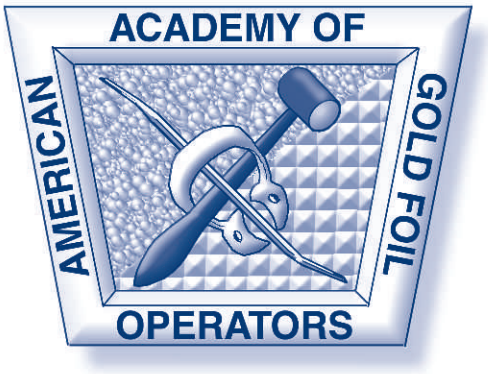
OPERATIVE DENTISTRY



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Reasons for Replacement of Restorations

INTRODUCTION

Surveys of the time spent on various procedures in general dental practice show that restorative dentistry, including caries diagnosis and preventive measures, comprises the major workload (Eklund, 1999; Gilbert, 2004). Non-implant restorative procedures alone represent almost 60% of treatment time in the US and, together with diagnostic and preventive measures, they comprise about 70% (Gilbert, 2004). Practice-based studies have shown that replacement of restorations involves from 50% to 80% of all restorative work done in general dental practice (Mjör, 1981; Tveit & Espelid, 1986; Klausner, Green & Charbeneau, 1987; Qvist, Qvist & Mjör, 1990a,b; Mjör & Qvist, 1997; Burke & others, 1999; Mjör, Moorhead & Dahl, 2000b; Mjör & others, 2002). Thus, "replacement therapy" constitutes a major part of general dental practice. A number of determining elements effect the replacement rate of restorations, including patient, material and clinician factors.

Detailed, practice-based, longitudinal studies on the selection of restorative materials, reasons for replacement and longevity of restorations in the primary dentition have recently been published (Qvist & others, 2004a,b; Qvist, Manscher & Teglers, 2004c). This review will be limited to restorations in the permanent teeth of adults and will focus on composite and amalgam restorations, because they are the most frequently used restorations.

FAILURE OF RESTORATIONS

Restorations diagnosed as failed will, in most cases, be replaced. The cost of this treatment must represent billions of dollars annually on a worldwide basis. Many factors play a role in determining the rate of the replacement of restorations, notably, the definition of what constitutes a failure. The date when studies of failures are carried out will also have a profound effect on the results. For example, reasons for the replacement of restorations using basically the same methodology have shown marked differences in the reasons for replacement of resin-based composite restorations in studies 20 years apart (Mjör, 1981; Mjör & others, 2000b). The increased use of composites and the wider indications for their use are included in these differences. The indications have changed markedly from being primarily used in small restorations in anterior teeth and in single-surface facial restorations to becoming an all-round material used in small and large, anterior and posterior restorations. Degradation, sometimes referred to as wear which was only a part of the problem, was the main reason for replacement of composite restorations in the 1970s, while secondary caries was cited as the main reason for failure in the 1990s. Two main reasons are considered important for the change in reasons for replacement of composite restorations; improved clinical techniques based on more adequate teaching of posterior composites at dental schools (Mjör & Wilson, 1998; Wilson & Mjör, 2000) and, on accumulated experience, largely by trial and error by clinicians in practice. The other main reason for the changes includes improved composite properties and adhesive technology.

The reasons for replacement of amalgam restorations have remained basically the same from the 1970s up to now (Mjör, 2001); in fact, they have not changed significantly during the last 100 years (Mjör, 2005). Moderate improvements in ditched margins as a reason for replacement has been noted in practice-based studies, possibly as a result of the introduction of high copper/"non-gamma-two" amalgams or due to the exclusion of amalgams with high creep (Mjör & Espevik, 1980). Presently, the reasons for replacement of amalgam and modern composite restorations are similar, with the exception of discoloration, which is not applicable for amalgam restorations (Mjör & others, 2000b).

Diagnosis of Failures

Some diagnoses of restoration failures are easy and cannot be disputed, for example, entire or partial loss of the restoration. Other diagnoses are more dubious and open to subjective opinion and interpretation. The clinical diagnosis of secondary (recurrent) caries is the most frequently cited, but a most ambiguous diagnosis for the replacement of restorations. The diagnosis of secondary caries is poorly defined, both in practice (Mjör & Toffenetti, 2000; Mjör, 2005) and in teaching programs (Clark & Mjör, 2001), and many studies have pointed to marked variations in this diagnosis among clinicians, both inter- and intra-individual variations (Merrett & Elderton, 1984; Tveit & Espelid, 1986; Maryniuk, 1990; Espelid & Tveit, 1991; Bader & Shugars, 1992, 1993; Tveit & Espelid, 1992; Setcos & others, 2004). This clinical diagnosis will be dealt with under a separate subheading. Part of the explanation for the variations noted may be because the studies did not discriminate between diagnosing and treatment planning (Özer & Thylstrup, 1995).

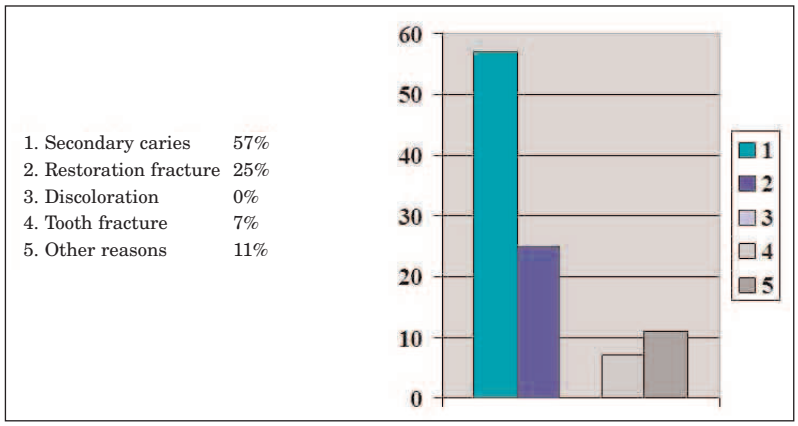

Figure 1. *Reasons for replacement of amalgam restorations (n=5,731). Data from Mjör and others (2000b).*

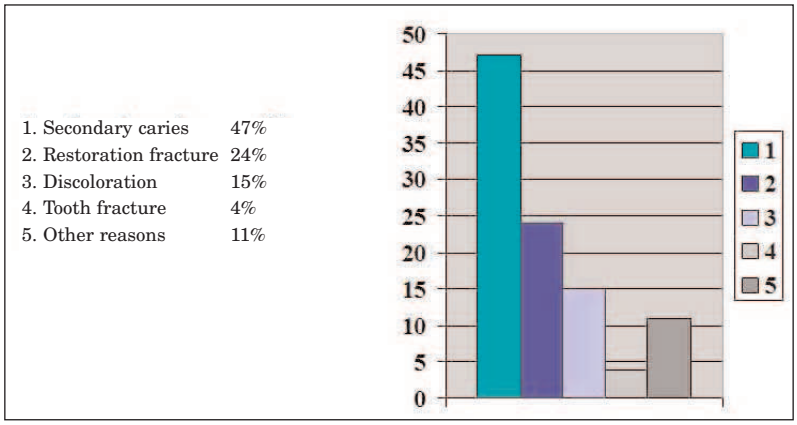

Figure 2. *Reasons for replacement of composite restorations (n=2,952). Data from Mjör and others (2000b).*

Some reasons for failure are only indirectly related to the restoration. Fracture of tooth, for example, is usually listed as a reason for replacement of restorations, but bulk fracture, such as cusp fracture, associated with restorations, may just as well be a failure of the tooth rather than a failure of the restoration *per se*. On the other hand, marginal fractures/degradation (or "ditching") differs in extent and degree, and the magnitude of marginal degradation is open to discussion as a reason for replacement. Degradations are commonly seen on the occlusal part of amalgam restorations. Such marginal defects do not predispose to secondary caries. Caries lesions are found equally often at non-ditched and ditched amalgam margins (Pimenta, Navarro & Consolaro, 1995), and ditching is not related to the consistency and color of the subjacent dentin (Kidd, Joyston-Bechal & Beighton, 1994; 1995). Only frank caries lesions at the margin of restorations constitute reliable diagnosis of secondary caries lesions (Kidd & Beighton, 1996).

Pain/sensitivity is a reason for replacement of only 1% to 2% of all restorations replaced, irrespective of the restorative material. "Change of material" of functional restorations without any defects has also become a reason for replacement, especially of amalgam restorations.

Discoloration of the bulk and/or margin of tooth-colored restorations are criteria that are subject to interpretation by the clinician and patient, either individually or together. The first resin-based composites introduced on the market were deficient in many respects, and bulk discoloration was a major problem. Marginal discoloration was also common before clinicians accepted acid etching as a legitimate and required part of the clinical procedure for placement of resin-based restorations. Improvements in adhesive technology and materials must also have played a role. These discoloration issues have largely been resolved, but extrinsic staining is still a factor to be considered.

A number of other defects result in the replacement of restorations. Some are of iatrogenic origin, for example, lack of adequate contact to adjacent teeth, overhangs at gingival margins, porosities in the restorative material and poor anatomical form of the restoration. Other defects, such as material degradation, were found to be associated with silicate cement restorations when they were in use. The first marketed composites also exhibited material degradation that predisposed them to wear and loss of anatomical form. Bruxism and unusual patient behavior may contribute to excessive wear.

In an early practice-based study where a list of reasons for replacement of restorations was provided to the clinicians, the last alternative was "more than one reason" (Mjör, 1979). Only about 2% of all replacements had more than one reason cited for replacement. Subsequent studies have not included this question, and all the infrequently cited reasons have usually been lumped into "other reasons;" the main reasons for failure have been limited to:

1. Secondary caries
2. Fracture of restoration
3. Discoloration of tooth-colored restorations
4. Fracture of tooth
5. Other reasons

Restoration and discoloration fractures have often been subdivided into bulk and marginal defects. Since the question regarding "more than one reason" was included in only the 1979 study, the topic should be repeated in a future investigation, with the restorative materials currently in use, and the information should be updated on a regular basis.

The relative frequency cited as reasons for replacement of amalgam and composite restorations in adults from a practice-based study carried out in the late 1990s are illustrated in Figures 1 and 2. Secondary caries, fracture of restorations, fracture of tooth and discoloration of tooth-colored materials comprise 82% to 90% of all reasons for replacement, leaving only 10% to 18% for a number of "other reasons." Other reasons include loss of restoration, poor anatomical form, pain/sensitivity, "change of material" and any undefined reason.

