kagi, 1991; Reinhardt, Boyer & Stephens, 1994; Ferracane & others, 1997). The ability of a material to resist indentation is referred to as hardness. It is measured as the relative resistance to indentation exhibited by a material with the application of a specific load (Mandikos & others, 2001). Asmussen (1982) found materials with decreased hardness also contained a higher amount of unreacted methacrylate groups. Chung and Greener (1990) found no correlation between the mechanical properties of posterior composites and the degree of conversion; however, Ferracane (1985) found a correlation between an increase in hardness and an increase in the degree of conversion for a specific resin. Hardness values may also be useful in assessing finishing and polishing characteristics and materials resistance to abrasion.

Greater hardness values may result from increasing the filler content of a composite material (McCabe, 1990; van Noort, 1994). In addition to filler content, attention has also been directed toward filler particle size (St Germain & others, 1985), filler matrix bond (Soderholm, 1984), polymerization method (Wendt, 1987; Cook & Johannson, 1987) and material chemistry (Beatty & others, 1993) to address some deficiencies of composite restorations. Packable composites containing larger filler particles than hybrid and microfill composite materials have been introduced more recently. In addition, these materials have incorporated irregular glass fibers (Kelsey & others, 2000; Cobb & others, 2000; Krause, Park & Straup, 1989; Vallittu, Lassila & Lappalainen, 1994), rough porous fillers (Kelsey & others, 2000) and irregular filler particles of different sizes (Perry, Kugel & Leinfelder, 1999; Kelsey & others, 2000) to obtain packability of the material.

With the advent of posterior packable composites and their various chemistries and reinforcing fillers, it has become necessary to compare the physical properties of these new materials with more conventional resin-based materials. This study compared the hardness and degree of conversion at the irradiated surface, as well as 2 mm and 4 mm from the surface. Information regarding the hardness and degree of conversion of these materials may be helpful in projecting their long-term durability in the oral environment.

METHODS AND MATERIALS

Six composite materials were tested in this study; product and manufacturer information is presented in Table 1. Three packable composites were evaluated: Solitaire (Heraeus Kulzer, South Bend, IN, USA), a hybrid composite which contains porous SiO_2 filler in a multifunctional methacrylate matrix; Alert (Jeneric/Pentron, Wallingford, CT, USA), a glass fiber-reinforced hybrid composite and SureFil (Dentsply Caulk, Milford, DE, USA), a microhybrid urethane-modified Bis-GMA based composite. Two direct composites were studied: Herculite (Kerr, Orange, CA, USA), a BisGMA-based microhybrid composite and Heliomolar (Ivoclar, Amherst, NY, USA), a non-homogenous microfill composite. One laboratory processed

Table 1: *Composite Materials Tested*

Material	Manufacturer	Type	Composition
Alert	Jeneric/Pentron, Inc Wallingford, CT, USA	**Direct** Packable	Glass fiber (10-12 mm dia) reinforced hybrid composite. Filler 84 wt %
Solitaire	Heraeus Kulzer, South Bend, IN, USA	**Direct** Packable	Porous SiO_2 (2-20 mm) hybrid composite Filler 66 wt %
SureFil	Dentsply, Caulk Milford, DE, USA	**Direct** Packable	Micro-hybrid (0.8 mm) urethane BisGMA-based composite. Filler 82 wt %
Belleglass	Belle de St Claire/Kerr, (dentin), Orange, CA, USA	**Indirect** Ceramic-Polymer Composite	Micro-hybrid (0.6 mm) BisGMA-based composite. Filler 78 wt %
Herculite	Kerr, Orange, CA, USA	**Direct** Micro-hybrid Composite	Micro-hybrid (0.6 μm) BisGMA-based composite. Filler 78 wt %
Heliomolar	Ivoclar, Amherst, NY, USA	**Direct** Non-homogeneous Microfill Composite	Prepolymerized organic filler (0.04 mm) in urethane BisGMA based composite. Filler <65 wt %

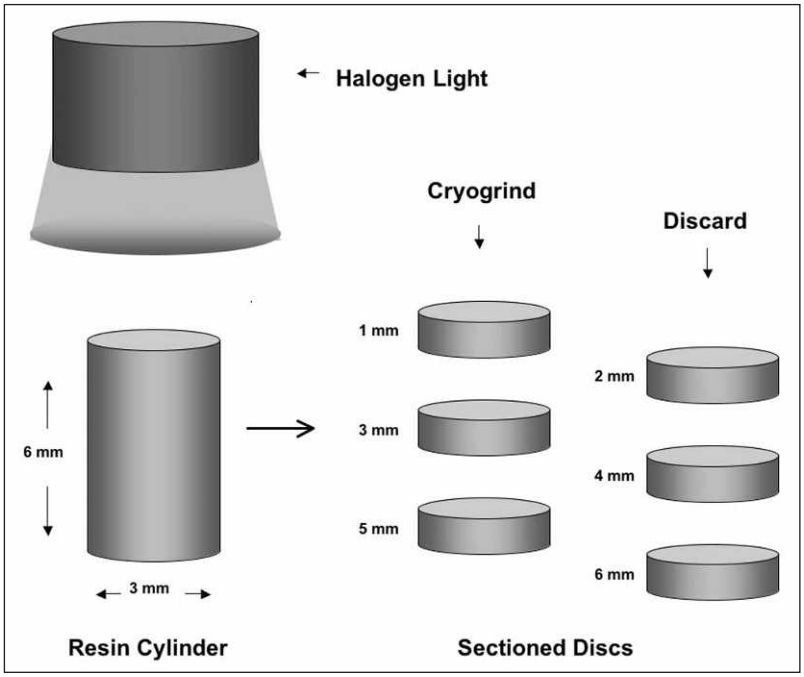

Figure 1. *Schematic representation of polymerized composite cylinder sectioned into six 1-mm segments. Segments in the middle column representing 0-mm, 2-mm and 4-mm depth of cure were maintained for testing. The remaining segments (right column) were discarded.*

composite, Belleglass (Belle de St Claire, Orange, CA, USA), was also studied. Belleglass dentin was utilized, which is a Bis-GMA-based composite similar to that used in Herculite.

Manufacturers' specifications as to specimen preparation and method of cure were carefully followed. Six cylindrical specimens [5.0 mm diameter (OD) x 6.0 mm length] of each composite were prepared using a Teflon split-mold. The packable and conventional composites were visible light cured for 60 seconds from one end in black-backed Teflon split molds. The indirect laboratory-processed composite was light, heat and pressure cured under N_2 according to the manufacturer's specifications. The cylindrical specimens were then sectioned into six equal parts, of which three were retained for testing (Figure 1).

Knoop Hardness (KHN)

Specimen surfaces at 0, 2 mm and 4 mm from the irradiated surface were sequentially polished to 0.5 μm with aluminum oxide and water. The specimens were maintained in sealed vials at 37°C ± 2°C for 24 hours prior to testing. Ten evenly spaced Knoop hardness measurements were then obtained from defined areas of each specimen at 0, 2 mm and 4 mm from the irradiated surface using a 50 gram load with an indenter dwell time of 20 seconds.

Degree of Conversion (DC)

The degree of conversion was determined for each composite material at increasing depths (Figure 1) from the irradiated surface. Fourier Transform Infrared Spectroscopy (FTIR) (MIDAC Corporation, Costa Mesa, CA, USA) was completed using a potassium bromide (KBr) pellet technique described by Ferracane and Greener (1984) and Urbanski and others (1977) and modified by Kerby and others (1999). Samples of the unpolymerized resin systems (n=3) were diluted in chloroform, smeared between two NaCl salt crystals, then placed into a cell holder in the spectrometer. The concentration of methacrylate carbon-carbon double bonds (C=C) was then determined by obtaining a spectrum from 16 scans over a bandwidth of 850-4000 cm^{-1} at a resolution of 4 cm^{-1}. Polymerized specimens (n=3) that had been stored in sealed polyethylene vials for 24 hours at 37°C were cryoground under liquid

Table 2: *Absorption Peaks*

Group	Wave Number (cm^{-1})
Methacrylate Peak	
C=C	1640
Reference Peak	
Ester	1730
$Aromatic_{11}$	1608
$Aromatic_2$	1582
N-H (urethane)	3350

Table 3: *Analysis of Variance Procedure for Knoop Hardness Values*

Source	Degrees of Freedom	Mean Square	F	P
Composite	17	1140.33	222.41	<.0001
Composite	5	2355.88	459.49	<.0001
Distance	2	2445.95	477.05	<.0001
Composite*Distance	10	271.42	52.94	<.0001
Error	36	5.13		
Corrected Total	53			

Table 4: *Analysis of Variance Procedure for Degree of Conversion Values*

Source	Degrees of Freedom	Mean Square	F	P
Composite	17	669.17	91.08	<.0001
Composite	5	1908.17	259.70	<.0001
Distance	2	709.20	96.52	<.0001
Composite*Distance	10	41.68	5.67	<.0001
Error	36	7.35		
Corrected Total	53			

Table 5: *Mean Knoop Hardness Values (KHN)*

Composite	Depth from Exposed Surface		
	0 mm	2 mm	4 mm
Alert	64.8 (3.7) A	40.6 (1.1) D,E	17.3 (1.0) H,I,J
Solitaire	42.7 (2.3) D	33.9 (0.3) E,F	24.2 (2.9) G,H
SureFil	56.4 (0.6) C	43.2 (0.8) D	31.1 (2.3) F,G
Herculite	57.6 (0.6) C,B	41.3 (0.9) D	18.7 (2.9) H,I
Heliomolar	26.8 (0.4) G	14.6 (0.43) I,J	11.1 (0.8) J
Belleglass	64.4 (2.4) A,B	67.5 (4.9) A	70.3 (3.9) A

**Values with the same letter denote no significant difference*

Table 6: *Degree of Conversion (%)*

Composite	Depth from Exposed Surface		
	0 mm	2 mm	4 mm
Alert	37.9 (1.1) E,D	31.4 (4.6) G,F,E	25.1 (4.4) F,G
Solitaire	47.2 (1.9) C	47.5 (0.7) C	36.0 (0.7) E
SureFil	52.6 (4.9) C	45.6 (3.5) D,C	46.0 (2.0) D,C
Herculite	45.2 (1.3) D,C	47.5 (1.8) C	32.9 (1.8) F,E
Heliomolar	45.9 (1.6) D,C	36.0 (2.4) E	24.5 (2.9) G
Belleglass	78.9 (1.8) A	70.6 (1.1) B	68.5 (3.9) B

**Values with the same letter denote no significant difference*

nitrogen into a fine powder. This technique, when compared to mortar or ball grinding, results in a smaller particle size (average 5 μm), increased transmittance and enhanced absorption peaks (Kerby & others, 1999). Subsequently, 3 mg of resin powder was blended with 60 mg of IR grade potassium bromide in a specimen holder and pressed into a transparent disk using a pellet-maker kit (KBr Port-A-Press, International Crystal Labs, Garfield, NJ, USA). The specimen-holder was then transferred to the spectrometer and spectra were obtained using the same parameters as with the unpolymerized specimens. A comparison was then made between the absorption peak intensities of the C=C methacrylate bonds and various internal reference peaks utilizing standard baseline technique (Table 2) before and after polymerization according to the following formulas:

$$\text{Residual (\% C=C)} = \frac{\text{[Abs (C=C)/Abs (reference peak)] Polymer}}{\text{[Abs (C=C)/Abs (reference peak)] Monomer}}$$

$$\text{Degree of conversion} = 100\% - \text{Residual (90 C=C)}$$

Statistical analysis using two-way ANOVA ($p<0.001$) (Tables 3 and 4) and the Tukey Multiple Comparison Test showed significant differences between several of the composites tested for both Knoop hardness ($\alpha=0.05$) and degree of conversion ($\alpha=0.05$). Significant interaction was shown between the composite material and the depth from the irradiated surface for both Knoop hardness and degree of conversion.

RESULTS

Knoop Hardness

The results of the Knoop hardness test with standard deviations are presented in Table 5, where groups denoted with the same letter are not significantly different from one another. The ANOVA ($p<0.001$) (Table 3) and Tukey Multiple Comparison Test ($p<0.05$) indicated significant differences among several of the composite materials tested (Table 5).

The mean hardness value of the indirect composite Belleglass at 2-mm and 4-mm depth from the irradiated surface and the fiber-reinforced packable

composite Alert at 0 mm were significantly higher than any of the other composites tested. At 4-mm depth, the mean hardness value of SureFil, a packable composite, was greater than any of the other direct placement composites tested. No significant difference was noted between the indirect composite Belleglass and the direct composite Alert at 0-mm depth.

Degree of Conversion

The results of the degree of conversion test with standard deviations are presented in Table 6, where groups denoted with the same letter are not significantly different from one another. The ANOVA ($p<0.001$) (Table 4) and Tukey Multiple Comparison Test ($p<0.05$) indicated significant differences among several of the composite materials tested (Table 6).

The indirect composite, Belleglass, exhibited a significantly greater degree of conversion than any of the other composites tested at all depths from the exposed surface. The mean degree of conversion of the packable composite SureFil was significantly higher than any of the direct composites at 4-mm depth. In addition, no significant difference was noted among the degrees of conversion of SureFil at 0, 2 and 4 mm depth.

The relationship between the degree of conversion and hardness for the materials tested at the various distances from the exposed surface was evaluated using Pearson Correlation Coefficients. A statistically significant correlation ($r^2=.5985$) ($p=.0002$) was observed between the degree of conversion and hardness.

DISCUSSION

In this study, composites with high filler fractions such as Alert (84 wt%), SureFil (82% wt), Belleglass (78 wt%) and Herculite (78 wt%) were more likely to demonstrate a greater degree of conversion and hardness than materials with decreased filler volume fractions, such as Solitaire (66 wt%) and Heliomolar (65 wt%). This is in agreement with previous investigations that have shown materials with large fractions of inorganic fillers exhibiting improved mechanical properties, including high flexural strength, hardness and elastic modulus (St Germain & others, 1985; Braem & others, 1989; Chung & Greener, 1990). In addition, the filler particles in the packable composites are larger than those in the conventional composites (Cobb & others, 2000). Larger filler particles have been associated with increased surface roughness and wear (Suzuki & others, 1995). The conventional composites in this study contained filler particles with an average particle size of 0.04 µm for the microfill composite and 0.6 µm for the hybrid composite. On the other hand, the packable composite Alert contains glass fibers 10-12 µm and Solitaire contains porous SiO_2 particles 2-20 µm. Although incorporation of the higher filler fractions and larger filler particles may help improve some physical properties, such as flexural strength, hardness, polymerization shrinkage and packability, a subsequent effect may be noted on the wear and finishing characteristics of a material (Ryba, Dunn & Murchison, 2002).

This degree of monomer conversion in direct visible light cured composite varies within the bulk of the specimen depending on the distance from the irradiated surface. This is because methacrylate conversion requires light energy for activation and may be affected by filler loading. As filler fraction and mean particle size are increased, light scattering and reflection are also enhanced, resulting in deeper penetration of visible light (Ruyter & Oysaed, 1982a). This may explain the relatively higher degree of conversion of the highly filled (82%) packable composite SureFil at 4-mm depth from the exposed surface. Subsequently, the mean degree of conversion for Heliomolar, a microfill composite, was lower at 4-mm depth than any other materials tested. This is in agreement with a previous study that found a low degree of conversion to be a function of distance for microfill composite (Eliades, Vougiouklakis & Caputo, 1987).

As the thickness of the composite material increases, there is a decrease in the transmission of light (Price, Murphy & Derand, 2000; Price & others, 2002). Previous studies have reported a decrease in hardness (Shortall, Wilson & Harrington, 1995; Pires & others, 1993) and the degree of conversion (Eliades & others, 1987; Nomoto, Uchida & Hirasawa, 1994) as the thickness of the composite increases; therefore, it is generally recommended that increments should not exceed 2.0 mm (Rueggerberg, Caughman & Curtis, 1994; Pilo, Oelgiesser & Cardash, 1999; Choi & others, 2000). Although the guideline of 2 mm for incremental composite build-up is generally agreed upon, some manufacturers of packable composites have claimed polymerization up to 5-mm increments. In this investigation, the mean degree of conversion value for each composite at the irradiated surface was generally greater but not always significant than at the 4-mm depth. The same trend was noted for the mean hardness values with the exception of Belleglass, the indirect composite. The mean hardness values for Belleglass increased, although not significantly, from the irradiated surface to 4-mm depth. Belleglass is light, heat and pressure cured; however, residual oxygen may be present, which could inhibit polymerization in the outer layers of the material. Residual oxygen may remain at the center of the bulk specimen, which was not dissipated under nitrogen atmosphere during bulk polymerization. This may tend to decrease the degree of polymerization at deeper levels.

Belleglass also demonstrated a significantly higher mean hardness value and degree of conversion when compared to the visible light cured direct placement composite materials. Heat curing, in addition to visible

light curing, has been shown to substantially increase the degree of polymerization (Dionysopoulos & Watts, 1989). In addition, Wendt demonstrated a substantial increase in hardness with a five-minute heat treatment after light curing (Wendt, 1987). Highly polymerized methacrylate materials characterized by increased crosslink density may also exhibit greater flexural strength and elastic modulus (Reinhardt & others, 1994).

Previous studies have reported on the correlation between hardness and the remaining unreacted methacrylate groups. A correlation between the increasing degree of conversion and hardness was demonstrated by Asmussen (1982) and Ferracane (1985); however, other investigations were unable to establish a correlation (Chung & Greener, 1990). In this investigation, the relationship between the degree of conversion and hardness for the materials tested at the various distances from the exposed surface was evaluated using Pearson Correlation Coefficients. Although the correlation was statistically significant (r^2=.5985) (p=.0002), other influential factors, such as degree of cross-linking, amount of filler and type of filler may be contributing to the results. There is a certain minimum threshold of cross-linking necessary to obtain a hard material from the gel state during polymerization. Above that minimum threshold, there may be a linear correlation between the degree of conversion and hardness.

Composite materials present the clinician with deficiencies that are often difficult to overcome. Consequently, the clinical success of posterior composite restorations is influenced by case selection (Ferracane, 1992). Several clinical studies have been reported on posterior composite restorations; however, minimal clinical data is available on packable composites (Barnes & others, 1991; Perry & others, 1999; Scheinbenhogen-Fuchsbrunner & others, 1999; Manhart & others, 2000; Wassell & others, 2000; Ernst & others, 2001). In addition, properties such as finishability and wear may be affected by the larger filler particles of packable composites (Perry & others, 1999; Reis & others, 2003). In a two-year study, 90% of direct composite restorations were rated as satisfactory; however, after three years, that percentage dropped to 87% (Scheibenbogen-Fuchsbrunner & others, 1999; Manhart & others, 2000). Another study reported five-year success rates at 90%; however, the success rate dropped to 77% at eight years (Barnes & others, 1991). SureFil packable composite exhibited clinical acceptability in all areas evaluated in a one-year study (Perry & others, 1999). Conversely, a three-year evaluation of Solitaire packable composite revealed a survival rate of only 79% and a fracture rate of 13.9% (Ernst & others, 2001). Overall, when evaluating the annual failure rate for composite, the results are comparable to amalgam except that amalgam studies can extend up to 20 years, whereas, posterior composite may be up to 10 years (Scheinbenhogen-Fuchsbrunner & others, 1999). In a recent survival analysis of posterior restorations, the results demonstrated that composite restorations exhibited a statistically greater chance of failing and therefore may not last as long as amalgam (Bogacki & others, 2002). More long-term clinical studies are necessary to evaluate the long-term durability of these materials.

CONCLUSIONS

Within the limits of this study, the following conclusions were drawn:

1. The indirect laboratory-processed composite Belleglass exhibited the highest mean values for both hardness and degree of conversion.
2. At 4-mm depth, the mean hardness value and degree of conversion of SureFil, a packable composite, was greater than any of the other direct placement composites tested.
3. No significant difference was noted between the degrees of conversion of SureFil at 0, 2 and 4-mm depth.
4. Lower mean degree of conversion values were found with the glass fiber reinforced packable composite Alert when compared to the other packable composites tested.
5. At 0-mm depth from the irradiated surface, the mean hardness values for Heliomolar, the microfilled urethane-based composite, were significantly lower than any of the other composites tested.

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Exploring the Nature of Acid-Resistant Hybrid Layer with Wet Bonding

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Clinical Relevance

The quality of the hybrid layer in the dentin/adhesive interface may be overestimated due to popular specimen preparation techniques that include polishing and acid-bleach treatment.

SUMMARY

The nature of the hybrid layer (HL) following chemical treatment has not been resolved. The effects of chemical treatments and polishing on HL morphology and composition were investigated using micro-Raman spectroscopy and SEM. Polished and unpolished SingleBond adhesive/dentin (a/d) interfaces from the same tooth were randomly selected for one of the following treatments: a) 5N HCl followed by 5%NaOCl; b) 5%NaOCl; c) 5%NaOCl and sonication; d) 5%NaOCl followed by 5N HCl. The results indicated that the HCl-NaOCl treatment could obscure the "true" HL in the unpolished a/d interfaces that were actually porous. The composition and morphology of the polished interface specimens were largely unchanged by the above treatments. Descriptions of HL quality are commonly based on the acid reactivity of polished specimens. The results of this study indicate that polishing dramatically alters the native composition, increasing its acid-resistance, and thus, this procedure could lead to false conclusions about HL quality.

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INTRODUCTION

The most popular techniques for determining the quality of the hybrid layer (HL) have relied on the morphologic characterization of this layer before and after acid-bleach treatments. Using these techniques, the existence of smooth, acid-resistant layers has been consistently reported for most of the adhesive systems (Nakabayashi, Kojima & Masuhara, 1982; Nakabayashi & Takarada, 1992; Perdigão & others, 1996; Vargas, Cobb & Armstrong, 1997; Yoshiyama & others, 1995). With these methods, the zone of demineralized dentin has been reported to be completely sealed by adhesives (Nakabayashi, 1992; Tay & others, 1994). In contrast, other studies using similar adhesives have suggested that the hybrid or a/d interdiffusion layer is likely to be complex, and this zone is not completely infiltrated by resin, leaving a potentially weak, multi-zoned structure (Li, Burrow & Tyas, 2000; Sano & others, 1995). Novel histomorphologic comparison of the optimum hybrid and a/d interface specimens suggests very poor adhesive encapsulation of the interfacial demineralized dentin collagen (Wang & Spencer, 2003). In contrast to an adhesive seal at the interface, micro-Raman spectral

analyses indicate a gradual decrease in adhesive penetration across the demineralized dentin (Spencer & others, 2000; Wang & Spencer, 2002a).

To understand these conflicting results, the impact of SEM preparatory procedures on the nature of the a/d interface must be carefully re-evaluated. Previous authors have criticized acid treatment of the a/d interface prior to scanning electron microscopic (SEM) analysis (Van Meerbeek & others, 2000), claiming that acid treatment dissolves both the mineral within the HL and the underlying "unaffected" dentin, inhibiting any conclusion about the completeness of resin infiltration. Polishing obscured the HL porosity that was clearly evident in fractured specimens (Carvalho & others, 1995). Although polishing is still commonly used in SEM investigations of the a/d interface, the nature of the acid-resistant layer and the effect of polishing on the morphology following these chemical treatments has not been studied. By using specimens from the same tooth, the effect of chemical treatments on the morphology and compositional changes in the a/d interfaces and differences in the morphology of polished and unpolished a/d interfaces after these same chemical treatments were studied using SEM and micro-Raman techniques. The two null hypotheses are that the appearance of the acid-resistant layer is real (1) and polishing has no effect on the SEM morphology of the chemically-treated a/d interface (2).

METHODS AND MATERIALS

Specimen Preparation

Six extracted, unerupted human third molars stored at 4°C in 0.9% w/v NaCl containing 0.002% sodium azide were used in this study. The teeth were collected after the patients' informed consent was obtained under a protocol approved by the UMKC adult health sciences IRB. The specimen preparation has been detailed in previous publications (Spencer & others, 2000). In brief, the occlusal one-third of the crown was removed by means of a water-cooled low-speed diamond saw (Buehler Ltd, Lake Bluff, IL, USA). A smear layer was created by abrading the dentin with 600 grit SiC under water. Six prepared dentin specimens were selected for treatment with Single Bond adhesive (3M, St Paul, MN, USA) using the wet bonding technique according to manufacturers' instructions. The adhesive was polymerized for 30 seconds using visible light. The treated dentin surfaces were sectioned perpendicular and parallel to the bonded surface. This provided a total of eight slabs with the following approximate dimensions: 1.5-mm width x 2-mm height x 5-mm length. Thus, there were eight slabs per tooth. Four slabs per tooth were polished with a series of silicon carbide papers (#600, #1000), then highly polished with microcloth impregnated with a water slurry of 1 µm and 0.05 µm alumina particles. The other four slabs/tooth were not polished. The surfaces of both the polished and unpolished specimens from the same tooth were treated, respectively, according to the following protocol: 1) 30 seconds 5N HCl followed by 30 minutes 5%NaOCl; 2) 30 minutes 5%NaOCl; 3) 30 minutes 5%NaOCl and one minute sonication; 4) 30 minutes 5%NaOCl followed by 30 seconds 5N HCl. Following rinsing, each specimen was analyzed with micro-Raman spectroscopy and SEM. Because micro-Raman is a non-destructive technique, the specimen was first analyzed using micro-Raman spectroscopy and the same specimen was then analyzed using SEM.

Micro-Raman Spectroscopy

As described above, separate slabs with/without polishing from the same tooth after different chemical treatments were prepared for investigation using micro-Raman spectroscopy. Since the micro-Raman spectroscopic technique is non-destructive, these same specimens were available for analysis using SEM. The micro-Raman spectrometer consisted of an argon ion laser beam (514.5 nm) focused through a 60x Olympus Plan Neofluor water-immersion objective (NA 1.2) to a ~1.5 µm beam diameter. Two consecutive scans of spectra (with 60 seconds accumulation time each) were obtained from each site. The laser power was approximately 3mW. Spectra were Raman shift frequency-calibrated using known lines of neon and silicon.

Scanning Electron Microscopy

Following micro-Raman analysis, the specimens described above were prepared for SEM examination. After drying, the prepared specimens were mounted on aluminum stubs and sputter coated with ~20 nm of gold-palladium. The specimens were examined at a variety of magnifications and tilt angles in a Philips XL30 ESEM-FEG (Philips Inc, Eindhoven, Netherlands) at 10kV.

RESULTS

Figure 1 presents representative SEM micrographs of the SB adhesive/dentin interfaces from the same tooth, which were cross-sectioned without metallo-graphical post-polishing. In Figure 1A, the sectioned interface specimen was treated with 5N HCl (30 seconds), followed by 5%NaOCl (30 minutes) and sonicated for one minute. Using this exposure technique, the HL appears resistant to the effects of acid/bleach treatments. Note the well-defined acid-resistant layer, the smooth surface of this layer and the funnel-shaped resin tags. In Figure 1B, the a/d interface from the same slab was only treated with 5%NaOCl (30 minutes). The surface of HL is irregular, with a very rough, granular and porous appearance, with sites of incomplete adhesive penetration readily identified in the micrograph. As shown in Figure 1B, voids at the a/d interface represent sites where collagen was removed

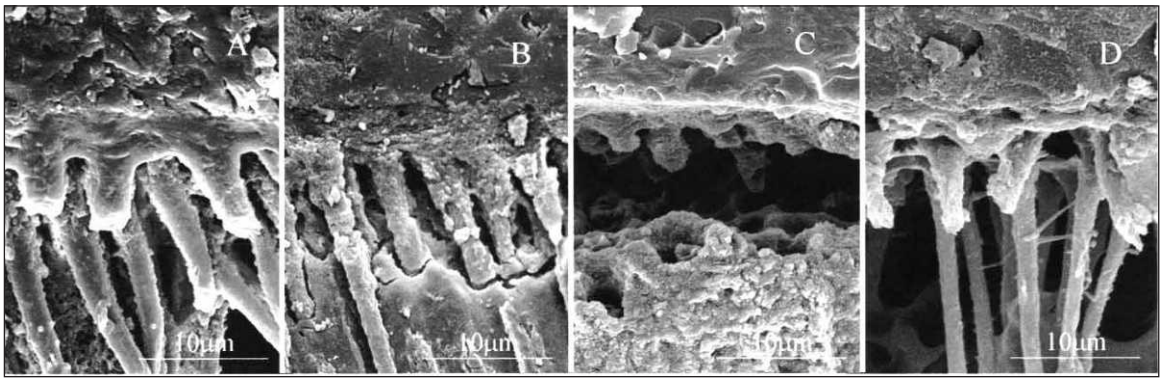

Figure 1: *Representative scanning electron micrographs of the SB adhesive/dentin interfaces of unpolished cross-sections from the same tooth with different treatments. A) the interface was treated with 5N HCl for 30 seconds followed by 5% NaOCl for 30 minutes; B) treated with 5% NaOCl for 30 minutes; C) treated with 5% NaOCl for 30 minutes and sonicated for one minute in an ultrasonic device(Ultrasonic Cleaners, Branson Model B-22-4); D) treated with 5% NaOCl for 30 minutes followed by 5N HCl for 30 seconds.*

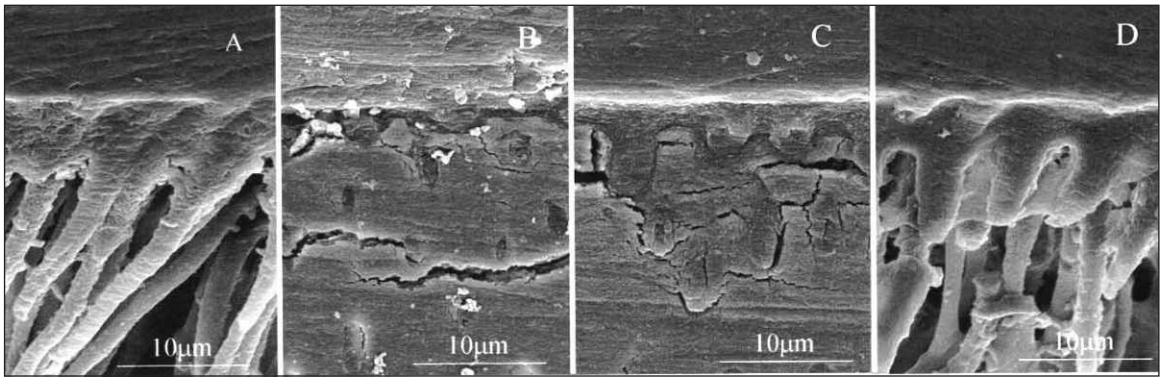

Figure 2: *Representative scanning electron micrographs of the SB adhesive/dentin interfaces of polished cross-sections from the same tooth as the sample in Figure 1. A,B,C and D are same as those in Figure 1.*

following exposure of the specimen to 5%NaOCl. More interestingly, if the interface specimen in Figure 1B was sonicated for one minute, the HL was lost and the resin tags fractured (Figure 1C). The large interfacial void and fracture of the resin tags suggest that the structural integrity of the HL and adhesive tags is not adequate to resist even gentle vibratory forces. In Figure 1D, the bonded interface was first treated with 5%NaOCl (30 minutes), followed by 5N HCl (30 seconds). This switch in the sequence of treatments produced dramatic differences in the morphology as compared to Figure 1A. The HL was susceptible to the effect of bleach-acid treatment and hardly visible in Figure 1D, with the diameter of the resin tags being much smaller. In combination, these results indicate that exposed collagen fibers were not protected by resin, otherwise, the sequence of treatments should not effect the morphology.

Figure 2 presents representative SEM micrographs of the SB adhesive/dentin polished specimen from the same tooth as the sample in Figure 1. The specimen has been highly polished to create a flat surface. The same chemical treatment procedures for unpolished specimens were performed on these polished specimens. Following polishing, the morphology is dramatically different from that of unpolished specimens after the same chemical treatments. The SEM micrograph of a polished interface treated with 5N HCl (30 seconds) followed by 5%NaOCl (30 minutes) revealed a relatively thick HL that was filled with an acid/bleach-resistant resin (Figure 2A). In this figure, the HL appears to provide a continuum of material connecting the bulk adhesive with the subjacent dentin. The effect of polishing on the appearance of the SEM micrographs was clearly observed in the interface treated only with bleach (Figure 2B) and/or bleach-acid treatments (Figure 2D). As compared with the unpolished interface (Figure 1B), there is no granular, porous appearance in the polished inter-face (Figure 2B). Following sonication, only the polishing debris left on the surface was removed, and the interface of the HL appears very smooth (Figure 2C). Compared to the differences in Figures 1A and 1D, there is minimal difference in the SEM appearance when the sequence of chemical treatments is reversed (Figures 2A and 2D). The above results indicate that polishing dramatically affects the SEM morphology of a/d interface specimens.

The corresponding Raman spectra of the above a/d interfaces with/without polishing after different chemical treatments is shown in Figure 3. All spectra were recorded at the same position, which is 2 microns below the a/d interfaces. The bands associated with the adhesive occur at 1720cm^{-1} (carbonyl), 1609 cm^{-1}

(phenyl C=C), 1454 cm^{-1} (CH_2 def), 1187 cm^{-1} (gem-dimethyl) and 1113 cm^{-1} (C-O-C), while the bands associated with collagen occur at 1667 cm^{-1} (amide I), 1454 cm^{-1} (CH_2 def) and 1245 cm^{-1} (amide III). The band at 961 cm^{-1} (PO_4^{3-}) is associated with mineral. Figure 3A represents a spectrum recorded from the unpolished interface without any treatment. Bands associated with both adhesive and collagen were noted in this spectrum. Following the different treatments (polishing and chemicals), major spectral changes (marked with arrows) were readily seen, especially in the region of 1780-1500 cm^{-1}, indicating changes in composition and structure of the a/d interface following the different treatments. By comparing the spectra of the unpolished interfaces following the different treatments (Figures 3A, B and D), the relative intensity of the Raman bands associated with collagen (amide I at 1667cm^{-1}) decreased substantially, indicating dentin collagen in the interface has been exposed and removed by these treatments. Interestingly, bleach-only treatment removed most of the collagen in the interface (Figure 3D), however, HCl-bleach treatment did not remove all the collagen (Figure 3B). These differences may be related to the effect of acid on the interface. These results are consistent with the SEM observations shown in Figure 1.