Fracture of tooth is recorded as a reason for replacement in 4% to 7% of all replaced restorations. A recent study has confirmed that tooth fracture occurs infrequently, and no significant difference exists between cusp fractures associated with amalgam and composite restorations (Wahl & others, 2004). Poor anatomic form, which may be associated with material degradation, is not a problem like it was for the composites used 20 to 30 years ago. Conventional glass ionomer restorations replaced at the 9% level based on poor anatomical form (Mjör & others, 2000b) indicate that material degradation is still a problem for certain types of restorative material. It is noteworthy that marginal discoloration is not much of a problem with present-day composite restorations, suggesting that proper clinical techniques have been adopted. Excess material at the gingival margin or "overhang" is rarely a reason for replacement of restorations, possibly because they can be removed without replacing the entire restoration. However, small overhangs may be difficult to detect and they are closely linked to plaque accumulations at the gingival margin of restorations (Özer, 1997).

This brief review of reasons for replacement of restorations highlights two issues:

1. The main reasons are few and easy to describe, but some are difficult to diagnose and open to subjective interpretation.
2. The need for definitions of what constitutes failures, to a degree that requires replacement of restorations, is imminent.

These issues should be at the forefront of tasks to be studied, evaluated, discussed and finalized within the field of Operative Dentistry. If we do not come to terms with these tasks, others, such as insurance companies or patient advocacy groups, may take over and dictate terms that may not optimally serve the public and the profession. It is important in this context to recognize that treatment decisions should be based on facts rather than on traditions (Boyd, 1989).

Secondary Caries

Practice-based studies over the last decades and, in fact, ever since GV Black's time more than 100 years ago, have shown that the clinical diagnosis of secondary caries is by far the most common reason for replacement of all types of restorations (Black, 1908; Healy & Philips, 1949; Dahl & Eriksen, 1978; Mjör, 1981; Qvist & others, 1990a,b; Wilson, Burke & Mjör, 1997; Mjör, 1997; Burke & others, 1999; Mjör & Toffenetti, 2000; Delgorgi & others, 2000; Mjör & others, 2000b; Mjör & others, 2002; Setcos & others, 2004). Depending on a number of variables, practice-based studies have shown the relative percentage of restorations replaced on adults due to secondary caries to vary from 32% (Qvist & others, 1990a,b) to 72% (Mjör, 1985). Most often, the percentage of replacements of amalgam restorations with the clinical diagnosis of secondary caries is at the 50% to 60% level. For tooth-colored restorations, this percentage is about 50%. Other rea-

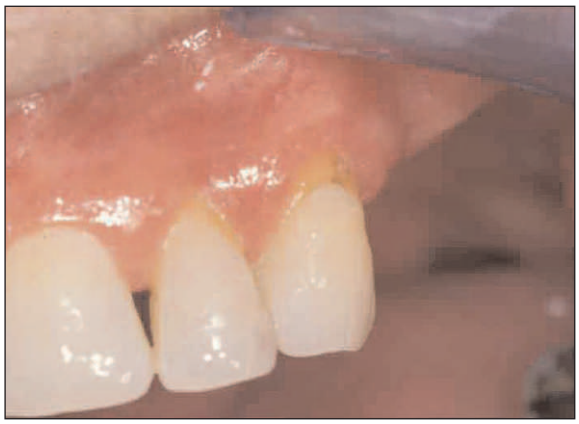

Figure 3. *Note two small lesions on the canine gingival to the composite restoration. The one at the margin of the restoration was diagnosed as a secondary caries lesion and the one further gingivally as a primary root caries lesion. (From the UFCD student clinic.)*

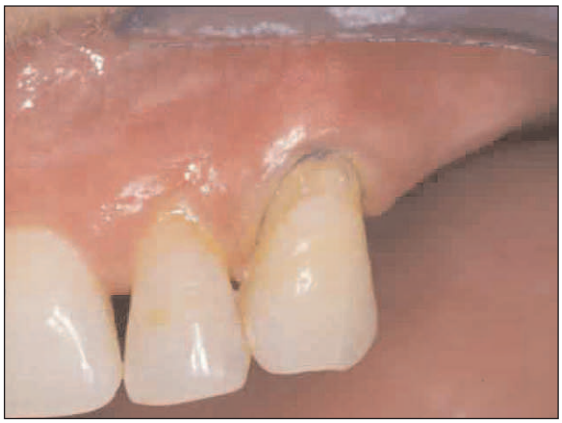

Figure 4. *The cavity preparation included the two lesions shown in Figure 3. Note that the "secondary caries lesion" was a limited, stained, localized, surface defect at the margin of the restoration.*

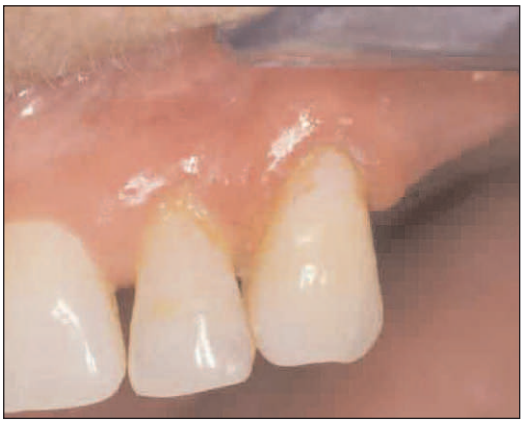

Figure 5. *The gingival defects shown in Figure 3 after the lesions were restored/repaired.*

sons for failure of all types of restorations include restoration fracture, bulk and marginal, poor anatomical form and fracture of tooth. Tooth-colored restorations may also fail as a result of bulk and marginal discolorations. It should be noted that the differential diagnosis of stained margins and secondary caries lesions (Figures 3 and 4) is difficult (Tyas, 1991; Kidd & Beighton, 1996), and these defects may often be repaired rather than lead to replacement of the restorations in order to save tooth tissues (Figure 5).

A number of additional reasons have also been cited in many studies, but since these generally comprise 2% to 4% and less than 10% of all replacements, they are included under "other" reasons.

A summary of multiple, longitudinal *controlled* clinical trials of amalgam restorations showed that 80% of the reasons for failures were due to some form of fracture of the restorations (Letzel & others, 1997), while secondary caries was only diagnosed in 2% of the cases after up to nine years' observation periods. After nine years, secondary caries comprised 11%, making a total failure due to secondary caries of 5%. The differences in the reasons for failure of restorations in controlled clinical trials and in practice-based studies are amazing (Mjör, 2001).

Although the clinical diagnosis of secondary caries is poorly defined and shows great variations in diagnostic agreement between clinicians, it has remained the main reason for replacement of all types of restorations over the last century. Relevant, modern textbooks provide little information on this major issue in clinical dentistry. Therefore, a short, critical assessment of the diagnosis will be presented.

Diagnosis of Secondary Caries Lesions

It has repeatedly been pointed out that the nature of secondary caries lesions has not been studied in detail (Özer & Thylstrup, 1995; Mjör, 2005). Review papers, supported by case reports, indicate that the lesions are limited and localized and, therefore, lend themselves to repair without the need for replacement of the entire restoration (Mjör & Toffenetti, 2000; Mjör & Gordan, 2002; Mjör, 2005). "Microleakage," which is often incorrectly associated with the clinical diagnosis of secondary caries, has no clinical support or even *in vitro* foundation related to secondary caries (Goldberg & others, 1981; Merrett & Elderton, 1984; Söderholm, Antonson & Fishlschweiger, 1989; Özer & Thylstrup, 1995; Mjör & Toffenetti, 2000). This term should be limited to results from *in vitro* permeability studies, and these should not be extrapolated to be valid for *in vivo* conditions without clinical evidence (Özer & Thylstrup, 1995; Sá & others, 2004).

Taking into account the magnitude, consequences and significance, including the financial implications of the diagnosis of secondary caries in general dental practice, it is astounding that few detailed studies have been devoted to the etiology of secondary caries.

Based on the literature reviewed in this paper and related publications (Mjör & Toffenetti, 2000; Mjör 2005), case reports and clinical experience, the most important points relevant to the clinical diagnosis of secondary caries may be summarized as follows:

1. Secondary caries lesions are, per definition, located at the tooth-restoration junction; the lesions start and develop at this surface location; it does not selectively progress into the tooth/restoration interface.
2. The lesions are diagnosed gingivally in about 90% or more of all cases recorded and usually as a result of an explorer "catch," often without the possibilities for direct visual inspection, rather than by carefully exploring the softness of the tissues involved.

3. The lesions can be diagnosed radiographically, provided a radiopaque restorative material does not obstruct the x-rays.
4. The bacteriology of secondary caries lesions is similar to that of primary caries lesions.
5. Overhangs of any size located interproximally predispose to the development of secondary caries lesions due to predisposition for plaque accumulation.
6. No well-defined, objective clinical criteria for diagnosing secondary caries lesions have been presented.
7. Poor agreement between clinicians has been found in *in vivo* and *in vitro* studies diagnosing secondary caries lesions.
8. Clinical studies have shown that the diagnosis of secondary caries associated with tooth-colored restorations is difficult to differentiate from stained margins.
9. Leaching of fluorides from restorative materials does not result in a lower frequency of clinically diagnosed caries lesions compared to non-fluoride releasing materials.
10. Crevices at the restoration-tooth interfaces are unrelated to the development of secondary caries lesions, except when the crevices are large.
11. "Microleakage" is unrelated to the development of secondary caries lesions.
12. Degraded, "ditched" restoration margins, which are characteristically found on the occlusal part of restorations, do not lead to the development of secondary caries lesions.

Based on the findings outlined in these 12 points, it has been concluded that secondary caries lesions are the same as primary caries lesions, the only difference being that the lesions are located adjacent to restorations (Özer, 1997; Mjör & Toffenetti, 2000; Mjör, 2005). The accumulation of cariogenic plaque is the fundamental etiological factor for the development of primary and secondary caries lesions (Özer & Thylstrup, 1995). As a consequence, the same diagnostic criteria should be used for primary and secondary caries lesions, including a differentiation between active and arrested lesions.

Radiolucency of the hard tissues involved is a common finding for both primary and secondary caries lesions, but radiopaque restorative materials may make the diagnosis of secondary lesions difficult or impossible. Active caries lesions are diagnosed clinically by careful probing to detect softening of the mineralized tissues, and by visual examination to assess discoloration and wetness of the tissues involved (Kidd, Joyston-Bechal & Beighton, 1993; Kidd & Beighton, 1996). Cavitation occurs as the lesion progresses. However, no investigations are available to show the progression of secondary caries lesions, and such studies are urgently needed. If the progression is like that for primary caries lesions (Shwartz & others, 1984), secondary caries will take up to four years before they progress through enamel in a population exposed to fluoride, for example, fluoride toothpaste. At any rate, it must be acknowledged that it takes time to develop caries lesions. Provided the cavosurface margins were caries-free at the time the restoration was placed, it is unlikely that caries will develop within a short time, especially when it is associated with fluoride-releasing materials. It is, therefore, unlikely that the diagnosis of secondary caries on glass ionomer restorations after less than two years (Mjör, Dahl & Moorhead, 2000a) really is caries. They probably represent localized defects other than caries. If all these defects diagnosed clinically as secondary caries lesions, in fact, were carious, fluoride release from restorative materials should have a beneficial effect.

Arrested caries lesions may not be radiolucent due to remineralization of a previously active lesion. Furthermore, an arrested lesion appears characteristically dark brown, hard and dry. It is important to differentiate between arrested and actively progressing lesions, because the arrested lesions do not require operative treatment except to improve esthetics when applicable.

The outlined criteria for the diagnosis of secondary caries are similar to that used to diagnose primary caries lesions. If they are adopted, they are likely to significantly reduce the number of lesions diagnosed and, therefore, fewer restorations will be replaced. A repair option must always be considered in the treatment of secondary caries lesions (Mjör & Gordan, 2002; Gordan & others, 2003; Blum & others, 2003; Mjör, 2005). Repair of restorations is also in line with the concepts of minimal intervention (Mjör, 2003).

LONGEVITY OF RESTORATIONS

The longevity of restorations is closely linked to their replacement rate; it being lower in the primary dentition and in the permanent teeth of adolescents rather than in adults (Qvist & others, 1990a,b; Mjör & others, 2000). The diagnoses of failures are critical in studies of longevity of restoration. However, no generally accepted, objective guidelines have been forwarded for what degree of failure constitutes conditions that will cause future damage to the tooth involved.

Apart from patient age, factors such as oral hygiene, access to fluorides and other caries preventive agents, oral habits, difficult conditions during placement of the restorations and the size of restoration will each potentially have an effect on the replacement rate of restorations. Furthermore, patient factors such as oral

hygiene seem to influence the choice of materials and possibly the replacement rate (Burke & others, 2001). The type of reimbursement system for services may also play a role (Mjör & others, 2000a; Burke & others, 2001). Clinician gender has also been indicated in one study to be a factor affecting the longevity of restorations, especially tooth-colored restorations (Mjör & others, 2000a), as well as the clinicians' experience, expressed as years since graduation (Mjör & others, 2002). The physical properties of the restorative material also play a role, but often not as decisively as it is often believed. In general, tooth-colored restorations have a shorter longevity than metal restorations. The single most important factor seems to be the skill of the operator, not only technically, but also diagnostically.

CONCLUDING REMARKS

The etiology of reasons for failures and reasons for the replacement of restorations will vary depending on many factors. These restorations will change with the continuous development of operative dentistry. Practice-based studies should, therefore, be conducted at regular, predetermined time intervals to monitor changes that occur. The results from such surveys are important, not only for the practitioners, but also for updating teaching programs and for information to manufacturers of restorative materials.

The total body of practice-based evidence emphasizes the importance of clinically diagnosed secondary caries lesions. However, little information is available on the nature of "secondary caries" and, based on literature reviews and practice-based data, it is evident that not all clinically diagnosed secondary lesions are true caries lesions. In this context it is important to keep in mind that the explorer, a major diagnostic tool in the detection of secondary caries lesions, will catch in any crevice and narrow space whether or not caries is present. Strict criteria for diagnosing secondary caries lesions must be established, and it should be basically similar to that used to diagnose primary caries. In this context, it is important to distinguish between diagnosing the lesions as a result of disease or as part of treatment planning, which refers to decisions about clinical procedures to remedy the lesion diagnosed.

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Clinical Research

Comparative Study of the Effects of Two Bleaching Agents on Oral Microbiota

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Clinical Relevance

While being a simple and effective procedure for achieving dental aestetics, 10% carbamide peroxide and 7.5% hydrogen peroxide agents do not provide changes in the *Streptococcus mutans* counts during bleaching procedures.

SUMMARY

This study evaluated the *in vivo* effects of bleaching agents containing 10% carbamide peroxide (Platinum/Colgate) or 7.5% hydrogen peroxide (Day White 2Z/Discus Dental) on *mutans Streptococcus* during dental bleaching. The products were applied on 30 volunteers who needed dental bleaching. In each volunteer, one of the two bleaching agents was used on both dental arches one hour a day for three weeks. Analysis of the bacterial counts was made by collecting saliva before (baseline values), during (7 and 21 days) bleaching treatments and 14 days post-treatment. The Friedman non-parametric analysis (α=0.05) found no differences in micro-organism counts at different times for each group for both agents (p>0.05). The Mann Whitney non-parametric test (α=0.05) showed no differences in micro-organism counts for both agents (p>0.05). Different bleaching agents did not change the oral cavity *mutans Streptococcus* counts.

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INTRODUCTION

Haywood and Heymann (1989) introduced the home vital tooth bleaching technique with 10% carbamide peroxide. Since then, this method has been widely used, because it is a simple and efficient procedure for removing intrinsic and extrinsic stains from teeth (Haywood, 1994).

Different products and tooth bleaching systems have been introduced, such as the use of other concentrations of carbamide peroxide (16% to 22%), hydrogen peroxide and over-the-counter products. However, carbamide peroxide at 10% seems to be the most effective and safest, as well as having the American Dental

Association (ADA) approval for some products (Haywood & Robinson, 1997).

Alterations in enamel and dentin mineral content, due to the acid properties of these materials (Ben-Amar & others, 1995; Ernst, Marroquin & Willerhausen-Zönnchen, 1996; Leonard, Bentley & Haywood, 1994; Murchinson, Charlton & Moore, 1992; Smidt, Weller & Roman, 1998; Zalkind & others, 1996), may occur. Loss of mineral content or demineralization alters the microhardness of enamel and dentin (Featherstone, ten Cate & Shariati, 1983; Rotstein & others, 1996) despite the presence of saliva, fluorides or other remineralizing solutions, which are capable of maintaining the balance between the de- and remineralization processes. However, few *in vivo* research reports have been found that focus on oral microbiota and on the use of bleaching agents, mainly on the pathogenic bacteria of dental caries (*Streptococcus mutans*, *total* and *Lactobacillus*).

Gürgan, Bolay and Alacam (1997) proved, *in vitro*, a significant decrease in the adherence of *Streptococcus mutans* on the surface of tooth enamel with the use of 10% carbamide peroxide. Bentley, Leonard and Crawford (2000) reported a bacteriostatic effect of on *Streptococcus mutans* and *Lactobacillus* when submitted to the action of 10% carbamide peroxide *in vitro* and a decrease in the salivary levels of *Lactobacillus* when analyzed *in vivo*.

Firestone, Schmid and Mühlemann (1982) assessed the action of hydrogen peroxide on periodontal tissues, tooth surfaces and the oral pH of rats. A reduction in the accumulation of bacterial plaque, the incidence of caries and decreased pH was noted.

However, *in vivo* studies regarding the action of 10% carbamide peroxide or 7.5% hydrogen peroxide on the bacteria responsible for dental caries are absent in the literature.

This study analyzed, *in vivo,* the effects of two bleaching agents containing 10% carbamide peroxide and 7.5% hydrogen peroxide on the oral microbiota (*Streptococcus mutans* and *total*) at different times (before, during and after bleaching treatment).

METHODS AND MATERIALS

Experimental Design

The factors under study were:

Bleaching Agents at Two Levels: a bleaching agent containing 7.5% hydrogen peroxide (Day White 2Z/Discus Dental Inc, Culver City, CA, USA) and one containing 10% carbamide peroxide (Platinum/Colgate-Palmolive Company, New York, NY, USA).

Bleaching Time at Four Levels: baseline, 7 and 21 days of treatment, and 14 days post-treatment period (corresponding to 35 days after beginning the bleaching treatment). At these times, saliva was collected to evaluate oral microbiota.

The experimental units consisted of stimulated human saliva collection from 30 volunteers, considering each volunteer as a block. The volunteers were randomly divided and submitted to a home-used dental bleaching treatment with 7.5% hydrogen peroxide agent or a 10% carbamide peroxide product for three weeks. The response variable, *Mutans Streptococci* colony forming units per mL saliva (CFU/mL), was quantitatively evaluated.

The basic principles of experimental design—repetition, randomization and blocking—were followed, according to Montgomery (1996).

Material Specifications

Two different brands of home-use bleaching agents were evaluated: Day White 2Z/Discus Dental (DW), which contains 7.5% hydrogen peroxide and Platinum/Colgate (PLA), which contains 10% carbamide peroxide. Table 1 presents their composition, batch number and manufacturer.

Preparation of the Volunteers for the Experimental Phase

The volunteers were 30 adults (23 women and 7 men) ranging in age from 18 to 25 years. Each volunteer was informed about the goals, benefits and possible risks involved in this experiment and participated only after providing formal written consent. This study had the approval of the Uniararas Ethical Committee

Table 1: *Composition, Batch Number and Manufacturer of Each Material Used in This Study*

Material	Composition*	Batch #	Manufacturer
Day White 2Z	7.5% hydrogen peroxide, carbopol, coloring, flavoring	02352012 02354056 02281025	Discus Dental, Inc Culver City, CA, USA
Platinum	10% carbamide peroxide, flavoring, water, calcium pyrophosphate, polossamero 407, PEG-12, PEG-2M, glycerin, calcium phosphate dihydrate, sodium pyrophosphate acid, sodium laurel Sulphate, sodium saccharine, EDTA- disodium dihydrate.	301002 208001	Colgate-Palmolive Company, New York, NY, USA

**According to the manufacturer.*

Guidelines, in agreement with the National Health Council (Brazil, 1996).

The volunteers who were candidates for bleaching treatment were graduate students from the Dentistry School of Uniararas, Araras, Brazil. Each volunteer's need for bleaching treatment was assessed, and only those who had esthetic or pathologic reasons were selected. The tooth color assessment was made by using a Vita shade guide (Vita Zahnbabrik, H= Rauter GmbH & Co KG, Bad Säckingen, Germany) and was also recorded through photographs to provide a comparison between the initial and final color achieved. Inclusion criteria included: tooth color A2 or higher, tetracycline stain degrees I and II, fluorosis with brown discoloration and physiological dark stains. Exclusion criteria for participation in this study included: volunteers who wore fixed or removable dentures or orthodontic appliances, pregnant or nursing women, smokers, volunteers with dentin sensitivity, those ingesting medicines that would decrease salivary flow or those individuals who used any microbial agent for the three months prior to the start of the study.