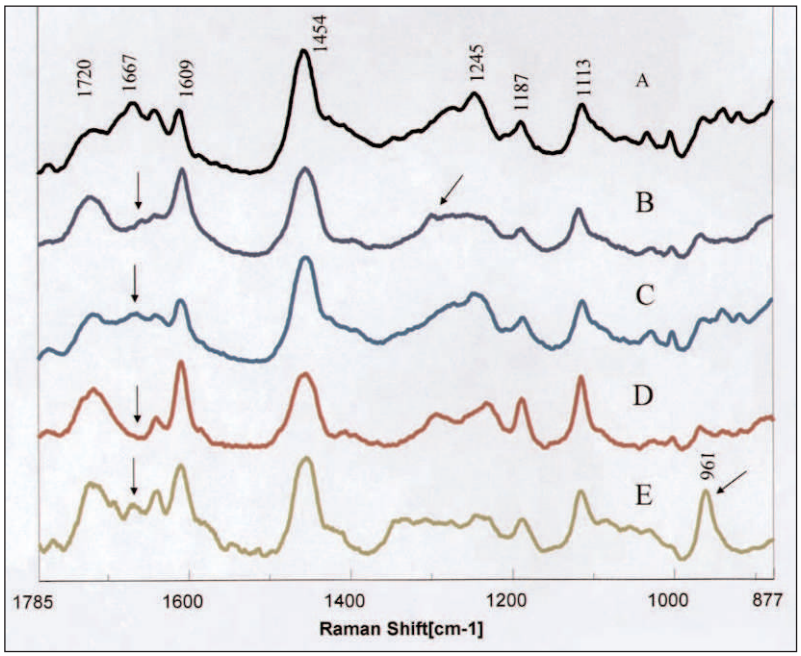

Figure 3: *Comparison of Raman spectra of SB/dentin interfaces w/o polishing after different treatments. Bands at 1720 cm^{-1}:carbonyl, 1609 cm^{-1}:phenyl C=C, 1113 cm^{-1}: C-O-C are associated with the adhesive and bands at 1667 cm^{-1}: amide I, 1245 cm^{-1}: amide III) are associated with dentin collagen. (A) unpolished, without treatment; (B) unpolished, treated with HCl-NaOCl; (C) polished, treated with HCl-NaOCl; (D) unpolished, treated with NaOCl; (E) polished, treated with NaOCl. All spectra were acquired at 2nd micron below the a/d interfaces.1A,B,C and D are same as those in Figure 1.*

There are dramatic differences between the unpolished and polished specimens in the spectra following the same treatments. After the HCl-bleach treatment, there is almost no change in the spectrum of the polished interface (Figure 3C) as compared to Figure 3A. In comparison, there is a tremendous decrease in the intensity of the collagen bands in the unpolished interface (Figure 3B), indicating that polishing the specimen protects the collagen from being removed. Similarly, the collagen was almost totally removed in the bleach-only treated unpolished interface (Figure 3D), but the collagen was not removed in the polished interface (Figure 3E). It was also noticed that there was a strong band at 961cm^{-1}, associated with dentin mineral in Figure 3E. This may be evidence that polishing the specimen could inadvertently smear the interface with debris from dentin and/or resin matrix and force this material into any porosities that originally existed at the interface.

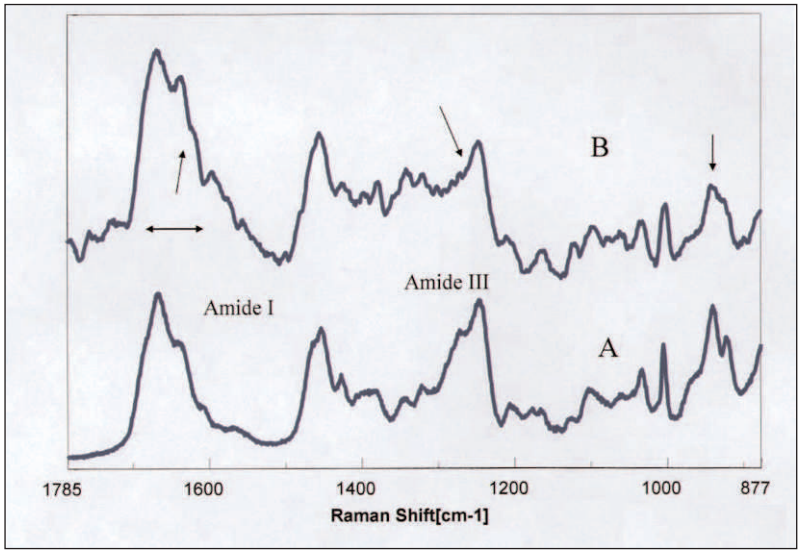

Figure 4: *Raman spectra of dentin collagen before (A) and after (B) treated with 5N HCl for 30 seconds. The relative intensities of the amide III and backbone decreased in the spectrum of HCL treated collagen, substantial broadening of the features associated with amide I, III and C-C backbone at 938 cm^{-1} was noted.*

Figure 4 shows the Raman spectra of dentin collagen before (A) and after (B) treatment with 5N HCl for 30 seconds. The major spectral changes have been marked with arrows. These changes suggest the loss of fine structure, increased disorder and the denaturing of dentin collagen after 5N HCl treatment (Wang & Spencer, 2002b).

DISCUSSION

A number of interface preparatory techniques have been utilized in SEM characterization studies of the HL. The formation of an HL was first demonstrated by Nakabayashi using highly concentrated HCl (Nakabayashi & others, 1982). Today, this acid treatment technique is commonly used in combination with NaOCl to identify the quality of the HL. As shown in Figure 1A, using this protocol, the quality of the HL appears ideal. However, when the a/d interface from the same slab was treated with NaOCl and sonicated for one minute, a large interfacial void was observed (Figure 1C), suggesting that without the acid treatment, the HL is severely damaged and disrupted by the NaOCl treatment. In corroboration with our micro-Raman data (Figures 3B, D and Figure 4), the differences in Figures 1A-D suggest that the acid treatment denatures the perturbed collagen (see Figure 4) such that it forms a gelatinous matrix that is resistant to removal with NaOCl (Figures 3B and D). In corroboration, these results indicate that the a/d interface is actually very porous; the exposed collagen that was easily removed by the bleach-only treatment was not protected by the resin (Figure 3D). Our ability to disrupt the resin in the bleach-only treated a/d interface using relatively gentle vibratory forces also points to a very porous adhesive phase.

One reason for the porous adhesive is the physical "oil and water" separation as the adhesive mixes with water in the demineralized dentin matrix during wet bonding (Spencer & Wang, 2002). This leads to partitioning of the adhesive components into hydrophobic BisGMA-rich and hydrophilic HEMA-rich phases. Such partitioning would compromise the structural integrity of the resultant HL. As shown in Figures 1B-D, this adhesive did not encapsulate the collagen fibrils, nor did it form a continuous uniform phase in the interface. Based on the above results, the combination of the HCl and NaOCl treatment could obscure the "true" HL in these unpolished samples. The effect of this treatment could be profound if the interfaces were actually very porous. Collagen in the dentin matrix may be perturbed by the heat generated during smear layer preparation and the subsequent acid pre-treatment. Further exposure to acid, as described in the technique for distinguishing the HL, denatured this disorganized collagen, converting it into a gelatinous matrix that resists treatment by sodium hypochlorite. Under these conditions, the gelatinous matrix would provide support to a porous HL. The differences with and without the HCl treatment of a porous HL are clearly evident in the SEM pictures (Figure 1).

Polishing bonded interfaces prior to SEM observation is a technique frequently used to improve the quality of the morphological analysis. Although previous authors have reported that the polishing technique could burnish material into porosities, thus, masking the a/d interface (Carvalho & others, 1995), the above chemical treatment protocol on polished interfaces continues to be a common practice. The appearance of the image and the existence of an acid-resistant layer have not been questioned, because it was thought that any protein or mineral within the HL that is not protected by resin would be removed using this aggressive acid-bleach treatment. The effect of polishing on SEM morphology following these chemical treatments was ignored and not studied. In this project, by using the specimens from the same tooth, the differences in morphology of polished and unpolished interfaces after the same chemical treatments were clearly noted (Figures 1 and 2). The compositional changes in the interfaces following different treatments were also determined by micro-Raman spectroscopy (Figure 3). It was shown that polishing the a/d interface has a dramatic influence on the SEM morphology. The polishing procedure could induce artifacts by destroying soft dentin collagen and smearing the polishing debris (mineral debris as shown in Figure 3E) across the interface, thus obliterating the actual interfacial structure. Acid treatment could remove the mineral debris in the interface caused by polishing (Figures 3C, E). However, the influence of polishing on morphology and composition of the interface still dominates and exists even after aggressive chemical treatments, which could lead to false conclusions about hybridization efficiency.

In a recent review of morphologic techniques used to evaluate the HL, the authors also described other disadvantages of this interface-preparation method (Van Meerbeek & others, 2000). Treatment with strong acid dissolves both the mineral within the HL that is not protected by resin and the underlying "unaffected" dentin, inhibiting any conclusion about the completeness of resin infiltration. In spite of this, other current methods also destroy the a/d interface in the process, which prohibits evaluation of this region using complementary analytical techniques. Since the hybrid zone consists of a complex composite structure with morphology and properties that are highly sensitive to both the demineralization process and specific characteristics of the bonding adhesive system, in this research protocol, the same interface specimens were used. These same interface slabs were first microtomed for the preparation of thin sections for the novel staining technique (Spencer & Swafford, 1999; Wang & Spencer, 2003) and FTIR imaging, then, the same slabs were analyzed by high resolution micro-Raman and SAM techniques (Katz & others, 2001) that allow direct, non-destructive *in situ* detection of chemical structure and micro-mechanics, after which they were observed by FE-SEM. Direct and comprehensive information regarding the quality of the interface could be obtained.

CONCLUSIONS

In this study, by using the same interface specimens, the effect of chemical treatments and polishing on the morphology and composition of the a/d interface was clearly demonstrated.

The problem associated with this commonly used HCl-bleach technique was explored using complementary spectroscopic and morphologic analyses. It was shown that this preparation technique in combination with the polishing procedure could alter the native interface composition, increasing its acid-resistance, thus, giving false conclusions about HL quality. Both null hypotheses were rejected. The results are very important, since this procedure is one of the first and most widely used tools to evaluate the quality of the HL. Based on these results, this exposure technique primarily provides evidence of resin infiltration into the demineralized layer; however, it cannot directly define the quality of this layer. This exposure technique and polishing procedure must be used with great caution.

Acknowledgement

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Influence of Different Composite Restoration Techniques on Cuspal Deflection: An *In Vitro* Study

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MA Vilchez-Díaz • C de Haro-Muñoz

Clinical Relevance

The utilization of a transparent plastic cone does not reduce cuspal deflection induced by polymerization shrinkage compared with conventional horizontal increment techniques.

SUMMARY

Cuspal deflection produced by polymerization shrinkage was measured after using different composite restoration techniques. This study included 30 healthy premolars embedded in acrylic resin connected to a system that simulated intrapulpal pressure. A small ball was attached to each cuspal vertex as a reference point for intercuspal distance measurements. A large mesio-occlusal cavity was cut in each premolar. All premolars were treated with the same adhesive (ScotchBond) and composite (Tetric Ceram). The teeth were randomly distributed among three study groups: Group 1, filled with two horizontal increments; Group 2, filled with two horizontal increments, the first up to half the cavity height and light cured using a transparent plastic cone (Cerana), and the second filling the remainder of the cavity and Group 3, filled in the same way as Group 2, except that the first increment only filled one-third of the cavity height. The intercuspal distance was measured before beginning the restoration and immediately after polymerization of the first and second increments. Under the experimental conditions used, none of the filling techniques utilized avoided the cuspal deflection phenomenon. Polymerization of the final increment, which binds occlusal enamel in the buccal-lingual plane, was the main cause of cuspal deflection and produced a statistically significant reduction in intercuspal distance vs the baseline measurement in the three study groups. The global deflection ranged from 4 µm to 6 µm, depending on the filling technique used, although the differences among techniques did not reach statistical significance ($p<0.05$).

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INTRODUCTION

Currently available composites offer excellent physical and mechanical properties. The greatest shortcoming of these materials remains the shrinkage (2.7–7-1% of volume, [Feilzer, de Gee & Davidson, 1988]) inherent to the polymerization reaction.

Polymerization shrinkage can produce two types of problems. When the filling material is weakly adhered to the dental tissues, a detachment of the enamel margins (and consequently, microfiltration) is produced and/or gaps can be formed in the dentin responsible for postoperative hypersensitivity. In contrast, if the adhesive strength exceeds the contraction stress, there is no detachment but the restoration maintains an internal tension that pulls the walls of the tooth together, reducing the intercuspal distance (cuspal deflection). Cuspal deflection may clinically manifest as changes in occlusion points, with the consequent onset of postoperative discomfort and may, in some cases, produce tooth fractures.

The degree of cuspal deflection ranges from 18 μm to 5 μm (Suliman, Boyer & Lakes, 1993), depending on the following factors: size and depth of the restoration (Meredith & Setchell, 1997), cavity shape, Young's module of the composite (Ausiello & others, 2001), the system of polymerization (auto or light- cured), intensity of the polymerization lamp and light-curing mode (Abbas & others, 2003), utilization of matrix in a retainer (Meredith & Setchell, 1997) and composite placement technique.

One of the methods proposed to minimize cuspid deflection is the combined use of glass ionomer cements and resin composites, which can reduce deflection by >55% (McCullock & Smith, 1986). Applying the filling in increments has also been recommended. Thus, filling in buccolingual increments produced a significantly reduced deflection compared with filling with gingivo-occlusal increments or in bulk technique (Segura & Donly, 1993). However, according to other studies (Abbas, & others, 2003), although filling in increments yields better microleakage results, it also gives rise to greater cuspid deflection compared with the bulk technique.

More recently, using flowable composites as liners has been proposed, because they have a low module of elasticity and can absorb the stress generated during polymerization (Alomari, Reinhardt & Boyer, 2001). Regarding composites with a low elasticity, it must be taken into account that they cannot always be used in stress-bearing areas (Ausiello, Apicella & Davidson, 2002). Furthermore, in restored teeth stressed by occlusal loading, although cusp displacement was higher with more rigid composites, recovery of the initial distance was lesser with flexible composites (Ausiello & others, 2001).

Another approach to minimize cusp deflection is the use of a cone-shaped transparent device (Ericson & others, 1994). It was hypothesized that insertion of the light-tip into the restorative resin during polymerization will alter the magnitude and direction of the contraction forces.

This study was designed to determine the magnitude of cuspal deflection caused by polymerization shrinkage in large mesio-occlusal (MO) cavities using different variants of the incremental technique (horizontal increments or utilization of a transparent plastic cone).

METHODS AND MATERIALS

The authors used 30 healthy premolars without cracks or fissures extracted during orthodontic treatment. They were preserved in physiologic saline with 5% chlorhexidine until their handling. All were embedded in individual molds of acrylic resin that covered the root up to 1-mm below the cemento-enamel junction. The samples were connected to a simulated pulpal pressure system using a previously described method (de Haro-Gasquet, 1996; Lucena-Martín & others, 1999), maintaining the tooth in realistic pressure and humidity conditions throughout the preparation and measurement period (Figure 1). Adhesive techniques were then used to fix a 1.5-mm diameter glass ball to each cuspal vertex as reference points for intercuspal distance measurements.

A large mesio-occlusal (MO) cavity was then cut into each tooth. The dimensions were always approximately the same: a buccal-lingual width of 3 mm at the occlusal box level, ensuring that the cuspal enamel remained supported by dentin. The gingival floor of the proximal box was 0.5 mm above the cemento-enamel junction and had a buccal-lingual width of 3.5 mm. The interior limit of the cavity was the distal marginal border, leaving no dentin remnant between the mesial proximal box and the pulpal floor (Figure 2).

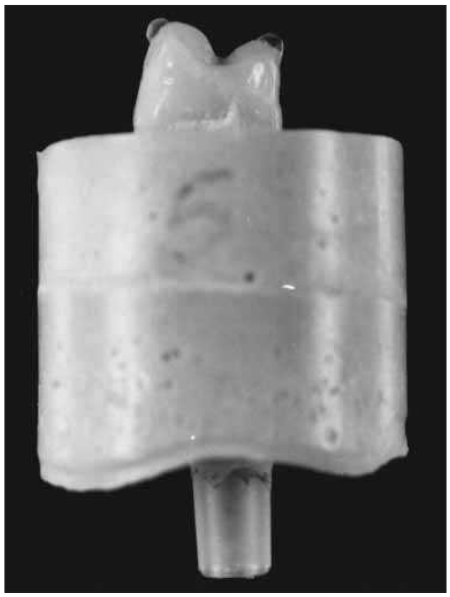

Figure 1: *Specimen prepared for connection to the simulated pulpal pressure system.*

The distance between reference balls was then measured

using a precision micrometer (Mitutoyo, 293- 561, Kanagawa, Japan) and recorded in the data collection tables as the "Initial distance" (D_i).

The teeth were randomly assigned to one of three groups (n=10). In all cases, the matrix band was placed without using a retainer to avoid any tension on the cusps. All cavities were treated with 37% orthophosphoric acid for 15 seconds; ScotchBond adhesive (3M Dental Products, St Paul, MN, USA) was then applied following the manufacturer's instructions. The composite used for the filling was the same for all teeth (Tetric ceram, Ivoclar, Schaan, Liechtenstein; color B3, batch number #922794). The only variable introduced was the technique used for placing the composite. In Group 1, the cavities were filled with horizontal layers in two increments: the first to one-half the cavity height and the second completing the restoration (Figure 3a). In Group 2, the first increment reached approximately half the cavity height and a transparent plastic cone was placed in the base of the proximal box (Cerana, Nordiska Dental AB Box 911, S-251 09) so that it displaced the restoration material against the cavity walls, where it remained adhered to the enamel and dentin (Figure 3b). Once light-cured, the cone was withdrawn and the remainder of the cavity was filled with the second increment of composite.

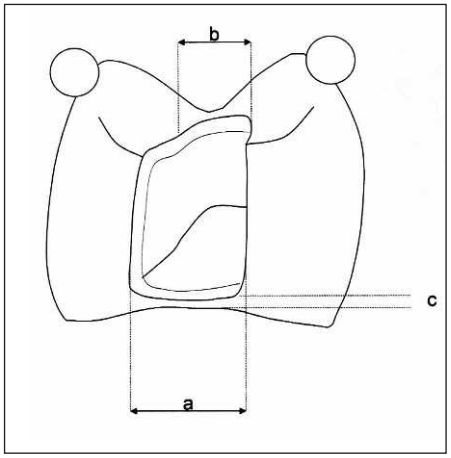

Figure 2: *Graphical representation of the cavity design: (a) bucco-lingual width: 3.5 mm (b) occlusal width: 3 mm. Gingival wall was 0.5 mm above the cemento-enamel junction; axial wall was not cut.*

In Group 3 (Figure 3c), after filling the cavity to one-third of its height with composite, the cone was placed deeply, displacing the composite against the walls so that it only covered the dentin and corresponding proximal enamel. The composite was light-cured, the cone was withdrawn and the remainder of the cavity was filled. The same photoactivation protocol was followed in all experiments: each increment was light-cured for 40 seconds with a standard halogen lamp (Optilux 401, Demetron Research Corp, Danbury, CT, USA) after confirming an output intensity >400 mW/cm^2 with a radiometer (Cure Rite, Efos Inc, Mississawga, Canada).

Cuspal displacement in the groups was determined after polymerization of the first increment (distance recorded as D_1), and again upon completion of the restoration (distance recorded as D_f).

For the global comparison among the three measurements (D_i, D_1, D_f) for each filling technique, the Friedman global test was used. After finding significant differences among the three groups, paired comparisons were made with the Mann-Whitney test. The global comparison among the study groups for the three measurements (D_i, D_1, D_f) was carried out using the Kruskal-Wallis test, $p<0.05$ was considered significant.

RESULTS

The description of the study groups and their comparisons is shown in Table 1. The first increment of composite produced a statistically significant reduction in intercuspal distance versus the initial distance in Group 1 ($p<0.05$), but there were no significant changes in this measurement in Groups 2 or 3.

Polymerization of the second increment produced a statistically significant reduction in all three groups with respect to the distance after the first increment and the baseline distance.

In the global comparisons among the three study groups, no significant differences were found in initial distance (D_i), distance after first increment (D_1) or final distance (D_f).

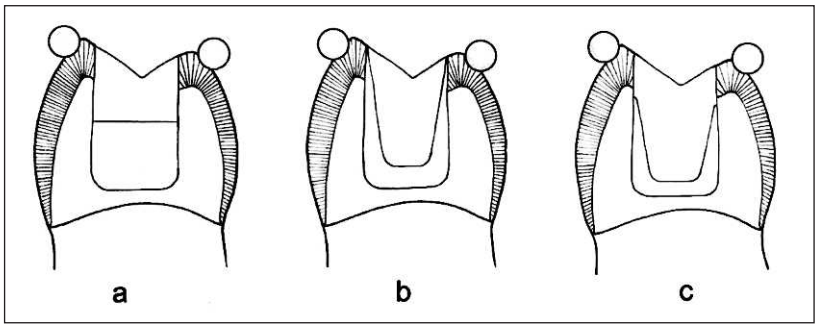

Figure 3: *Schematic representation of study groups. (a) first horizontal increment reaches half the cavity height; (b) first increment, applying the light cone so as to displace the composite against cavity walls where it remained adhered to enamel and dentin; (c) first increment, applying the light cone so as to displace the composite against cavity walls, covering only the dentin and corresponding proximal enamel.*

DISCUSSION

A restoration is designed to achieve the anatomic and functional restitution of the tooth. Because contraction stress can cause adhesion failures at the interface between the tooth and the restoration material, easily applicable filling techniques that minimize these effects are required. The authors investigated the influence of filling technique on cuspal displacement secondary to polymerization shrinkage.

Table 1: *Description and Comparison of Study Groups*

Technique	n	D_i, (X ± DS) (μ)	Δ_1 (X ± DS) (μ)	Δ_2 (X ± DS) (μ)	Paired Comparisons (Mann-WhitneyTest)[c]
Group 1	10	9742.4 ± 678.2	-0.9 ± 1.2	-6.3 ± 1.8	$\Delta1, \Delta2 \neq 0; \Delta1 \neq \Delta2$
Group 2	10	9841.8 ± 776.1	-0.6 ± 1.3	-4.0 ± 3.2	$\Delta2 \neq 0; \Delta1 \neq \Delta2$
Group 3	10	9636.3 ± 567.7	-0.5 ± 0.7	-5.4 ± 1.6	$\Delta2 \neq 0; \Delta1 \neq \Delta2$
Global comparison (Kruskal-Wallis test)		H= 0.477 *p*= 0.788	H=0.815 *p*= 0.665	H= 2.703 *p*= 0.259	

$\Delta_1 = D_1 - D_i$

$\Delta_2 = D_f - D_i$

[c]*Statistically different pairs are indicated (p<0.05). The comparison by pairs was performed after global test (Friedman) showed significant differences among the measurements (D_i, D_1 and D_f) in the three techniques.*

All experiment groups in this study showed a reduction in intercuspal distance, both after polymerization of the first increment of composite and after completion of the restoration. Nevertheless, the values of this displacement were lower than previously reported (Suliman & others, 1993; Causton, Miller & Sefton, 1985), probably because of differences in experimental design. The degree of cuspal deflection is known to be directly related to the amount of dental structure lost, given that this loss causes a reduction in the resistance of the tooth (McCullock & Smith, 1986; Blaser & others, 1983) and requires an increase in the volume of composite required for the filling, thus producing greater contraction forces (Suliman & others, 1993).

In this context, previous studies (Pearson & Hegarty, 1989) showed that the sequential loss of tooth structure significantly increases the cuspal deflection of teeth subjected to loads (Panitvisai & Messer, 1995). Thus, intact premolars subjected to an occlusal load of 100 N show scarcely perceptible cuspal movements. The loss of margin (mesial or distal) produces small increases in cuspal deflections of 2 mm to 5 mm, and cuspid deflection in cavities for endodontic access is two-fold in MOD cavities compared with MO cavities.

In this study, the intercuspal distance was measured immediately after polymerization, and it has been demonstrated that maximum displacement occurs within the first 15 minutes after polymerization (Suliman & others, 1993), although displacement can continue for up to 48 hours later (Douglas, 1985). Moreover, the application of pulpal pressure ensured hydration of the tooth throughout the experiment. These conditions may explain the lesser deflection compared to other authors' findings.

The deflection produced after polymerization of the first increment only reached statistical significance in Group 1, where the filling was applied in horizontal increments. In Groups 2 and 3, insertion of the light cone displaced the filling material towards the walls, increasing the free surface area of composite that would facilitate the release of contraction stress through deformation of this surface (Figure 4).

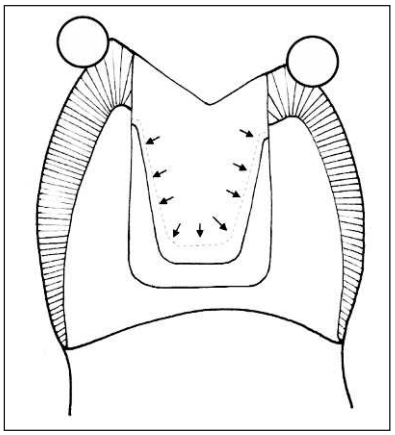

Figure 4: *Graphical representation of the direction of the contraction when light cones are used. The reduction in volume is due to deformation of the free surface.*

Polymerization of the second increment produced a greater reduction in intercuspal distance compared with the first increment, and the deflection (4 μm to 6 μm, depending on the technique used) was statistically significant in all three groups. This result could be expected, because the free surface area of composite is smaller. Furthermore, because of the loss of dentin above the chamber, which acts as a buttress between the buccal and lingual cusps, the increase in composite that binds the enamel in a vestibular-lingual plane might have produced greater deflection.

Global comparisons among the groups (Kruskal-Wallis test) showed no significant differences in the intercuspal distance in the cavitated tooth, indicating homogeneity of the cavity dimensions among the samples. Comparisons among the groups after polymerization of the first and final increments show that the magnitude of the deflection was independent of the filling technique used. This finding contrasts a report by Ericson and others (1994), whose use of the Light-tip light cone reduced cuspal deflection by 24% compared with conventional light-curing.

None of the filling tests evaluated in this study avoided cuspal deflection. In principle, a gradual relaxation of internal stress may be produced by the hygroscopic expansion of the composite through its exposure to the

oral medium (Suliman & others, 1993; Feilzer, de Gee & Davidson, 1990; Alomari & others, 2001), thus contributing to a recovery of the initial situation. The design of this experiment did not allow the authors to evaluate the long-term effects. However, some studies (Suliman & others, 1993; Segura & Donly, 1993) have shown that the total or near-total recovery of the initial intercuspal distance is a slow process that may last for up to two weeks and is never complete in medium-size and large restorations.

CONCLUSIONS

Under the conditions of this experiment, none of the filling techniques studied, including the use of a transparent plastic cone, reduced the cuspal deflection induced by polymerization shrinkage compared with conventional horizontal increment techniques.

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Effects of Artificial Saliva and APF Gel on the Surface Roughness of Newer Glass Ionomer Cements

H-K Yip • WM To • RJ Smales

Clinical Relevance

An acidic oral environment may adversely affect the long-term clinical performance of newer viscous glass ionomer cements.

SUMMARY

Objectives: To investigate the surface roughness changes of newer esthetic dental restorative materials with aging and acid erosion in a simulated oral environment. Methods: The materials included two viscous conventional glass ionomer cements originally marketed for the ART approach, one resin-modified glass ionomer cement and two resin composites. Ten specimens for each material were prepared according to the manufacturers' instructions, then each specimen was immersed in 2 ml of buffered artificial saliva at 37°C for three weeks. For each material, five specimens (Group B) were then coated with 1.23% acidulated phosphate fluoride (APF) gel for four minutes, rinsed and immersed again in artificial saliva for another three weeks. Gel was not applied to the Group A specimens. For each material, the surface roughness of an additional three fresh specimens and those from Groups A and B were evaluated using a profilometer and SEM. Results: The resin composites showed the least effects of acidic corrosion on their surface texture. The viscous glass ionomer cements showed the greatest changes, with significantly increased surface roughness $p<0.001$). Conclusions: The immersion of two newer viscous GICs in a buffered artificial saliva and the single application of APF gel resulted in significantly rougher surfaces over a relatively short six-week period.

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INTRODUCTION

Restorative dentistry has undergone many changes during the past 20 years, with the introduction of a wide range of newer esthetic materials for directly placed intra-coronal restorations, such as improved resin composites, polyacid-modified resin composites (compomers) and various glass ionomer cements (GICs). Several viscous conventional GICs have been specifically developed and marketed for the atraumatic restorative treatment (ART) approach (Frencken & Holmgren, 1999). Their mechanical strengths, chemical wear resistance and esthetics should be sufficient to ensure an adequate clinical performance. However, the topical application of acidulated phosphate fluoride (APF) gel to remineralize initial carious lesions has also

been shown to cause surface erosion or corrosive wear of several restorative materials (Mair & others, 1996), with increased surface roughness (Yip, Peng & Smales, 2001a) and reduced surface hardness (Yap & Mok, 2002).

With earlier conventional GICs, the glass particles were either lost or left protruded after APF gel treatment for four minutes (Neuman & García-Godoy, 1992). The amount of surface material lost was proportional to the time of exposure to non-buffered acid (Walls, McCabe & Murray, 1985). The dissolution of GICs and resin-modified GICs occurs by acid diffusion and surface reaction at low pH and predominantly by diffusion near neutral pH (Gao & others, 1997). It has been suggested that the phosphoric acid in APF gel is capable of forming large, stable complexes with the metal ions in GICs, resulting in increased surface roughness (El-Badrawy, McComb & Wood, 1993). Dissolution of the salt gel matrix occurs at a slightly acidic pH (6), but does not affect the glass particles (Gao & others, 1997). This leads to dislodgement of the glass particles, leaving voids on the surface (Yip & others, 2001a). In resin-modified GIC restorations, "plucking-out" the filler particles is also related to an increase in surface roughness (Sidhu, Sherriff & Watson, 1997). In strong acids without buffer systems, the elution of matrix-forming cations such as aluminum, calcium and strontium occurs (Forss, 1993; Williams, Billington & Pearson, 2001).

The surface hardness of GIC is also reduced in an acid medium (McKinney, Antonucci & Rupp, 1987). However, human saliva has been reported to significantly increase the surface hardness of Fuji IX GP, probably due to the ionic exchange between saliva and the surface of the GIC (Okada & others, 2001). Such a phenomenon does not occur with resin-modified GICs (Kanchanavasita, Anstice & Pearson, 1998) and resin composites (Okada & others, 2001). There is scant literature on the changes in surface hardness of esthetic restorative materials after acid erosion, and no correlations have been made with the changes in surface roughness. While nanoindentation has been used to study the hardness of tooth substrates (Urabe & others, 2000) and the filler particles of restorative materials (Willems & others, 1993), it has not been adopted for the study of changes in the surface hardness of restorative materials.

Surface degradation of resin composites has been studied using microhardness tests and surface profilometry (Van Groeningen, Jongebloed & Arends, 1986; Chadwick & others, 1990). The surface degradation that results from acid erosion appears related to the type of filler particles present (Kula & others, 1986). The erosion pattern in resin composites has been studied with pharmacological slow-release devices such as drug-containing cylindrical polymer matrices made of several layers of different polymers. The proposed two-stage matrix erosion mechanism involves the bulk eroding of polymer before erosion can move into the core of the resin composite (Göpferich, 1997). This process is further complicated by the dislodgment of particles embedded in the matrix. The erosion is more severe in a pH-cycling model than in either distilled deionized water or artificial saliva (Turssi & others, 2002).

Because of improvements in several of their physical properties (Yip & others, 2001b), the newer viscous GICs may show less surface degradation than earlier GIC restorative materials. Therefore, the objective of this study is to investigate the short-term surface roughness changes of two of these viscous GICs as compared with three other esthetic restorative materials in a simulated oral environment using profilometry and SEM. The null hypothesis is that there are no significant differences present for surface roughness with aging in buffered artificial saliva and APF gel application among the five restorative materials evaluated.

METHODS AND MATERIALS

Specimen Preparation

Two GICs (Fuji IX GP Fast, Ketac-Molar) marketed for the ART approach, one resin-modified GIC (Vitremer) and two resin composites (UniFil, Z250) were evaluated. Table 1 shows the details of the five restorative materials.

Table 1: *Details of Restorative Materials*

Material	Type	Manufacturer	Shade	Batch #	Expiry (yy/mm)
Fuji IX GP Fast (Capsule)	Conventional GIC (P:L, 3.5:1.0)	GC Int Corp, Tokyo, Japan	A3	9906214	01/06
Ketac-Molar (Hand-mixed)	Conventional GIC (P:L, 2.9:1.0)	3M ESPE, FGR, D-82229 Seefeld, Germany	A3	FW0050325	00/04
Vitremer (Hand-mixed)	Resin-modified GIC (P:L, 2.5:1.0)	3M ESPE, St Paul, MN, USA	A3	19990922	01/02
UniFil (Syringe)	Resin composite	GC Int Corp, Tokyo, Japan	A3	9906011	01/06
Z250 (Syringe)	Resin composite	3M ESPE, St Paul, MN, USA	A3	19990609	02/04

Initially, 10 specimens of each material were prepared according to manufacturers' instructions. The materials were injected into disposable cylindrical Teflon molds measuring 3.0-mm diameter x 2.7-mm height. After filling, each mold was then manually pressed between two Mylar strips sandwiched with two glass slides to ensure a smooth specimen surface. The resin-based materials were photopolymerized on both sides of the molds for 40 seconds using a VCL 200 visible light unit (Demetron/Kerr, Orange, CA, USA). The light intensity was checked with a curing radiometer (Demetron/Kerr) and found to be 320 mW/cm^2.

After setting for one hour in a humidor, the 50 specimens were carefully removed from their molds and placed separately in small polypropylene vials containing 2 ml of artificial saliva solution (0.05 M acetate buffer with 2.2 mM $CaHPO_4$ adjusted with glacial acetic acid to pH 5.0). The vials were stored at a constant temperature of 37°C.

The solution was replaced daily for three weeks using fresh vials. Each time, the specimens were also rinsed thoroughly with distilled water. At the end of the third week, five specimens of each material selected at random (Group A) were rinsed with distilled water then evaluated for their surface roughness. The remaining five specimens of each material (Group B) were coated with 2 ml of 1.23% acidulated phosphate fluoride (APF) gel (Protect; John O Butler Co, Chicago, IL, USA) for four minutes, rinsed in deionized water to remove any visible remnants of gel, then immersed again in artificial saliva for another three weeks. As before, the solution was replaced daily during these three additional weeks prior to repeating the same evaluations. For each material, two additional fresh specimens for evaluating surface roughness and one additional fresh specimen for SEM examination was also prepared for comparison.

Surface Roughness Evaluation

The two additional fresh specimens of each material and those from Groups A and B were evaluated for surface roughness using the Alpha-Step 200 profilometer (KLA-Tencor Instruments, San Jose, CA, USA). A custom-made holder allowed for the rotation of each specimen for surface measurements along different random axes. The traveling distance of the diamond stylus was set to 400 µm and the measured results were displayed on a digital screen. Eight arithmetic average surface roughness (R_a) measurements were made for each fresh and each aged specimen after three weeks, and those coated with APF gel after a total of six weeks. The Ra was automatically determined using the graphical-centerline method with a cut-off of 80 µm according to the ASME Standard Y14.36M (2002).

Scanning Electron Microscopy Evaluation

One new, fresh specimen of each material and one specimen selected at random from Group A after three weeks and from Group B after six weeks was examined using the Philips XL30CP (Philips Electron Optics, Eindhoven, The Netherlands) scanning electron microscope (SEM). The surfaces of the specimens were sputter coated with a thin layer of gold before being examined, then photographed at 500x magnification.

Statistical Analysis

Raw data for the surface roughness measurements of the materials were analyzed using the software package Prism 3.0 (GraphPad Software Inc, San Diego, CA, USA). Because of unequal group variances, statistical analysis for material differences was performed at each time period using the non-parametric Kruskal-Wallis ANOVA and Tukey-Kramer post-tests, when appropriate. Comparisons of the fresh and the three- (Group A) and six-week (Group B) findings for each material were analyzed using the non-parametric Mann-Whitney U-test. The probability level for statistical significance was set at $p \leq 0.05$.