Top and bottom dental arch impressions were taken with alginate (Jeltrate/Dentsply Caulk, Milford, DE, USA) and stone cast molds were made (Vigodent, Rio de Janeiro, RJ, Brazil). The maxillary casts were horseshoe shaped without a palate to avoid interference with vacuum pull efficiency on the hot thermoplastic sheet. In the molds, vestibular reservoirs with three coatings of nail varnish (Colorama/CEIL Com Exp Ind Ltda, São Paulo, SP, Brazil) were prepared for all teeth for bleaching agent deposition. A scalloped tray was manufactured for each volunteer in a vacuum forming machine (P7/Bio-Art Equip Odontológicos Ltda, São Carlos, SP, Brazil) using a 1.0-mm thick flexible ethyl vinyl acetate polymer (Bio-Art Equip Odontológicos Ltda). The trays were trimmed and polished up to the cervical margin of the teeth, restricting the bleaching agent action to the teeth.

Two weeks before the experiment began, toothbrushes (Oral B P30/Gillette do Brasil Ltda, Manaus, AM, Brazil) and sodium fluoride dentifrices (1100 ppm) (Sorriso/Colgate Ltda, Osasco, SP, Brazil) were given to the volunteers. They were instructed on how to perform the Bass dental hygiene technique to standardize the toothbrushing method and fluoride levels in the mouth. This phase was called the *run-in* period and lasted for two weeks. The volunteers were asked for a daily diet form for mapping their food habits starting from the run-in period through to final saliva collection.

The 30 volunteers were randomly divided into two equal groups of 15. One group received the 7.5% carbamide peroxide bleaching agent (DW), while the other received the 10% carbamide peroxide bleaching agent (PLA).

Saliva Collections

Microbiological Samples

The morning prior to starting bleaching treatment, whole saliva samples were collected in sterile tubes from each of the volunteers after stimulation in order to assess the *Mutans Streptococci* (MS) levels in saliva. Volunteers were asked to chew on a piece of paraffin wax until it attained the soft consistency of chewing gum. They swallowed any saliva present in the mouth, chewed on the paraffin for an additional minute, using both sides of the mouth and intermittently spat saliva into a sterile glass tube (Dasanayake & others, 1995).

All the samples were placed on ice and plated within three hours. Most samples were processed within 90 minutes of collection.

Bacteriological Methods

The samples were dispersed by vortex (Wan & others, 2001a,b; 2003) for 60 seconds to disperse bacteriam, then submitted to 10-fold dilutions in saline solution (0.9% NaCl) through 10^{-4}. Aliquots of 5 µL of each dilution were spread on small Petri dishes (5x2 cm), in duplicate, containing Mitis Salivarius Agar (Difco Co, Detroit, MI, USA) supplemented with two IU of bacitracin/ml (Sigma Chemical Co, St Louis, MO, USA), 1% potassium tellurite (Sigma Chemical Co) and 15% sucrose (Synth, Diadema, SP, Brazil) for MS cultivation (MSB) (Gold, Jordan & Van Houte, 1973). All plates were incubated under anaerobic conditions (37°C, 10% CO_2) for 48 hours.

Thereafter, a stereoscopic microscope was used to verify the presence of colony-forming units (CFU) resembling MS. They were distinguished by their molar and granular frosted glass-like formation, adherent growth in agar and production of glucans surrounding colonies (Emilson, 1983). MS Counts were considered as baseline.

Bleaching Agents Protocol

The volunteers were instructed to put the corresponding bleaching agents in the trays and wear them for one hour during the day, concurrently for both arches. Directions were given to avoid consuming foods or beverages while using the tray and cleaning it after taking it out of the mouth and storing it in the container provided. The volunteers were instructed to apply the bleaching agent for 21 days and were monitored weekly for the presence of tooth sensitivity or gingival irritation.

On the seventh and twenty-first days, saliva collections were performed. Upon completion of the treatment, another saliva collection was performed after 14

days (corresponding to 35 days from the start of the treatment).

Statistical Analysis

To compare differences among the various saliva collection times for each group, the data of volunteers who missed some of the collections were excluded. For PLA, seven volunteers were excluded due to problems with sensitivity or lack of compliance, while one volunteer was discarded from group DW.

Means, standard deviations and confidence intervals (95% CI) were determined for all data. The MS levels in CFU/mL of volunteers' saliva were converted into logarithm (log_{10}) for statistical analyses. Non-parametric analysis were used to compare bacteriological counts for each bleaching agent over time and between products.

RESULTS

The Friedman non-parametric test was used to verify MS counts over time for each bleaching agent, showing that there were no differences ($p>0.05$). The Mann Whitney non-parametric test showed that there were no differences among MS counts at each time interval for both bleaching agents ($p>0.05$). Table 2 shows the mean and standard deviation for PLA and DW at each saliva collection time and Figure 1 shows the linear diagram for MS samples over time for both bleaching agents.

DISCUSSION

Home dental bleaching procedures have become increasingly frequent because they are simple and efficient. However, due to the bleaching agent's close contact with tooth structure, microscopic alterations to the enamel surface and dentin have been reported (Ben-Amar & others, 1995; Bitter, 1998; Bitter & Sanders, 1993; Ernst & others, 1996; Flaitz & Hicks, 1996; Josey & others, 1996; Shannon & others, 1993; Smidt & others, 1998; Zalkind & others, 1996), although there has been no clinical harm to tooth structure. These superficial alterations to the teeth, such as the presence of erosion and porosity, and the increase in dentin tubule diameter, have been related to by-products of bleaching agent oxidation reaction (Arends & others, 1984; Goldberg & others, 1983; Hegedüs & others, 1999).

With regard to the micro-organism responsible for caries infection, the complexity of the microbiota present in biofilm makes it difficult to determine a single bacterial agent in its etiology. However, there is considerable evidence that gram positive, acidogenic, uric acid microorganisms, with a capacity to adhere to tooth surfaces, belonging to a heterogeneous group called the *streptococcus mutans* group, are especially involved in the dynamics of the caries process (Loesche, 1986).

The oral *Streptococcus mutans* group is comprised of seven species: *Streptococcus cricetus*, *S downei*, *S ferus*, *S macacae*, *S mutans*, *S rattus* and *S sobrinus* (Whiley & Beighton, 1998), classified on the basis of genetic, antigenic and biochemical characteristics. Among the species of this group, *S mutans* and *S sobrinus* are isolated with greater frequency from dental plaque and are strongly related to human dental caries (Seow, 1998), as well as forming part of the oral microbial ecology of the great majority of human beings (Li, Wang & Caufield, 2000).

Due to the multi-factorial characteristic of caries disease, in order for it to start and develop, it is necessary for an inter-relation between time and behavioral, cultural, social and biological factors inherent to the host. Thus, the harmonious coexistence of microbiota with

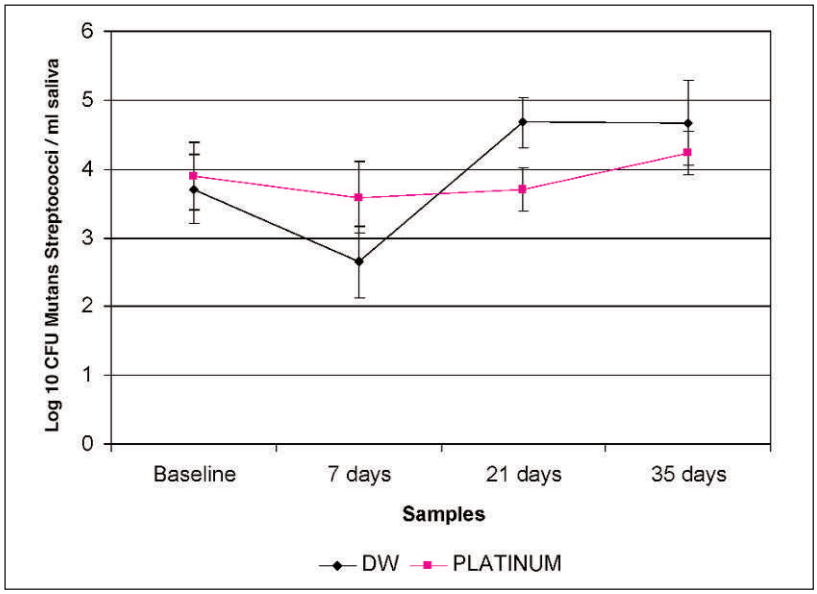

Figure 1. *Linear diagram of* Mutans Streptococci *samples (Log 10/mL saliva) related to the bleaching agent used (Mann Whitney test,* p*>0.05; Friedman test,* p*>0.05).*

Table 2: *Mean and Standard Deviation (SD) for PLA and DW Over Time*

Bleaching	Baseline		7 Days		21 Days		35 Days	
Agent	Mean	SD	Mean	SD	Mean	SD	Mean	SD
PLA	1.88 x 10^5A	3.10 x 10^5	2.15 x 10^5A	4.69 x 10^5	4.27 x 10^4 A	9.07 x 10^4	5.76 x 10^4 A	5.71 x 10^4
DW	1.96 x 10^5 A	4.62 x 10^5	3.84 x 10^4 A	8.89 x 10^4	5.77 x 10^5 A	8.63 x 10^5	4.51 x 10^5 A	8.27 x 10^5

*Equal letters indicate mean values that are not significantly different (*p*>0.05).*

the host is disturbed the moment environmental alterations influence the balance of the oral eco-system and makes the micro-organisms with better adaptive capability begin to act as pathogens, predisposing the host to disease (Marsh, 1991).

The formation of dental biofilm occurs in two distinct phases. During the first phase, proteins from the bacterial surface interact with the host or with bacterial products adsorbed on the tooth surface. In the second phase, bacteria accumulate in the biofilm by cell proliferation, the interaction between receptors and adesines between micro-organisms of the same species or different species and through the production of an extracellular polysaccharide matrix (Kolenbrander, 2000). *S mutans* secrete various proteins that may be involved in tooth surface colonization mechanisms in processes dependent on and independent of sucrose (Hamada & Slade, 1980; Loesche, 1986; Steinberg & others, 1999).