RESULTS

Surface Roughness of Fresh and Aged Specimens

The surface roughness of the fresh, three- and six-week immersion specimens increased with the length of aging in buffered artificial saliva and acid erosion by APF gel (Figure 1). The increase in mean R_a was greatest for Fuji IX GP Fast, followed by Ketac-Molar. Vitremer and the two resin composites showed relatively slight increases in surface roughness values after the same treatments (Table 2). Z250 had the smoothest surface initially and

Table 2: *Mean Surface Roughness (R_a) Values for the Restorative Materials*

	Mean (Standard Deviation) Microns*		
Material	**Fresh (N=10)**	**3 Weeks (N=25)^**	**6 Weeks (N=25)#**
Fuji IX GP Fast	0.19 (0.05)	0.86 (0.68)	1.82 (1.25)
Ketac-Molar Aplicap	0.17 (0.02)	0.59 (0.20)	0.94 (0.43)
Vitremer	0.14 (0.04)	0.23 (0.07)	0.33 (0.16)
UniFil	0.15 (0.05)	0.22 (0.09)	0.25 (0.07)
Z250	0.06 (0.03)	0.10 (0.03)	0.14 (0.03)
Kruskal-Wallis	18.169	25.792	30.608
p values	0.001	< 0.0001	< 0.0001

**Materials joined by the same vertical line are not significantly different at the 5% probability level. ^Group A. #Group B (APF gel applied at three weeks).*

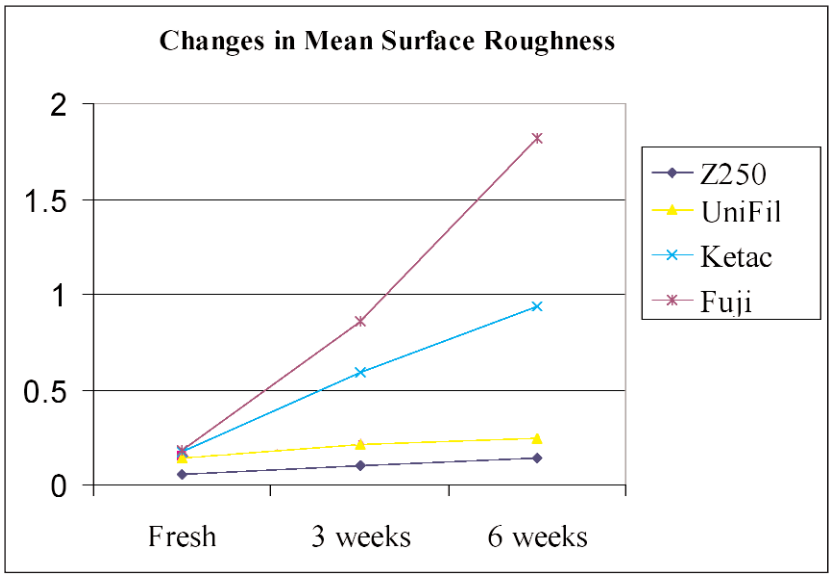

Figure 1: *Changes in mean (R_a) surface roughness for fresh, three-week and six-week immersion specimens (microns).*

also showed the lowest roughness values during the study. By contrast, after six weeks, Fuji IX GP Fast showed significantly greater surface roughness changes compared to all of the other materials ($p<0.0001$). After six weeks, the surface roughness of Fuji IX GP Fast had increased 9.6 times and Ketac-Molar by 5.5 times. The increases for UniFil, Z250 and Vitremer were 1.7, 2.3 and 2.4 times, respectively. Even before APF gel application at three weeks, the surface of Fuji IX GP Fast was already significantly rougher than the surfaces of the three resin-based materials. With the exception of UniFil resin composite at three weeks ($p=0.14$), each of the other four materials showed significantly rougher surfaces at three and six weeks compared to when freshly prepared ($p\leq 0.05$).

SEM of Fresh and Aged Specimens

SEM confirmed visually the surface profilometry results for the different materials at each observation period. Z250 showed the smoothest surfaces (Figure 2) and Fuji IX GP the roughest (Figure 6). The other three restorative materials tested were between these two extremes in the restorative spectrum (Figures 3-5). Small voids and the protrusion of filler particles were most apparent for the GICs and increased with time. Cracks on the surfaces of the GICs were artifacts caused by vacuum dehydration during processing.

DISCUSSION

An increase in surface roughness has been used as a criterion to assess and predict the clinical deterioration of restorations of different types of materials (Smales, Webster & Leppard, 1992; Larsen & Brunn, 1994). This increase encourages plaque retention (Quirynen &

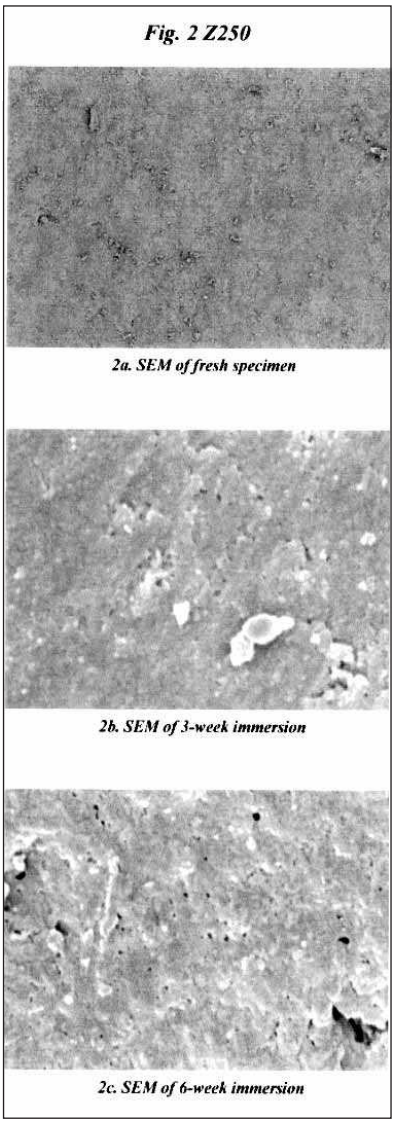

Figure 2: *Scanning electron micrographs of Z250 at different stages of aging: a. fresh; b. three-week; c. six-week. There is an increase in the surface roughness and surface voids of the specimens with aging (magnification 500x).*

Bollen, 1995) and susceptibility to gingival inflammation, surface staining and wear of both the restorative material and the opposing tooth substance. The critical mean surface roughness (R_a) for the adhesion and colonization of bacteria on restorative materials has been reported to be 0.2 µm (Bollen, Lambrechts & Quirynen, 1997), which is considerably less than for the mature GICs in this study.

Currently, lactic acid impingement is used to test the *in vitro* solubility of restorative materials. However, relatively few studies have reported on clinically relevant changes to the surfaces of restorative materials that may take place when restored teeth are subjected to

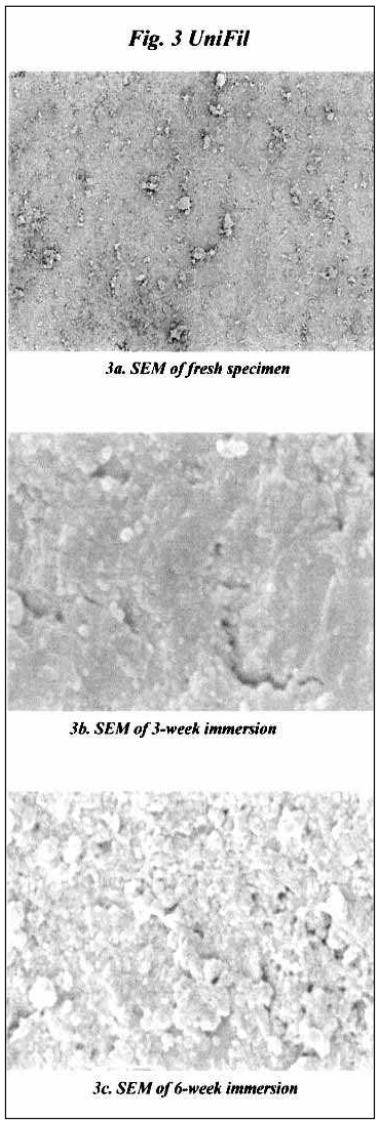

Figure 3: *Scanning electron micrographs of UnFil at different stages of aging: a. fresh; b. three-week; c.six-week (magnification 500x).*

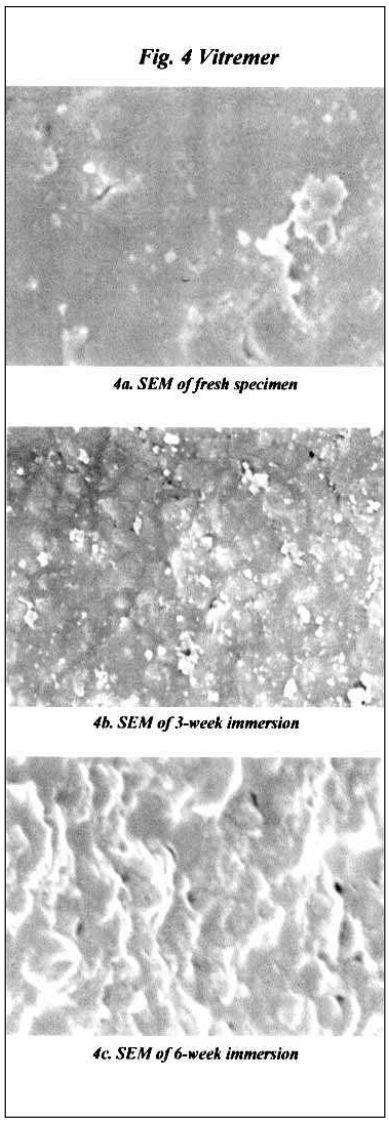

Figure 4: *Scanning electron micrographs of Vitremer at different stages of aging: a. fresh; b. three-week; c. six-week (magnification 500x).*

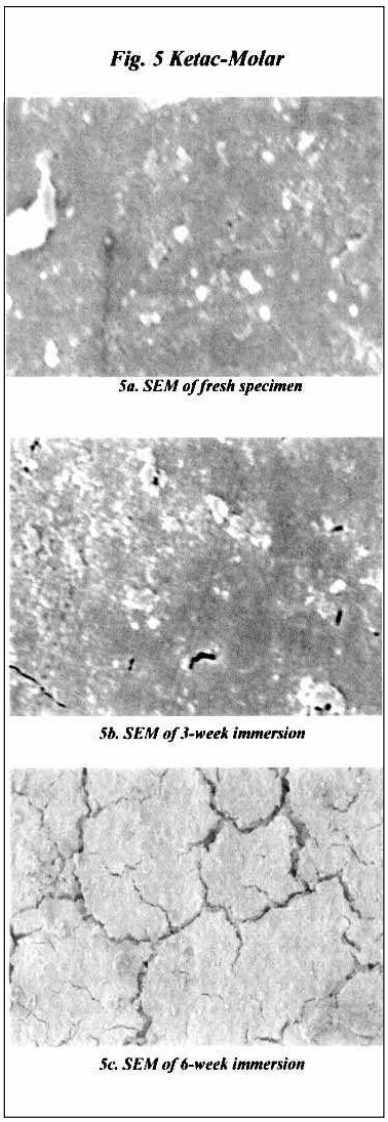

Figure 5: *Scanning electron micrographs of Ketac-Molar Aplicap at different stages of aging: a. fresh; b. three-week; c. six-week. The surface cracks present are preparation artifacts (magnification 500x).*

repeated preventive and therapeutic topical applications of APF gel (Yip & others, 2001a; El-Badrawy & others, 1993; Triana & others, 1994; El-Badrawy & McComb, 1998). Even fewer studies have examined the surface changes across a spectrum of restorative materials (Bapna & Mueller, 1999; Yip, Lam & Smales, 1999).

The simple artificial saliva substitute used in this study (0.05M acetate buffer with 2.2 mM $CaHPO_4$ adjusted with glacial acetic acid to pH 5.0) and stored at 37°C has also been used previously in several other *in vitro* studies of fluoride-releasing esthetic restorative materials (Yip & others, 1999, 2001a; Gao, Smales & Gale, 2000; Peng & others, 2000; Gao & Smales, 2001). Different artificial salivas and other storage media, such as lactic acid, deionized water and water can affect the chemical and physical characteristics of esthetic restorative materials (Crisp, Lewis & Wilson, 1980; El-Mallakh & Sarkar, 1990; Forss, 1993; Geurtsen, Leyhausen, & García-Godoy, 1999; Gao & others, 2000). The use of a more complex artificial saliva system for evaluating restorative materials has also been proposed but not evaluated (Leung & Darvell, 1997).

Surface hardness is defined as the surface resistance of materials to indentation or penetration (McCabe, 1990). When stored in either human saliva (Okada & others, 2001) or distilled water (Yap, Cheang & Chay, 2002), the surface hardness of Fuji IX GP has been

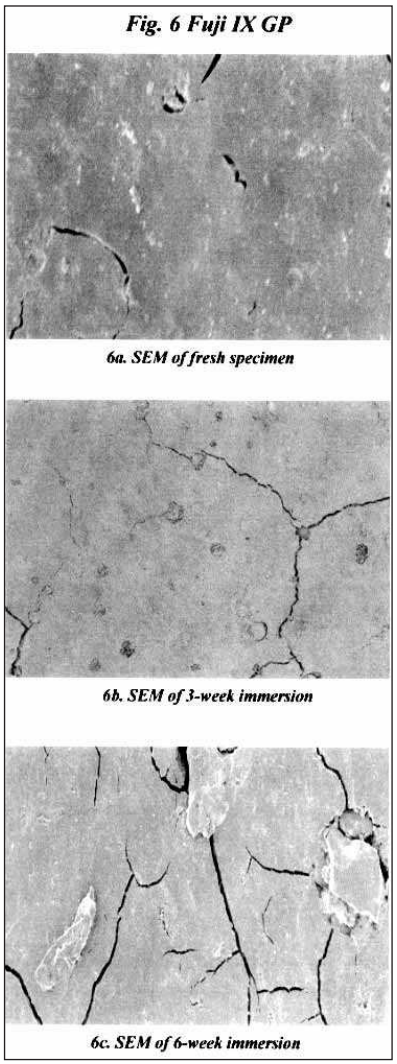

Figure 6: *Scanning electron micrographs of Fuji IX GP Fast at different stages of aging: a. fresh; b. three-week; c. six-week. The surface cracks present are preparation artifacts (magnification 500x).*

shown to increase significantly over one week during the cement's maturation phase. After one week in distilled water, the surface hardness of Fuji IX GP Fast was also found to be significantly higher than for Fuji IX GP (Yap, Pek & Cheang, 2003). The GICs used in these studies and in the current study are high powder-to-liquid ratio viscous materials with improved compressive and flexural strengths and reduced *in vitro* wear as compared to earlier conventional GICs (Yip & others, 2001b). When exposed to APF gel, even resin-based materials were found to have reduced surface hardness (Yap & Mok, 2002). However, there is no significant correlation between surface wear and hardness for resin composites (Manhart & others, 2000), which is probably also true for GICs, and the clinical wear of resin composites is best predicted from their modulus of elasticity, modulus of resilience and flexural strength (Lewis, 1993). The relationship of these parameters to the wear of newer GICs also requires investigation. The type, proportion and size of the largest fillers and the nature of the filler-matrix interface appears to dominate resin composite wear behavior.

Three weeks maturation followed by the single application of APF gel produced a surface roughness on the GICs that was significantly different from their fresh specimens. Instead of using the longer six- and 12-week immersion periods as adopted in previous studies (Yip & others, 1999, 2001a), the authors found that a three-week period was adequate to observe significant changes in surface roughness. The clinically recommended single four-minute application of APF gel (Ripa, 1981), followed by an additional three-week aging period in buffered artificial saliva, appeared to accentuate the earlier three-week surface roughness trend present between the GICs and the resin-based materials. One limitation of this study was the lack of a group included to measure the surface roughness at six weeks without APF gel application. It is possible that the roughness that was found may have increased significantly even without the application of APF gel. However, the most marked increase in surface roughness for Fuji IX GP, especially, occurred during the second three-week period following application of the APF gel.

The mean sizes of the filler particles and the voids left following their dislodgment are greatest in GICs, less in resin-modified GICs and least in resin composites (Gladys & others, 1997). SEMs of the five materials in this study agreed with these observations (Figures 2-6). A clinical trial (van Dijken, 1999) and several laboratory studies (Yip & others, 1999, 2001a) have shown a decreasing surface roughness from GICs to resin-modified GICs, to resin composites. Subsequent to surface wear, the surface roughness of restorative materials is also related to the number and size of voids incorporated during mixing and placement of the materials. Although machine-mixed restorative materials have been shown to contain less porosity than hand-mixed materials (Covey & Ewoldsen, 2001), the initial surface roughness of the encapsulated Fuji IX GP Fast and the hand-mixed Ketac-Molar were very similar (Table 2). Despite the improved mechanical properties of the newer viscous conventional GICs (Yip & others, 2001b), including improved wear resistance, there appeared to be no significant improvements in their resistance to erosion in a buffered artificial saliva and after the single application of APF gel.

CONCLUSIONS

The null hypothesis was rejected. Aging in a buffer artificial saliva, and the single application of 1.23% APF gel to all materials for four minutes resulted in significantly increased surface roughness over a six-week period, which was particularly severe for the two conventional GICs. The effects were less pronounced for the three resin-based materials. APF gel should not be applied routinely to the esthetic restorative materials investigated in this study.

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Importance of Water Sorption and Solubility Studies for Couple Bonding Agent—Resin-based Filling Material

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Clinical Relevance

Among the mechanical and physical characteristics needed for a long-term restoration, the behavior of resin-based restoration materials in the oral environment depends on the resistance to solubility and, consequently, to water intake ability. In this way, resin composites and ormocers appear to be more suitable than compomers and resin-modified glass-ionomer cements in meeting longevity requirements. Water sorption and desorption of bonding agents are also to be taken into account, because their solubilization may deteriorate the bonded interface and open a gap to salivary fluids.

SUMMARY

This study investigated the water sorption and solubility of two light-cured resin composites (Filtek P60 and Solitaire 2), one compomer (Compoglass F), one ormocer (Admira) and the associated bonding agents (Scotchbond 1 [Scotchbond 1 = Scotchbond Single Bond in USA], Gluma One Bond, Excite and Admira Bond, respectively) and of a RMGIC (Fuji II LC). Five disks of each product type were subjected to water sorption and solubility tests based on ISO 4049 requirements. The data were subjected to Kruskal-Wallis and non-parametric multiple-comparison tests using ranked sums at 95% confidence interval. Fuji II LC showed the highest water sorption (167.5 µg/mm^3). Fuji II LC and Compoglass F had higher solubility values (8.3 and 10.0 µg/mm^3, respectively) than the other materials. Bonding agents have very high water sorption and solubility values (between 77.4 and 355.4 µg/mm^3 and between 75.9 and 144.9 µg/mm^3, respectively) compared to the restorative materials. Gluma One Bond and Admira Bond showed lower sorption and solubility than Excite and Scotchbond 1.

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INTRODUCTION

In oral conditions, filling materials are in contact with salivary fluids that contain a wide range of organic and inorganic substances, together with numerous and complex bacterial flora. Approximal polymer fillings are often close to the gingival crevice and are in contact with the sulcular fluid. Therefore, their water sorption and solubility behavior is of considerable importance from a clinical point of view.

The absorption of water into polymer materials results in a volume expansion that potentially compensates for the initial setting contraction and, consequently, decreases both the interfacial gap width and resultant microleakage (Torstenson & Brännström, 1988; Feilzer, de Gee & Davidson, 1990). However, expansion that exceeds dimensional polymerization shrinkage may result in expansion stress in the material and adjacent dental tissues (Feilzer & others, 1995; Huang & others, 2002a). Moreover, if the polymer network is developed in a few minutes or a few hours, depending on the material family, hygroscopic expansion, which potentially reduces the marginal gap, takes several days, during which the interfacial space may be contaminated by salivary microorganisms (Huang & others, 2002b). Water absorption affects the mechanical properties of the material, particularly by decreasing the elastic modulus and resistance to wear or flexion, which may affect the quality of the marginal joint (Øysaed & Ruyter, 1986; Cattani-Lorente & others, 1999; Örtengren & others, 2000; Musanje, Shu & Darvell, 2001). In addition to these effects, Iwami and others (1998) found that water sorption leads to color change in fillings: thus, materials in an aqueous environment may also interfere with the aesthetic characteristics of polymer restorations.

The conversion of monomers into a polymer network is never complete for resin composites, polyacid-modified resin composites (so-called compomers) or resin-modified glass ionomer cements (RMGICs). Moreover, different factors such as an insufficient light intensity or the persistence of oxygen result in significant amounts of unreacted monomers in the bulk polymer and on the material surface (Matsumoto & others, 1986). Consequently, the material could release chemical substances into the environment, bringing into question the biocompatibility of dental restoration products (Øysaed, Ruyter & Sjovik Kleven, 1988; Ferracane, 1994; Geurtsen, 1998; Hamid & others, 1998; Geursten, Spahl & Leyhause, 1998). Residual monomers (Tanaka & others, 1991; Palmer, Anstice & Pearson, 1999), fillers (Söderholm, 1990), activators and inhibitors of polymerization (Rathbun & others, 1991) or degradation products such as formaldehyde (Øysaed & others, 1988) or methacrylic acid (Munksgaard & Freund, 1990) are thus able to leach out from resin-based materials. These molecules are potentially hazardous to the surrounding soft tissues (Stankey, 1992; Katsuno & others, 1995; Heil & others, 1996; Mac Dougall & others, 1997; Söderholm & Mariotti, 1999).

Although numerous studies have examined the sorption and solubility of resin-based filling materials, few have investigated these properties in adhesive systems (Burrow, Inokoshi & Tagami, 1999). Nevertheless, bonding agents are exposed to salivary fluids on the external surface and to dentinal fluids on most of the internal surface. The capacity to absorb water and dissolve are determining factors related to the quality and duration of the marginal bond, which determines the success of a filling when the restoration material will not deteriorate. In addition, released molecules could reach the pulp through the dentinal tubules (Tay & others, 1994; Hebling, Giro & Costa, 1999) and may have harmful effects (Jontell & others, 1995; Schweikel & Schmaltz, 1997; Schweikel, Schmaltz & Bey, 1994; Gwinnett & Tay, 1998).

It is of particular interest to determine whether the bonding agent behavior is identical to the filling material in an aqueous environment. This study comparatively determined the water sorption and solubility of four restoration materials and their associated adhesives against the same characteristics of a resin-modified glass-ionomer cement material.

METHODS AND MATERIALS

Table 1 lists the five restoration materials used in the study (two resin composites, one ormocer, one compomer and one RMGIC), while Table 2 lists the four bonding agents.

Five disks of each restoration material were made according to ISO specification 4049: 12-1998. Samples were made in a jig consisting of a Teflon mold (15-mm in diameter by 1-mm in thickness) compressed between two glass slabs with 50-µm thick polyester separating sheets; the filling materials were packed into the molds, being careful to minimize air inclusion. The materials were light-cured from both sides with a visible-light unit (XL 3000 Curing Light, 3M Dental Products, St Paul, MN, USA), the light intensity was tested at regular intervals. The light tip was first directed over the center of the sample for 40 seconds, and then irradiated for eight peripheral overlapping sectors for 20 seconds each. The specimens were immediately stored in an incubator at 37°C for 15 minutes. The excess material was removed with a scalpel blade. The rim was trimmed and polished with 1000 grit silicone carbide grinding paper until the diameter of the final product was maintained within a 14.9 ± 0.1 mm average diameter obtained from two measurements at perpendicular planes with an electronic caliper (Digimatic, model 500-181U, Mitutoyo Corporation, Tokyo, Japan). As for the adhesive materials, which, unlike the restorative materials, cannot be packed into the molds, each disk was made with the same volume of bonding agents. Once the mold was filled with adhesive, a first irradiation was carried out over the sample center for 40 seconds, then the polyester sheet and glass slab were put down, being careful to minimize air inclusion. The bonding agents were then polymerized similar to the restorative materials, as described above.

The specimens were then immediately placed in a desiccator with silica gel (Silicagel, Prolabo, Paris,

Table 1: *Materials Used*

Code	Material (Manufacturer)	Type	Main Components	Batch #
ADM	Admira (Voco D-27457 Cuxhafen Germany)	Organically modified ceramic (ormocer)	*monomers:* Bis-GMA, di-UDMA, TEGDMA *fillers (78 wt.% = 56% vol.)*: Ba-Al-B-silicate glass (90%, ca. 0.7 µm), SiO_2 (10%) 3-dimensionally curing anorganic-organic copolymers, additive aliphatic and aromatic dimethacrylates	03655
COF	Compoglass F (Vivadent Ets FL-9494 Schaan Liechtenstein)	Polyacid-modified resin composite (compomer)	*monomers:* (22.5 wt.%): UDMA, poly-EGDMA, CADCADM *fillers (77 wt.%)*: Ba-Al-fluorosilicate glass (1 µm), ytterbium trifluoride, Spheroid mixed oxide *Additional contents (0.25 wt.%)*: catalysts, stabilizers, pigments	B06383
FLC	Fuji II LC (GC Corp Tokyo, Japan)	Resin-modified glass ionomer	*powder*: aluminofluorosilicate glass *liquid*: polyacrylic acid, HEMA, 2-2-4 trimethyl hexamethylene dicarbonate, TEGDMA, water	powder 030461 liquid 080771
P60	Filtek P60 (3M ESPE Dental Products St Paul, MN, USA)	Resin composite	*monomers:* Bis-GMA, Bis-EMA *fillers (81 wt.% = 61% vol.)*: $ZrSiO_4$	9AN
SOL2	Solitaire 2 (Heraeus Kulzer D-41538 Dormagen, Germany)	Resin composite	*monomers*: UDMA, TEGDMA, Bis-GA *fillers (75 wt.%)*: Ba-Al-B-fluorosilicate glass (mean diameter: 0.7 µm and 5 µm), porous SiO_2 glass	010225

Bis-GMA= Bisphenol-A glycidylmethacrylate; Bis-GA= Bisphenol-A glycidylpolyacrylate; UDMA= urethane dimethacrylate; TEGDMA= triethyleneglycol dimethacrylate; poly-EGDMA= polyethyleneglycol dimethacrylate; CADCADM= cycloaliphatic dicarboxilic acid dimethacrylate; Bis-EMA= ethoxylated bisphenol-A glycol dimethacrylate

HEMA= 2-hydroxyethyl methacrylate

Table 2: *Bonding Agents Used*

Code	Bonding Agent (Manufacturer)	Associated Resin-Based Material	Main Components	Batch #
ADB	Admira Bond (Voco D-27457 Cuxhafen Germany)	ADM	Bis-GMA, HEMA, organic acids complex 3-dimensionally curing anorganic-organic copolymers acetone	03653
EXC	Excite (Vivadent Ets FL-9494 Schaan Liechtenstein)	SOL2	Bis-GMA, HEMA, dimethacrylate phosphonic acid acrylate highly dispersed silica ethanol (25 wt.%)	B33276
SCO	Scotchbond 1* (3M ESPE Dental Products St Paul, MN, USA)	P60	Bis-GMA, HEMA, Bis-phenol A glycerolate dimethacrylate, copolymer of polyacrylic and polyitaconic acids water, ethanol	0EE
GLU	Gluma One Bond (Heraeus Kulzer D-41538 Dormagen Germany)	COF	HEMA, UDMA (ratio = 40/60), 4-META: 10 wt.% acetone	105641

Bis-GMA= Bisphenol-A glycidylmethacrylate; UDMA= urethane dimethacrylate; HEMA= 2-hydroxyethyl methacrylate

4-META= 4- methacryloxyethyl-trimelittic acid

**Scotchbond 1 = Scotchbond Single Bond in USA*

France). The entire set of samples was stored in an incubator maintained at 37°C ± 1°C. According to the ISO standard, the disks were weighed daily for 35 days using an analytical balance (Model M-120, Denver Instrument, Arvada, CO, USA) with a repeatability of 0.1 mg until a constant mass (m_1) was obtained (until the mass loss or gain of each sample was less than ± 0.2 mg in any 24 hour period). The volume V of the specimens was then determined by measuring the specimen diameter from two perpendicular planes and the thickness from five measurements, one at the center and four at equally spaced points on the specimen circumference using an electronic caliper. This initial cycle allows free water to be removed as recommended in the ISO standard test.

Each sample was then suspended in a flask containing 40 ml of distilled water and stored in the incubator at 37°C ± 1°C for seven days. The samples were then removed, washed briefly with water, wiped with absorbent paper and shaken in air for 15 seconds. The samples were weighed for one minute after removal from the incubator to record the mass (m_2). This second cycle gives a combination of water sorption and dissolution of the soluble components from the sample.

After weighing, the disks were reconditioned at a constant mass (m_3) in a desiccator and incubator following the stages of initial cycling described above. This third cycle allowed for measuring of the mass loss.

The values for sorption and solubility W_{sp} and W_{sl}, respectively, in µg/mm^{-3} were calculated for each specimen using the following equations:

$$W_{sp=} \frac{m_2 - m_3}{V} \qquad W_{sl=} \frac{m_1 - m_3}{V}$$

Where m_1 is the mass in µg before immersion in water; m_2 is the mass in µg after immersion in water for seven days; m_3 is the reconditioned mass in µg and V is the volume of the sample in mm^3. Statistical analysis of the results was carried out with Kruskal-Wallis tests and non-parametric multiple-comparison tests using rank sums.

RESULTS

Table 3 presents the results of the water sorption and solubility for all materials tested. According to the Kruskal-Wallis tests (p=0.05), the filling materials sorbed and solubilized in a significantly different way (p=0.0002). Non-parametric multiple-comparison procedures showed that FLC had significantly higher water sorption values compared with all the other materials, which did not differ from each other. Regarding solubility, ADM, P60 and SOL2 did not differ from each other but had significantly lower values than FLC and COF, which also did not differ from each other.

Concerning bonding agents, the Kruskal-Wallis tests showed that the materials behaved differently, in terms of either water sorption (p=0.0011) or solubility (p=0.0031). Non-parametric multiple-comparison procedures showed that the water sorption values of all the materials tested were significantly different from each other. Concerning solubility, ADB and GLU did not significantly differ from each other and had significantly lower values than EXC and SCO, which significantly differed from each other.

Table 3: *Mean Water Sorption (W_{sp}) and Water Solubility (W_{sl}) in µg/mm^{-3} and Standard Deviation (SD)*

Material	W_{sp} (SD)	W_{sl} (SD)	Bonding Agent	W_{sp} (SD)	W_{sl} (SD)
ADM	23.3 (0.8)[a]	1.2 (0.3)[a]	ADB	106.2 (15.2)[a]	82.2 (20.8)[a]
COF	31.5 (3.2)[a]	10.0 (2.1)[b]	GLU	77.4 (19.0)[b]	75.9 (13.0)[a]
P60	19.2 (1.0)[a]	2.4 (1.1)[a]	SCO	355.4 (29.7)[c]	133.6 (12.6)[b]
SOL2	20.2 (0.4)[a]	4.9 (0.7)[a]	EXC	273.4 (66.1)[d]	144.5 (32.8)[c]
FLC	167.5 (12.4)[b]	8.3 (0.8)[b]	*no bonding agent required*		

(In each column, results with the same superscript letter are not statistically different.)

DISCUSSION

With a radius of about 0.158 nm, water molecules are capable of diffusing through polymers, because the size of the molecules is smaller than the interchain distance in the matricial resin (Tamai, Tanaka & Nakanishi, 1995). The effect of water sorption and solubility on the short- and long-term thermomechanical behavior of polymer materials cannot be disregarded for clinical reasons. From an atomic viewpoint, mechanisms of diffusion are a step-wise migration of atoms from site to site (Callister, 1994). In fact, two attitudes to these problems have been amply mentioned in the literature. In the "free volume theory," it has been proposed that the water equilibrium concentration is mainly ruled by the available free volume fraction, in which fluids diffuse through microvacancies or other morphological defects without any mutual connection with the polar sites of the material. In the "interaction theory," water molecules would be bound successively to polar groups of polymer chains, especially those forming hydrogen bonds, mainly hydroxyls. Lately, it has been assumed that both approaches could be valid, each for a defined sample family or both simultaneously (Bellenger, Verdu & Morel, 1989). However, polymers absorb water to different degrees, relying on their microstructural and molecular aspects (Venz & Dickens, 1991); for example, polarity of the molecular structure; presence of pendant hydroxyl groups capable of forming hydrogen bonds with water, degree of cross-linking of the continuous

matrix; presence of residual water attracting species or type dimension, volume, diffusivity and solubility of filler particles (Marcovich, Reboredo & Aranguren, 1999).

In this study, it was not surprising that the most significant water intake occurred in the RMGIC material (FLC). This kind of material has, in fact, a dual setting reaction involving mainly the acid-base reaction of conventional glass-ionomer cement and the free radical polymerization process used in resin composite systems. In this case, the polymerized structure contains numerous hydrophilic functional groups in a highly entangled matrix and behaves as a synthetic hydrogel (Anstice & Nicholson, 1992) that can absorb an extensive amount of water, up to 80% in mass for compounds obtained from HEMA-copolymers (Pedley, Skelly & Tighe, 1980). Therefore, materials with less HEMA content are expected to present with water sorption (Huang & others, 2002a). Compared to FLC, COF showed five times less sorbed water. Compomers, also known as polyacid-modified resin composite, differ from RMGIC, because of their composition and structure. They are fabricated of ion-leachable glass particles identical to those of glass-ionomer cements embedded in a polymeric matrix. The matrix is formed principally during the photopolymerization of different kinds of monomers: modified methacrylates, such as bis-GMA, UDMA or TEGMA, and bifunctional monomers comprising two carboxylic groups and two double-bond functions (Meyer, Cattani-Lorente & Dupuis, 1998). The functional monomers are supposed to react concurrently with methacrylates by common free-radical polymerization and with cations liberated from glass fillers in an acid-base reaction. The acid-base reaction is initially limited in compomer, because of the anhydrous structure. Once water infiltrates the compomer material, a belated acid-base reaction is likely to occur (Meyer & others, 1998). Water uptake in compomers thus involves hydration of the glass filler within the polymerized material on top of the water absorbed in a larger amount by the resin matrix (Huang & others, 2002b). This phenomenon is likely responsible for the greater amount of water absorbed by the compomer compared to the resin composites (Table 3).

The samples of P60, SOL2 and ADM clearly absorbed less water than the compomer and RMGI materials. Water permeates resin composite material according to three mechanisms: direct diffusion into the material phase, penetration by voids or damage already present in the material generated by water attack (Lekatou & others, 1997) or through inclusion in the filling (Øysaed & Ruyter, 1986) or flow of water molecules along the filler-matrix interface. Therefore, the differences in water sorption observed among the three resin composites experimented on may primarily come from the type of resin and filler. For an equal volume, the more filled the material, the less important the matrix volume and the less water sorption. Moreover, the hydrophobic nature of the constituent monomers is a major factor (Øysaed & Ruyter, 1986). ADM contains Bis-GMA, which has two hydroxyl groups per molecule that can hydrogen bond to absorbed water (Venz & Dickens, 1991). P60 contains Bis-EMA and SOL2 includes bis-GA monomers, both of which are molecules where many hydroxyl groups were substituted. Thus, the elimination of pendant polar groups leads to a reduction in water sorption (Yap & others, 2000; Sideridou, Tserki & Papanatasiou, 2003). In addition, the nature and quality of links between the fillers and matrix are also important: a weak adhesion between fillers and the matricial phase may determine ways of a capillary diffusion that carries and holds water (Kalachandra, 1989; Feilzer & others, 1990).

These solubility results are consistent with data found in the literature: resin composites dissolve less than RMGICs and compomers (Iwami & others, 1998). Among the resin composite family, P60 presents solubility two times less than SOL2, whereas, its water intake is practically the same. One explanation lies in the fact that fillers in P60 are made of few soluble $ZrSiO_4$ particles, while the glass Ba-Al-B-F-silicate filler particles of SOL2 are liable to dissolve easily (Øysaed & Ruyter, 1986; Söderholm, 1990). Another factor for resin composite solubility is the monomer conversion rate; residual monomers solubilize easily and a lower rate of polymerization entails much more solubility (Pearson & Longman, 1989; Yap & Lee, 1997; Palmer & others, 1999). The voids entrapped in bulk polymer contain oxygen that may inhibit polymerization and consequently facilitate solubility. Ormocer-based materials such as ADM consist of an inorganic SiO_2 three-dimensionally coiled structure. Supplementary fillers are incorporated into the structure, together with the resin matrix, and that complex structure, which allegedly protects fillers and residual monomers, may explain the lowest solubility found in ADM (Table 3). COF and FLC are materials with the highest solubility, particularly during the first week (Friedl & others, 1997), and this leads to decreased material strength. Among the substances leaking out, released fluorides present a cariostatic effect, which is very useful when the sandwich technique is used to restore teeth. However, the amount of substances released from these products cannot predetermine the material behavior in the oral cavity, since El Mallakh and Sarkar (1990) showed that solubility is more important in distilled water than in artificial saliva.

Bonding agents have been developed that improve the adhesion of polymer fillings to dental hard tissues, with the intention of thwarting the polymerization contraction and forming a gap-free interface. If tooth obturation durability must be achieved, the tooth-restoration

material interface is to be considered when the filling material involves no drawback. The deterioration of bonding agents opens a gap interface where microorganisms may penetrate and colonize. If adhesives expose a very small surface area to the oral fluids, they have a significant contact with dentin. Dentin tubules occupy about 20% to 40% of the mid-coronal dentin surface (Mjör, 1984) and water represents approximately 22% of the volume of dentin (Trowbridge, 1984). Regarding water, 75% of its total amount is in the tubules and 25% is bound in the mineralized matrix around mineral crystals and/or collagen (Panighi & others, 1997). With time, the tubular fluids may consequently damage the bond between dentin and the restoration.

Dentin adhesion mechanisms are thought to rely upon an intermediary layer between the sound bulk dentin and the resin composite restoration. Etching procedures demineralize the dentin surface to expose a collagen network into which the adhesive system infiltrates to form the so-called hybrid layer (Nakabayashi, Kojima & Masuhara, 1982). Resin bonding agents must present some adequate viscosity to penetrate the demineralized dentin and link with the resin composite monomers. To maintain viscosity, these compounds usually contain no filler particles, but if present, the filler content is sufficiently low to keep the product fluid and hydrophobic resin monomers are diluted in a solvent such as ethanol, acetone or water. To overcome some of the problems due to the hydrophobic character of resin monomers, the molecules have been modified to comprise hydrophobic and hydrophilic elements, and often a more hydrophilic molecule such as HEMA has been incorporated into the mixture. However, these modifications augment the potential ability of resins to absorb water.

In this study, adhesives' water sorption values are much higher than filling materials. All products contain HEMA, but much of the time the proportion is unknown. Only GLU, which presents the lowest sorption measurements, has a HEMA/UDMA ratio of 40/60. Moreover, GLU contains 4-META acid (10% by weight), partly hydrophobic, owing to the presence of methacryloxyethyl groups and acetone as diluting agent. SCO, which has the highest sorption value, is only known to contain water as a solvent, probably indicating a more hydrophilic nature. EXC, although containing a small amount of fillers (0.5% by weight), shows an important water intake value. In the absence of recognized data, a plausible explanation is that the solvent volatilization during the initial desiccation leads to more water absorbed.

The solubility of the bonding agents is 30 to 150 times higher than the filling materials (Table 3). In the former, the absence of fillers leads to a weaker polymerization rate, which leaves numerous monomers uncured and potentially soluble. GLU, which has the lowest solubility, contains UDMA, which is less soluble than Bis-GMA (Pearson & Longman, 1989). The three-dimensionally inorganic-organic complex structure of ADB may contribute to lower solubility than EXC and SCO. The absence of or low amount of fillers increases polymerization contraction at the tooth-restoration interface (Feilzer & others, 1990), and this phenomenon may deteriorate the quality of the bond joint, particularly in areas where the bonding agent is thicker (Burrow & others, 1999). Water intake may also damage the interface by "over-compensation" of the setting shrinkage (Feilzer & others, 1990). Conversely, this effect cannot exist for the RMGICs, such as FLC, which do not require an adhesive system to bond to hard dental tissues. Therefore, if we want to compare the water sorption and solubility characteristics of restorative materials, the filling material and its adhesive system must be compared as a whole; in this case, restoration water impermiability seems to be a good parameter.

The disposition of bonding agents to absorb and dissolve, to a massive extent, may lead to the development of coloration of the bond joint, affecting the aesthetic qualities of the restoration. Opening a gap at the interface may also facilitate penetration of microorganisms stimulated by contact with monomers such as TEGDMA (Hansel & others, 1998). Moreover, HEMA has been shown, because of its low molecular weight and high solubility, to diffuse easily through dentin tubules toward the pulp, where it may have adverse effects (Hamid & others, 1998; Bouillaguet & others, 2000).

CONCLUSIONS

The behavior of resin-based materials in water varies according to composition characteristics. Composites, such as P60 and SOL2, present the lowest water intake, followed by the ormocer ADM, the compomer COF and the resin-modified glass-ionomer cement FLC. The lowest solubility was found in the ormocer ADM, followed by the two composites, the RMGIC and the compomer.

The absence or a low amount of fillers and the high portion of hydrophilic monomers in bonding agents provoke very high water sorption and solubility values. Fluid modifications, combining sorption and desorption, potentially deteriorate the bonded interface and allow released chemical substances to have adverse effects when close to the pulp.

This experiment demonstrates that the water sorption and solubility values of the unique filling material are insufficient to take into account the interfacial outcomes. Bonding agents contribute to the interface damage to a major extent. The capacity of filling polymer

materials to restore teeth for long periods depends principally on the material-bonding agent couple.

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Radiopacity of Resin-based Materials Measured in Film Radiographs and Storage Phosphor Plate (Digora)

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Clinical Relevance

Although wide variations in radiopacity were observed among the same group of resin-based materials, most of the composites investigated had higher radiopacity than enamel. When used in posterior restorations, some packable and flowable composites with low radiopacities may cause some confusion regarding the diagnosis of secondary caries.

SUMMARY

This study compared the radiopacity of 41 resin-based materials using conventional dental x-ray film (Ultraspeed-D) and a digital system (Digora) based on storage phosphor plate technology.

For the film-based technique, optical density measurements were carried out using an X-Rite densitometer. Al equivalents (mm) were calculated as described in the literature using a calibration curve of Optical Density versus the thickness of aluminum. Regarding the digital system after exposures of 0.16 and 0.32 seconds, the images were exported to an image processing software (NIH Image Engineering). An approach similar to that used for optical density was used to generate a calibration curve for gray pixel values.

Linear correlations were found between the percentage of fillers by weight and x-ray film radiopacity and the Digora system, and the same coefficient of estimation was recorded (r=0.60; $p \leq 0.05$). A linear correlation was also observed between the conventional x-ray film technique and the Digora system (r=0.93; $p \leq 0.05$). Using two different exposure times did not affect the radiopacity.

Considerable differences were found among materials of the same category. Flowable resin composites were more radiopaque than dentin, while microfine composites were "radiolucent." Most of the available resin-based materials were more radiopaque than enamel. The radiopacity of resin composites depended on their fillers (percentage and type). Using elements with low atomic numbers (Si) resulted in radiolucent materials, while adding elements with high molecular numbers (Ba, Y, Yb), resulted in radiopaque resin composites.

Despite the numerous benefits offered by the digital imaging system (low irradiation dose,

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instant image, image manipulation), the conventional x-ray film technique seems to be more accurate for radiopacity measurements.

INTRODUCTION

Improvements in resin composite formulations and, consequently, their mechanical properties, have increased their use in dental practice. The radiopacity of a composite is an important property to consider, especially when used for posterior restorations (ADA, 1983). A material with a high radiopacity or high density is a strong absorber of x-rays and causes the radiographic image to be light (White & Pharoah, 2000). Objects with low densities are weak absorbers and are referred to as radiolucent.

Resin-based materials are used to replace dental hard tissues, so theoretically, their radiopacity must be compared to enamel and dentin. According to ISO 4049 (1988), the radiopacity of a resin composite must be at least greater than the radiopacity of dentin to allow for the detection of recurrent caries to occur (Roberson, Heymann & Swift, 2002), as well as the detection of voids within the restoration or excess material (overhangs) and steps on the margins of proximal surface in direct and indirect restorations (Cook, 1981).

However, it has been shown that the radiopacity of human teeth varies considerably depending on the individual, age, site and storage conditions (Williams & Billington, 1987). In order to make comparisons between the different studies possible, aluminum step-wedge was chosen as a standard for measuring radiopacity, because its linear absorption coefficient (μ) is of the same order as dental enamel (Cook, 1981).

Many authors have used different techniques to study the radiopacity of resin composites (Cook, 1981; van Dijken, Wing & Ruyter, 1989; Bouschilcher, Cobb & Boyer, 1999). Radiographic images using a transmission or photographic densitometer were the most widely used (Cook, 1981; Stanford & others, 1987; Curtis, von Fraunhofer & Farman, 1990; Akerboom & others, 1993; Marouf & Sidhu, 1998).

A few years ago, digital imaging systems (DIS) were introduced to dental practice. They claim to be more beneficial than conventional radiography, because with less exposure time, an immediate image can be obtained on the screen. Two main systems are now available for direct digital dental radiology. The first, based on charge coupled devices (CCD), uses an intra-oral sensor connected directly by a cable to the computer such as CDR (Computed Dental Radiology), RVG (Radiovisiography) and SenS-A-Ray (Mason & Bourne, 1998). The second system is based on storage phosphor technology and uses a photostimulable phospor (PSP) imaging plate such as Digora and DenOptix. The conventional film-based x-rays suffer from the constraint that neither brightness nor contrast can be changed after the film has been processed, except by alterations of the viewing light (Börg, 1999). With the Digora system, imaging plates can be reused thousands of times, their size is the same as conventional film x-rays and the plates are inexpensive.

Many authors have described the Digora system in detail (Brettle & others, 1996; Van Der Stelt, 1996; Börg, 1999; Hildebolt, Couture & Whiting, 2000). Other researchers compared film characteristics such as limiting spatial resolution (lp/mm), noise and signal-to-noise ratio to those of the digital systems (Börg & Gröndahl, 1996; Huda & others, 1997; Yoshiura & others, 1999).

Two studies compared the optical densities of dental resin composites using different systems: CCD, storage phosphor and Ektaspeed Plus radiographic film (Farman & others, 1996; Wenzel, Hintze & Horsted-Bindslev, 1998). Radiopacities were expressed in optical density for conventional films and in pixels and gray shades for digital systems. To date, no study has tried to establish any correlation between the systems or convert pixel values into equivalent aluminum or determine the exact relationship between the two systems.

This study compared the radiopacity of resin-based materials using conventional dental x-ray film (Ultraspeed D) and a digital system (Digora) based on storage phosphor plate technology. The x-ray film technique, most widely used by researchers and manufacturers to study the radiopacity of a material, was considered as a gold standard technique. The authors tried to investigate the extent to which the results of Digora could be compared to those of the x-ray film technique. The pixel values obtained by the digital system were converted into equivalent millimeters of aluminum. Correlations were also established between both techniques and between the percentage of fillers by weight.

METHODS AND MATERIALS

Thirty-six commercially available light-curing composites were investigated in this study. Two chemically cured resin composites and three compomers were also examined. All materials and their specifications are listed in Table 1.

Specimen Preparation

For each material, three circular specimens were prepared in a cylindrical stainless steel mold with a diameter of 6 mm and a thickness of 2 mm. After filling the mold to capacity, the material surface was covered with a mylar strip and a glass slide, then pressure was applied to force out excess material. The specimens were light-cured using two visible hand-held curing

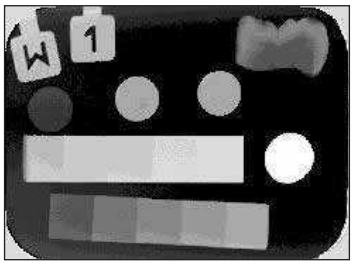

Figure 1. *Image of a radiographic film-setting showing a section of tooth, aluminum step-wedges (1 to 10 mm) and in the center from left to right: samples of Silux-Plus (2 mm), Solitaire-II (2 mm), Admira (2 mm) and lead (10 mm).*

lights (XL-3000, 3M, St Paul, MN, USA, tip diameter 8 mm) that were applied for 60 seconds on each side. Before separation of the specimens, the mold was ground flat with carbide paper (500 grit). The specimens were then stored in a dry place until testing. Enamel and dentin specimens were obtained from 2-mm thick longitudinal sections of recently extracted human molars. Just before testing, the specimens were measured with a digital micrometer to ensure their standardization.

Conventional Ultraspeed D Film

Radiopacity was first measured using conventional dental film (Kodak Ultra speed D DF-57 C, 31x41 mm, Kodak Co, Rochester, NY, USA) in accordance with the ISO-4049 (1988). Forty-two films from the same box were used so that they had the same emulsion numbers. Pure Aluminum (99.99%) step-wedge (1 to 10 mm) was used as a standard. Each x-ray included three specimens from three different materials, enamel, dentin and aluminum step-wedge (Figure 1). A 10-mm cylinder of lead was also placed on each film to ensure an unexposed part of the film that would be used as a correction for inherent film fog. A special holder was mounted to ensure a fixed focus/film distance. Films were exposed for 0.32 seconds at 70 kV and 8 mA using a Siemens dental x-ray unit (Bensheim, Germany). All films were processed immediately in an automatic processor (XR24 Nova Dürr Dental, D-74321, Germany) to minimize any variation in the process.

Radiographic densities, defined as the overall degree of darkening of an exposed film (White & Pharoah, 2000), were measured with a transmission densitometer (X-Rite model 331, MI, USA) with an aperture of 1 mm. Three readings were made for each specimen, including the aluminum step-wedge. For each x-ray, a graph of the optical density versus the thickness of the aluminum was

Table 1: *List of Materials Used in This Study. VLC: Visible Light Cured, CC: Chemically Cured, CS: Composite, CM: Compomer, Flow: Flowable, Pack: Packable, Univ: Universal, Hyb: Hybrid*

Material	Classification	Manufacturer	Batch and Shade
Admira	VLC "Ormocer"	Voco, Cuxhaven, Germany	94545 (A3)
Aeliteflo	VLC Hyb Flow CS	BISCO, Inc, Itasca, IL, USA	039317 (A3)
Amelogen	VLC Hyb Univ CS	Ultradent Products, South Jordan, Utah, USA	2CPM (A2)
Arabesk	VLC Hyb Univ CS	Voco, Cuxhaven, Germany	70500 (A3)
Arabesk-Flow	VLC Hyb Flow CS	Voco, Cuxhaven, Germany	82777 (A3)
Arabesk-Top	VLC Hyb Univ CS	Voco, Cuxhaven, Germany	81594 (A3)
Ariston-pHc	VLC "Smart Material"	Vivadent, Schaan, Liechtenstein	A00001 (-)
Brilliant-Dentin	VLC Hyb Univ CS	Coltène Whaledent, Alstatten, Switzerland	GE931 (A3)
Brilliant-Enamel	VLC Hyb Univ CS	Coltène Whaledent, Alstatten, Switzerland	GE902 (A3)
Charisma-F	VLC Hyb Univ CS	Heraeus Kulzer, Wehrheim, Germany	23 (A20)
Charisma-PPF	CC Hyb Univ CS	Heraeus Kulzer, Wehrheim, Germany	2 (A10)
Clearfil Photo Posterior	VLC Hyb Univ CS	Kuraray, Osaka, Japan	0035A (UL)
Clearfil Photo Anterior	VLC Hyb Univ CS	Kuraray, Osaka, Japan	0024C (A3)
Coltène-SE	VLC Hyb Univ CS	Coltène Whaledent, Alstatten,Switzerland	FBJ01 (A3)
Concise	CC Conventional CS	3M, St-Paul, MN, USA	19970303 (U)
Durafill VS	VLC Microfine CS	Heraeus Kulzer, Wehrheim, Germany	030122 (A3)
Dyract-Flow	VLC Flow CM	Dentsply De Trey, Konstanz, Germany	9809000103 (A2)
Elan	VLC CM	Sybron/Kerr, Orange, CA, USA	805872 (A3,5)
F-2000	VLC CM	3M, St Paul, MN, USA	19970905 (A3)
Flow-Line	VLC Hyb Flow CS	Heraeus Kulzer, Wehrheim, Germany	010021 (A2)
Glacier	VLC Hyb Univ CS	Southern Dental Industries, Victoria, Australia	60506 (B3)
Metafil-CX	VLC Microfine CS	Sun Medical, Shiga, Japan	71201 (A3,5)
P-60	VLC Hyb Pack CS	3M, St Paul, MN, USA	030998 (A3,5)
Pertac-II	VLC Hyb Univ CS	ESPE, Seefeld, Germany	00634764 (A3)
Polofil-Molar	VLC Hyb Univ CS	Voco, Cuxhaven, Germany	63596 (U)
Point-4	VLC Hyb Univ CS	Sybron/Kerr, Orange, CA, USA	003156 (A3)
Prodigy Condensable	VLC Hyb Pack CS	Sybron/Kerr, Orange, CA, USA	904665 (A2)
Pyramid	VLC Hyb Pack CS	BISCO, Inc, Itasca, IL, USA	009803 (A2)
Quadrant Anterior	VLC Hyb Univ CS	Cavex Haarlem, Holland	22C (A2)
Quadrant Posterior	VLC Hyb Pack CS	Cavex Haarlem, Holland	30C (A2)
Revolution	VLC Hyb Flow CS	Sybron/Kerr, Orange, CA, USA	710669 (A3)
Silux-Plus	VLC Microfine CS	3M, St Paul, MN, USA	6DH (U)
Solitaire	VLC Hyb Pack CS	Heraeus Kulzer, Wehrheim, Germany	26 (A30)
Solitaire II	VLC Hyb Pack CS	Heraeus Kulzer, Wehrheim, Germany	VP150499 (A1)
Spectrum	VLC Hyb Univ CS	Dentsply De Trey, Konstanz, Germany	9608244 (A3)
Surefil	VLC Hyb Pack CS	Dentsply De Trey, Konstanz, Germany	980818 (A2)
Tetric-Ceram	VLC Hyb Univ CS	Vivadent, Schaan, Liechtenstein	900513 (A3)
Tetric-Flow	VLC Hyb Flow CS	Vivadent, Schaan, Liechtenstein	901232 (A3)
Wave	VLC Hyb Flow CS	Southern Dental Industries, Victoria, Australia	80608 (A3)
Z-100	VLC Hyb Univ CS	3M, St Paul, MN, USA	19960229 (UD)
Z-250	VLC Hyb Univ CS	3M, St Paul, MN, USA	030998 (A3,5)

drawn and a calibration curve was generated using best-fit logarithmic regression (Cook, 1981; Stanford & others, 1987; Shah & others, 1997). These gave straight-line plots from which the mean net radiographic density values of the materials and their equivalents related to the thickness of the aluminum were derived. The optical density value for each material was the arithmetic mean of the values of the three radiographs.

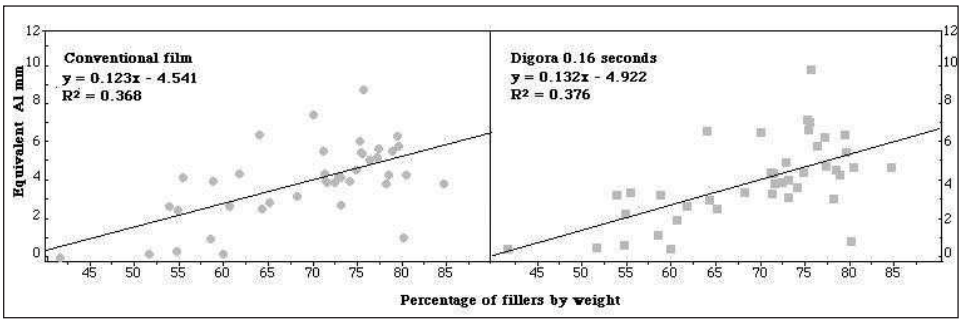

Figure 2. *Correlation between the percentages of fillers by weight and the radiopacity obtained with conventional x-ray films and Digora at 0.16 seconds. Linear regression curve was obtained with r=0.60 and* p≤ *0.05.*

Digital Imaging

The photostimulable phosphor plates (Digora FMX) (n° 2, 30-mm x 40-mm size) were exposed using the same process described above. The same PSP plate was used for all exposures to avoid possible differences between plates. Two different exposure times were used (0.32 and 0.16 seconds). These values were chosen in order to investigate whether reducing the exposure time by half affected the radiopacity. After exposure, gray values of the images had to be calibrated, so images were sent as TIFF files to an image processing software, NIH Image Engineering (http://rsb.info.nih.gov/nih-image). This software allows the transformation of pixel values directly from a linear scale into a scale that correlates with OD (Optical Density).

An approach similar to that used for optical density was used to produce a calibration curve for gray pixel values, then convert those values to equivalent mm Al.

Statistical Analysis

The mean values of the radiopacity of the different materials were compared using a one-way ANOVA and post-hoc Scheffe's tests at $p<0.05$ level. These were performed separately for each technique. Simple regression analyses were carried out to study the relationship between the radiopacity and the percentages of fillers by weight determined in a previous experiment (Sabbagh, Vreven & Leloup, 2002) and also to study the relationship between both techniques.

RESULTS

Table 2 shows the mean values and standard deviations of the radiopacities of the materials investigated and their percentages of fillers by weight. Radiopacities were expressed in millimeters of aluminum (Al), and higher values represented greater radiopacity, ranging between 0.0 for Metafil-CX and 8.8 mm Al equivalent for Tetric-Ceram.

Using conventional x-ray film, considerable differences were found between materials of the same category. Radiopacities of the flowable composites ranged between 2.5 (Aeliteflo) and 6.5 (Tetric-Flow) mm of Al equivalent. Packable resin composites ranged between 2.6 (Solitaire) and 6.4 (Surefil). The same observation was made for the universal hybrid composite, with radiopacities ranging between 2.8 (Polofil-Molar) and 8.8 (Tetric-Ceram) mm of Al equivalent.

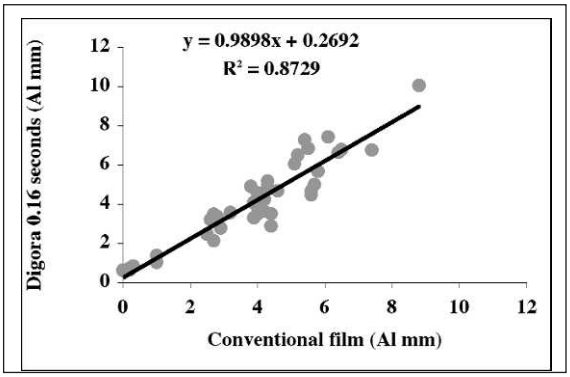

Figure 3. *Correlation between the radiopacity of conventional film and Digora at 0.16 seconds. Linear regression curve was obtained with r=0.93 and* p≤*0.05.*

With the Digora system, considerable variations were also observed among materials of the same category. Values ranged between 0.6 for Clearfil Anterior and Metafil-CX and 10.1 mm Al equivalent for the Tetric-Ceram, respectively.

As a result of regression analyses, linear correlations were found between the percentages of fillers by weight and conventional radiopacities (r=0.60; $p\leq 0.05$) and between the percentages of fillers by weight and Digora radiopacities (r=0.60; $p\leq 0.05$ for both exposure values) (Figure 2).

Radiopacity values measured with Digora, at both exposure times, were nearly identical. This is illustrated by the regression curve (r=0.99; $p\leq 0.05$).

Radiopacities measured with conventional x-ray films correlated significantly with those measured with Digora at 0.32 and 0.16 seconds (r=0.93; $p\leq 0.05$) (Figure 3).

DISCUSSION

This study included a large number of materials representing all the categories of resin-based materials used for direct restorative techniques.

Using conventional x-ray films, all the materials, except the microfine anterior composites Concise (chemically cured resin composite) and Quadrant-Anterior, showed greater radiopacity than dentin. According to the manufacturer, Quadrant-Anterior contains SiO_2 and a small percentage of Ba-Al-F-Si fillers in its formulation (Table 2). Therefore, it is classified as a hybrid rather than a microfine composite. All the hybrid universal (with the exception of Quadrant-Anterior) packable and flowable composites and compomers met ISO-4049 (1988) recommendations. Only a few hybrid composites were less radiopaque than enamel. Apart from Solitaire and Quadrant Posterior, the radiopacity of all packable composites was greater than enamel. Wide variations

Table 2: *Radiopacity mean values expressed in mm Al with standard deviation (SD), obtained with conventional film and Digora at 0.16 and 0.32 seconds, percentages of fillers by weight, type and range of fillers. A separate test (Anova-one factor) was performed for each group. Values represented by the same letters are not significantly different. (Type and range of fillers were obtained from manufacturers. NA : Not Available).*

Materials	Conventional Film Radiopacity	Digora 0.32 Sec	Digora 0.16 Sec	% Fillers Weight	Range of Fillers (mean)	Type of Fillers
Admira	4.2 (0.4)^{e,f,g,h,i,j,k}	4.3 (0.2)^{e,f,g,h,i,j,k}	4.2 (0.1)^{f,g,h,i,j}	73	NA	Quartz, SiO_2
Aeliteflo	2.5 (0.2)^{a,b,c,d,e,f}	2.6 (0.2)^{a,b,c,d,e,f}	2.5 (0.3)^{b,c,d,e}	54.9	0.7 µm	Ba
Amelogen	4.3 (0.6)^{e,f,g,h,i,j,k}	5.3 (0.4)^{g,h,i,j,k,l,m,n}	5.2 (0.0)^{i,j,k,l,m}	72.9	0.7 µm	NA
Arabesk	3.9 (0.2)^{e,f,g,h,i,j,k}	4.5 (0.6)^{e,f,g,h,i,j,k,l}	4.1 (0.2)^{f,g,h,i,j}	71.6	0.5-2 µm (0.05)	NA
Arabesk-Flow	4.4 (1.5)^{e,f,g,h,i,j,k}	2.9 (0.1)^{a,b,c,d,e,f,g}	2.9 (0.1)^{c,d,e,f}	61.8	0.7 µm	SiO_2, Ba, Borosilicate, Sr
Arabesk-Top	4.0 (0.3)^{e,f,g,h,i,j,k}	4.1 (0.5)^{e,f,g,h,i,j}	4.6 (0.2)^{g,h,i,j,k}	71.5	0.05-0.7 µm	NA
Ariston-pHc	4.6 (0.4)^{f,g,h,i,j,k,l}	4.6 (0.2)^{e,f,g,h,i,j,k,l}	4.7 (0.1)^{g,h,i,j,k}	74.8	NA	Ba-Al-F Si, YtF_3
Brilliant-Dentin	5.4 (0.0)^{g,h,i,j,k,l}	7.8 (1.4)^{n,o}	7.3 (0.4)^{n}	75.6	0.04-2.8 µm (0.5)	SiO_2, Ba
Brilliant-Enamel	5.5 (0.4)^{h,i,j,k,l}	7.0 (0.2)^{l,m,n}	6.9 (0.2)^{n}	75.4	0.04-2.8 µm (0.5)	SiO_2, Ba
Charisma-F	5.1 (0.3)^{f,g,h,i,j,k,l}	6.2 (0.6)^{i,j,k,l,m,n}	6.1 (0.2)^{k,l,m,n}	76.4	0.02-2 µm (0.7)	AlF, Ba, SiO_2
Charisma-PPF	3.2 (0.2)^{d,e,f,g,h,i,j}	3.4 (0.3)^{b,c,d,e,f,g,h}	3.6 (0.3)^{d,e,f,g,h}	68.3	0.02-2 µm	Si-F-Al-Ba, SiO_2
Clearfil Photo Anterior	0.2 (0.1)^{a,b}	0.6 (0.0)^{a}	0.7 (0.1)^{a}	59.9	0.04-10 µm (2.8)	SiO_2, Glass, prepolymerized fillers
Clearfil Photo Posterior	3.8 (0.1)^{e,f,g,h,i,j,k}	4.7 (0.1)^{e,f,g,h,i,j,k,l,m}	4.9 (0.1)^{g,h,i,j,k,l}	84.7	0.1-20 µm (4)	SiO_2, Ba, Si
Coltène-SE	4.4 (0.6)^{e,f,g,h,i,j,k}	3.6 (0.1)^{c,d,e,f,g,h,i}	3.5 (0.2)^{d,e,f,g,h}	71.3	0.04-2.7 µm (0.7)	SiO_2, Sr, Ba
Concise	1.0 (0.1)^{a,b,c,d}	0.8 (0.1)^{a,b}	1.1 (0.1)^{a,b}	80.2	1-40 µm (9)	Quartz
Dentin	1.8 (0.2)^{a,b,c,d,e}	1.4 (0.2)^{a,b,c,d}	1.4 (0.1)^{a,b,c}	-	-	-
Durafill VS	0.2 (0.1)^{a,b}	0.9 (0.1)^{a,b,c}	0.7 (0.1)^{a}	51.7	0.01-0.04 µm	SiO_2
Dyract-Flow	4.2 (0.2)^{e,f,g,h,i,j,k}	3.7 (0.1)^{d,e,f,g,h,i}	3.6 (0.3)^{d,e,f,g,h,i}	55.4	(1.6)	Sr-Al-F, Si
Elan	5.6 (0.2)^{i,j,k,l}	4.8 (0.1)^{e,f,g,h,i,j,k,l,m}	4.7 (0.1)^{g,h,i,j,k}	71.2	(1.4)	NA
Enamel	3.1 (0.3)^{c,d,e,f,g,h,i,j}	2.6 (0.2)^{a,b,c,d,e,f}	2.7 (0.2)^{c,d,e,f}	-	-	-
F-2000	4.3 (1.0)^{e,f,g,h,i,j,k}	4.9 (0.1)^{e,f,g,h,i,j,k,l,m}	4.9 (0.1)^{g,h,i,j,k,l}	80.5	1-10 µm (3)	Zr/Si
Flow-Line	4.0 (0.4)^{e,f,g,h,i,j,k}	3.7 (0.3)^{d,e,f,g,h,i,j}	3.5 (0.2)^{d,e,f,g,h}	58.8	0.7 µm	NA
Glacier	3.9 (0.4)^{e,f,g,h,i,j,k}	3.5 (0.5)^{b,c,d,e,f,g,h,i}	3.3 (0.0)^{d,e,f,g}	78.2	40nm-1µm (0.7)	NA
Metafil-CX	0.0 (0.2)^{a}	0.7 (0.2)^{a,b}	0.6 (0.1)^{a}	41.7	20 µm	SiO_2, TMTP
P-60	5.6 (0.1)^{i,j,k,l}	4.6 (0.1)^{e,f,g,h,i,j,k,l}	4.5 (0.2)^{g,h,i,j,k}	78.9	0.01-3.5 µm (0.6)	Zr/Si
Pertac-II	7.4 (1.0)^{l,m}	6.8 (0.4)^{k,l,m,n}	6.8 (0.2)^{m,n}	70	0.1-2 µm	SiO_2, Quartz,YtF_3
Point-4	4.3 (0.3)^{e,f,g,h,i,j,k}	5.0 (0.2)^{f,g,h,i,j,k,l,m}	4.8 (0.1)^{g,h,i,j,k}	73.1	0.4 µm	SiO_2, Ba Al-Si,
Polofil-Molar	2.8 (0.2)^{b,c,d,e,f,g,h}	3.3 (0.4)^{b,c,d,e,f,g,h}	3.4 (0.2)^{d,e,f,g}	78.5	0.05-25 µm	NA
Prodigy Condensable	5.2 (0.4)^{f,g,h,i,j,k,l}	6.4 (0.7)^{j,k,l,m,n}	6.5 (0.4)^{l,m,n}	77.2	0.6 µm	SiO_2, Ba
Pyramid	4.0 (0.1)^{e,f,g,h,i,j,k}	4.2 (0.6)^{e,f,g,h,i,j}	3.9 (0.1)^{e,f,g,h,i}	74.1	NA	NA
Quadrant Anterior	1.0 (0.2)^{a,b,c,d}	1.4 (0.2)^{a,b,c,d}	1.4 (0.1)^{a,b,c}	58.6	<0.1 µm	SiO_2, Ba-Al-F-Si,
Quadrant Posterior	2.9 (0.2)^{b,c,d,e,f,g,h,i}	2.8 (0.0)^{a,b,c,d,e,f,g}	2.8 (0.1)^{c,d,e,f}	65.2	0.1-10 µm	SiO_2,Ba-Al-F-Si, SrF2
Revolution	2.7 (0.1)^{b,c,d,e,f,g}	3.3 (0.2)^{b,c,d,e,f,g,h}	3.5 (0.4)^{d,e,f,g,h}	53.9	(1.7)	Ba
Silux-Plus	0.3 (0.0)^{a,b,c}	0.8 (0.1)^{a,b}	0.8 (0.1)^{a}	54.8	10-50 µm (0.04)	SiO_2
Solitaire	2.6 (0.3)^{a,b,c,d,e,f}	3.5 (0.4)^{c,d,e,f,g,h,i}	3.2 (0.3)^{d,e,f,g}	64.3	0.7-22 µm	SiO_2, Ba-Al-B-Si-F, SrF2
Solitaire II	3.9 (0.4)^{e,f,g,h,i,j,k}	4.1 (0.1)^{e,f,g,h,i,j}	4.1 (0.4)^{f,g,h,i,j}	72.4	0.7-25 µm	SiO_2, Ba-Al-B-Si-F
Spectrum	6.1 (0.5)^{k,l,m}	7.4 (0.1)^{m,n}	7.4 (0.8)^{n}	75.3	0.04-5 µm	SiO_2, Ba, Al, B-Si
Surefil	6.4 (0.3)^{k,l,m}	6.8 (0.2)^{j,k,l,m,n}	6.7 (0.2)^{l,m,n}	79.4	0.04-0.1 (0.8)	Ba-F-Al-BSi
Tetric-Ceram	8.8 (0.3)^{m}	10.1 (0.7)^{o}	10.1 (0.1)^{o}	75.7	0.04-3 µm (0.7)	SiO_2, Ba-Al-F, YbF_3
Tetric-Flow	6.5 (0.1)^{k,l,m}	6.9 (0.2)^{l,m,n}	6.8 (0.4)^{m,n}	64	0.04-3 µm (0.7)	SiO_2, Ba-Al-F, YbF_3
Wave	2.7 (0.4)^{b,c,d,e,f,g}	2.2 (0.0)^{a,b,c,d,e}	2.1 (0.2)^{a,b,c,d}	60.7	(1.5)	Sr
Z-100	5.8 (0.2)^{j,k,l}	5.5 (0.9)^{h,i,j,k,l,m,n}	5.7 (0.3)^{j,k,l,m,n}	79.6	0.01-3.5 µm (0.6)	Zr/Si
Z-250	5.7 (0.2)^{j,k,l}	5.2 (0.2)^{f,g,h,i,j,k,l,m,n}	5.0 (0.4)^{h,i,j,k,l}	77.4	0.01-3.5 µm (0.6)	Zr/Si

in radiopacity were observed among the same group of materials (Table 2).

According to Langland and Langlais (1997), radiopacity is influenced by several factors classified as primary and secondary factors. Primary factors include milliampere, exposure time, kilovoltage and source-film distance, while secondary factors concern development conditions, type of film, intensifying screens and grids. Since two completely different systems were used in this study, factors relating to the imaging system were expected to differ.