Bacterial adherence to tooth surface is also dependent on properties of the surface on which it will be adsorbed. Some factors that may facilitate bacterial accumulation on the tooth structure are brought about by the presence of surface roughness and electrostatic and hydrophobic interactions (Steinberg & others, 1999). Bleaching agents may affect tooth structure, altering the biofilm formation process (Steinberg & others, 1999). Although Mc Guckin, Babin and Meyer (1992) had observed an increase in enamel surface roughness after using 10% carbamide peroxide, Gürgan and others (1997) showed that there were no differences between a bleached and non-bleached surface. Furthermore, the effects of 10% carbamide peroxide seem to minimize the adherence of *Streptococcus mutans*, *Streptococcus sobrinus* and *Actinomises viscosus* (Steinberg & others, 1999), present a bacteriostatic effect *in vitro* for *Streptococcus mutans* (Bentley & others, 2000; Gürgan, Bolay & Alacam, 1996), *Streptococcus mitis*, *Streptococcus sanguis* (Gürgan & others, 1996) and *Lactobacillus* (Bentley & others, 2000; Gürgan & others, 1996) and reduce the salivary levels of *Lactobacillus in vivo* (Bentley & others, 2000). These results indicate that bleaching agents containing carbamide peroxide may present a beneficial effect to be used as an anti-cariogenic agent. In opposition, Hosoya and others (2003), assessing the effect of vital bleaching in an *in vitro* study, found an increase in both enamel surface roughness and *S mutans*' adherence to the tooth surface.

In this *in vivo* study, the sample plan comprised 30 dentistry graduate students, making up a homogenous sample with regard to adequate oral hygiene and fitting into a context of being motivated with regard to oral health. Therefore, the mean prevalence of the *streptococcus mutans* group in this population during baseline collection was shown to be reduced, as noted in Table 2 and Figure 1. This factor may have masked the more realistic results related to reductions in microbial counts after application of the bleaching agents at their different times.

The study was carried out for five weeks—baseline (prior to bleaching treatment), 7 and 21 days of bleaching treatment and 14 days after bleaching—in which saliva samples were collected to assess oral microbiota. The choice for quantifying the *streptococcus mutans* group in saliva was made because of its association with the prevalence and activity of dental caries (Gibbons & Houte, 1975; Loesche, 1986; Dasanayake & others, 1995; Bowden, 1996). The reduction in the number of these bacteria has been related to the control and prevention of the disease (Zickert, Emilson & Krasse, 1982; Gisselsson, Birkhed & Bjorn, 1988; Lindquist & others, 1989; Isokangas & others, 1991; Bowden, 1996). Furthermore, saliva is considered a niche representative of the oral colonization standard, as the count of its micro-organisms is pointed to as the main method used for the quantification of this microbial group (Dasanayake & others, 1995), there being a direct relation between its number and the number of intra-oral sites colonized (Togelius & others, 1984; Dasanayake & others, 1995). Thus, the number of micro-organisms having been reduced or not as a result of carrying out the treatment may indicate a condition of reduction in the cariogenic potential of the bacterial biofilm or of caries disease, although these parameters have not been measured.

The results obtained show that there were no differences among the different times or between products, bearing in mind the lack of homogeneity of variance of the results. It is emphasized that, in the literature, there are no studies using this *in vivo* methodology for assessing the effect of bleaching agents containing 10% carbamide peroxide and 7.5% hydrogen peroxide on the *streptococcus mutans* group. In an *in vivo* study, Bentley and others (2000) did not find the effects of reduction in the *streptococcus mutans* group counts with the use of 10% carbamide peroxide, which may be the result of the short period of exposure to peroxide (one hour per day for six weeks), loss of the active agent or even the possibility of the number of volunteers being insufficient (10 adults), as plausible justifications for this study.

Results of *in vitro* studies, although they contribute very important findings, may not be representative of *in vivo* oral conditions since, under these conditions, the oral cavity is continually bathed by organic and inorganic salivary components that influence bacterial adhesion (Gürgan & others, 1997).

An important factor to consider for not finding alterations in the salivary microbial count relates to the tooth faces on which bleaching occurs. With special

esthetic purposes, the elected faces are restricted to the vestibular surfaces of front teeth. It is known that the *streptococcus mutans* group, weak initial colonizers, present greater affinity of adherence to retentive surfaces, especially occlusal ones (Gonçalves & Flório, 2003). This being the case, bleaching gel contact with those tooth surfaces most colonized by the group occurs through the compound diluted in oral fluids. Different results may have been obtained if the collection of bacterial plaque had been done on different tooth surfaces and if the volunteers had halted the procedures of mechanical dental biofilm removal during the treatment period.

CONCLUSIONS

Based on the results obtained, no alterations were found in the *streptococcus mutans* group count during bleaching treatment with bleaching agents containing 10% carbamide peroxide or 7.5% hydrogen peroxide.

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Eighteen-month Clinical Performance of a Self-etching Primer in Unprepared Class V Resin Restorations

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Clinical Relevance

The use of acid etching or a self-etching primer for the minimum specified time is not, alone, sufficient to retain resin restorations of unprepared Class V lesions.

SUMMARY

This study evaluated the clinical performance of unprepared Class V resin composites, placed using a self-etching primer and a single-bottle adhesive, over a period of 18 months. Thirty-eight pairs of restorations of Renew hybrid resin composite (BISCO, Inc) were placed using adhesives from the same manufacturer in caries-free cervical erosion/abfraction lesions. Based on insensitivity to air, the dentin in 76% of these lesions was considered to be sclerotic. The restorations were placed without abrasion of tooth surfaces, except for cleaning with plain pumice. One of each pair was placed using Tyrian, a self-etching primer and the other was placed using One-Step, a single-bottle adhesive placed after acid etching. Both the etchant and self-etching primer were applied for 20 seconds. The restorations were clinically evaluated at baseline, 6, 12 and 18 months, using modified Ryge/USPHS criteria. For both adhesives, very low retention of 50% to 56% of the restorations was observed over 18 months, leading to the conclusion that tooth surfaces must receive some additional treatment prior to restoration with these adhesives. No statistically significant difference (p=0.75) between the two adhesives was observed in overall performance, and dentinal sclerosis and axial depth did not appear to be important factors in the study.

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INTRODUCTION

The adhesion of resin to dentin became feasible with the advent of hydrophilic resins capable of infiltrating into and polymerizing within a moist decalcified dentinal surface to form a hybrid layer (Nakabayashi,

Ashizawa & Nakamura, 1992). Although nomenclature varies, in this report, dentin-adhesive resins that use etching and rinsing prior to application have been called "total-etch" and "single-" or "two-bottle," depending on whether the primer and adhesive are combined. Self-etching resins have been termed "self-etching primers" when there is a separate adhesive or "self-etching adhesives" when etchant, primer and adhesive are combined.

The adhesive restoration of non-carious cervical lesions with resin composites is considered problematic, because the dentinal surface of such lesions is often hypermineralized and resistant to acid etching (Van Meerbeek & others, 1994). Laboratory studies consistently demonstrate reduced efficacy of resin-based adhesives in bonding to dentin of this type (Yoshiyama & others, 1996; Tay & others, 2000; Kwong & others, 2002), which has led some investigators to advocate the mechanical removal of the surface with rotary instruments or more aggressive etching (Kwong & others, 2002). Laboratory bond strength studies also indicate that some self-etching resins do not adhere as strongly to unprepared enamel as total-etch materials (Kanemura, Sano & Tagami, 1999; Perdigão & Geraldeli, 2003).

Clinical studies of non-retentive Class V resin composites typically include lesions in both sclerotic and non-sclerosed dentin with an incisal or occlusal enamel margin. Prior abrasion of tooth surfaces in such studies is not always reported, and it is controversial whether the use of this method constitutes restoration with "no cavity preparation," as is specified in the protocol for clinical studies of the 2001 American Dental Association acceptance program for dentin and enamel adhesive materials.

Surprisingly, dentinal sclerosis does not appear to be a factor in the retention of resin restorations in non-carious cervical lesions when total-etch resin adhesives are used and when dentinal surfaces are abraded with a diamond bur (Bayne & others, 2003). A two-year study of three adhesives, both total-etch and self-etching, has also shown neither sclerosis nor roughening of restored surfaces as being important factors in the retention of unprepared Class V resins (van Dijken, 2004), although a previous study by the same author showed both roughening and sclerosis to be detrimental to the retention of one acetone-based single bottle adhesive (van Dijken, 2000).

Non-retentive Class V resin restorations placed in unabraded lesions and retained with two-bottle adhesives have demonstrated high retention rates in representative two-year studies, ranging from 86% to 100% (Van Meerbeek & others, 1996; Browning, Brackett & Gilpatrick, 2000; De Munck & others, 2003). More recent adhesives, with various components combined, demonstrate reduced laboratory dentin bond strengths relative to earlier formulations (Bouillaguet & others, 2001; Inoue & others, 2001; Molla, Park & Haller, 2002). In clinical studies, however, Class V retention rates for single-bottle adhesives are variable, with some products retaining Class V resins and two-bottle adhesives (Swift & others, 2001; Turkun, 2003) and some at lower rates (Brackett & others, 2003; van Dijken, 2004). Among these studies, there seems to be no correlation between adhesive solvent (acetone, alcohol, water), dentinal surface abrasion or dentinal sclerosis and clinical performance.

One acetone-based adhesive, One Step (BISCO, Inc, Schaumburg, IL, USA), has been widely studied and varies widely in clinical performance according to dentinal sclerosis and application technique. The retention of resins placed with this adhesive appears to worsen in sclerotic lesions, with two authors reporting up to a 40% loss over two years (van Dijken, 2000; Baratieri & others, 2003). This effect appears to be ameliorated by the application of three or four separately-dried coats of the adhesive, as needed, to attain a shiny dentinal surface, according to an article published after the start of this study, which reported 87% retention over two years using this method (Tyas & Burrow, 2002).

Peer-reviewed reports on the clinical performance of self-etching resins are rare, and many products appear to not have been evaluated in controlled clinical trials, although some studies have been reported in abstract form. Two different adhesives with self-etching primers have demonstrated retention greater than 90% over two years in unprepared Class V restorations, while a self-etching adhesive has demonstrated relatively poor results, with retention ranging from 79% to 84% (Friedl & others, 2004; van Dijken, 2004).

Over a period of 18 months, this study compared the clinical performance in non-retentive Class V resin restorations of a well-studied acetone-based single-bottle adhesive to that of an adhesive with a self-etching primer for which no clinical trials have been published.