Filler percentages and their types are the most important factors that influence materials' radiopacity. In fact, as a result of regression analysis, linear correlation was found between radiopacity values and the percentages of fillers by weight (Figure 2), which is in line with the findings of Toyooka and others (1993). A closer look at this curve shows two opposite groups of resin-based materials deviating from the center of the curve. The first group includes Pertac-II, Tetric-Ceram and Tetric-Flow, with high radiopacities, while the second one contains materials with low radiopacities (Metafil-CX, Durafill-VS, Silux-Plus, Clearfil Anterior and Concise). As shown in Table 2, these materials contain different kinds of fillers. SiO_2 is the only filler used in Metafil-CX, Durafill-VS, Silux-Plus and Clearfil Anterior. The quartz fillers used in Concise are also based on silicium. This element has a low atomic number (14) and does not provide radiopacity. On the other hand, Pertac-II, Tetric-Ceram and Tertic-Flow contain Yttrium (Y atomic number 39) and Ytterbium (Yb atomic number 70). These elements have high atomic numbers and provide a high level of radiopacity. Barium (Ba atomic number 56) is the element most commonly incorporated into composite-based resins to increase radiopacity (Table 2).

Watts (1987) found that radiopacity values higher than those in enamel can be achieved in composites with a filler loading of approximately 70% by volume when the mass percentage of radiopaque oxide in the filler particles exceeds about 20%. Toyooka and others (1993) demonstrated that radiopacity is linearly proportional to the amount of radiopaque oxide in the filler.

Some of the materials used in this study were investigated by other researchers (Toyooka & others, 1993; Bouschilcher & others, 1999; Murchison, Charlton & Moore, 1999; Choi & others, 2000; Lutz & Krejci, 2000). In some cases, the authors did not follow the ISO-4049 (1988) recommendations. Murchison and others (1999) and Choi and others (2000) observed lower radiopacities than those obtained in this study. Using higher exposure times or kV or mA may explain the differences observed.

Although variations in test methodologies render comparisons between the studies difficult, the results of this investigation are in line with some other studies. Toyooka and others (1993) had similar radiopacities for Clearfil-Photo Posterior, enamel and dentin. Bouschlicher and others (1999) used the same parameters as recommended by the ISO-4049. Apart from Pertac-II, Tetric-Flow and Z-100 (which differed slightly), the tested resin composites and dentin and enamel had radiopacity values similar to those in this study. Recent changes in the formulation of these materials may be responsible for these variations. Other variables such as speed of the film or processing solution (temperature and age) could have influenced the results.

A digital imaging technique, the Digora system, was also used to study the radiopacity of resin-based materials using two exposure times (0.16 and 0.32 seconds). Similar results were observed with both exposure times, as shown in Table 3. Having carried out regression analysis, linear correlation was found between the radiopacity values and percentages of fillers by weight.

As with conventional x-ray films, differences between the same groups of composites were observed. Although a strong correlation exists between both techniques (Figure 3), for some materials radiopacity measured with the Digora system was lower or higher than that obtained with the conventional technique. Enamel and dentin showed lower values with Digora.

In order to understand the variations observed between the conventional and digital methods, some information and explanations regarding their technologies need to be noted.

A conventional radiographic image consists of the arrangement of silver grains in photographic emulsion. The density of the silver grains depends on the intensity of the x-ray beam (Van der Stelt, 2000). The radiation contrast is an analog signal with continuous intensities.

In digital systems, output of the measurements is stored on the computer as numbers. These values are absolute numbers of available gray shades (from 0 to 255) in contrast to the continuous density curve in the analog film image (Van der Stelt, 2000). The Digora PSP system has a much wider dynamic range than film, giving this system a much broader exposure latitude (White & Pharoah, 2000). An important characteristic of a receptor is its latitude. Latitude refers to the range of useful densities that can be recorded (Miles, 1992). In practical terms, this means that Digora can provide a similar quality of image irrespective of whether the exposure is relatively low or high and, thus, eliminates all under and over exposure (Hayakawa & others, 1998).

This explains why other exposures times (0.32 and 0.16 seconds) produced similar radiopacities.

Moreover, Digora has an autoranging function whereby signal intensities from an exposed phosphor are obtained during a prescan and are used to adjust gains

for the internal amplifiers. As a result, there is no direct relationship between the measured pixel value and phosphor exposure (Huda & others, 1997).

The image "noise" is not the same in an exposed and unexposed plate as it is in a film (background fog) since, in an exposed plate, the program will find sufficient information to extract (Stamatakis, Welander & McDavid, 1999). Thus, it was not necessary to perform any subtraction (as is done with conventional x-ray films) when calculating the radiopacity.

Stamatakis and others (1999) studied the dose response of the Digora system. Special software was required to obtain information about the values of the internal registers of the scanner's microcontroller. They concluded that the gray levels may be used as a relative measure of exposure.

Wenzel and others (1998) compared the radiopacity of some resin-based materials to amalgam using a conventional film (Ektaspeed-Plus) and a phosphor plate system (Digora). Amalgam had the highest density with Ektaspeed-Plus film, while with the Digora system, it was not significantly different from Herculite. With regard to the digital systems, differences between the distributions of the gray shade values for the various materials were less significant than those observed when using film. As stated above, digital systems operate with an absolute number of available gray shades (256) in contrast to the continuous density curve in the analog film image. Wenzel and others (1998) concluded that when using conventional film radiographs, filling materials can be differentiated with a high level of probability, while the digital system is less reliable.

This study shows that gray pixel values could be converted into millimeters of aluminum using special software and this aluminum could be used to measure the radiopacity of resin-based materials.

CONCLUSIONS

1. Most of the materials investigated were complied with ISO-4049 and were more radiopaque than enamel when specimens 2-mm thick were used. Different levels of radiopacities were observed among materials of the same group.
2. Some flowable and packable composites are less radiopaque than enamel and may cause some confusion regarding the diagnosis of secondary caries when used in posterior cavities.
3. The radiopacity obtained with the Digora system correlated with conventional x-ray films. Consequently, the conversion of pixel values into equivalent millimeters of aluminum seemed adequate.
4. Many factors affect radiopacity measurements. The type and percentage of fillers by weight determine the radiopacity of resin-based materials. Exposure time did not affect digital radiopacity, because of the wide latitude of the phosphor storage plate.
5. There is a great need for the standardization of radiopacity measurements using digital imaging techniques.

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Effect of Conditioner on Bond Strength of Glass-ionomer Adhesive to Dentin/Enamel With and Without Smear Layer Interposition

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Clinical Relevance

Although bonding of a glass-ionomer adhesive to dentin can be achieved without the use of a polyalkenoic acid conditioner, even with the interposition of a smear layer, instrumented and non-instrumented enamel requires separate conditioning to provide sufficient micro-mechanical retention.

SUMMARY

The effect of a polyalkenoic acid conditioner pretreatment on the bond strength of a glass-ionomer adhesive to tooth substrates with or without smear layer was evaluated. Smear-layer covered and smear-layer free dentin and enamel surfaces were prepared from 24 extracted human molars. Resin composite was bonded to the surfaces using FujiBond LC with or without a polyalkenoic acid conditioner and subjected to microtensile bond strength (μTBS) testing. Failure modes were determined using scanning electron microscopy. For dentin, smear-layer coverage and conditioner treatment did not reveal significant differences in μTBS, which ranged from 20 to 29 MPa. For enamel, smear-layer coverage did not significantly affect μTBS, whereas,

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the use of conditioner significantly improved μTBS, reaching the same μTBS-values as when bonded to dentin. Regarding failure mode, most dentin specimens failed mixed adhesive cohesively. For enamel, adhesive failures mostly occurred when no conditioner was used, though mixed failures were predominant when the specimens were conditioned beforehand. Bonding of the glass ionomer adhesive to dentin can be achieved without the separate use of a polyalkenoic acid conditioner, even with the interposition of a smear layer. However, instrumented and non-instrumented enamel requires separate conditioning to provide sufficient micro-mechanical retention.

INTRODUCTION

In addition to the interpositioning of a resin-based adhesive between the restorative material and the remaining tooth structure, bonding to tooth tissue can also be clinically achieved directly using glass-ionomer cements (Mount, 1994). Both glass-ionomer and resin technology have converged in the latest development of glass-ionomer-based adhesives. Some recent research reports have illustrated the beneficial and unique characteristics of a commercially available glass-ionomer adhesive (FujiBond LC, GC, Tokyo, Japan). This system was reported to have a fairly high tensile and shear bond strength (Gordan, Boyer & Söderholm, 1998; Inoue & others, 2001b) and was highly successful in retaining resin composite in non-undercut cervical cavities for a minimum of five years (Burrow & Tyas, 1998; Tyas & Burrow, 2001, 2002). Moreover, like other conventional and resin-modified glass ionomer cements, fluoride release from this adhesive was reported to be greater than from resin-based materials. Increased resistance to demineralization from an acid attack was confirmed as well (Francci & others, 1999).

The glass-ionomer adhesive has been documented to produce a hybrid layer with a thickness of about 0.5-1 μm (Van Meerbeek & others, 1998, 2000). This hybrid layer was formed upon partial demineralization achieved through the use of 20% polyalkenoic acid conditioner. This conditioner "cleans" the dentin surface (removal of smear debris) without completely unplugging the dentin tubules (Inoue & others, 2001). Within the hybrid layer, hydroxyapatite (HAp) crystals are not completely removed from collagen (Van Meerbeek & others, 1998; 2000). Consequently, these HAp-coated collagen fibrils offer not only micro-mechanical retention sites for hybridization (Van Meerbeek & others, 1998, 2000), but they also serve as receptors for primary chemical (ionic) bonding with the carboxyl groups of polyalkenoic acid (Yoshida & others, 2000). Thus, a two-fold micromechanical and chemical bonding mechanism has been demonstrated for this particular adhesive.

So far, the effect of bonding to ground versus fractured substrate surface and the effect of the separate use of a polyalkenoic acid conditioner on the bonding effectiveness of a glass ionomer adhesive have not been investigated. Such information may help to better understand how much each of the two bonding mechanisms, micromechanical versus chemical, contributes to the final bond strength and stability. Therefore, the objective of this study was to evaluate the effect of a polyalkenoic acid conditioner pre-treatment on the bond strength of the glass ionomer adhesive FujiBond LC (GC) to both dentin and enamel with or without smear layer interposition, using a microtensile bond strength testing methodology (Sano & others, 1994).

METHODS AND MATERIALS

Twenty-four caries-free, non-restored human third molars stored in an aqueous solution of 0.5% chloramine at 4°C were used within one month of extraction. Four kinds of substrate surfaces were prepared:

1. Smear Layer-covered Dentin

Mid-coronal dentin parallel to the occlusal surface was exposed by removing occlusal enamel using an Isomet low-speed diamond saw (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA). A standard smear layer was produced by wet-sanding the dentin surface with 600-grit silicon carbide sandpaper for 60 seconds.

2. Smear Layer-free Dentin

A 4-mm deep groove was made around the tooth crown 2-to-3 mm above the cement-enamel junction using the low speed diamond saw (Isomet 1000) under water. Then, the tooth was fractured at the groove using forceps to expose the mid-coronal flat dentin.

3. Smear Layer-covered Enamel

Two-thirds of the root of the molars was removed perpendicular to the long axis of the tooth, after which the remaining part was cut in half mesio-distally using the Isomet. Buccal or lingual enamel was flattened with a diamond disk (918B.104.220, Komet, Lemgo, Germany) in a low-speed straight handpiece prior to being wet ground using 600-grit silicon carbide paper for 60 seconds in order to produce a standard smear layer.

4. Smear Layer-free Enamel

Two-thirds of the root of the molars was removed perpendicular to the long axis of the tooth, after which the remaining third was cut in half mesio-distally using the Isomet. The intact buccal or lingual enamel surface was cleansed using water-pumice slurry and a rubber cup in a slow speed handpiece. The flattest surfaces were selected for bonding to the adhesive.

Bonding Procedures

All surfaces were first thoroughly washed with water and immediately dried with moisture-free air. The

Table 1: *Composition and Manufacturer's Recommended Bonding Protocol of the Glass Ionomer Adhesive Tested*

Components	Lot #	Composition	Application
Conditioner	020981	20% polyalkenoic acid, 3% aluminum chloride	10 seconds, rinse, air-dry without desiccation
Powder	250881	Fluoro-alumino-silicate glass	10 seconds mix, apply by brush, 20 seconds light-cure
Liquid	010981	Polyalkenoic acid, 2-hydroxyethyl methacrylate, Dimethacrylate, Camphoroquinone, Water	

glass-ionomer adhesive (FujiBond LC) was used either with or without the polyalkenoic acid conditioner (Cavity Conditioner, GC) strictly following the manufacturers' instructions (Table 1). For the specimen in the group "with conditioner," the dentin and enamel specimens were conditioned with Cavity Conditioner (Table 1) for 10 seconds, rinsed thoroughly and air-dried without desiccation. FujiBond LC was then applied onto the conditioned surface. Two drops of liquid and one level spoonful of powder were placed in a disposable dish and mixed with a brush for 10 seconds. The cement mixture was then applied to the conditioned surface using the brush and light-cured for 20 seconds using a light-curing device (Optilux 500, Demetron/Kerr, Danbury, CT USA). For the specimen in the group "without conditioner," conditioning of the dentin and enamel surfaces using Cavity Conditioner (as mentioned above) was not performed.

Microtensile Bond Strength (μTBS) Testing

Immediately following bonding procedures, four layers of resin composite (Z-100, shade A3, Lot 9EE, 3M, St Paul, MN, USA) were incrementally built up to a height of approximately 5 mm. Each layer was light-cured for 20 seconds.

After completing the resin composite build-up, all specimens were stored at 37°C in water for one week to allow the acid-base reaction to proceed. For dentin, the specimens were sectioned into three to five slabs approximately 0.7-mm thick parallel to the long axis of the tooth using the diamond saw (Isomet) under water. For enamel, the specimens were sectioned into two-to-three rectangular forms 1-mm thick and 2.5-mm wide perpendicular to the resin-enamel interface (Inoue & others, 2003). The dentin slabs and the rectangular enamel specimens were trimmed into an hourglass shape using a high-speed superfine diamond bur (835KREF, Komet, Lemgo, Germany), ensuring that the narrowest portion was located at the bonding interface. Consequently, the bonded surface area to be pulled apart was approximately 1 mm^2. The trimmed specimens were then attached to "Ciucchi's device" (Pashley & others, 1999) using a cyanoacrylate glue (Model Repair II Blue, Sankin Kogyo KK, Otahara, Japan) and subjected to microtensile bond testing (Sano & others, 1994; Pashley & others, 1999) in a computer-controlled desktop material testing machine (LRX, Lloyd, Hampshire, UK) with a crosshead speed of 1 mm/minute.

After testing, the width and thickness of the specimen were precisely measured using a three-dimensional precision measuring instrument transformed from an x-y multipurpose modular microscope (Leitz, Wetzlar, Germany) (Lambrechts & others, 1984). Using the same instrument, the remaining dentin thickness (RDT) was measured for the dentin specimens and recorded as the distance vertically from the center of the tested interface to the pulp chamber. In this study, only "mid-coronal" dentin surfaces, of which RDT was between 2 and 3 mm, were included.

Failure Analysis Using Scanning Electron Microscopy

To determine the mode of failure, the broken interface specimens were immersed in 2.5% glutaraldehyde in 0.1M sodium cacodylate buffer at pH 7.4 for 12 hours at 4°C. After fixation, they were rinsed in 0.2M sodium cacodylate buffer at pH 7.4 for one hour, with three changes, followed by distilled water for one minute. They were then dehydrated in ascending grades of ethanol (25% for 20 minutes, 50% for 20 minutes, 75% for 20 minutes, 95% for 30 minutes and 100% for 60 minutes), immersed in hexamethyldisilazane (HMDS, Electron Microscope Sciences) for 10 minutes (Perdigão & others, 1995), placed on filter paper inside a covered glass vial and left to air-dry at room temperature. The specimens were then mounted on aluminum stubs using a conductive carbon cement (Leit-C, Neubauer Chemikalien, Munster, Germany) coated with gold at 15 mA for 90 seconds and observed using a field-emission scanning electron microscope (Fe-SEM) (XL-30, Phillips, Eindhoven, The Netherlands) at an accelerating voltage of 10 kV.

Statistics

One-way and two-way ANOVA and Scheffe's multiple comparison test were used to statistically analyze the μTBS data at a confidence limit of 95%.

RESULTS

Figure 1 shows the results of μTBS to smear-layer covered and smear-layer free dentin and enamel with or without prior polyalkenoic acid conditioning. For dentin, both experimental variables, the smear layer and conditioner did not reveal statistically significant differences in μTBS, which ranged from 20 to 29 MPa (two-way ANOVA, $p>0.05$). For enamel, while smear layer coverage did not affect μTBS, the use of condi-

tioner significantly improved the μTBS. Moreover, when comparing the μTBS to dentin with that to enamel, there were no significant differences between the respective μTBSs when the conditioner was used, although when the conditioner was not used, the μTBS to dentin was significantly higher than that to enamel. There were no pre-testing failures for any of the experimental groups.

The results of the failure mode analysis are shown in Table 2. Figures 2 through 5 are representative photomicrographs illustrating the different failure patterns. For dentin, most specimens failed mixed adhesive-cohesively, except for a high number of adhesive failures that occurred between FujiBond LC and composite when a smear layer was interposed and the conditioner was used. For enamel, most failures were adhesive in nature when no conditioner was used. When the specimens received polyalkenoic acid conditioning beforehand, mixed adhesive-cohesive failures were predominant.

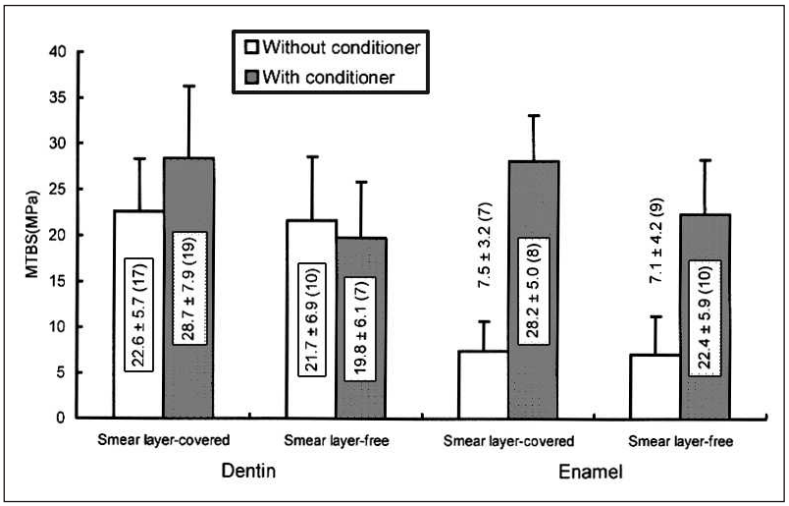

Figure 1. *Microtensile bond strength to smear layer-covered and free dentin and enamel with or without Cavity Conditioner treatment. Values are presented as mean ± SD (number of specimens).*

DISCUSSION

According to a classification based on the number of clinical steps and the underlying mechanism of bonding to tooth substrate, the glass-ionomer adhesive FujiBond LC should be regarded as belonging to a separate category next to those of "etch&rinse" and "self-etch" resin-based adhesives (Van Meerbeek & others, 2000, 2001, 2003; Inoue & others, 2000a). With regard to its composition, FujiBond LC is a diluted version of the restorative resin-modified glass-ionomer cement Fuji II LC "(GC)". One major feature of this adhesive is its two-fold micromechanical and chemical bonding mechanism to enamel and dentin (Van Meerbeek & others, 1998; Yoshida & others, 2000).

Micromechanical interlocking occurs through hybridization, which means that micro-porosities are created at the tooth surface and become inter-diffused by adhesive components that, *in situ,* set partially by polymerization and partially by acid-base reaction. In this way, a relatively thin hybrid layer is formed that consists of a zone of partially demineralized dentin with residual HAp crystals still attached around individual collagen fibrils. Transmission electron

Table 2: *Failure Modes in Function of Tooth Substrates, Smear Layer and Use of Conditioner*

Tooth	Smear Layer	Conditioner	Adhesive Failure		Cohesive Failure			Mixed Failure	Total
			Tooth-FBLC	FBLC-Comp[1]	Dentin	FBLC	Comp[1]		
Dentin	Covered	Without	4 (24%)	1 (6%)	0 (0%)	1 (6%)	1 (6%)	10 (59%)	17 (100%)
		With	2 (11%)	9 (47%)	0 (0%)	3 (16%)	0 (0%)	5 (26%)	19 (100%)
	Free	Without	1 (10%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	9 (90%)	10 (100%)
		With	1 (14%)	0 (0%)	0 (0%)	2 (29%)	0 (0%)	4 (57%)	7 (100%)
Enamel	Covered	Without	6 (86%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	1 (14%)	7 (100%)
		With	0 (0%)	0 (0%)	0 (0%)	1 (12%)	0 (0%)	7 (88%)	8 (100%)
	Free	Without	8 (89%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	1 (11%)	9 (100%)
		With	0 (0%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	10 (100%)	10 (100%)

[1]*Comp = Resin composite*

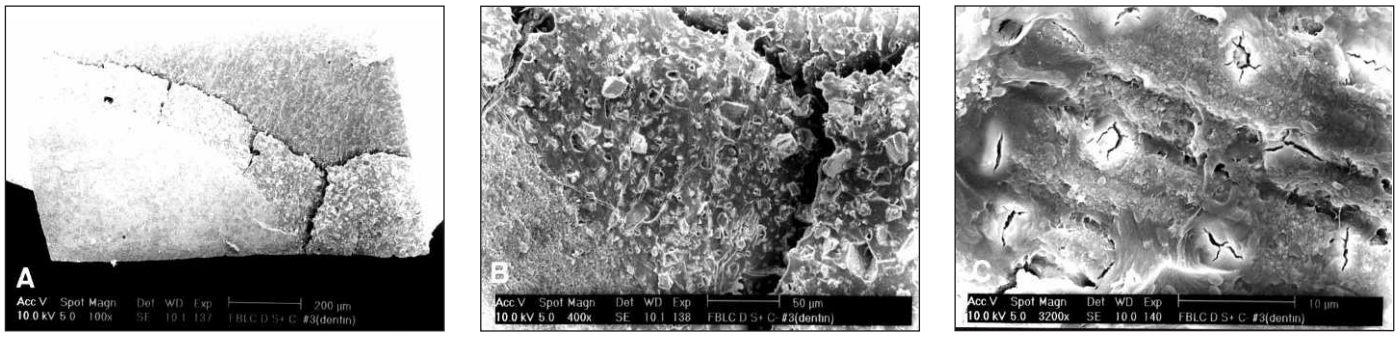

Figure 2. *Fe-SEM photomicrograph of the dentin failure side of a specimen (A) that was bonded using FBLC without polyalkenoic-acid conditioning to smear layer-covered dentin. The interface mainly failed partially cohesively within the resin composite, cohesively in FBLC and adhesively between dentin and FBLC. (B) shows a higher magnification of the cohesive failure of FBLC, in which the glass-ionomer filler are clearly observable. (C) shows an area where the specimen failed adhesively. The tubules are occluded with debris representing the smear layer that was prepared, but not removed as no conditioner was used.*

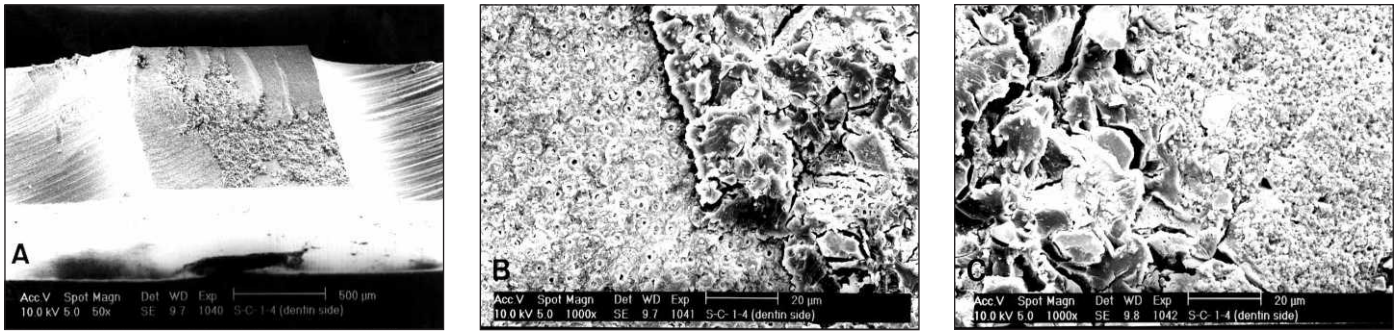

Figure 3. *Fe-SEM photomicrograph of the dentin failure side (A) of a specimen that was bonded using FBLC without polyalkenoic acid conditioning smear layer-free (fractured) dentin. The interface failed mixed adhesive-cohesively. Higher magnification in (B) shows a cohesive failure of FBLC (right side) and an adhesive failure between dentin and FBLC (left side). Open tubules can be seen on the latter failure pattern. The right side shows an irregular surface, representing the glass-ionomer material that crumbled inside the microscope due to dehydration and the high vacuum. The high-magnification photomicrograph in (C) shows that in this particular area resin composite was fractured cohesively.*

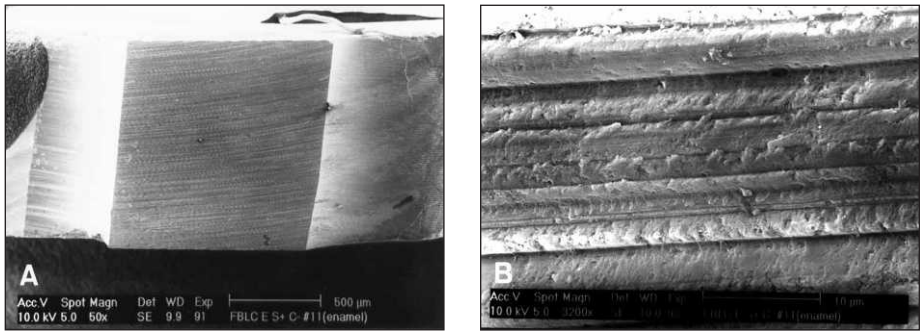

Figure 4. *Fe-SEM photomicrograph of the enamel failure side (A) of a specimen that was bonded using FBLC without polyalkenoic acid conditioning to smear layer-covered enamel. A typical homogeneous adhesive failure pattern was revealed between enamel and FBLC. In the higher magnification image of (B), scratches disclose the presence of a smear layer on enamel that was not removed.*

microscopy (TEM) (Van Meerbeek & others, 1998, 2001; Inoue & others, 2000a), atomic force microscopy (AFM) (Yoshida & others, 1999) and scanning electron microscopy (SEM) (Inoue & others, 2001a) demonstrated a so-called "gel phase" deposited as a submicron amorphous phase on top of the thin hybrid layer. This phase was found to be the morphological manifestation of a gelation reaction of the polyalkenoic acid with calcium that was extracted from the underlying dentin. The role of this phase, either contributing or weakening the bond stability, needs further in-depth study. The hybrid layer itself is much thinner than the typical 3-5 µm hybrid layer produced by etch&rinse adhesives (Van Meerbeek & others, 2000, 2001, 2003; Inoue & others, 2000a). Except for the gel phase, the interfacial ultrastructure produced by FujiBond LC at dentin rather resembles that observed with "mild" (resin-based) self-etch adhesives (Inoue & others, 2000a; Van Meerbeek & others, 2003). Such adhesives have a pH of about 2 and also only partially demineralize dentin up to a depth of about 1 µm.

The chemical bonding component is based on the formation of ionic bonds between the carboxyl groups of

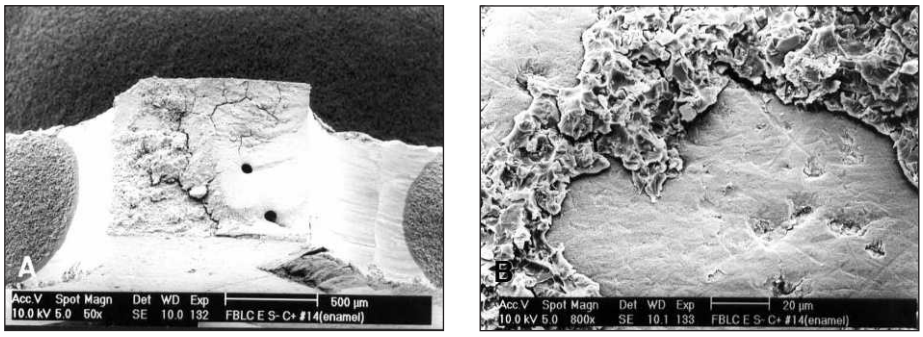

Figure 5. *Fe-SEM photomicrograph of the enamel failure side (A) of a specimen that was bonded using FBLC including beforehand polyalkenoic acid conditioning to smear layer-free enamel. This image shows a mixed adhesive-cohesive failure pattern. In the higher magnification image of (B), parts of FBLC were still attached to enamel that clearly was not prepared as no smear layer scratches could be observed.*

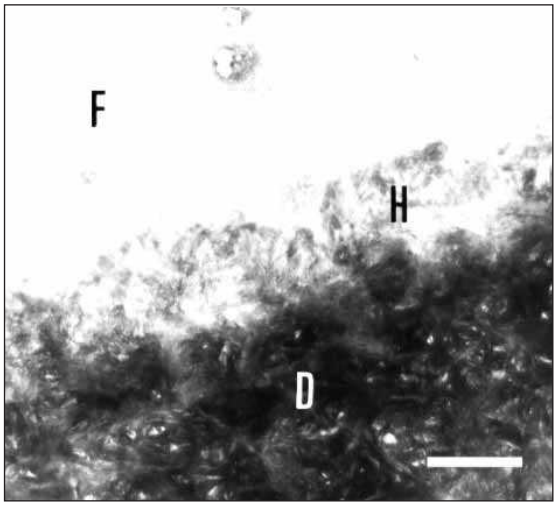

Figure 6. *TEM photomicrograph of the interface between FBLC (F) and smear-layer covered dentin (D) without polyalkenoic acid conditioning. Partial demineralization of the dentin surface still resulted in hybrid-layer (H) formation (0.5 to 1 µm), although a "gel phase" was not deposited on top of the hybrid layer. Bar = 0.5 µm.*

the polyalkenoic acid and calcium of residual HAp that is kept around the exposed collagen fibrils (Van Meerbeek & others, 1998, 2001, 2003; Inoue & others, 2000a). Cavity Conditioner and FujiBond LC contain a 10:1 acrylic/maleic polyalkenoic acid co-polymer (Yoshida & others, 2000) and, thus, both can contribute to this primary chemical bonding potential.

From the results of this study, it appeared that whether or not dentin was covered with a smear layer, the conditioner treatment did not reveal statistically significant differences in µTBS. When the conditioner was not used, TEM in another study revealed that partial demineralization of the dentin surface still resulted in hybrid-layer formation (0.5 to 1 µm), although a "gel phase" was not deposited on top of the hybrid layer (Figure 6) (Inoue & others, 2000b). Such shallow hybridization must be attributed to the self-etching ability of FujiBond LC, which is rather weak. Shimada and others (1999) reported that acid-containing cements such as zinc phosphate and glass-ionomer cements have self-etching properties that are effective in removing the smear layer and promoting close adaptation to dentin surfaces. In resin-based adhesives, particularly self-etch adhesives, the interposition of a smear layer is thought to be one of the major problems affecting their bonding efficacy. A substrate covered by a relatively thick smear layer is more difficult for the self-etch adhesive to penetrate through and find adequate micromechanical interlocking in the tooth substrate underneath (Miyasaka & Nakabayashi, 1999). For instance, coarse diamond burs operated at high speeds have been reported to lower the bond strength of some self-etching priming systems, mainly due to insufficient self-etch capacity (Ogata & others, 2001). Thin and relatively permeable smear layers are less likely to hinder acidic monomers in dissolving the smear layer up to the underlying dentin (Tay & others, 2000a,b; Inoue & others, 2001a,b). In this study, both the polyalkenoic-acid conditioner (Cavity Conditioner, pH= 1.2) and FujiBond LC have a rather weak etching potential. Nevertheless, FujiBond LC on its own and FujiBond LC in combination with a polyalkenoic acid conditioner resulted in µTBSs comparable to those when dentin was fractured and no smear layer was interposed.

Following failure mode analysis, a high number of adhesive failures between FujiBond LC and resin composite were observed when the conditioner was used on smear-layer covered dentin. Although the authors do not currently have any logical explanation for this effect, inadequate matching between the glass-ionomer and resin composite may be a plausible reason.

At dentin, the µTBS was not affected by the interposition of a smear layer, which could be explained by the fact that FujiBond LC itself is sufficiently acidic and has sufficient self-etching potential to achieve adequate micromechanical interlocking (Figure 6). The contribution of the chemical bonding component to the overall bond strength remains unknown, but most likely is less relevant.

On enamel, the additional application of Cavity Conditioner prior to application of the glass-ionomer adhesive FujiBond LC had a significant positive effect on the µTBS. This most likely means that though FujiBond LC might have sufficient self-etching potential on dentin, it might not be sufficiently effective on enamel. Probably, FujiBond LC, alone, may not be

capable of (in case of smear layer-free enamel) providing sufficient micro-retention at aprismatic enamel or (in case of smear layer-covered enamel) removing the smear layer and exposing a micro-retentive etch pattern underneath. Hence, a separate conditioning step with a polyalkenoic acid is required, as was confirmed by the failure-mode analysis (Table 2). When no conditioner was used, most failures were "adhesive" in nature, while mixed "adhesive-cohesive" failures were predominant when the specimens received treatment by Cavity Conditioner prior to the application of FujiBond LC (Table 2). When FujiBond LC was bonded to smear-layer free enamel, the surface should be ideally receptive to chemical bonding. Nevertheless, the μTBS of FujiBond LC (applied without Cavity Conditioner) to the smear-layer free enamel was rather low, so that the authors may conclude that the contribution of the chemical self-adhesive mechanism of FujiBond LC to the overall bond strength might be relatively low.

These findings are in agreement with a report (Nitta, 1992) on the bond efficacy of the restorative material Fuji II LC (GC) to enamel. That study also revealed that the use of a conditioner significantly increased the tensile bond strength of Fuji II LC (GC) to enamel. The conditioner used in that study was Dentin Conditioner (GC), which contained 10% polyalkenoic acid in water. According to SEM analysis in that report (Nitta, 1992), the conditioner partially dissolved the smear layer (#600-grit SiC paper) and partially exposed the structure of enamel rods. Due to the higher concentration (20%) of polyalkenoic acid, Cavity Conditioner used in this study has a more aggressive etching ability than Dentin Conditioner. Cavity Conditioner most likely removed not only the smear layer, if any, but also somewhat demineralized the enamel surface. FujiBond LC could then readily adapt to the irregular enamel surface and find sufficient micro-mechanical interlocking by resin-tag formation into the created etch pits at enamel.

From the results of the μTBS to enamel, the overall bond strength of 22-28 MPa should mainly be attributed to the combined effect of micromechanical interlocking (due to etching by Cavity Conditioner and the self-etching ability of the adhesive) and additional chemical bonding. A bond strength of about 7 MPa resulted from micromechanical interlocking (only due to the self-etching ability of FujiBond LC) and additional chemical bonding. Consequently, the major factor determining the overall bond strength of FujiBond LC to enamel appears to be achieved through micromechanical retention induced by the conditioner, rather than by chemical interaction with the substrate surface.