METHODS AND MATERIALS

Tyrian (BISCO, Inc) was the self-etching primer chosen for this study, while One-Step (BISCO, Inc) was the single-bottle adhesive chosen. The use of both adhesives preceded restoration with the same manufacturer's hybrid resin composite, Renew (BISCO, Inc). The study was conducted according to the protocol for clinical studies set forth in the 2001 American Dental Association acceptance program for dentin and enamel adhesive materials.

Thirty-eight pairs of equivalent-sized cervical erosion/abfraction lesions, primarily in premolar and anterior teeth, were identified in 25 healthy patients presenting for treatment at student clinics of the Facultad

Table 1: *Distribution of Restorative Materials to Teeth Restored; Axial Depth and Preoperative Sensitivity of Erosion/Abfraction Lesions*

Tyrian SPE

	Incisors						Canines						Premolars						Molars					
	n	s	o	a	b	c	n	s	o	a	b	c	n	s	o	a	b	c	n	s	o	a	b	c
Maxillary	2		2	1	1		4		4		4		13	5	8	5	8							
Mandibular	3		3	3									14	3	11	5	9		2		2		2	

One-Step

	Incisors						Canines						Premolars						Molars					
	n	s	o	a	b	c	n	s	o	a	b	c	n	s	o	a	b	c	n	s	o	a	b	c
Maxillary	2		2	2			3		3		3		10	3	7	4	6							
Mandibular	3	1	2	1	2		2	1	1	2			17	6	11	3	14		1		1	1		

s = sensitive to air
o = insensitive to air

a = axial depth < 1 mm
b = axial depth 1-2 mm
c = axial depth > 2 mm

Table 2: *Adhesives, Manufacturer's Instructions/Technique*

Adhesive	Technique
One-Step Universal Dentin Adhesive	Etch, 32% phosphoric acid, 20 seconds, Rinse, Briefly air dry, leaving tooth surfaces moist, Apply two thick coats, agitate 10-15 seconds, Air dry, Verify shiny surface, Light cure, 10 seconds
Tyrain SPE Self-Priming Etchant	Shake and activate unit-dose capsule, stir, Air dry tooth surfaces, apply two coats with agitation, 20 seconds, Blot dry, verify shiny surface, Apply two thick coats One-step Plus, agitate 10-15 seconds, Air dry, verify shiny surface, Light cure, 10 seconds

Table 3: *Modified USPHS Rating System*

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

de Estomatologia, Benémerita Universidad Autónoma de Puebla. The study was conducted in accordance with all local regulations for the ethical treatment of human subjects. The median age of these patients was 52 years, while the age range was 28 to 69 years. Each pair of cervical erosion/abfraction lesions received one restoration placed using each adhesive, assigned randomly. No patient received more than two pairs of restorations. All of the lesions included in the study were 1-2 mm in axial depth. The approximate size of each lesion and any sensitivity of the lesion to air from the dental unit was recorded prior to restoration (Table 1). Seventy-six percent of the lesions were insensitive to air and considered sclerotic.

Two experienced investigators familiar with adhesive dentistry placed all the restorations. Isolation was accomplished using cotton rolls, with gingival retraction cord placed to expose any subgingival margins. All restorations were placed according to manufacturers' instructions and are summarized in Table 2. Other than cleaning with plain pumice and water in a rubber prophylaxis cup, no mechanical preparation or abrasion of tooth surfaces was done. The application of acid or self-etching primer was not extended beyond the manufacturer's recommended 20 seconds, which the authors consider minimal.

Because the lesions were of minimal axial depth, each restoration was placed in one increment and light-

Table 4: *Results of Clinical Evaluation of Class V Resin Composites Placed with Self-etching Primer and Single-bottle Adhesive (%)*

Tyrian SPE

		Retention**			Color Match			Marg Disc		Sec Caries		Anat Form		Marg Adapt		Surf Texture	
	n*	alpha	charlie	n*	alpha	bravo	alpha	bravo	alpha	charlie	alpha	bravo	alpha	bravo	alpha	bravo
baseline	38	100	0	38	100	0	100	0	100	0	100	0	94	6	100	0
6 months	38	92	8	35	100	0	100	0	100	0	100	0	85	15	97	3
12 months	38	79	21	30	100	0	100	0	100	0	100	0	71	29	97	3
18 months	36	56	44	20	100	0	90	10	100	0	100	0	80	20	90	10

One-Step

		Retention**			Color Match			Marg Disc		Sec Caries		Anat Form		Marg Adapt		Surf Texture	
	n*	alpha	charlie	n*	alpha	bravo	alpha	bravo	alpha	charlie	alpha	bravo	alpha	bravo	alpha	bravo
baseline	38	100	0	38	100	0	100	0	100	0	100	0	100	0	97	3
6 months	38	89	11	34	97	3	97	3	100	0	97	3	91	9	100	0
12 months	38	79	21	28	96	4	96	4	100	0	96	4	82	18	89	11
18 months	36	50	50	18	95	5	94	6	100	0	94	6	72	28	100	0

*Sample size larger for retention than for other criteria because lost restorations were unavailable for evalation.

**Cumulative throughout the study.

cured for 40 seconds. Light output of the SL3000 curing light used (3M ESPE, St Paul, MN, USA) was found to exceed 450 mW/cm^2 prior to and after the study and was verified during placement of the restorations with the unit's built-in radiometer. For all restorations, the shade considered to be the closest match using a Vita shade guide (Vita-Zahnfabrik, Bad Säckingen, Germany) was selected. Restorations, shaped with a plastic instrument prior to light curing, were contoured with finishing burs using air/water coolant and polished with wet abrasive rubber points and cups.

At baseline, 6, 12 and 18 months, the restorations were clinically evaluated by two other calibrated investigators using modified Ryge/USPHS criteria (Cvar & Rype, 1971) which are listed in Table 3. The examiners were unaware of which adhesive had been used for any restoration, and any discrepancy between examiners was resolved before the patient was dismissed.

For purposes of statistical analysis, restorations receiving a score of "charlie" in any category were classified as failed restorations, The incidence of failures was analyzed as a pairwise comparison using McNemar's Chi square test at a confidence level of 5%.

RESULTS

At the end of 18 months, 36 pairs of restorations were available for evaluation, a recall rate of 95%. Overall retention was 50% for One-Step and 56% for Tyrian, with lost restorations the only scores of "charlie" assigned in the study. Of the 22 pairs of lesions from which restorations were lost, 12 showed loss of both restorations. There was no difference in the percentage of retained restorations between the two operators. Of the 18 One-Step-retained restorations lost, three were lost prior to the six-month recall, five during the 6 to 12 month interval and 10 during the 12 to 18 month interval. Of the 16 Tyrian-retained restorations lost, two were lost prior to the six-month recall, six during the 6 to 12 month interval and eight during the 12 to 18 month interval.

None of the teeth with retained restorations that exhibited sensitivity to air at the beginning of the study were sensitive at any recall. Twenty-four percent of the lost restorations in this study had been placed in lesions which were sensitive to air, while 75% of the lost restorations had been placed in lesions with a 1-2 mm axial depth. This was nearly the same as the incidence of sensitive and moderate depth lesions in the overall study populace, 25% and 77%, respectively.

The hybrid resin composite displayed good color match and surface texture over the course of the study, with scores of 90% alpha or more for both categories. No statistically significant difference in the incidence of failed restorations was found between the materials (McNemar's Chi square test; p=0.75). Complete results are presented in Table 4.

DISCUSSION

The low rate of retention for both adhesives observed in this study was disappointing and unexpected, as both demonstrate good bond strengths in laboratory studies. Neither axial depth nor sclerosis appears to have been important factors, given their relative incidence preoperatively and among lost restorations.

Performance of the One-Step-retained restorations was similar to data reported for sclerotic lesions in a previous study (van Dijken, 2000) and would likely have been better had the authors known to apply more layers than specified by the manufacturer according to the protocol since established by Tyas and Burrow (2002). Although the phosphoric acid supplied by this manufacturer contains benzalkonium chloride and is of a lower concentration than some other manufacturers, the same etchant was used by Tyas and Burrow and probably was not a factor. Although Tyas and Burrow

abraded dentinal surfaces, this did not improve adhesion in the previously cited study of One-Step (van Dijken, 2000), so the protocol of restoration without tooth abrasion in this study probably was not detrimental to retention. Both operators who placed the restorations for this study were aware that acetone-based adhesives, such as One-Step, must be applied to moist dentin and used care not to over-dry tooth surfaces.

Although no clinical studies of Tyrian have been published, similar products employing self-etching primers perform better in laboratory and clinical studies than self-etching adhesives and approach the efficacy of total-etch adhesives. Because the pH of this material is about 1, inadequate demineralization of tooth surfaces probably did not contribute to the relatively poor adhesion observed, although retention might have improved had the enamel margin been ground. Since a filled version of One-Step serves as the adhesive for Tyrian, the application of more coats of primer or more separately dried coats of the adhesive might have improved the performance of Tyrian.

It is not possible to determine the primary mechanism by which restorations failed in this clinical study. If failure involved the loss of adhesion to dentin, this could have occurred because the bonding procedure did not produce sufficient infiltration of the adhesives into the dentinal surface, because there was not sufficient adhesion between the resin composite and hybrid layer or because the hybrid layer or bond were not sufficiently stable in the oral environment. The loss of the greatest number of restorations for both adhesives after six months to a year in place indicates that stability of the bond under oral conditions may be an important factor.

The authors believe that the results of this and previous studies indicate that separate manufacturers' instructions for the restoration of erosion/abfraction lesions should be given for all resin-based adhesives. Since laboratory studies do not appear to be adequately predictive, these instructions should be verified via clinical studies.

CONCLUSIONS

Under the protocol used in this study, neither adhesive was effective in retaining unprepared Class V resin composite restorations.

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Laboratory Research

Effect of Desensitizers on the Bond Strength of a Self-etching Adhesive System to Caries-affected Dentin on the Gingival Wall

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Clinical Relevance

The application of Micro Prime and Gluma Desensitizer to caries-affected dentin showed no negative effect on bond strength. Therefore, they can be used as desensitizing and antibacterial agents after caries removal.