However, the authors currently hypothesize that the two-fold micromechanical and chemical bonding mechanism still is beneficial. They speculate that micromechanical attachment is essential to resist acute debonding stress (such as that which resin-dentin bonds are subjected during μTBS testing). Chemical bonding may be important, especially in achieving and preserving better sealing of margins and thus extending the clinical longevity of adhesive restorations. Further investigation is needed to confirm this hypothesis.

CONCLUSIONS

Bonding of FBLC to dentin can be achieved through micromechanical interlocking by means of hybridization following demineralization of dentin by a separate polyalkenoic acid conditioner or by the self-etching effect of the adhesive, itself, even with the interposition of a smear layer. However, instrumented and non-instrumented enamel absolutely requires separate beforehand conditioning using a polyalkenoic acid copolymer to provide sufficient micromechanical retention for this glass-ionomer adhesive.

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An *In-vitro* Microtensile Test of Scotchbond Multi-Purpose Adhesive Applied at Different Priming Times

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Clinical Relevance

This study investigated the microtensile bond strength of a water-based adhesive (Scotchbond Multi-Purpose) whose primer was allowed to dwell on the surface of etched dentin according to manufacturers' directions and 30, 60 and 120 seconds longer than manufacturers' directions to determine whether longer dwelling times would result in higher microtensile bond strengths of adhesive to dentin at the gingival wall of Class II resin composite restorations.

SUMMARY

Adhesive bonding to dentin can fail if the dentin is too wet during application of the bonding resin. This study compared the *in vitro* 24-hour microtensile bond strength of teeth restored at four different priming times at the gingival cavity wall of Class II resin composite restorations. After IRB approval, six pairs of extracted third molars (yielding 12 teeth) received a proximal Class II prep/restoration in each tooth. Each pair was from the same patient. Four treatment groups were randomly assigned for each pair. The treatment groups were: TM–primer applied and dried according to manufacturer's directions; T30–primer allowed to dry for an additional 30 seconds; T60–primer dried for an additional 60 seconds; T120–primer dried for an additional 120 seconds. The teeth were restored with 3M ESPE Scotchbond Multi-Purpose Dental Adhesive and 3M ESPE Z100 Restorative. Manufacturers' directions were followed except for the additional primer dwelling times. The teeth were sectioned to obtain rectangular specimens with a surface area of approximately 0.5 mm². Samples were tested on the Instron at 1.0 mm/minute until failure. The results in megapaschals were TM (n=15) 25.5 ± 12.2; T30 (n=14) 22.7 ± 13.6; T60 (n=15) 28.1 ± 14.7; T120 (n=20) 27.7 ± 15.2. Samples that debonded during the preparation phase and could not be tested from each group were TM=5, T30=6, T60=5, T120=1. A one-way ANOVA found no statistically significant difference between groups. Ninety percent of the samples broke through the adhesive layer as observed under the scanning electron microscope at 2000x. A chi square analysis found no difference in the

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number of debonds between groups. Increasing the primer drying time did not increase the microtensile bond strength of adhesive bonded to the dentin gingival wall.

INTRODUCTION

A previous study, which used a water-based adhesive, Scotchbond Multi-Purpose (SBMP), bonded to axial vs gingival walls in Class II preparations, found that the bond to the gingival wall was weaker than the bond to the axial wall (Purk & others, 2004). This weaker bond strength may be due to an increased amount of voids at the gingival wall, possibly caused by greater wetness at the gingival rather than the axial wall. If the dentin surface is too wet, an overwet phenomenon occurs, whereby the adhesive resin does not fully penetrate the dentin tubules or the demineralized dentin (Pereira & others, 2001; Spencer & others, 2000; Tay, Gwinnett & Wei, 1996a, 1996b, 1996c, 1996d; Van Meerbeek & others, 1998). Excessive surface moisture may result in voids at the resin-dentin interface (Walshaw & McComb, 1996). Water that is not completely removed within the interdiffusion zone can interfere with polymerization of the resin (Tanumiharja, Burrow & Tyas, 2000). The increased wetness may come from either intrinsic (dentin tubules) or extrinsic sources (water primer, sulcular fluid, humidity of the operatory or water left on the dentin after the acid is washed off). Any solvent remaining on a primed dentin surface will prevent complete adaptation of bonding resin and may result in non-attachment (Walshaw & McComb, 1998).

The anatomy of dentin consists of dentin tubules filled with fluid, peritubular dentin surrounding the tubules and intertubular dentin between the tubules. The adhesive resin bonds primarily to the intertubular dentin (Eick & others, 1997). Dentin close to the pulp has a concentration of dentin tubules of approximately 50,000 tubules/mm, while dentin closer to the dentino-enamel junction has a density of approximately 20,000 tubules/mm (Eick & others, 1997). The higher concentration of tubules close to the pulp can increase the amount of fluids at the gingival floor. The deeper dentin also has less intertubular dentin available to bond with the adhesive. Both factors result in weaker bonds to deeper dentin than to superficial dentin. This increased wetness at the gingival floor can result in weaker bonding between the adhesive resin and this deeper dentin (Tay & others, 1996a,b,c,d). As the amount of superficial intertubular dentin increases, the bond strength of the adhesive resin to dentin also increases (Prati & Pashley, 1992). In addition, the dentinal tubule density 1.0 mm above the cemento enamel junction (CEJ) is 49% more dense on the gingival floor compared to the axial wall (Garberoglio, 1994). This explains why the gingival wall, with more tubules and less collagen, will be intrinsically wetter than the axial wall. By nature, dentin is a very heterogeneous substrate made of 55% mineral by volume, 30% collagen and 15% water, while enamel consists of 97% inorganic matter and 1-2% water (Marshall, 1993). The intrinsic wetness of dentin could be a reason why adhesive bonding to dentin compared to enamel results in weaker bond strengths (Ogata & others, 2001).

Other reasons for the variability of bond strengths to dentin include whether the dentin surface is wet or dry (Tay & others, 1996b); the region of the tooth that one is bonding to (Bouillaguet & others, 2001; Prati & Pashley, 1992; Purk & others, 2004); whether the surface being bonded to is flat or an actual cavity preparation wall (Bouillaguet & others, 2001; Prati & Pashley, 1992); the configuration-factor (c-factor) of the tooth preparation (Yoshikawa & others, 1999); the adhesive system used (Prati & Pashley, 1992); the thickness of the composite increment placed in the cavity, where a 1.0 mm increment will bond better than a 2.0 mm increment (Eick & others, 1997) and the operator placing the material (Shono & others, 1999).

Jacobsen and Söderholm (1995) reported that increasing the amount of time a water-based primer dries on the dentin surface improved its bond strength using the shear bond test. However, this study was performed on saw cut flat surfaces and not on cavity wall preparation surfaces. The gingival cavity wall of a Class II preparation is the most frequent place for failure of a resin composite restoration (Mjör, 1998). If this failure is due to an increased wetness from the primer, perhaps a longer priming time could yield a better bond of the adhesive to the gingival cavity wall.

The purpose of this study is to determine whether longer dwelling times after application of the primer will result in increased microtensile bond strengths to the gingival wall of Class II cavity preparations. The null hypothesis is that there will be no statistically significant difference in microtensile bond strength between groups that allowed the primer to dry (dwell) at increasing times before applying the adhesive resin.

METHODS AND MATERIALS

The research design chosen to test this hypothesis was a controlled *in vitro* trial with one independent variable containing four levels. The independent variable was the time that the primer was allowed to dwell on the gingival cavity wall surface. The four treatments included allowing the primer to dry according to manufacturer's directions and allowing the primer to dry for 30, 60 and 120 additional seconds. The dependent variable was microtensile strength. The microtensile strength was measured on a Universal Instron testing machine Model 1125 (Instron Corp, Canton, MA, USA) using a 500kg load cell at a crosshead speed of 1.0 mm/minute. Before testing on the Instron, the area of cross section in mm^2 of the sample was measured.

Table 1: *Microtensile Bond Strength*

Condition	N	Mean Microtensile Bond Strength (MPa + Stan Dev)	Range (MPa)	Mean Area (mm^2)	# Debonds	% Debonded
Manuf Dir	15	25.5 + 12.2	8.2-50.5	0.54	5	25%
30 seconds	14	22.7 + 13.6	8.0-47.7	0.51	6	30%
60 seconds	15	28.1 + 14.7	5.6-58.6	0.53	5	25%
120 seconds	20	27.7 + 15.2	4.4-65.7	0.57	1	5%
Total	64					

ANOVA – no significant difference found between groups (p>0.05)
Chi Square – no significant difference found between observed and expected number of debonds between groups (p>0.05)

After approval from an Institutional Review Board to collect the extracted teeth, paired interproximal surfaces of a pair of third molars from the same dental patient were randomly assigned to the four treatment groups. The extracted teeth had no previous restorations, caries or pathology. Before preparing the teeth, they were steam autoclaved. The interproximal surface of each molar was prepared with a box-only interproximal preparation. The gingival depth of the box was 4.0-mm deep, the facial-lingual width was approximately 5.0-mm wide and the axial depth was approximately 1.5-mm deep into the dentin. The preparation was made 1.0 mm above the CEJ. A #56 carbide bur (Brasseler USA, Savannah, GA, USA) was used to prepare the teeth. The enamel was not beveled. Each proximal preparation from each pair of teeth received all four different priming times applied in random order. A hydrophilic primer (3M-ESPE, St Paul, MN, USA) was applied to the wet dentin followed by application of a hydrophobic Bis-GMA adhesive, Scotchbond Multi-Purpose (SBMP) adhesive (Lot #20001201) (3M-ESPE). The adhesive was applied over the primed enamel and dentin and was light cured for 10 seconds after each of the four primer dwelling treatment times. An XL 3000 (3M-ESPE) halogen light-curing unit was used to cure all specimens. The unit produced an intensity at least 500 mW/cm^2 within three seconds, and light output was measured before and after placement of the restorations using the Demetron light meter (Kerr Corp, Orange, CA, USA). All materials were applied according to manufacturer's directions. Dessication of the dentin was avoided. The restorative material Z-100 (Lot #20001128, shade A-3) (3M-ESPE) was placed with an initial increment of approximately 1.0-2.0 mm placed at the gingival wall. Three increments (in layers no thicker than 2.0 mm) were placed at oblique angles until the preparation was restored to its original contours and the tooth was restored. After restoration, the teeth were stored for 24 hours in tap water.

Six sets of paired third molars were obtained. Samples were cut to obtain a flat rectangular plane shape with a cross sectional area of ~ 0.55 mm^2 with no notch at the adhesive junction. Three to four gingival sections were taken from each of the proximal boxes. A total of 81 specimens were obtained. The thickness of each section was determined with a Mitutoyo digital micrometer (Mitutoyo, Aurora, IL, USA) (1 micron resolution). Samples were mounted on the Instron using Zapit, a cyanoacrylate adhesive (Dental Ventures of America Inc, Corona, CA, USA), (Lot #DS99060012-6/30/02) and accelerator (Lot #DS99060015-6/29/02) to attach the microtensile specimen to the opposing arms of a Bencor Multi-T testing device (Danville Engineering Inc, San Ramon, CA, USA). Representative samples from each group were observed under the scanning electron microscope XL30 (FEI Company, Hillsboro, OR, USA) to determine the mode of failure. A total specimen size of 20 per group was determined to be necessary with alpha = 0.05, power = 0.80 and a percent change in means of 25% being a clinically statistically significant difference in bond strengths.

RESULTS

Microtensile bond strength and debond data are presented in Table 1. All three of the experimental groups had a microtensile bond strength similar to the group applied according to manufacturer's directions. Increasing the dwelling time on the gingival wall of Class II resin composite preparations did not increase the microtensile bond strength of the adhesive to the dentin. Of the 81 samples obtained, 17 debonded during specimen preparation. This left 64 samples that could be tested for microtensile strength. An analysis of variance showed no significant difference in microtensile bond strength among the groups, $F(3, 60) = 0.44$. A chi square analysis found no difference in the number of debonds between groups, $\chi^2(3,N=81) = 4.57$. The null hypothesis was not rejected; there was no statistically significant difference in microtensile bond strengths between treatment groups that had different primer dwelling times before application of the adhesive resin.

In order to determine the mode of failure of each treatment group, representative samples were observed under the scanning electron microscope. Ninety percent of the samples broke through the adhesive layer as observed under the scanning electron

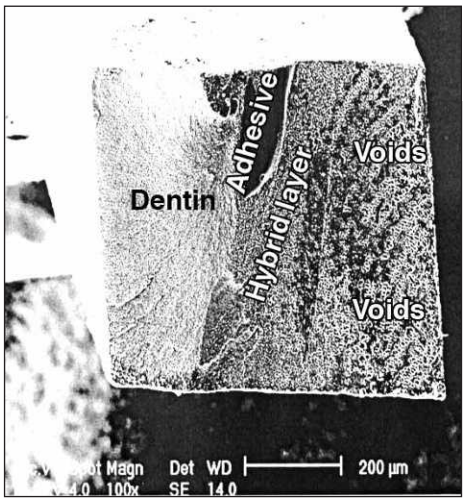

Figure 1. *Manufacturer's directions.*

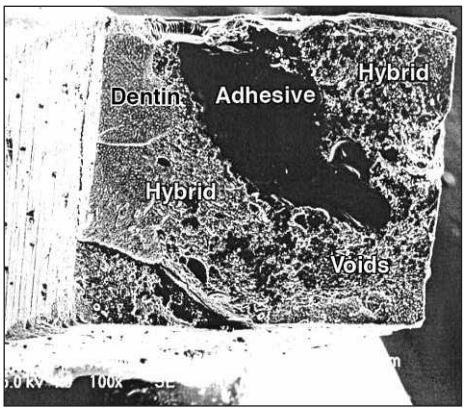

Figure 2. *120-second group.*

microscope at magnifications up to 5000x. Figure 1 is a representative sample from the manufacturer's directions group. It shows a mixed fracture pattern involving the dentin, adhesive and hybrid layer. Within the hybrid layer are voids, probably due to wetness. Figure 2 is a representative sample from the 120 second dwell time group. It shows a very similar fracture pattern to the group that followed the manufacturer's directions. Groups that had a dwell time of 30 and 60 seconds looked very similar to Figures 1 and 2.

DISCUSSION

Increasing the adhesive bond strength to the dentin gingival wall and reducing the number of debonded samples is a difficult task. If the dentin has been acid etched and the smear layer removed without hybridizing the demineralized dentin, then the dentin could be forever "wounded." This unhybridized dentin may be more susceptible to further degradation from hydrolytic breakdown and susceptible to penetration by bacterial enzymes or other toxic substances (Spencer & Swafford, 1999). A debond rate (17 out of 81 specimens) as high as 21% is also clinically unacceptable.

The water content of the dentin surface plays a very important role in the success of the adhesive bond at the gingival wall. A previous *in vitro* study (Jacobsen & Söderhold, 1995) found that increasing the priming time of a water based adhesive improved bond strength. However, the test used was a shear bond strength test and the adhesive was bonded to a flat surface. In contrast, in the study reported here, the adhesive was bonded to box cavity walls. Additionally, increasing the primer dwelling time had no effect on bond strength. It is also difficult to distinguish whether the gingival floor is too wet or too dry before placing the adhesive bonding agent. Placing the primer on a flat surface as Jacobsen did might be more conducive to water evaporating than placing a water-based primer in a cavity wall box. A moist collagen mesh may enhance primer infiltration, but the presence of water could also weaken the bond strength if not removed before the resin is cured (Jacobsen & Söderhold, 1995).

Perhaps increasing the air drying time of the primer might improve bond strengths. Similarly, blotting the primer to remove excess water from the dentinal surface before application of the adhesive may also be worthy of investigation.

CONCLUSIONS

Allowing the primer to dwell for an extended period of time beyond what the manufacturer recommends does not result in greater microtensile bond strengths of adhesive to the dentin at the gingival wall of Class II resin composite preparations.

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Correlation of Bottom-to-Top Surface Microhardness and Conversion Ratios for a Variety of Resin Composite Compositions

MR Bouschlicher • FA Rueggeberg • BM Wilson

Clinical Relevance

Bottom-to-top surface Knoop hardness ratios (B/T-KHN) of resin composite samples were highly correlated with bottom-to-top degree of conversion ratios (B/T-DC) and were independent of filler type and filler loading for the three composites tested. B/T-KHN ratios, therefore, provide an accurate, simple method of assessing the efficacy of photoinitiation strategies (curing light/exposure duration) instead of using more complex FTIR methods to determine degree of conversion.

SUMMARY

Knoop microhardness (KHN) and degree of conversion (DC) values were obtained from the bottom and top surfaces of 1-, 2- and 3-mm thick samples of three types of resin composite: an anterior microfill, an anterior hybrid and a posterior hybrid, all having differing filler size and loading but similar shade (A2) and basic monomeric content. Sample infrared spectra were obtained using attenuated total reflectance (ATR) in a Fourier transform infrared (FTIR) spectrometer. The samples were exposed using a 40-second exposure to a quartz-tungsten-halogen light source with an irradiance of ≈ 560 mW/cm². They were stored for 24 hours in complete darkness at 37°C and 100% humidity prior to obtaining cured spectra and KHN readings. KHN and DC values were obtained from the same sample specimen made at similar surface depths, but separate groups were made for obtaining top and bottom values. Cure and hardness data were analyzed with one- and two-way ANOVAs followed by the Tukey-Kramer post-hoc test. Linear regression was also applied. Statistical testing was performed at a pre-set 0.05 level of significance. KHN and DC were significantly different according to composite type and depth (p=0.0001), with an interaction effect (p=0.0022). KHN, DC and corresponding bottom/top surface (B/T) ratios decreased with depth. Regression revealed a linear relationship between DC and KHN for each composite type, with no r^2 less than 0.96. When B/T ratios were correlated, a B/T-KHN ratio of 0.80 corresponded to a narrow range of B/T-DC

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ratios (between 88 to 91%) for the three composites tested. When combining results from composite types, linear regression of B/T-DC and B/T-KHN produced a very predictable relationship (r^2=0.959), for which a B/T-KHN ratio of 0.80 corresponded to a B/T-DC ratio of 0.90. As a measure of completeness of conversion, B/T-KHN was approximately 2.5 times more sensitive than the B/T-DC ratio. In summary, while KHN cannot be used to directly compare conversion of the different composites tested, the use of B/T ratios for both hardness and conversion resulted in a linear relationship independent of filler size or loading.

INTRODUCTION

The physical properties of resin composites are related to filler type, size and loading (Chung, 1990; Kim, Ong & Okuno, 2002; Suh, Ferber & Baez, 1990) and are tailored for the intended clinical use as either anterior or posterior restorations. The intended area of usage has traditionally included a tradeoff between composite polishability and strength, based on filler size (microfill or hybrid) and loading. Composite physical properties are also dependent on the degree of conversion of the resin matrix (DC) (Asmussen, 1982a; Ferracane & Greener, 1986; Rueggeberg & Craig, 1988). DC can be assessed by indirect measures, such as scrape-back length (Cook, 1980) and microhardness testing (DeWald & Ferracane, 1987), or by direct methods, such as infrared spectroscopy (Asmussen, 1982a,b; Eliades, Vougiouklakis & Caputo, 1987; Ferracane & Greener, 1984; Rueggeberg & Craig, 1988; Ruyter & Svendsen, 1978). However, the indirect methods cannot be used to directly compare composites with differing monomer composition, filler type and size or loading (Chung 1990; Ferracane, 1985). Additionally, differing comonomers in the resin matrix may result in composition-dependent variation in the maximum DC achievable (Ferracane & Greener, 1986). Both matrix and filler characteristics affect absolute post-cure DC and hardness (Asmussen, 1982; Chung 1990). A positive correlation has been established between composite hardness and inorganic filler content (Chung, 1990; Raptis, Fan & Powers, 1979). Ferracane (1985) demonstrated good correlation between increasing hardness and increasing DC but concluded that an absolute hardness number could not be used to predict DC when different composites were compared. In fact, DC declines more rapidly than hardness with increasing sample depth (DeWald & Ferracane, 1987; Eliades & others, 1987; Rueggeberg & Craig, 1988).

Curing light irradiance, exposure duration and composite light transmission are variables significantly affecting hardness and conversion profiles with sample depth (Halvorson, Erickson & Davidson, 2003). Bottom-to-top (B/T) hardness ratios ranging from 0.80-0.90 have been used as criteria for adequate conversion at a specific sample depth (DeWald & Ferracane, 1987; Johnston, Leung & Fan, 1985). Johnston and others (1985) used composite samples with significantly different hardness and described the use of B/T ratios as a means of circumventing composite-specific hardness properties. These ratios are considered to reflect the relative extent of conversion of a deeper surface to that of the top exposed surface, but this assumption has never been validated directly.

To date, B/T-hardness ratios have not been directly correlated with B/T-DC ratios. B/T-hardness or DC ratios may be independent of filler content if they are normalized relative to the maximum obtainable value for a specific material (maximal hardness or DC achieved at the sample's top, exposed surface).

This study explores the relationship between B/T-microhardness and B/T-degree of conversion ratios of three commercially available composites selected for having similar co-monomer composition but different filler content (particle size and loading). The correlation of B/T ratios for DC with those for hardness may establish a composition-independent relationship between B/T-hardness and B/T-DC ratios and validate use of the simpler microhardness hardness test over that of a more complex method used to determine monomer conversion.

METHODS AND MATERIALS

Samples 1, 2 and 3 mm in thickness (n=5) were fabricated from a variety of commercially available, photocured resin composites having similar shade (A2), resin matrix and photoinitiator but differing in filler size proportion and weight percent loading (Table 1): anterior hybrid (AH), anterior microfill (AM) and posterior hybrid (P) composites (Matrixx AM, AH and P, Discus Dental, Culver City, CA, USA). Composite was expressed into 6-mm diameter cylindrical brass rings atop a Mylar strip on a glass slide. The uncured samples were inverted and pressed against the diamond element of a horizontal attenuated total reflectance (ATR) attachment (Golden Gate, SPECAC, Inc, Smyrna, GA, USA) in a Fourier Transform Infrared (FTIR) spectrophotometer (FTS-40, Digilab, Bio-Rad, Cambridge, MA, USA). A quartz-tungsten-halogen light curing unit (Optilux 501, Kerr/Demetron, Danbury, CT, USA) with an 8-mm diameter light guide (irradiance ≈ 560 mW/cm^2) was positioned 1 mm above the Mylar surface of each sample, and an individual, uncured spectra consisting of 16 scans was obtained at 2 cm^{-1} resolution. Irradiance was monitored periodically by using a hand held digital dental curing radiometer (Hilux Curing Light Meter, pn 950-700, Benlioglu Dental Inc, Ankara, Turkey). Samples were exposed directly on the ATR for 40 seconds. After five minutes,

the samples were removed and stored for 24 hours in complete darkness at 37°C and 100% humidity prior to determining the degree of conversion (DC) and Knoop hardness (KHN). After 24 hours, cured spectra and microhardness values were obtained from the readily identifiable smooth-surface area left by contact of the composite with the ATR crystal. To ensure adaptation of the cured samples to the diamond surface when obtaining cured spectra, a standardized torque of 85 cN-M was applied using a torque driver (Torqueleader, Model: Quickset Minor, MHH Engineering Co LTD, Surrey, UK). DC was calculated by comparing the ratio of aliphatic carbon-to-carbon double bonds (C=C) at 1636 cm^{-1} and aromatic C=C at 1608 cm^{-1} in the cured and uncured states (Rueggeberg & Craig, 1988).

Table 1: *Approximate wt-% Compositions of Composites Tested**

Components (wt-%)	Anterior Hybrid (AH)	Anterior Microfill (AM)	Posterior Hybrid (P)
Bis-GMA	<10	<10	<10
UDMA	<20	<15	<10
TEGDMA	<10	<20	<5
Ba-glass filler	<65	<40	<65
Silica filler	<25	<40	<25
Others	0.5	0.5	0.5
Lot #	02085016	01201020	02098027

**approximate wt-% data supplied by manufacturer; vol-% unavailable from manufacturer*
Products are all part of the Matrixx Composite system, distributed by Discus Dental, Culver City, CA, USA

Surface microhardness indentations for Knoop hardness (KHN) were made with a Tukon hardness tester using a load of 0.5Kg and 10x magnification (Knoop attachment, model MO, Wilson Instrument Division, American Chain and Cable Co, Inc, New York, NY, USA) within the sampling area used for DC measurements. After measuring DC values, the samples were flush mounted with heated dental compound (impression compound, Type 1, Gray, pn 00455, Kerr Manufacturing, Romulus, MI, USA) in a gimbaled holder, allowing the test surface to be easily positioned rigidly at right angles to the hardness indenter. Indentation lengths were measured digitally from stored images (NIH Image software, version 1.61, National Institutes of Health, Bethesda, MD, USA). Knoop hardness values were then calculated from the lengths of the indentation readings using standard formulas.

Since heat from the molding compound could have altered the DC and KHN values if both the top and bottom surfaces of the same sample had been tested, a separate group of 1-mm thick, top-surface-only samples was made. These samples were prepared and treated as described above, except that the bottom surface was embedded in compound and cured spectra and KHN readings were obtained from the top surface only. The two different sample groups (bottom and top) were then used to calculate B/T surface ratios of 1-, 2- and 3-mm samples for both DC and KHN.

Data were analyzed using one-way and two-way analysis of variance with the Tukey-Kramer post-hoc test for pair-wise comparison. Linear regression analysis was also performed. All statistical testing was made at a pre-set 0.05 level of significance.

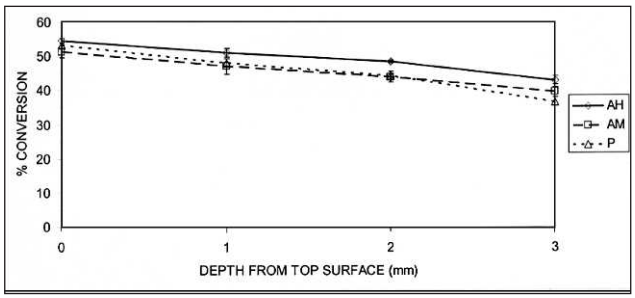

Figure 1A. *Composite conversion as a function of depth from top surface (1, 2, and 3 mm) for AH, AM and P composites. Degree of conversion (DC) decreased with depth for all composites. Vertical bars = ±1 standard deviation. N = 5 samples per group.*

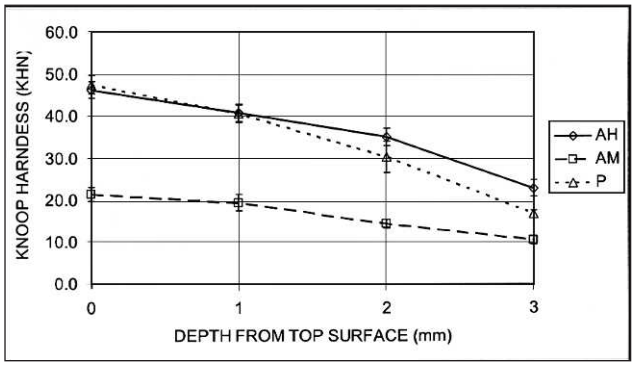

Figure 1B. *KHN as a function of depth from top surface (1, 2 and 3 mm) for AH, AM, and P composites. KHN of all composites decreased with depth. AM had the lowest KHN at all depths. Vertical bars = ±1 standard deviation. N = 5 samples per group.*

RESULTS

The two-way ANOVA showed that both DC and KHN were significantly different according to composite type and depth (p=0.0001), with a significant interaction effect (p=0.0022). Both KHN and DC decreased with increasing sample thickness (depth) (Figures 1A and 1B). One-way ANOVA showed that DC was significantly different among composite types at each depth, with the exception of the 1- and 2-mm depths of AH (Table 2). KHN also decreased significantly with an increase in depth for all materials, with the exception of the top surface and 1-mm depth of AM (Table 2).

Table 2: *One-way ANOVA and Tukey Post-hoc Statistical Results* Summary for Effects of Depth*

Depth	Effect of Composite on DC			Effect of Composite on KHN			Effect of Composite on BT/DC			Effect of Composite on BT/KHN		
Composite	AH	P	AM	P	AH	M						
Top Mean	54.6	53.3	51.3	47.6	46.4	21.4	-			-		
(SD)	(0.7)	(0.7)	(1.7)	(2.4)	(1.8)	(1.7)						
Composite	AH	P	AM	AH	P	AM	AH	AM	P	AM	AH	P
1 mm Mean	51.2	48.1	47.1	41.0	40.6	19.4	93.9	92.0	90.2	90.6	88.2	85.6
(SD)	(1.6)	(1.4)	(2.1)	(2.0)	(1.9)	(2.1)	(3.0)	(4.2)	(2.6)	(9.0)	(4.3)	(4.3)
Composite	AH	P	AM	AH	P	AM	AH	AM	P	AH	AM	P
2 mm Mean	48.4	44.4	44.1	35.0	30.4	14.2	88.9	85.9	83.2	75.7	67.0	63.7
(SD)	(0.4)	(1.3)	(1.7)	(2.0)	(3.8)	(1.1)	(0.7)	(3.3)	(2.4)	(4.2)	(5.4)	(7.9)
Composite	AH	AM	P	AH	P	AM	AH	AM	P	AH	AM	P
3 mm Mean	43.0	39.5	36.6	22.8	16.6	10.4	78.8	77.1	68.5	49.0	48.3	34.7
(SD)	(1.2)	(1.4)	(1.5)	(1.9)	(1.1)	(0.9)	(2.3)	(2.6)	(2.8)	(4.1)	(4.4)	(24.0)

**Pre-set level of significance = 0.05; horizontal bar indicates groups that were not significantly different.*

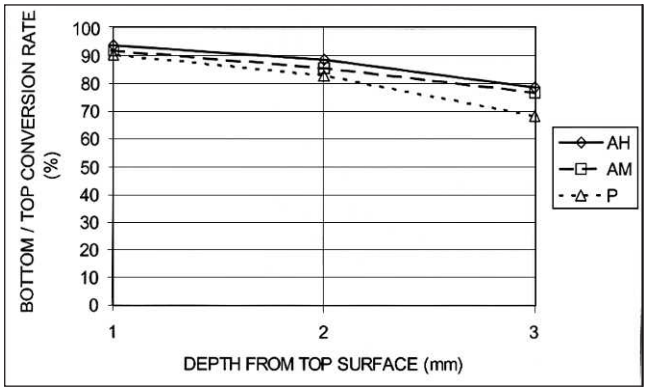

Figure 2A. *B/T degree of conversion (DC) as a function of depth from top surface (1, 2 and 3 mm) for AH, AM, and P composites. B/T-DC decreased with depth for all composites. DC of P was lowest and decreased with depth faster than AM or AH.1*

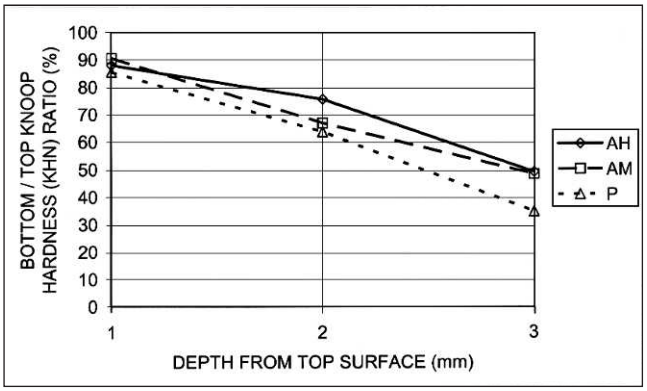

Figure 2B. *B/T-KHN as a function of depth from top surface (1, 2 and 3 mm) for AH, AM, and P composites. B/T-KHN decreased with depth for all composites. B/T-KHN of P was lowest and decreased with depth faster than AM or AH.*

Table 3: *One-way ANOVA and Tukey Post-hoc Statistical Results* Summary for Effects of Composite Type*

Composite		Effect of Depth on DC				Effect of Depth on KHN				Effect of Depth on BT/DC			Effect of Depth on BT/KHN		
Combined	**Depth**	Top	1 mm	2 mm	3 mm										
	Mean	53.1	48.8	45.7	39.5										
	(SD)	(1.8)	(2.4)	(2.4)	(3.0)										
AH	**Depth**	Top	1 mm	2 mm	3 mm	Top	1 mm	2 mm	3 mm	1 mm	2 mm	3 mm	1 mm	2 mm	3 mm
	Mean	54.6	51.2	48.0	43.0	46.4	41.0	35.0	22.8	93.9	88.9	78.8	88.2	75.7	49.0
	(SD)	(0.7)	(1.6)	(0.4)	(1.2)	(1.8)	(2.0)	(2.0)	(1.9)	(3.0)	(0.7)	(2.3)	(4.3)	(4.2)	(4.1)
AM	**Depth**	Top	1 mm	2 mm	3 mm	Top	1 mm	2 mm	3 mm	1 mm	2 mm	3 mm	1 mm	2 mm	3 mm
	Mean	51.3	47.1	44.0	39.5	21.4	19.4	14.2	10.4	92.0	85.9	77.1	90.7	67.0	48.3
	(SD)	(1.7)	(2.1)	(1.7)	(1.4)	(1.7)	(2.1)	(1.1)	(0.9)	(4.2)	(3.3)	(2.6)	(9.0)	(5.4)	(4.4)
P	**Depth**	Top	1 mm	2 mm	3 mm	Top	1 mm	2 mm	3 mm	1 mm	2 mm	3 mm	1 mm	2 mm	3 mm
	Mean	53.0	48.1	44.4	36.6	47.6	40.6	30.4	16.6	90.2	83.2	68.5	85.6	63.7	34.7
	(SD)	(0.7)	(1.4)	(1.3)	(1.5)	(2.4)	(1.9)	(3.8)	(1.1)	(2.6)	(2.4)	(2.8)	(4.3)	(7.9)	(2.0)

**Pre-set level of significance = 0.05; horizontal bar indicates groups that were not significantly different.*

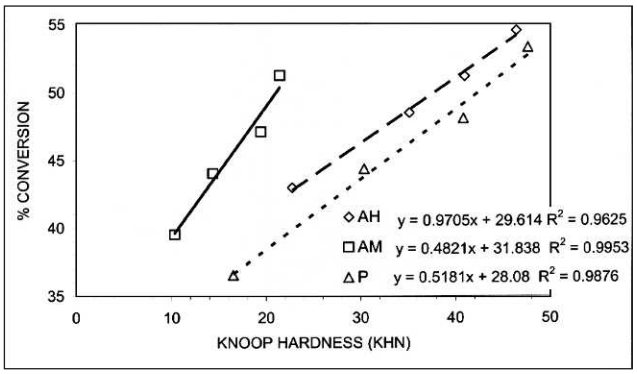

Figure 3. *Relationship between microhardness (KHN) and degree of conversion (DC) at top surface (0 mm), 1-, 2- and 3-mm depths for AH, AM and P composites.*

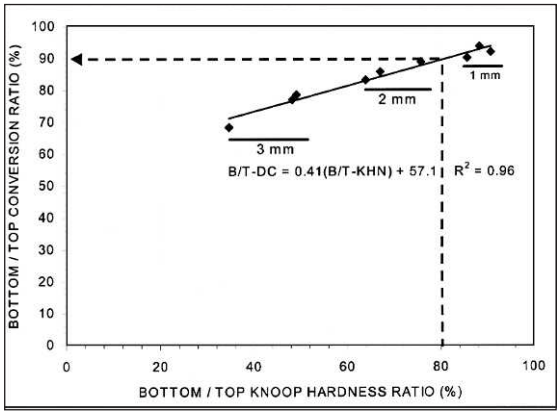

Figure 5. *Combined linear regression, relationship between B/T-KHN and B/T-DC at 1-, 2- and 3-mm depths for three composites: AM, AH and P. A B/T-KHN of 0.80 (80%) corresponds to a B/T-DC of 0.90 (90%).*

Bottom/top degree of conversion ratios (B/T-DC) decreased with increasing depth (sample thickness) (Figure 2A). B/T-DC ratios were significantly different at each depth among all materials (Table 2). Bottom/top hardness ratios (B/T-KHN) also decreased with increasing depth (sample thickness) (Figure 2B) and were significantly different at each depth among all materials (Table 2).