SUMMARY

A self-etching dentin adhesive was evaluated for its ability to bond to caries-affected and sound dentin after applying three desensitizers to the gingival walls. Sixty extracted human molars, with approximal dentin caries, were cut horizontally on the long axis of the tooth through caries-affected gingival walls. Carious dentin was removed with SiC paper by means of a caries detector to expose caries-affected dentin. The molars were randomly assigned to four groups: control and three experimental groups—Micro Prime, Glauma Desentizer and Cervitec. Desensitizers were applied to the dentinal surfaces according to manufacturers' instructions. A resin composite was bonded to both the caries-affected and sound dentin of each tooth using a bonding system and plastic rings. The restoration was debonded by shear bond strength. The application of Micro Prime and Gluma Desensitizer to caries-affected dentin did not show any effect on bond strength testing. However, Cervitec caused a decrease in bond strength to caries-affected dentin. The effect of desensitizers on the bond strength of the self-etch bonding agent to caries-affected dentin changed according to the chemical composition of the materials. Desensitizer application on sound dentin is recommended with self-etch bonding systems.

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INTRODUCTION

The clinical consequence of leaving residual bacteria under restorations is still a subject of considerable debate (Yoshiyama & others, 2002). Although it was

assumed that bacteria were dormant in arrested caries under restorations (Björndal & Larsen, 2000), a study by Weerheijm and Groen (1999) demonstrated that amalgam or resin-modified glass-ionomer restorations that were placed over carious dentin still contained viable, cultivable bacteria flora after two years.

Anaerobic and protolytic bacteria may be active in already-established dentin caries. The metabolic products of oral microbiata can also contribute to the advancement of dentin carious lesions (Baca & Liébana, 1995). Besides affecting the evaluation of dental caries, oral bacteria play a role in the problems that may arise after restorative therapy, such as dental sensitivity or recurrent caries (Brännström, 1986).

In-vitro studies (Duran & others, 2003; Settembrini & others, 1997; Chan & Lo, 1994) report the possible residual antibacterial effects of antimicrobial etchants, cavity disinfectants and dentin desensitizers following tooth preparation. Conclusions from these studies revealed that these pre-treatments exhibit varying degrees of antimicrobial inhibition. Any material and/or procedure that infers resistance to intraoral bacterial assault will improve the clinical performance and longevity of adhesive bonding systems and the accompanying composite restorations. While components of these pre-treatment solutions can eliminate caries-producing bacteria, currently, the same components can possibly induce leakage by interfering with the mechanical bond at the tooth/material interface (Owens, Lim & Arheart, 2003).

A recent study confirmed that most recently developed desensitizers have different antibacterial effects (Duran & others, 2003). Specifically, fluoride and/or chlorhexidine components of these materials have been found to reduce or eliminate bacteria in dentinal tubules (Vahdaty, Pitt Ford & Wilson, 1993; Munshi, Reddy & Shetty, 2001). However, the potential difficulty of desensitizing pre-treatment use in cavity preparation with dentin bonding systems could adversely affect hydrophilic adherence of the adhesive components to dentin. However, there is no literature detailing the effect of desensitizers on the bond strength of adhesive systems to caries-affected dentin.

When different desensitizers are used for caries-affected dentin on the gingival wall, the bond strengths of bonding agents may be different in Class II cavities. This study determined whether desensitizer pre-treatment solutions have a detrimental effect on the bonding process of a self-etching dentin adhesive.

METHODS AND MATERIALS

Specimen Preparation

Sixty extracted human molars with approximal dentin caries stored in isotonic saline at +4°C were employed in this study. The teeth had only mesial or distal

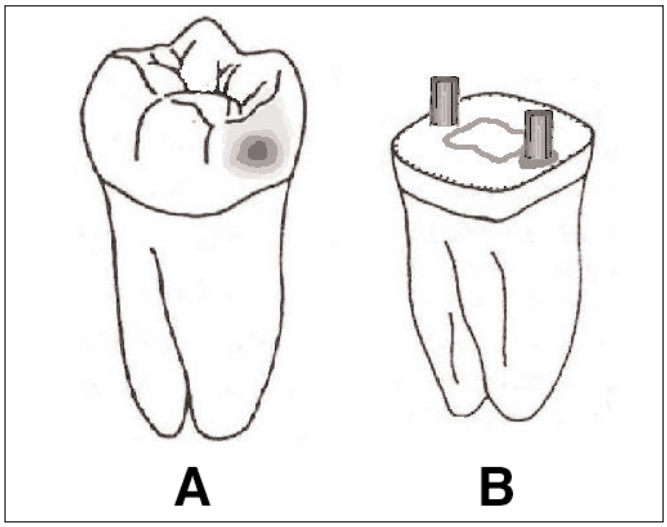

Figure 1: *A: The tooth with approximal caries.*
B: Cylindrical composite attachments at gingival wall of the tooth.

approximal caries, and the gingival wall of caries lesions were 1 mm±D_1 (the D_1 diagnostic threshold encompasses initial, enamel lesions and dentinal caries) above the cemento-enamel junction. Both caries-affected and sound dentin surfaces were obtained on the same tooth (Figure 1A). All teeth were cut perpendicular to the long axis of the tooth from the gingival border of the carious lesion by using an Isomet saw (Buehler Ltd, Lake Bluff, IL, USA) under water to expose a flat dentin surface. Grinding with 320 silicon carbide abrasive papers was performed under running water to remove caries-infected dentin. It was determined that caries-infected dentin was removed by using the combined criteria of visual examination, hardness to a sharp excavator and staining with a caries detector solution (Kuraray Co, Ltd, Osaka, Japan). The relatively soft, dark-red stained dentin was classified as caries-infected dentin, while the discolored, harder dentin that stained pink was classified as caries-affected. Yellow, hard dentin was classified as sound dentin.

The roots of the teeth were mounted in a 3-cm diameter circular mold using chemically cured acrylic resin. The teeth were positioned so that their bonding surface was parallel to the base of the molds.

Shear Bond Strength Analysis

The teeth were randomly divided into four groups, three experimental and one control, of 15 teeth. The flat dentin surfaces were hand polished with 600-grit silicon carbide abrasive papers under running water. The surfaces were examined after polishing to ensure that the orientation was not altered. Three commercially available desensitizers, Micro Prime (Danville Engineering Inc, San Ramon, CA, USA), Cervitec

Table 1: *Contents and Manufacturers' Information of the Materials Used*

	Materials	Contents	Manufacturers
Desensitizers	Micro PrimeTM	Benzethonium chloride and HEMA	Danville Engineering Inc, San Ramon, CA, USA Lot #1424
	Cervitec	Chlorhexidine and thymol	Ivoclar-Vivadent, Konstanz, Germany Lot #C02356
	Gluma Desensitizer	HEMA, glutardialdehyde, and purified water	Heraeus Kulzer, Hanau, Germany Lot #010032
Bonding Agent	Optibond Solo Plus Self-etch system	**Primer:** HEMA, GPDM, MMEP, Ethanol, Water **Adhesive:**Bis-GMA, HEMA, GPDM,silicate glass filler	Kerr, Orange, CA, USA Lot #205187
Com-posite	Point 4	Optimized Particle Composite	Kerr, Orange, CA, USA Lot #202688

Abbreviations: Bis-GMA, bisphenyl-glycidyl-methacrylate; HEMA, 2-hydroxyethyl methacrylate; GPDM, glycerophosphoric acid dimethacylate; MMEP, mono 2-methacryloxy ethyl phthalate

(Ivoclar-Vivadent, Konstanz, Germany) and Gluma Desensitizer (Heraeus Kulzer, Hanau, Germany) (Table 1) were applied to the dentinal surfaces according to the manufacturers' instructions. Next, a self-etch primer (Optibond Solo Plus SE, Kerr Corporation, Orange, CA, USA) was applied to dentin surfaces with a brushing motion for 15 seconds and gently air-dried for three seconds. Then, a bonding system (Optibond Solo Plus SE) was applied to the dentin surfaces with a light brushing motion for 15 seconds and gently air-dried for three seconds. After the bonding system was reapplied to the dentin surfaces for 15 seconds and dried gently with an air syringe for three seconds, the bonding agent was cured for 20 seconds with a curing light (Hilux 250, Benlioglu, Ankara, Turkey). A resin composite (Point 4, Kerr Corporation) was built-up on the caries-affected and sound dentin regions of the same tooth by packing the material into a cylindrically shaped plastic apparatus with an internal diameter of 2.34 mm and a height of 3 mm (Ultradent Products, Inc, South Jordan, UT, USA) (Figure 1B). Excess composite was carefully removed with an explorer from the periphery of the apparatus. The resin composite was light-cured for 40 seconds. The intensity of light was at least 400mW/cm^2. The specimens of the control group were prepared without any application of desensitizers on dentin. The specimens were stored in distilled water at 37°C for 24 hours, then mounted in a universal testing machine (Testometric Micro 500, Lancashire, England) for shear bond testing. A notched blade attached to a compression load cell traveling at a crosshead speed of 1 mm/minute was applied to each specimen at the interface between the tooth and the composite until failure occurred. The maximum load (N) was divided by the cross-sectional area of the bonded composite blocks to determine shear bond strength in MPa.

Fracture Analysis

Fracture analyses were performed at 20x magnification using an optical stereomicroscope (Olympus SZ4045 TRPT, Osaka, Japan). The failure modes were designated as adhesive fracture if 90% to 100% of the bonded interface failed between the dentin and resin composite, cohesive fracture if 90% to 100% of the failure was in the resin composite or dentin or mix fracture if the failures were partially adhesive and partially cohesive resin fractures and/or dentin fracture.

Statistical Analysis

Since the data were homogenous and parametric, one-way ANOVA and Tukey HSD Post Hoc tests were used to test shear bond strength differences between desensitizers for both caries-affected and sound dentin. Paired Samples *t*-test was used to test shear bond strength differences between caries-affected and sound dentin in the same tooth for each adhesive material. The frequency of fracture modes was analyzed using Kruskal-Wallis and Mann-Whitney U tests. The level of statistical significance was set at 0.05 for all statistical analyses.

RESULTS

Table 2 summarizes the results of shear bond testing. The bonding system alone gave the highest bond strength value (29.25±6.40 MPa) on sound dentin. Micro Prime and Gluma Desensitizer application to caries-affected dentin did not show any effect on bond strength when compared to the control group ($p>0.05$). Alternately, Cervitec caused a decrease in bond strength to caries-affected dentin ($p<0.05$). Bonding to sound dentin was negatively influenced by all desensitizers when compared to the control group ($p<0.05$). Although bond strengths to sound dentin in the control and Cervitec groups were higher than those to caries-affected dentin, the application of Micro Prime and Gluma Desensitizer on sound dentin significantly decreased bond strengths.