AH had the highest DC at 1- to 3-mm depths, while AM had the lowest hardness at all depths (Table 3). The B/T-KHN and B/T-DC ratios of AH were statistically similar to AM at all depths. The B/T-KHN and B/T-DC ratios of P were statistically different from AH at 2 mm and P<AH and AM at 3 mm. The plotted relationship between KHN and DC varies widely by composite type as shown in Figure 3. However, when B/T-KHN ratios were plotted against B/T-DC ratios (Figure 4), the resulting linear regression equations for all three composites had similar intercepts and slopes with r^2 values ranging from 0.9695 to 0.9999. The 80% B/T hardness ratio, often used as a criterion for adequate depth of

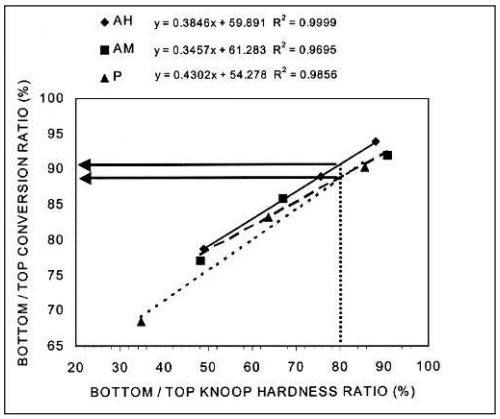

Figure 4. *Relationship between B/T-KHN and B/T-DC ratios among 1-, 2- 3-mm depths for the three composites AM, AH, and P. A B/T-KHN ratio of 0.80 (80%) corresponded to a B/TDC ratio between 0.89 and 0.91 (89 to 91%). Regression equations resulted in $r^2 \geq 0.97$.*

cure, corresponded to B/T-DC ratios between 0.89 and 0.91 for all composites tested. Linear regression of B/T-DC and B/T-KHN, when combining results from all three composite types (Figure 5), resulted in a relationship where a B/T-KHN ratio of 80% corresponded to a B/T-DC ratio of 90% (r^2=0.959).

DISCUSSION

As expected, Knoop hardness and degree of conversion decreased with increasing sample depth. Both parameters also varied with composite type. Most clinicians are aware that microfills are typically more difficult to adequately cure than their hybrid counterparts, requiring additional exposure duration. The smaller filler particles in microfills are most likely to scatter light, especially those similar in size to wavelengths of light emitted from the curing source. It was assumed that the 0.04 µm diameter fumed silica filler particles used in the AM would result in higher light attenuation than the filler particles used in hybrids. Surprisingly, the DC of AM was similar to the P hybrid at 1-mm and 2-mm depths and was statistically higher than P at 3-mm depth. While the resin matrix constituents (Bis-GMA, UDMA, TEGDMA) were similar for all three composites, the wt-% concentration of TEGDMA varied the most between P (<5 wt-%) and AM (<20 wt-%). This higher TEGDMA concentration, a low molecular weight comonomer diluent that increases the monomer's mobility/reactivity by decreasing resin viscosity, was probably responsible for the unexpectedly high DC of the AM relative to P (Lovell, Newman & Bowman, 1999).

The relationship between monomer conversion and inorganic filler loading is inversely proportional, as light transmission decreases with increased filler loading (Barron, Rueggeberg & Schuster, 1992). Since the AH and P products had similar filler loading, one would

have expected similar KHNs and DC based on their similar resin matrix and filler compositions (Table 1). However, anterior hybrid (AH) had the highest DC of the three materials at all depths. with DC at the top surface and at 1-mm depth of AH being statistically similar. While the anterior hybrid's DC was greater than the posterior hybrid (P) at 1-, 2- and 3-mm depths, the DC of the top surface of these two materials was similar. Similar DC of these two materials at the top surface, where equivalent radiant energy was applied, suggests that the lower DC of the posterior hybrid at depth is the result of lower levels of available radiant energy, as the intensity of transmitted light versus composite thickness obeys the Lambert equation (Cook, 1980). The higher overall degree of conversion and the similarity in conversion between the top surface and 1-mm depth of AH is probably due to the higher translucency (light transmission) of this material, which was designed to serve primarily as an enamel replacement in anterior teeth.

As expected, AM had the lowest KHN at all depths. There is a significant correlation between volume fraction of filler and Knoop hardness (Chung & Greener, 1990), and since AM filler loading (<80 Wt-%) was lower than that of AH and P hybrids (<90 Wt-%), it would be expected that AM would have the lowest KHN at all depths. Also, what constitutes "filler particles" in a microfilled material is often nebulous. One could measure only inorganic material, or also take the combined inorganic content along with the amount of prepolymerized ground fillers, as well. Doing so would result in vastly different values for filler content in the same material.

Accordingly, hardness numbers cannot be used to directly compare composites with differing filler type, size or loading. This concept is illustrated by the plots of the relationship between KHN and DC for the three composites tested (Figure 3). The slope of the regression equation for AM was decidedly different from the others and occupies an entirely different location distinct from those of AH and P. AH and P had similar slopes due to similar filler size and loading values. Again, the effects of sample depth and, thus, light attenuation, had a more dramatic effect on P, as evidenced by the more extended range of KHN vs DC values plotted. The markedly lower range of hardness values plotted for AM does not overlap the lower end of the range of AH values, even though most of the DC values overlap. This finding is in agreement with Chung and Greener (1990), who found no correlation between degree of conversion and mechanical properties of resin composite.

The use of B/T ratios normalizes the plotted relationship between hardness and DC relative to the maximal parameter value obtainable at the sample's top surface, irrespective of composite composition. The relationship between B/T-KHN and B/T-DC (Figure 4) supports the use of B/T hardness as an accurate indirect measure of B/T-DC for individual composites. Linear regression, showing the relationship between B/T-KHN and B/T-DC at 1-, 2- and 3-mm depths for all three composites (Figure 5,) demonstrates that a 0.80 B/T-KHN ratio corresponds to 0.90 B/T-DC or 90% of maximum conversion possible at the composite's top surface. Figure 5 also illustrates that adequate physical properties only coincide with higher degrees of conversion as B/T-DC increases from 0.70 to 0.95, while B/T-KHN increases from 0.35 to 0.90. Within the range tested, B/T-KHN is approximately 2.5 times (1.0/0.41—slope of the regression analysis in Figure 5) more sensitive than B/T-DC as an index of percent maximum cure obtainable at the composite's top surface.

Since the plotted relationship between ratios in Figure 5 was independent of composite filler type, one can make direct comparisons of the adequacy of composite cure with similar resin matrix but different filler content by using B/T-KHN ratios. This method allows for rapid but accurate assessment of photoinitiation strategies with the more easily performed B/T-KHN ratios. The ease of sample preparation and the use of less costly test equipment make this technique more widely applicable than complex, costly FTIR methods. B/T hardness ratios can be used to compare the relative extent of cure of different composites with different curing strategies, as long as each composite is normalized for maximal hardness (conversion) at the sample's top surface. Maximal hardness would be obtained by ensuring the top surface conversion process could go no further, requiring exposure duration exceeding that which is recommended by the manufacturer. Thus, caution should be exercised with use of ratioed values. If the top surface demonstrates higher hardness than that of a deeper surface, but the to surface has not yet reached a maximal value, both the numerator and denominator are increasing concurrently and high ratios may occur over a range of radiant exposures (mJ/cm^2) before the stop surface reaches an asymptotic value. Also, only microhardness testing was used in this study to evaluate the correlation between hardness and conversion values. The use of other hardness methods may not provide similar results and should be tested as well.

CONCLUSIONS

The simpler methodology used to obtain the bottom-to-top surface microhardness hardness ratios of a variety of composite materials proved to be an accurate, indirect reflection of bottom-to-top degree of conversion ratios. Both bottom-to-top microhardness and degree of conversion ratios were independent of composite composition, with respect to the three composites tested.

Thus, bottom-to-top surface microhardness ratios can be used as a simple test for adequate polymerization at a given sample depth independent of composite composition, with respect to the composites tested.

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Resin-Dentin Interfacial Ultrastructure and Microtensile Dentin Bond Strength After Five-year Water Storage

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Clinical Relevance

As a result of long-term exposure to an aqueous environment, the hybrid layer degrades and the dentin-adhesive resin bond weakens.

SUMMARY

Objective: **To evaluate a total-etch three-step adhesive system's resin-dentin interfacial ultrastructure and microtensile dentin bond strength (μTBS) after multi-year storage in water.**

Methods: **Resin composite crowns were formed on 600 grit SiC flattened extracted human molars using a total-etch three-step adhesive system (Optibond FL, Kerr) and a hybrid resin composite (Prodigy, Kerr). μTBS specimens were fabricated and placed in water with 0.5% chloramine T at 37˚C until respective static load to failure testing at one-month, six-months and five-year storage. Failure modes were determined by scanning electron microscopy. The interfacial ultrastructure of the resin-dentin interface was analyzed by transmission electron microscopy (TEM) at 48-hours and 44-months storage. μTBS was modeled with Weibull distribution for survival analysis and failure curve distributions were analyzed by the Wald chi-square statistic for significant differences at α=0.05.**

Results: **The characteristic tensile strength (σ_o) at one-month, six-months and five-year storage was 52.63, 14.77 and 23.57 Mpa, with a Weibull modulus of 3.04, 1.56 and 1.28, respectively. Failure distributions for all groups were significantly different ($p<0.0001$) with one-month > five-year > six-months. TEM interfacial morphology demonstrated hydrolytic degradation of hybrid layer components at 44-months storage.**

Significance: **The decrease in tensile strength and changes in ultrastructure may be caused by water sorption and resultant hydrolytic degradation of the adhesive joint.**

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INTRODUCTION

A durable bond between dentin and resin composite is essential if the subsequent development of dentin sensitivity, marginal staining, marginal defects, recurrent caries and restoration loss is to be avoided. Time-dependent *in vitro* changes in bond strength and failure modes between dentin-adhesive resin/resin composite bonded joints have been investigated by several laboratories (Armstrong & others, 2003; Burrow, Tagami & Hosoda, 1993; De Munck & others, 2003; Kiyomura, 1987; Nakajima & others, 2003; Okuda & others, 2002; Shono & others, 1999). In general, bond strength decreases over time and the failure location shifts from predominantly cohesive substrate failures to more failures in the adhesive joint. For obvious reasons, fewer *in vivo* studies have been accomplished but have generally reported similar observations related to marginal degradation (Hashimoto & others, 2000; Sano & others, 1999; Takahashi & others, 2002).

Degradation may occur in the biological tissues, polymer components of the resin composite-dentin joint or both. Several authors have supported theories of dentin collagen degradation (Burrow, Satoh & Tagami, 1996; Hashimoto & others, 2003b; Kiyomura, 1987; Maciel & others, 1996; Van Strijp, Klont & ten Cate, 1992), while others have proposed theories of adhesive degradation (Burrow, Inokoshi & Tagami, 1999; Burrow & others, 1996; Gwinnett & Yu, 1995; Hashimoto & others, 2003a; Sano & others, 1999; Tay & others, 2003) in the resin composite-dentin bonded joint.

This study evaluated and correlated the interfacial ultrastructural appearance and μTBS of resin-dentin bonded samples after multi-year storage.

METHODS AND MATERIALS

Specimens remaining from a previous study involving paired μTBS and fracture mechanics testing (Armstrong, Keller & Boyer, 2001), in which only one μTBS specimen per tooth was utilized, were prepared for TEM and μTBS analysis after continuous water storage for 44-months and five-years, respectively. The remaining dentin-Optibond FL-Prodigy resin composite sticks were stored in separate containers by tooth in unchanged water containing 0.5% chloramine T (pH 7.0) at 37°C until utilized for this investigation. Twenty-one storage containers had lost their seal in storage, leaving 19 sectioned teeth available for μTBS testing and electron microscopy in this study. The storage media maintained a neutral pH throughout all storage periods.

Cylindrical tensile specimens were formed as described in the previous study, with the exception that a shorter gauge length (1 mm) was used to preserve a remaining dentin thickness >1.5 mm from the pulp and a pin vice was used to hold the stick for trimming and testing in lieu of a Plexiglas rod. Four specimens from three teeth (#s 2, 4 and 38) broke while trimming and were excluded from the analysis, giving a sample size of 30. Interestingly, the premature failures all came from the teeth used in the six-months storage testing with μTBS values of 5.82, 5.95 and 6.57 MPa, all well below the median μTBS for that test period. μTBS testing was completed as described previously (Armstrong, Boyer & Keller, 1998; Armstrong, Keller & Boyer, 2001) by gluing the composite side of the test specimen to the lower Plexiglas fixture with cyanoacrylate (Zapit, Dental Ventures of America, Inc, Corona, CA, USA) and applying a tensile force at 1-mm minute^{-1} until failure.

Once broken, each sample was mounted on aluminum stubs with cyanoacrylate adhesive and gold-sputter coated. Failure modes were observed with a scanning electron microscope (AMRAY 1820, AMRAY, Inc, Bedford, MA, USA) and recorded as either joint, mixed, composite or dentin failure modes. Joint and mixed mode failures were also examined to identify the predominate region of the adhesive joint comprising the fractured surface.

Fresh teeth were bonded for short-term storage controls as previously described, with the exception that a less-filled resin composite (Clearfil Protect Liner, Kuraray) was utilized to minimize damage to the diamond knife during TEM preparation. Two sticks were randomly selected from each of these control teeth and four additional sticks were randomly obtained after 44-months storage from those that remained from the previous study. The sticks were then fixed in 3% glutaraldehyde/formaldehyde in 0.1M sodium cacodylate buffer (Electron Microscopy Sciences, Fort Washington, PA, USA) at pH 7.4 at 4°C before partially demineralizing four hours in Surgipath decalcifier I (Surgipath Medical Industries, Inc, Richmond, IL, USA). Specimens were then rinsed in 0.1M sodium cacodylate buffer for one minute with three changes, dehydrated in ascending grades of ethanol (50%, 75%, 90%, 95%, 100%) for two hours each with two changes, followed by immersion in 100% propylene oxide (PO), 2:1 PO:Epon, 1:1 PO:Epon, 1:2 PO:Epon and 100% Epon for three hours before embedding in fresh 100% Epon (812 Epoxy resin, Tousimis, Rockville, MD, USA). The specimens were oriented for embedding so that ultra-thin sections through the resin-dentin interface could be obtained from the central portion of each tooth. The epoxy blocks were polymerized in an oven at 60°C for a minimum of 48 hours.

Ultra-thin sections (approximately 90 nm) were made by a diamond knife (Diatome, Bienne, Switzerland) using an ultra-microtome (Ultracut E, Reichert-Jung, Wein, Austria). The sections were examined unstained or stained with saturated uranyl acetate for 20 minutes, lead citrate for three minutes and phosphotungstic acid/uranyl acetate for two minutes. Due to the

slight acidity of conventional TEM stains, it is imperative to also observe unstained sections to examine the hybrid layer in its unaltered state. The resultant interfaces were examined with TEM (Zeiss EM10, Oberkochen, Germany) for the following micro-morphologicial characteristics: depth and uniformity of hybrid layer, acid-resistance of the hybrid layer, transition between the hybrid layer and unaffected dentin, collagen fibril morphology, presence of hydroxyapatite in the hybrid layer, tubule content, tubule wall hybridization and lateral tubule wall hybridization.

Fisher's exact test was used to determine whether there was a possible association between storage time and failure modes. Failure data was modeled with Weibull distribution for survival analysis. Wald chi-square test was conducted for comparison of failure curves over time for each experimental condition. All tests have a 0.05 level of statistical significance.

RESULTS

The one-month, six-months and five-year characteristic tensile strengths (σ_O) were 52.63, 14.77 and 23.57 Mpa, respectively, with a Weibull Modulus of 3.04, 1.56 and 1.28, respectively. The stress levels at which one would expect 5% ($\sigma_{0.05}$) and 95% ($\sigma_{0.95}$) of the specimens to fail for each group are also included in Table 2, with 95% confidence intervals. Failure distributions for TBS groups (Figure 1) were significantly different with a *p*-value <0.0001.

Failure modes in the one-month μTBS group demonstrated a propensity to fracture in either the dentin (6/20) or resin composite (5/20), with six failures within the adhesive joint and three involving both the joint and a substrate (mixed). Debond pathways that involved the joint (9/20) included all potential cohesive or interfacial zones. All failures in the six-month μTBS group included the bottom of the hybrid layer (BHL), 18/20 entirely within the joint and 2/20 mixed failures including the joint and resin composite. Two-thirds of the failures in the five-year μTBS group fractured in the joint, with the lower stress-to-failure specimens predominantly involving BHL, with stronger joint failures occurring in the top of the hybrid layer (THL). The remaining failures were dentin substrate (9/30), with one resin composite substrate failure.

TEM investigation of the control group revealed normal ultramorphological features consistent with previous investigations (Van Meerbeek & others, 1996) (Figures 2A and 2B). The 44-month water storage group presented areas of relatively normal appearance similar to the control group mixed with unstained areas or "ghost hybrid layer" (Figures 2C and 2D). In some sections, the entire thickness of the hybrid layer was devoid of staining. Some collagen fibrils in the 44-month storage group appeared to be smaller in diameter and the typical cross-banding was harder to distinguish.

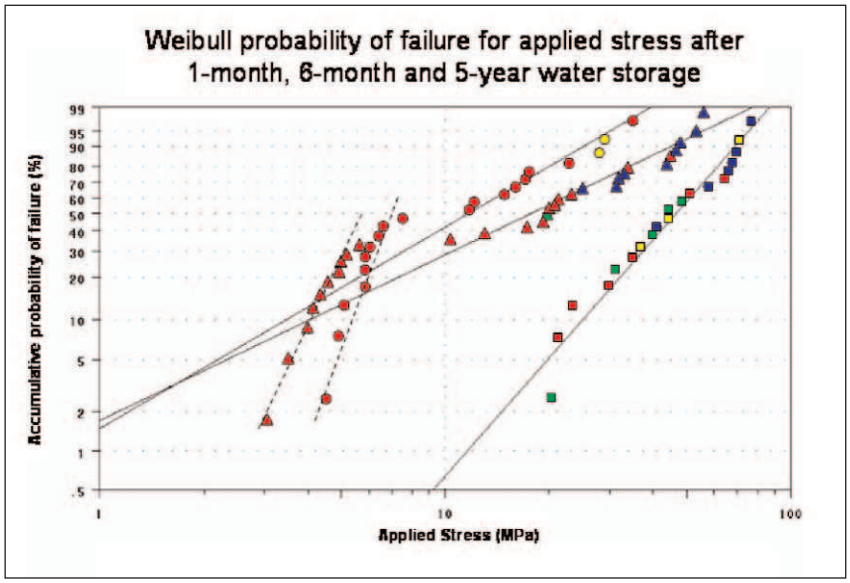

Figure 1. *Weibull plot of probability of failure (%) against stress to failure (MPa). □ = 1-month storage; O= 6-months storage; Δ = 5-year storage. Symbol colors represent failure mode: red = joint, yellow = mixed, green = resin composite, blue = dentin. Dashed lines represent possibility of two independent modes of failure in six-month and five-year storage groups, that is, bimodal Weibull distribution.*

Table 2: *Failure Distributions (MPa) and Group Comparisons*

Storage Time	n	Median	Min	Max	σ_O	m	$\sigma_{0.05}$ (95% CI)	$\sigma_{0.95}$ (95% CI)	*
1-month	20	46.66	20.41	76.26	52.63	3.04	19.84 (13.08-30.10)	75.46 (64.06-88.89)	A
6-month	20	11.68	4.51	35.33	14.77	1.56	2.21 (1.01-4.82)	29.81 (21.85-40.66)	C
5-year	30	17.72	3.06	55.39	23.57	1.28	2.33 (1.03-5.26)	55.43 (40.29-76.25)	B

*σ_o: normalizing parameter, characteristic strength of material, or scaling factor, m: Weibull modulus, indicator of reliability of material, or shape factor, $\sigma_{0.05}$: stress expected to induce 5% failure rate, $\sigma_{0.95}$: stress expected to induce 95% failure rate; indicator of attainable bond strength, Reference: McCabe and Walls (1986). *Groups with the same letter are not significantly different at p<0.05.*

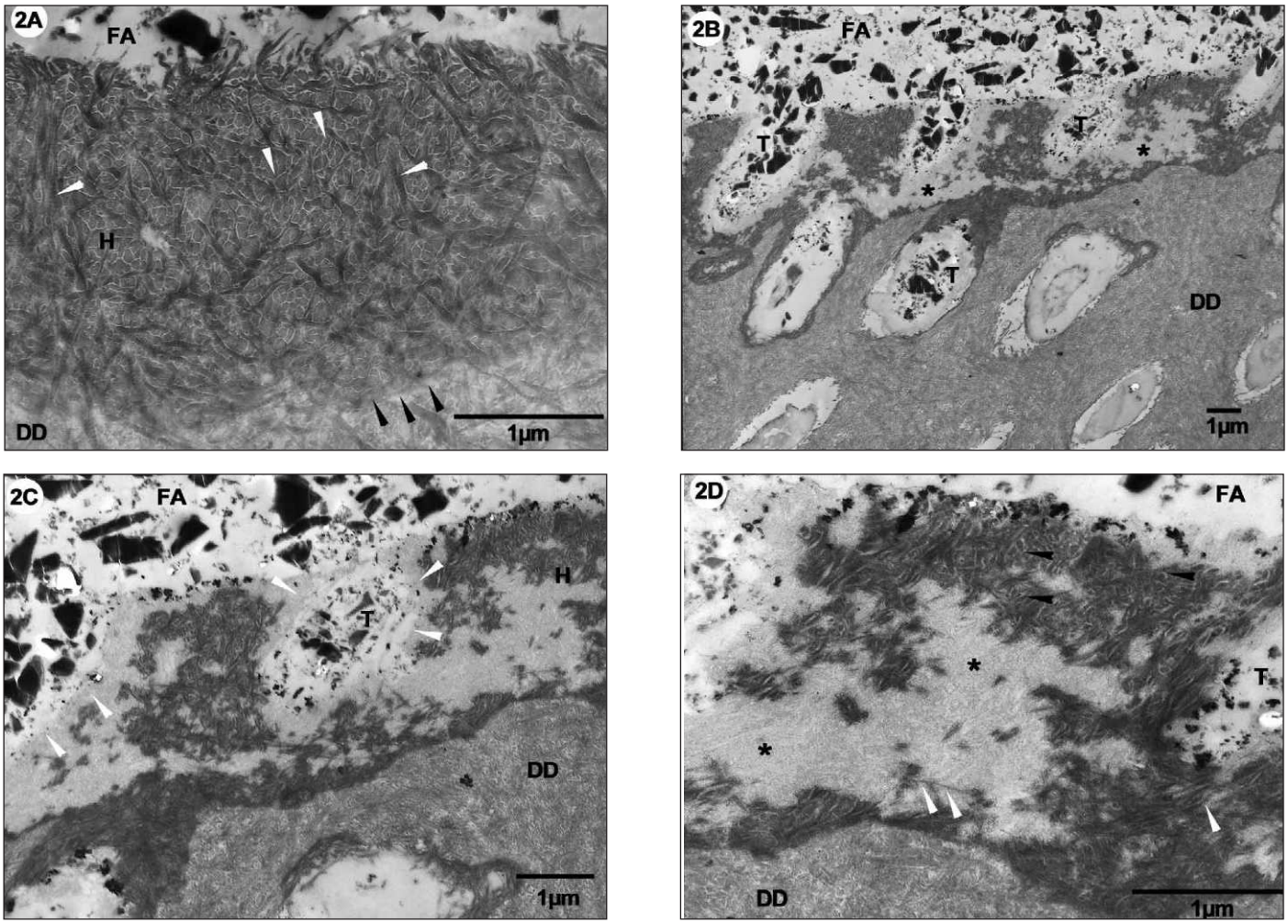

Figure 2. *Transmission electron micrographs. Specimens were demineralized and stained with lead, uranyl acetate and phosphotungstic acid.*
Figure 2A
High magnification of a control specimen soaked in water for 48 hours. The filled adhesive (FA) wetted the top of the hybrid layer (H). Within the hybrid layer, randomly oriented collagen fibrils could be seen (white arrows). These cut in cross-section revealed uniform 10-20-nm wide interfibrillar spaces. The depth of resin infiltration was defined by the abrupt change in electron density (black arrows) between acid-etched resin-infiltrated dentin fibrils in the hybrid layer and an electron-lucent laboratory-demineralized dentin matrix (DD). Original magnification 12500x.
Figure 2B
Low magnification of a bonded specimen incubated for 44 months. Little change is apparent in the filled adhesive (FA), but there was a loss of heavy metal staining of collagen fibrils in nearly half of the hybrid layer (*). The regions in the hybrid layer that contain filler particles are actually the edges of resin-filled dental tubules (T) passing through the hybrid layer. There were some resin tags beneath the hybrid that also contained adhesive filler particles. Original magnification 4000x.
Figure 2C
Intermediate magnification of a 44-month bonded specimen showing the loss of staining of the dentin matrix on either side of resin filled dentinal tubules. Loss of staining was also seen near the top and bottom of the hybrid layer. Original magnification 8000x.
Figure 2D
High magnification of a 44-month bonded specimen showing a loss of staining of the collagen fibrils with in hybrid layer. Original magnification 12500x.

DISCUSSION

Strength, marginal seal and biodurability of the union between adhesive resins and dentin is of critical importance for the clinical success and longevity of adhesively bonded direct and indirect restorations. This study continued the investigation of resin-dentin bonded samples (Armstrong & others, 2001) to describe the interfacial ultrastructural appearance after 44-months water storage and the µTBS after five-year water storage. A significant decrease in dentin bond strength over long-term water storage occurred and morphological changes in the hybrid layer were readily apparent. Damage or degradation of the adhesive bond occurred due to hydrolytic attack of organic tissues or elution of and/or alterations in mechanical properties of resinous components.

The five-year storage group was significantly weaker than the one month, but oddly, the overall stress-to-failure distribution was stronger than six-months storage. This may have occurred due to slight modifications in specimen preparation timing. One- and six-month specimens were stored as trimmed cylindrical tensile specimens, whereas the leftover specimens were stored as untrimmed "sticks" and trimmed immediately before testing at five-year storage. However, this four and one-half years of additional storage time combined with the unprotected short diffusional distance at the adhesive joint (1.5-mm square stick trimmed into an 0.8-mm diameter cylindrical specimen), combined with the observed drop in bond strength between one and six months storage, makes this very unlikely. As demonstrated by the lower Weibull modulus, there was more variability in μTBS after six-months and five-year storage compared to one-month storage. After longer periods of storage in water, all lower strength values were associated with adhesive joint failures and the failure distributions suggested a bimodal failure pattern (Figure 1).

A bimodal Weibull distribution reveals the possibility of two independent modes of failure (Robin & others, 2002). When evaluating the stress levels that one would expect 5% of the specimens to fail ($\sigma_{0.05}$) for six-months (1.03 – 4.82 MPa) and 5-year (1.03 – 5.26) storage, one sees that they are equivalent. Table 3 shows that these lower strength values are predominately associated with failure modes involving the BHL. This could represent a region of the adhesive joint where adhesive resins inadequately infiltrate, phase separate or poorly polymerize. It could also be argued that any inadequately infiltrated and enveloped collagen fibrils in this region of the hybrid layer are susceptible to hydrolytic degradation (Hashimoto & others, 2003b). Why the upper-strength distribution region after five-years of storage was stronger and had more dentin substrate failures is unknown. Cohesive failures in dentin may have occurred at five-year testing due to endogenous and/or exogenous enzymatic challenges under the storage conditions imposed to the dentin matrix (Tay & others, 2003). One must remember that only global or average bond strength values are obtained in such testing and the local stresses are unknown (van Noort & others, 1989).

The overall observation that dentin bond strength declines over time concurs with previous studies (Burrow & others, 1996; De Munck & others, 2003; Hogan & Burrow, 2001; Kiyomura, 1987). A recent study by Hashimoto and others (2003b) found a 67% drop in μTBS between the 24-hour control and the one-year aged specimens. Their failure specimens revealed no real changes in the adhesive resin layer or tags; however, the hybrid layer had regions devoid of adhesive resin and the collagen fibrils, especially near the BHL, were of lower density, disorganized and lacked the typical 67-nm cross-banding of normal collagen. They concluded that the reduction in bond strength came from degradation of the hybrid layer components and not from the overlying adhesive resin or resin composite. The authors observed a 72% decline in μTBS between the one- and six-month storage periods and their failure analysis and TEM observations confirm their findings. Synthetic (adhesive resin) and biological (collagen) polymers make up the hybrid layer, both of which are susceptible to hydrolytic degradation. Analytical techniques beyond those employed in this study will be required to determine the exact degradative mechanisms occurring in this adhesive joint, with additional adhesive strategies required to prevent its occurrence.

The one-month μTBS group demonstrated a propensity to debond in dentin or resin composite substrates. However, after six-months and five-years, debonds occurred primarily in the adhesive joint involving either THL or BHL, with the lower strength values occurring in the BHL. Armstrong and others (2001) and Li, Burrow and Tyas (2001) reported that interfacial fracture mechanics, microtensile bond strength and tracer studies revealed a shifting debond pathway from the interphase region between the top of the hybrid layer and the adhesive resin to the BHL after several months of water storage. Kiyomura (1987) reported that the debond pathway shifted from the AR-THL interface to the base of the hybrid layer (BHL). Utilizing a different total-etch three-step system, Tay and others (1995) observed that the adhesive resin-hybrid layer interface (AR-THL) is the weak link and stated that microleakage may progress from this point to involve the entire hybrid layer. Imai and others (1998), looking at the resistance to contraction gap formation by three reduced bottle dental adhesive systems, also demonstrated failure at the AR-THL interface. Burrow and others (1996) reported that failure was initially cohesive in dentin but shifted to the base or top of the hybrid layer after one year. Using a total-etch two-step system, Tay and others (2003) studied the nanoleakage patterns over one-year storage in artificial saliva and initially found silver deposits within the hybrid layer that, over time, accumulated in the top half of the hybrid layer and the hybrid layer-adhesive resin interface, with water trees forming into the adhesive resin. The course of these fluid channels throughout the hybrid layer and into the adhesive resin cover the range of failure patterns in Table 3. This study observed that as the specimens age, more joint failures are observed, with the weaker values predominately from failures involving the bottom of the hybrid layer, intermediate values at the top of the hybrid layer and the strongest bond strengths from mixed or substrate failures. It is very important to recognize that the

Table 3: *Failure Modes and Predominately Involved Region of Joint Failures After One-month, Six-month and Five-year Storage*

One-month			**Six-month**			**Five-year**		
μTBS (MPa)	Failure Mode	Predominate region of joint-involved failures	μTBS (MPa)	Failure Mode	Predominate region of joint-involved failures	μTBS (MPa)	Failure Mode	Predominate region of joint-involved failures
20.41	RC		4.51	Joint	Bottom HL	3.06	Joint	Bottom HL
21.28	Joint	Top HL	4.92	Joint	Bottom HL	3.56	Joint	Bottom HL
23.53	Joint	Top HL	5.14	Joint	Bottom HL	4.00	Joint	Bottom HL
29.88	Joint	Top HL	5.80	Joint	Bottom HL	4.10	Joint	Top HL
31.04	RC		5.82	Joint	Bottom HL	4.39	Joint	Bottom HL
34.72	Joint	Top HL	5.82	Joint	Bottom HL	4.61	Joint	Bottom HL
36.77	Mixed	Top HL	5.95	Joint	Bottom HL	4.92	Joint	Top HL
40.06	RC		6.43	Joint	Bottom HL	4.98	Joint	Top HL
41.55	Dentin		6.57	Joint	Top HL	5.20	Joint	Bottom HL
44.26	Mixed	Top HL	7.50	Joint	Top HL	5.64	Joint	Middle HL
44.63	RC		11.64	Joint	Bottom HL	10.39	Joint	Top HL
47.98	RC		12.08	Joint	Bottom HL	12.92	Joint	Top HL
51.04	Joint	Top HL	14.85	Joint	Bottom HL	17.15	Joint	Top HL
57.02	Dentin		15.98	Joint	Top HL	19.32	Joint	Top HL
63.71	Joint	Top HL	17.00	Joint	Bottom HL	19.80	RC	
65.90	Dentin		17.59	Joint	Top HL	19.99	Joint	Top HL
67.14	Dentin		22.96	Joint	Adhesive	20.96	Joint	Top HL
69.80	Dentin		28.07	Mixed	Adhesive	21.24	Joint	Top HL
70.26	Mixed	Adhesive	29.08	Mixed	Adhesive	23.21	Joint	Top HL
76.26	Dentin		35.33	Joint	Adhesive	24.50	Dentin	
						31.41	Dentin	
						31.87	Dentin	
						32.80	Dentin	
						33.44	Joint	Top HL
						43.77	Dentin	
						45.26	Joint	Top HL
						46.36	Dentin	
						47.29	Dentin	
						53.16	Dentin	
						55.39	Dentin	

HL = hybrid layer; RC = resin composite

Totals

	Dentin	RC	Joint	Mixed	Substrates	Joint-involved	Total
One-month	6	5	6	3	11	9	20
Six-month	0	0	18	2	0	20	20
Five-year	9	1	20	0	10	20	30

failure initiation site is the most critical fractographic piece of information. However, this is very difficult to discern due to the viscoelastic properties of the dentin-adhesive resin-resin composite joint. Results are also assuredly adhesive system-dependent.

Transmission electron microscopy has added greatly to the authors understanding of the resin-dentin interdiffusion zone (Tay, Gwinnett & Wei, 1998; Tay & others, 1994; Van Meerbeek & others, 1993; 1996). Hydrolytic degradation within the hybrid layer occurs due to water penetration through nanoleakage channels (Tay & others, 2003), resulting in lower bonding strengths (Okuda & others, 2002) and ultrastructural changes (Hashimoto & others, 2003b). The ultrastructural appearance of specimens stored for 44-months were distinctly different from short-term controls (Figure 2). Varying from section to section, regions of the hybrid layer failed to acquire stain and the fibrils

Table 1: *Materials, Components, Batch Numbers and Manufacturers*

Materials	Components	Batch #	Manufacturer
Optibond FL	*Etchant:* 37% phosphoric acid liquid	612595 011038*	Kerr
	Primer: HEMA, GPDM, mono phthlalate, CQ, ethanol, water	704263 101308*	
	Adhesive: BisGMA, HEMA, GDMA, CQ, fillers (48%)	704264 101911*	
Prodigy shade B1 Resin Composite	*Filler:* barium aluminoborosilicate glass, fumed silicon dioxide, zinc oxide, titanium oxide, pigments	704438 704440	Kerr
	Matrix: EBADM, TEGDMA, BisGMA, CQ		

Abbreviations: *BisGMA = bisphenol A diglycidylmethacrylate, CQ = camphorquinone, EBADM = ethoxylated bis-phenol-A-dimethacrylate GDMA = gylcerol dimethacrylate, GPDM = glycerol phosphate dimethacrylate, HEMA = 2-hydroxyethyl methacrylate, TEGDMA = tri[ethylene glycol] dimethacrylate.*

**= batches used for TEM preparation of control groups, Clearfil Protect Liner (Kuraray) used for short-term TEM sample preparation.*

appeared to be smaller and less organized. When using a total-etch three-step system, the hybrid layer should be composed of monomers primarily from the primer. In most adhesive systems, HEMA is a common monomer, including the primer and adhesive used in this study, and is susceptible to hydrolysis as a linear hydrophilic polymer. However, this primer (Optibond FL) also includes a dimethacrylate resin (GPDM) (Table 1) and therefore should crosslink to form a polymer network that should reduce swelling and increase polymer stability. Unprotected collagen fibrils in the hybrid layer may also be susceptible to hydrolysis (Hashimoto & others, 2003a,b). Several theories may be proposed to describe specific degradative mechanisms of the dentin-resin adhesive joint; however, additional chemical analytical methods will be necessary to accurately describe these mechanisms and continued studies are warranted.

CONCLUSIONS

The characteristic tensile strength at five-year storage was significantly lower than one-month storage, and significant alterations in the ultrastructural appearance of the hybrid layer were readily apparent over long-term water storage with this total-etch three-step adhesive. The interphase regions between the hybrid layer and the dentin and resin composite substrates (BHL, THL) are the weak zones of the adhesive joint, with the BHL weaker than the THL.

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The Effect of Flowable Resin Composite on Microleakage and Internal Voids in Class II Composite Restorations

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Clinical Relevance

Flowable resin composites provided a reduction in the formation of voids and marginal microleakage. Fewer voids were observed when a flowable composite was used in conjuction with a condensable composite. Also, a correlation between the extent of microleakage and the presence of voids in a restoration was found.

SUMMARY

This *in vitro* study evaluated the influence of two flowable resin composites on marginal microleakage and internal voids in Class II composite restorations with the margins below the cementoenamel junction (CEJ). Class II cavities randomly divided into four groups: Group I–Filtek with Filtek Flow lining; Group II–Filtek; Group III–Tetric Ceram with Tetric Flow lining; Group IV–Tetric Ceram. After thermocycling tests (5-60 x 1500) and dye soaking, the teeth were sectioned in a mesiodistal direction along their longitudinal axis. Gingival-marginal microleakage and internal voids in three separate portions of the restoration (interface, cervical and occlusal voids) were observed with a microscope. Statistical analyses indicated that the use of flowable resin composites (Groups I and III) provided a reduction in marginal microleakage and a reduction in some parts of the internal voids or total voids ($p<0.05$). The condensable material (Filtek) in combination with the flowable liner showed fewer voids (interface, occlusal, total) than the hybrid resin (Tetric) ($p<0.05$). There was a correlation between the number of internal voids or total voids and the marginal microleakage ($p<0.05$). It was concluded that a composite lining in a Class II resin composite with margins below the cementoenamel junction may reduce marginal microleakage and voids in the interface and the total number of voids in the restoration.

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INTRODUCTION

Tooth-colored posterior restorations are now the first choice of many patients. One of the most popular is the composite restoration. However, microleakage is one of the important problems at the margins of the proximal box of Class II cavities restored with resin composites (Crim & Chapman, 1994; Schuckar & Geurtsen, 1997). It is likely that this cervical microleakage contributes to the high incidence of secondary caries in this region and accounts for many clinically failed restorations (Mjör, 1998).

To improve the marginal sealing of a composite restoration, the use of low-modulus lining materials such as glass ionomers (Aboushala, Kugel & Hurley, 1996), resinous liners (Kakaboura, Eliades &

Palaghias, 1996) or new-generation dentin bonding agents (Chan & Swift, 1994) have been proposed. However, these lining materials did not completely eliminate microleakage.

Recently, flowable composites were introduced (Estafan & Estafan, 2000; Payne, 1999; Unterbrink & Liebenberg, 1999). They have a filler size similar to hybrid composites but a lower filler content (weight: 60%-70%; volume: 46%-65%) than their hybrid analogs (weight: 70%-80%; volume: 60%-75%) (Chuang & others, 2001a). Some flowable composites have compressive strengths comparable to those of more conventional composites.

An advantage of flowable composites is their lower Young's Modulus in comparison with other hybrids (Bayne & others, 1998; Estafan & Estafan, 2000). This could contribute to the dissipation of contraction stresses during polymerization. However, the utilization of occlusal irradiation could explain the poorer results obtained with this technique (Lutz, Krejci & Barbakow, 1991). Occlusal irradiation tends to pull out composite from the margins as it shrinks toward the light source. However, this theory has recently been contested by afinite-element analysis (Versluis, Tantbirojn & Douglas, 1998), which showed that the direction of the polymerization contraction is more influenced by the cavity than by the position of the light source. However, this type of polymerization still has problems, such as the distance of the increment to the light, leading to subpolymerization of the cervical increment mainly at its inner part (Pires & others, 1993; Rueggeberg & others, 1994), resulting in poor adhesion. This situation is even worse in deep cavities, such as those with margins apical to the CEJ. Even with those potential problems, utilization of a flowable composite as the first increment in a Class II restoration can be effective with the help of its lower Young's modulus. Several authors have reported encouraging results in reducing microleakage with the use of flowable composite restorative materials (Ferdianakis, 1998; Mazer & Russell, 1998).

When used in lining materials beneath resin composite restorations, flowable composites may improve marginal adaptation (Alomari, Reinhardt & Boyer, 2001; Payne, 1999; Belli & others, 2001). Compared with injectable glass ionomers, Payne (1999) reported a reduction in microleakage of flowable composite in Class II restorations, especially at the cavosurface margin of the proximal box. Tung, Estafan and Scherer (2000) evaluated microleakage in Class II cavities restored with a composite placed with or without a liner and found that a flowable composite should be used as a liner.

Beznos (2001) evaluated microleakage at the cervical margins of slot cavities of Class II resin composite restorations restored with different techniques, such as directed-shrinkage, sited resin modified glass ionomer cement or a flowable resin composite. Results showed that all the techniques worked well for enamel, with almost no leakage. However, on cementum, all the techniques demonstrated moderate to severe leakage.

Bedran de Castro and others (2002) evaluated the microleakage of Class II box cavities restored with various restorative systems and found that flowable resin composite decreased the values of microleakage only for the ormocer-based resin.

Tung and others (2000) found that a flowable composite should be used as a liner when a condensable composite material was used to prevent microleakage in Class II cavities. However, Jain and Belcher (2000) compared the microleakage of Class II hybrid resin-based composite (RBC) restorations with and without low viscosity (flowable) RBC in the proximal box and found that none of the four flowable RBCs tested influenced microleakage significantly in Class II RBC restorations.

Loguercio and others (2002) reported the extent of marginal leakage with Class II resin composite restorations associated with different materials, such as flowable or glass-ionomer cements. It was concluded that using glass-ionomer material led to the best sealing of the gingival margins based upon the lowest degree of microleakage observed.

There are other potential clinical problems that can arise when using traditional hybrid resin-based composites in Class II cavity preparations. Voids and gingival marginal areas can result from the inability to adequately adapt the materials to margins before curing (Nash, Lowe & Leinfelder, 2001). Condensable composites with high viscosity may increase the possibility of internal voids (Opdam & others, 1996).

Recently, some *in vitro* studies have reported a reduction in microleakage but an increase in the presence of internal voids in conservative Class I and II flowable resin composite fillings (Ferdianakis, 1998; Payne, 1999). However, Chuang and others (2001a) and Chuang, Liu and Jin (2001b) determined the influence of a flowable composite lining on microleakage and internal voids in a Class II composite restoration. It was decided that a flowable composite lining in a Class II resin filling could effectively reduce voids in the restoration but may not necessarily improve marginal sealing.

Few studies include a correlation between microleakage and the presence of voids. Chuang and others (2001a, 2001b) found a reduction in the presence of internal restoration voids when using flowable resin composites (Tetric Flow) as a lining material for Class II resin composites. The incidence of internal voids was significantly reduced at both the restoration's interface

and within its mass. In addition, no significant difference in the likelihood of marginal microleakage between pairs with or without flowable resin composite lining was found and again no significant correlation between the number of voids detected in any part of the restoration and marginal microleakage was found, but the margins were located above the CEJ in those studies.

This study investigated the influence of two commercial flowable composite linings on marginal microleakage and the presence of internal voids in Class II composite restorations (hybrid or condensable) when the margins were below the cemento-enamel junction. A correlation between microleakage and the presence of voids was also investigated.

METHODS AND MATERIALS

Forty freshly extracted human molars were scaled, cleaned and examined with a microscope to eliminate any decayed or cracked teeth. To simulate clinical posterior teeth alignment, the molars were mounted in stone jigs with one premolar and one molar each on mesial and distal sides. Two Class II box-only cavities (a bucco-lingual width of 4 mm, an occluso-gingival height of 5 mm and a pulpal depth of 2 mm) were prepared on the mesial and distal surfaces. The proximal box margins were placed 1.0 mm below the cemento-enamel junction (CEJ). All the margins were kept as close as possible to a 90° cavosurface angle. These preparations were accomplished with diamond burs (Shofu 411, Shofu Inc, Kyoto, Japan) and carbide burs (#330, Beavers Dental, Morrisburg, Ontario, Canada). All specimens were rinsed with tap water. The cavity preparations were tightly sealed with a metal matrix and wooden wedges (Hawe Neos Dental, Bioggio, Switzerland). Two flowable resin composites, Filtek Flow and Tetric Flow, and their compatible composites, Filtek and Tetric Ceram, were chosen as lining and restorative materials. Table 1 summarizes the experimental materials used in this study.

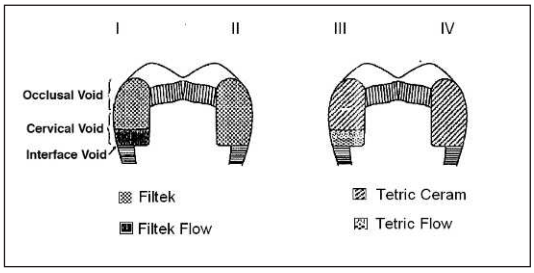

Figure 1. *Design of the study.*

The 80 cavities on 40 teeth were divided into four groups according to the materials used for their restoration:

Group I–Filtek composite with Filtek Flow lining

Group II–Filtek composite without lining

Group III–Tetric Ceram composite with Tetric-Flow lining

Group IV–Tetric Ceram composite without lining

Figure 1 illustrates the experimental design. Each tooth had two cavity preparations; one was restored with a flowable composite liner, whereas, the other was restored without a liner, resulting in each tooth being restored with a Group I/Group II or a Group III/Group IV restoration. Each group (n: 20) was equally divided and restored. One well-trained specialist undertook the filling and testing procedures.

For Group I and II restorations, the cavities were etched with 35% phosphoric acid for 15 seconds, washed for 10 seconds and dried. Single Bond bonding system was applied to the cavity and air dried for two-to-five seconds, then light cured for 10 seconds. According to the manufacturer's instructions, a second layer of Single Bond was applied in the same manner. For Group I, the cavities were filled with Filtek Flow to about 1-mm occlusal gingival thickness and light cured from the occlusal aspect for 20 seconds. Filtek was placed in the cavities and light cured in four increments of 40 seconds each. For Group II, the cavities were filled with Filtek and light cured in five increments of 40 seconds each.

For Group III and IV restorations, the entire cavity (enamel and dentin) was also etched with 35% phosphoric acid for 15 seconds, washed and air dried. The compatible bonding system Syntac Single Component was applied and allowed to stand for 20 seconds before air drying and light curing for 20 seconds. According to the manufacturer's instructions, a second layer of Syntac Single component was then applied in the same manner. For Group III, the cavity preparations were filled with Tetric Flow to about 1-mm occlusal-gingival thickness and light cured from the occlusal aspect for 20 seconds. Tetric Ceram was placed in cavities and light cured in four consecutive increments of 40 seconds each. For

Table 1: *Materials Used*

Material	Manufacturer	
Syntac Single Component	Vivadent Ets, Schaan, Liechtenstein	Single-component DBA
Tetric Flow	Vivadent Ets, Schaan, Liechtenstein	Flowable composite lining
Tetric Ceram	Vivadent Ets, Schaan, Liechtenstein	Hybrid composite
Filtek P 60	3M Dental Products, St Paul, MN, USA	Condensable composite
Filtek Flow	3M Dental Products, St Paul, MN, USA	Flowable composite lining
Single Bond	3M Dental Products, St Paul, MN, USA	Single-component DBA

Group IV restorations, the cavities were filled with Tetric Ceram and light cured in five increments of 40 seconds each.

After removing the metal matrix and wooden wedges, all the restorations were light cured from both the buccal and lingual sides for 20 seconds.

The restorations were finished with a scalpel and fine diamond burs, then polished with paper disks (Sof-Lex, 3M ESPE, St Paul, MN, USA). After finishing and polishing, the experimental teeth were removed from the stone mounting jigs and placed in isotonic saline at 37°C in a water bath for 24 hours (Crim & García-Godoy, 1987). The teeth were then placed in a thermocycling machine for 1,500 cycles ranging from 5°C to 60°C (each dwelling time for 20 seconds), after which the root apex was sealed with utility wax. The teeth were coated with nail varnish except for the restoration and one millimeter beyond the margins, then soaked in a 2% basic-fuchsin dye for 24 hours. They were then removed from the bath, embedded in epoxy resin and sectioned in a mesio-distal direction along their longitudinal axis using a double-face diamond disc. The sectioned teeth were observed with a 50x stereomicroscope (Prior, England) attached to a computer and scored for the degree of dye penetration and internal porosity. The accessor was blind to which group the teeth belonged. The scoring scales for marginal adaptation are shown:

0 = no leakage

1 = leakage extending to the half of the cervical wall: light

2 = leakage to the full extension of the cervical wall, but not including the axial wall: moderate

3 = leakage to the full extension of the cervical wall and including the axial wall: severe

Two main examiners scored the restorations independently and any discrepancies between the two examiners were reevaluated by both and a consensus reached. For each restoration, one score by convention, the worst was used for the analysis. For purposes of dye penetration analyses, only the gingival floor of the tooth/restoration interface was considered.

The scoring system for internal voids was modified from Ferdianakis' study (Ferdianakis, 1998). For the description of the location and semi-quantitative analysis of the internal porosity in the restoration, the assessment of the voids present was performed in three different parts of the restoration (Figure 1):

The gingival margin-resin interface (Interface void)

The cervical half (Cervical void) or

The occlusal half (Occlusal void) of the entire restoration.

Scores for recording voids in the three parts were shown as:

Score 0 = no void

Score 1 = some voids exist

The sum of the scores for each of the three parts (Interface void, Cervical void and Occlusal void) was expressed as Total voids. Scores for Total voids ranged from 0 to 3, depending on the sum of the three individual void scores.

Friedman-nonparametric test statistically examined the data for microleakage and internal voids (three separated parts and the Total voids) in four groups. The computation of significant differences was assigned at a $p<0.05$ level. Correlation between marginal microleakage and internal voids in any of the three restoration regions was examined by the Pearson test at a $p<0.05$ level.

RESULTS

None of the groups showed complete prevention of dye penetration. Table 2 demonstrates the frequency of the

Table 2: *Scores of Marginal Microleakage*

Group		Score			
		0	1	2	3
I-Filtek Flow	(n=20)	20	4	8	8
II-Filtek	(n=20)	8	12	8	12
IIITetricFlow	(n=20)	24	10	6	0
IV-Tetric	(n=20)	6	10	14	10

No significant difference (p>0.05) between Group II-IV.

Table 3: *Scores of Three Parts of Internal Voids and Total Voids*

Group		Interface Void		Cervical Void		Occlusal Void		Total Void			
		0	1	0	1	0	1	0	1	2	3
I-Filtek Flow	(n=20)	40	0	16	24	40	0	16	24	0	0
II-Filtek	(n=20)	32	8	28	12	28	12	16	16	8	0
III-Tetric Flow	(n=20)	40	0	20	20	16	24	12	12	16	0
IV-Tetric	(n=20)	16	24	12	28	12	28	0	20	0	20

Interface void: No significant difference (p>0.05) between Groups I and III.

Cervical void: No significant difference (p>0.05) between Groups and III, I and IV, II and III.

Occlusal void: No significant difference (p>0.05) between Groups III and IV.

Total void: No significant difference (p>0.05) between Groups II and III.

scores for marginal microleakage. Group III (Tetric Flow) showed the best marginal sealing compared with the other groups ($p<0.05$). Although there was no statistically significant difference between Group II (Filtek) and Group IV (Tetric) ($p>0.05$), flowable composite linings showed better results than resin composites.

The value for interface voids (Table 3) in Groups I (Filtek Flow) and III (Tetric Flow) was zero, suggesting effective elimination of voids within the composite in the restoration-tooth interface region for groups using flowable composite linings ($p<0.05$). There was no statistically significant difference between Groups I and III ($p>0.05$), though statistical differences were found among the other groups (0.05). Group II (Filtek) was superior to Group IV (Tetric) in reducing the void. Thus, the use of flowable composite linings reduces the likelihood of interface voids. In the cervical area, results showed no significant differences between Groups I and III, I and IV, II and III comparisons ($p<0.05$). Group II (Filtek) showed the best results, as the restorations with flowable composite linings had similar results. Occlusal voids showed significant differences among the groups ($p<0.05$), except Groups III and IV comparison. While Filtek Flow had the best results, it exhibited less void formation compared to Tetric or Tetric Flow ($p<0.05$). Total voids were lower for restorations utilizing flowable composite linings. Filtek Flow showed the best results. Although Filtek and Tetric Flow showed similar results ($p>0.05$), they were superior to the Tetric group in reducing total voids ($p<0.05$).

The Pearson test examined the correlation between marginal microleakage and internal voids in the three restoration regions. There was a correlation between marginal microleakage and the presence of total voids or voids in any region of the restoration ($p<0.05$).

DISCUSSION

The C-factor is the relationship between the number of bonded surfaces and the number of unbonded surfaces in a restoration (Kanca & Suh, 1999). The lower the C-factor, the lower the internal stresses. When the internal stresses are low, there is less competition between the contraction forces arising from monomer conversion and the efforts of the adhesive agent to keep the composite bonded to the surface (Tung & others, 2000). The porosities reduce the relation between the adhering/non-adhering surfaces (Factor C), because the oxygen present in the porosities impairs the polymerization of the resin that contacts it so that this subpolymerized mass could flow around and compensate for the contraction with less stress. However, these mechanisms were not sufficient to avoid the formation of gaps mainly at the cementum margins (Beznos, 2001; Hilton, Schwartz & Ferracane, 1997). When the margins were located below the CEJ, none of the techniques demonstrated a good sealing capacity. Based on *in vitro* studies with similar results, some authors do not recommend direct composites when the margins are located below the CEJ, as inlays have shown a better sealing capacity (Puy & others, 1993; Dietschi & others, 1995). However, Van Dijken, Horstedt and Waern (1998) reported that even with margins below the CEJ, Class II composites showed excellent adaptation.

This study also obtained good results with flowable composites. In this study, there were statistical differences among the groups but there was a clear tendency for better results with the flowable technique.

Also in this study, even though margins were below the cementoenamel junction, a significant difference in microleakage and voids was found between the cavity pairs (with/without flowable composite linings). Thus, the use of a flowable composite lining in Class II restorations effectively reduced marginal microleakage and interface or total voids. This confirms other authors who proved that flowable composites under resin composites can effectively reduce microleakage (Tung & others, 2000; Leevailoj & others, 2001; Yazıcı, Baseren & Dayangaç, 2003). The use of a low modulus flowable composite may have increased the flexibility of the bonded assembly, allowing it to act as a stress breaker (Van Meerbeek & others, 1993).

A correlation between marginal microleakage and the presence of total voids or any region of the voids was found. Large voids will reduce the mechanical strength of the restoration. Extensive internal porosity distributed throughout the restoration will decrease the contact surface area between the restoration and the tooth. Porosity of the external surface of the restoration results in surface roughness and may lead to staining porosity at the inner interface or may elicit unpredictable results within the body of the restoration.

Interface voids were completely eliminated by using flowable composite linings. This result suggests that the low viscosity of the composite linings improved marginal adaptation at the gingival wall interface and concurrently reduced the formation of total voids. It is also possible that no void developed, because of the low filler content and/or incremental placement technique that was used. Although there was no significant difference between Filtek or Tetric in marginal microleakage, Tetric showed the worst result in reducing total or any part of the voids. Voids at gingival marginal areas can result from the inability to adequately adapt the materials to margins before curing. Filtek's good performance can be due to its type as being condensable.

All tested techniques used incremental insertion. When the increments were thicker than 2 mm, an increase in polymerization stress occurs, including a poorer rate of conversion, mainly cervical, where the distance from the source of light is greater than the ideal (Rueggeberg & others, 1994).

To obtain good interproximal contact, a metallic matrix is used, therefore, the composite can only be light cured from the occlusal surface. As a result, polymerization shrinkage is directed away from the gingival margin of the preparations (Hilton, Schwartz & Ferracane, 1997). To simulate clinical conditions, a metallic matrix and incremental insertions were applied in this study.

Careful application of the restorations to the gingival margin will result in success. It should be ascertained that no air is trapped when expressing the material. Any violent injection or stirring motion will lead to undue void formation.

This study revealed that the use of flowable resin composites as a lining material should result in a reduction in the likelihood of the formation of voids and a reduction in marginal microleakage. As flowable composites are more resin rich, they have low viscosity and flow and adapt at least as well as resin composites.

CONCLUSIONS

Within the limitations of this study, it was concluded:

1. For marginal microleakage, Group III (Tetric Flow) showed the best marginal integrity ($p<0.05$).
2. Flowable resin composites (Filtek Flow or Tetric Flow) were superior to resin composites (Tetric Ceram or Filtek) in preventing microleakage, though the margins were below the cementoenamel junction ($p<0.05$).
3. The value for interface voids in flowable resin composites suggested effective and similar elimination of voids.
4. The condensable material (Filtek) produced fewer voids (interface, occlusal, total) than the hybrid resin in combination with the flowable liner ($p<0.05$).
5. There was a correlation between marginal microleakage and the presence of total voids or voids in any region of the restorations ($p<0.05$).

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Departments

Abstracts

***In vivo* caries formation in enamel following argon laser irradiation and combined fluoride and argon laser treatment: A clinical pilot study. Hicks J, Winn II D, Flaitz C & Powell L (2004) *Quintessence International* 35(1) 15-20.**

(University of Texas Dental Branch at Houston, Texas, USA; Academy Laser Dentistry, Colorado Springs, Colorado, USA; University of Utah, Department of Pathology, Salt Lake City, Utah, USA)

This study compared the effect of argon laser irradiation and topical fluoride treatment combined with argon laser irradiation on caries development in sound enamel surfaces beneath plaque-retentive orthodontic bands.

Fourteen teeth with caries-free buccal surfaces from three females and two males were divided into three groups: (1) argon laser irradiation (12 J/cm^2, 250 mW, 10 seconds, ARGO-MOD, Premier Laser Systems); (2) topical fluoride (0.5% fluoride ion, 1.1% neutral sodium fluoride, four minutes, Thera-Flur-N, Colgate Oral Pharmaceuticals) followed by argon laser irradiation; (3) no treatment controls. Stainless steel orthodontic bands with buccal plaque retention slots were placed on teeth scheduled for extraction. The teeth were extracted within five weeks after *in vivo* caries formation. Orthodontic bands and cement were removed and the teeth were stored in 10% buffered formalin. Serial longitudinal sections were performed with 12 sections obtained per tooth. Sections were imbibed with water and images of lesions captured with polarized light microscopy. A computer-interfaced program was used to determine the mean lesion depths.

The mean lesion depths were significantly decreased for both the argon laser irradiation and combined fluoride and argon laser treatment groups when compared with the no treatment control group. Argon laser irradiation and fluoride combined with argon laser irradiation resulted in mean lesion depth reductions of 44% and 62%, respectively. There was a 32% decrease in mean lesion depth for the combined fluoride and argon laser treatment compared to argon laser treatment alone. This study and previous studies have shown that argon laser exposure increases enamel microhardness, while fluoride uptake by "laser-hardened" enamel enhances caries resistance. The synergistic effect of argon laser irradiation and fluoride treatment may favorably influence the critical pH where tooth mineral undergoes dissolution and resistance to subsequent caries development. Low fluence argon laser irradiation may be of clinical importance in the prevention of caries formation and progression.

Eight-year study on conventional glass ionomer and amalgam restorations in primary teeth. Qvist V, Laurberg L, Poulsen A & Teglers PT. *Odontologica Scandinavica* (2004) 62 37-45.

(School of Dentistry, University of Copenhagen, Copenhagen, Denmark)

The objective of this study was to continue a previous three-year investigation of conventional glass ionomer (GIC) and amalgam restorations placed in primary teeth in Denmark. The original study assessed the potential costs associated with a ban on amalgam, with GIC used as the alternative to amalgam as the main restorative material in primary teeth. This article outlines the differences in the longevity and cariostatic effects of these different materials up to eight years after placement.

Children from two different Danish towns in need of treatment for active caries had, after parental notification, either conventional glass ionomer (Ketac-Fil) or amalgam (Dispersalloy) restorations placed in primary teeth by one of 14 providers. While the providers were informed of the requirements of the study, they were not calibrated in terms of diagnosing caries or assessing restorations. The materials were used in alternate weeks for seven months in 1991-1992. Restoration totals included 515 GIC and 543 amalgam in 666 children ages 2.8 to 13.5 years. Eighty-six percent of the restorations were for primary caries and 14% were replacement restorations. Fifteen percent were Class I restorations, 79% Class II and 5% Class III/IV. Eighty-four percent of restorations were lined with Dycal, but at no time were rubber dam, acid etch, conditioners or bonding agents used. The restorations were followed until the teeth were exfoliated or extracted or until they were repaired or replaced. The 592 unrestored surfaces adjacent to the restorations were also monitored and recorded. The restorations were considered *failed* if they were replaced, repaired or the tooth was extracted for endodontic reasons. The adjacent surfaces were considered *failed* if they required operative caries treatment.

Due to natural tooth loss, the median observation period for all restored teeth was 1.8 years for GIC and 2.3 years for amalgam. Ten percent of the restorations were present after five years; 2% were present at the end of the study. In total, 31% of the restorations failed, nearly one-half of which were due to approximal caries

lesions. The GIC failed at twice the rate of amalgam, mostly due to bulk fracture in Class II GIC restorations. GIC restorations survived on average for 42 months, while the median longevity of amalgams was at least 7.8 years. Thirty-seven percent of surfaces in contact with amalgam developed caries, while only 19% of the surfaces adjacent to GIC.

The results for GIC restorations (3.5 years average longevity, fracture as the main cause of failure) are similar to those in other studies involving primary teeth. Although the amalgams lasted at least twice as long, they only need to last five to six years in primary teeth. Due to this fact, the authors estimated an increase in cost of at least 20% if GIC restorations were to be placed exclusively. However, the authors also noted the importance of the reduced need for operative treatment in teeth adjacent to GIC, leading to a cost reduction of about 8% when compared to the cost of amalgams and the subsequent restorations placed in adjacent teeth. Finally, the authors noted that the individual clinician plays an important role in placing the restorations and that final conclusions should be based upon results taken from several different clinicians and studies.

Durability of three simplified adhesive systems in Class V non-carious cervical lesions. van Dijken, JMV (2004) *American Journal of Dentistry* 17(1) 27-32.

(Department of Odontology, Dental School, Umeå University, Umeå, Sweden)

This *in vivo* study compares the durability of three different simplified adhesive systems with resin-based composite materials in Class V non-carious abrasion/erosion lesions over a 24-month period.

The three adhesive systems were: 1) *Clearfill Liner Bond 2/Clearfil APX–a 2-step self-etching primer system* with primer liquid A & B mixed, applied twice for 30 seconds each then light cured for 20 seconds. 2) *One Coat Bond/Synergy–a one-bottle total-etch adhesive system* using a 15 second etch with 15% phosphoric acid followed by a 20 second application of the primer and 30 second light cure. 3) *Promt L-Pop/Pertac Hybrid–an "all –in-one" self-priming system* applied to the dentin for 15-20 seconds followed by a 10 second light cure.

One hundred and forty-four Class V restorations were placed in 90 patients. One experienced operator familiar with adhesive dentistry placed all restorations. No more than 50% of the cavosurface margin involved enamel and at least 90% of the surface area of the restoration was in contact with dentin. Pre-operatively, the lesions were categorized in terms of depth (superficial, moderate, deep) and degree of dentin sclerosis (none, <50%, > or =50%). Sixty-five of the lesions were randomly chosen to be lightly roughened with a diamond bur at low speed and with water-cooling prior to conditioning. Plaque covered lesions were cleaned preoperatively with a polishing paste before conditioning. Application of the adhesive systems was done according to the manufacturer's instructions. In order to secure unrestricted contamination-free access to the field, gingival retraction instruments or matrix bands retracted the adjacent gingiva when necessary. Adjacent enamel was not beveled or additionally etched.

Only three of the 144 restorations were not evaluated at all recalls of 6, 12, 18 and 24 months. The patients reported no post-operative sensitivity. A total of 21 restorations (14.6%) were lost during the follow-up: 4 Clearfil Liner Bond 2 (8.7%), 6 One Coat Bond (13.0%) and 11 Promt-L-Pop (21.2%). Prompt-L-Pop showed significantly higher loss rates compared to Clearfil Liner Bond 2. The cumulative loss rates of the materials in sclerotic lesions (15.7%) versus non-sclerotic lesions (14.0%) were not significantly different. Restorations placed in roughened sclerotic lesions showed a loss rate of 14.5%, while for the non-roughened lesions the frequency was 14.8%. At two years the slightly modified USPHS criteria scores for marginal adaptation, color match, marginal discoloration and secondary caries revealed no differences between the groups of acceptable restorations.

It was concluded that the three systems provided acceptable initial clinical retention, but that the increasing loss rates observed during the two-year follow-up indicated that the simplification of adhesive systems seems to restrict the efficacy of the bonding.

Candidates must possess a doctoral degree, and/or a DDS/DMD degree, and documented history of significant academic accomplishments in teaching and scholarly activity. Demonstrated evidence of funded research, publications and presentations at significant meetings is strongly preferred.

NYU offers an excellent benefits package with opportunities to participate in its Faculty Practice in the heart of Manhattan. Salary and academic rank are commensurate with credentials and experience. Send curriculum vitae, statement of academic objectives, names and addresses of four references to: Dr Van P Thompson, NYU College of Dentistry, 345 East 24th Street, Room 804S, New York, NY 10010-4086. The search will continue until an appropriate candidate has been selected.

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University of California, San Francisco School of Dentistry

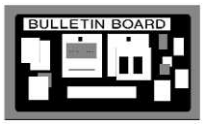
Chair, Division of Clinical General Dentistry

The UCSF School of Dentistry invites applications and nominations for a full-time, faculty position as chair of the Division of Clinical General Dentistry within the Department of Preventive and Restorative Dental Sciences. The division is the largest in the School of Dentistry and is responsible for the teaching of comprehensive care in the predoctoral DDS program. The chair is responsible for the smooth running of the division, scheduling of faculty, assigning of faculty duties, and liaising with specialty discipline. The successful applicant will have proven leadership skills, an outstanding reputation as a clinical educator, and as a clinician, and will likely have clinical research experience. He/she will lead the division in embracing the philosophy of minimally invasive dentistry.

The individual selected must have a DDS/DMD or equivalent, be currently licensed in the USA, preferably in California and have a proven record of outstanding clinical education over several years. A national reputation as a clinical educator is essential. Experience in minimally invasive dentistry is required. Academic rank and salary will be commensurate with the applicant's achievements, qualifications and experience.

Interested applicants should submit a letter of intent, a current curriculum vitae and three letters of reference to Dr David Graham, Department of Preventive and Restorative Dental Sciences, Box 0758, UCSF School of Dentistry, 707 Parnassus Avenue, San Francisco, CA 94143. Review of applicants will begin immediately and continue until the position is filled by a qualified candidate.

Announcements



34th Annual Meeting of the Academy of Operative Dentistry

23-25 February 2005
Fairmont Hotel, Chicago, IL

Come one, come all to The Academy of Operative Dentistry's 34th Annual Meeting in beautiful downtown Chicago. See and hear the amazing, educational essay presentations. Interact with the practical, knowledgeable table clinicians. Immerse yourself in the popular and entertaining social programs...O.K. that may be a bit over the top, but we do have an outstanding, clinically relevant program this year.

SCIENTIFIC SESSION: Thursday's lineup begins with Dr Michael Miller, the editor of *Reality*, speaking on "Dual-Cure Materials–Have We Been Misled?" Dr Dennis Fastbinder follows with "Chairside CAD/CAM Ceramic Restorations: the CEREC 3D System." This year's Buonocore Memorial Lecturer is Dr Franklin Tay, who will offer a practical, in-depth appraisal entitled "Reducing Steps in Dentin Bonding–What Have We Really Gained?" The highlight of Thursday's luncheon will be the presentation of the Hollenback Memorial Prize to Dr Stephen C Bayne. Thursday afternoon features Dr Jeff Rouse presenting "The Science and Art of Ultraconservative, Full-Porcelain Laminate Veneers" followed by Dr Kevin B Frazier with a topic of great interest to both dentists and patients, "Maintenance Considerations for Esthetic Restorations."

Dr Newton Fahl leads off on Friday morning with a two-hour presentation on "Mastering Anterior Composite Restorations." The essay sessions conclude with Dr Tom McDonald speaking on "Functional Considerations in Esthetic Dentistry." Friday lunch will be held in the Mid-America Club and will feature the presentation of the Academy's Award of Excellence to Dr James B Summitt. The 2005 annual session will conclude with Friday afternoon's table clinics. These are always concise, focused presentations that provide a wealth of "pearls" for us to take back to our offices.

Activities Program is particularly exciting this year. Thursday offers a tour of a fascinating exhibit at the Field Museum titled "Jacqueline Kennedy: The White House Years" followed by a wonderful three-course lunch at Pili Pili, a popular upscale bistro offering traditional French dishes with a southern European twist.

Friday morning offers a "Continental Buffet Breakfast at the Fairmont" featuring Jeanne Elledge and "Sal Capone" in "Chicago's Famous (or Infamous) Characters." You will find this a very entertaining and informative program on the history and prominent individuals of Chicago's Roaring Twenties.

RECEPTION: Finally, our Gala Reception on Thursday evening will once again provide a wonderful, once-a-year, platform for socializing with all our friends and colleagues from across the country and around the world.

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

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(University of West Virginia School of Dentistry)

September 28-October 1, 2005

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For details and information, contact:

Dr. Ronald K. Harris, Meeting Coordinator
256 Sand Brook Drive
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Phone: 317/867-0414
Fax: 317/867-3011
E-mail: pipedoc@sbcglobal.net

Announcement for Direct Gold Operators

Please note that Ivoclar-Vivadent is no longer manufacturing or selling Direct Gold materials of any kind. EZ Gold and Gold Foil can still be obtained from the following supplier:

LB Dental Center
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Telephone: 909/799-3773
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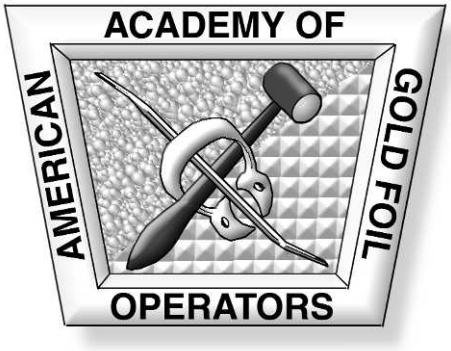

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