The fracture patterns of the materials involved are shown in Table 3. In general, a greater percentage of the fractures were adhesive at the tooth-composite junction. That is, the fractures failed in the dentin/composite interface in both caries-affected dentin and sound dentin.

DISCUSSION

Caries-affected dentin may contain deposits of whitlockite in dentinal tubules (Daculsi & others, 1979). Dentinal tubules beneath the dentin surface may be completely obliterated with mineral deposits, confirming the quantitative results reported by Pashley and others (1991) that caries-affected dentin, even after excavation and removal of the smear layer, has very low permeability. The tubules include acid-resistant crystals and extrinsic proteins that have permeated into the mineral phase during cycles of remineralization and demineralization. This tends to result in low bond strengths (Nakajima & others, 1999; Kwong & others, 2000; Sengun & others, 2002). The results of this study are in agreement with previous studies where a higher bond strength to sound dentin was found in the control group compared to that observed in caries-affected dentin.

After removing the infected dentin, caries-affected dentin may have remaining micro-organisms. Cariogenic bacteria under the sealed restoration could multiply through dentinal tubules and cause secondary caries (Yoshiyama & others, 2002).

In some studies, the antibacterial effectiveness of dental materials was found to be related to either the pH or chemical composition of the materials (Imazato & others, 1997; Imazato, Imai & Ebisu, 1998). For example, current desensitizers include antibacterial components such as fluoride, triclosan, benzalkonium chloride, ethylene dianinetetraacetic acid and glutaraldehyde (Duran & others, 2003). Similarly, the materials used in this study have some antibacterial components.

Micro Prime is used as a desensitizing agent under dental cements and temporary, provisional or final restorative materials, as well as on abrasions, cervical erosions and preps. The antibacterial activity of Micro Prime may be related to the chemical composition, benzalkonium chloride. In a previous study, it was found that Micro Prime had a significant inhibitory effect on most oral bacteria (Duran & others, 2003). The antibacterial activity of Gluma desensitizer may be related to its glutaraldehyde composition. Only a few products containing gluataraldehyde exhibited antibacterial effectiveness, but these products were shown to depend upon the leaching of glutaraldehyde from cured materials (Fraga, Siqueira, Jr & de Uzeda, 1996; Meiers & Miller, 1996). Both Micro Prime and Gluma Desensitizer contain HEMA, while the bonding agent primer also contains HEMA. Thus, HEMA produced an acidic effect twice on the caries-affected dentin. This acid, applied to the dentinal surface, may have caused sufficient priming/etching of the surface prior to placement of the bonding agent. This caused higher bond strength values on caries-affected dentin.

Cervitec contains chlorhexidine. Chlorhexidine, delivered in a varnish form, has been shown to be effective in the control and prevention of caries (Bowden, 1990). Chlorhexidine is a most effective antibacterial agent in the reduction of dental plaque and is particularly potent against *S mutans* (Murata, Miyamoto & Ueda, 1990). However, the varnish form of chlorhexidine probably prevented adequate diffusion of the bonding agent and, therefore, resulted in the lowest bond strength to caries-affected dentin.

The principle of dentin bonding relies on the formation of a resin-infiltrated layer in intertubular and peritubular conditioned dentin. Following polymerization, these monomers may form a micro-mechanical bond with primed dentin, forming the hybrid layer that is the principle mechanism of bonding (Perdigão, Swift & Cloe, 1993; Santini & Mitchell, 1998). However, to obtain reliable dentin adhesion, the open tubules and exposed collagen-rich meshwork must be completely

Table 2: *Mean±SD (MPa) Shear Bond Strength Values to Caries-affected and Sound Dentin (n=15)*

Desensitizers	Sound Dentin*	Significance (P)**	Caries-affected Dentin*
Micro Prime	20.44±7.11c	0.001	25.15±5.58a
Gluma Desensitizer	18.51±5.88c	0.001	22.67±5.85a
Cervitec	20.23±6.08c	0.001	16.95±5.78b
Control	29.25±6.40d	0.011	21.98±7.92a

**There is no statistical difference between same letters in same column.*

***The differences between caries-affected and sound according to paired samples* t*-test.*

Table 3: *Failure Modes of the Bonding Agents Used After Shear Bond Testing*

	Sound Dentin				Caries-affected Dentin			
Desensitizers	A (%)	M (%)	C (%)	Grouping	A (%)	M (%)	C (%)	Grouping
Micro Prime	10 (66.6)	4 (26.6)	1 (6.6)	a	8 (53.3)	6 (40)	1 (6.6)	a
Gluma Desensitizer	11 (73.3)	1 (6.6)	3 (20)	a	14 (93.3)	1 (6.6)	0	a, b
Cervitec	12 (80)	0	3 (20)	a	12 (80)	3 (20)	0	a, b
Control	15 (100)	0	0	a	15 (100)	0	0	b

(A: Adhesive, M: Mixed, C: Cohesive).

There is no statistical difference between same letters in same column (p>0.05).

and homogeneously infiltrated by resin monomers. Otherwise, a weak layer, formed by non-encapsulated collagen, remains at the bottom of the hybrid layer, propagating fracture. This reduces the bond strength and durability of adhesion (Santini & Mitchell, 1998; Titley & others, 1994; Gwinnett, 1993).

Many factors contribute to the reduction of dentin bond strength through incomplete resin infiltration. Among them, dentin permeability is one of the most important (Tagami, Tao & Pashley, 1990; Hamid, Sutton & Hume, 1996). Several studies observed that desensitizing agents effectively reduced dentin permeability between 60% and 80% (Jain & others, 1997; Camps & others, 1998). In a study by Pashley, Tao and Pashley (1993), the effect of a dentin desensitizer on the bond strength on three different dentin bonding systems was evaluated. In another study by Seara and others (2002), a dentin desensitizer was used to evaluate the effect of bond strength of two different dentin bonding systems. Seara and others (2002) reported that dentin desensitizers significantly decreased the bond strength of dentin bonding agents. These observations confirm the results obtained in this study. The application of desensitizers before the bonding system significantly reduced shear bond strengths to sound dentin. Effective bonding requires acid etching, rinsing and drying of dentin before applying the primer. Self-etching primers succeeded in eliminating the dentin-conditioning steps before priming. However, self-etching primers have low acidic components. One possible reason for the decreased bond strengths to sound dentin may be the result of desensitizer deposition blocking the dentin tubule orifices and intertubular diffusion channels. The precipitation of desensitizers on the dentin surface may chemically and/or physically prevent complete penetration of the bonding system resin components through the demineralized zone, thus decreasing bond strength values.

Most published reports on the performance of bonding systems have used sound dentin on the pulp chamber as a substrate, even though caries-affected dentin on the gingival wall is often the clinically relevant bonding substrate. Dentin tubule orientation is different between the pulpal and gingival walls of Class II cavities. When different adhesive systems were used on caries-affected dentin on the gingival wall, bond strengths may change according to the bonding mechanism and chemical composition of the adhesives. In this study, the application of Cervitec, a chlorhexidine and thymol-containing varnish to caries-affected dentin decreased bond strengths compared to sound dentin. However, Micro Prime and Gluma Desensitizer increased bond strengths to caries-affected dentin.

CONCLUSIONS

Based on the results obtained in this study, the authors feel that desensitizers containing the acidic ingredient HEMA can be used safely on caries-affected dentin. However, care should be taken with chlorhexidine containing varnishes.

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Color and Translucency of A2 Shade Resin Composites After Curing, Polishing and Thermocycling

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Clinical Relevance

Color changes from pre-cure to post-cure shades were perceptible in all composites studied (ΔE^*_{ab}>3.8), and polishing caused perceptible color changes in some composites (ΔE^*_{ab}=1.9-4.5). This study supports the clinical practice of curing a small amount of composites in the tooth to select the desired shade before esthetic restorative procedures.

SUMMARY

This study determined the differences in values and changes of color and translucency of eight brands of A2 shade resin composites after curing, polishing and thermocycling (TC). The color of specimens 10-mm in diameter and 2-mm thick was measured on a reflection spectrophotometer with SCE geometry under D65 illumination over a white and black background. Measurements were obtained before curing, after curing, after polishing and after TC. The color change (ΔE^*_{ab}), translucency parameter (TP) and contrast ratio (CR) were then compared. The range of ΔE^*_{ab} after curing was 3.8 to 10.2 (average ΔE^*_{ab} for the eight composites = 6.4), which was deemed perceptible to the observer. Polishing caused ΔE^*_{ab} of 1.9 to 4.5, which was perceptible in five of the eight composites. After 2,000 TC, ΔE^*_{ab} was 0.4 to 1.3. TP values tended to increase after curing and decrease after TC (range before curing was 7.1 to 17.2). Changes in TP values after curing were statistically significant in all composites (p<0.05). Changes in CR values were similar to the translucency changes in TP. Though the composite shades were all designated as A2, color coordinates, TP and CR values, changes in color and translucency after curing, polishing and thermocycling varied by brand.

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INTRODUCTION

With increasing patient demand for an esthetic dentition, resin composite restorations enjoy great popularity due to their excellent esthetics, acceptable longevity and relatively low cost. However, the relatively significant

color changes of resin composites after curing and aging indicate that these changes must be taken into account during initial clinical shade match at placement of the uncured composite (Johnston & Reisbick, 1997).

Several studies on the color changes of resin composites after curing and aging have reported significant color changes, and the magnitude of color change varied by the characteristics of the composites (Eldiwany, Friedl & Powers, 1995; Lee & Powers, 2001). Also, the color change of resin composites after curing showed characteristic chromatic shifts (Seghi, Gritz & Kim, 1990). Since human enamel has inherent translucency, esthetic restorations should reproduce the translucency of natural teeth (Paul & others, 1996; Arikawa & others, 1998). Relatively high variability in translucency changes after curing and aging has been shown among resin composites, with some products increasing and others decreasing (Johnston & Reisbick, 1997). The significant changes in color and translucency that occur when resin composites are cured, stored or processed under various conditions indicate that such changes should be taken into account when these materials are used.

The optical properties of dental resin composites are also influenced by surface changes during the restorative procedures of finishing and polishing (Chung, 1994). Colors change with the varied surface conditions of materials and this difference was perceptible in some of the tested materials (Lee, Lim & Kim, 2002; Kim & others, 2003).

There also may be differences among composites with the same shade designation in the changes of optical properties after curing, polishing and aging. This study evaluated the values and changes of color and translucency after curing, polishing and thermocycling of eight resin composites.

METHODS AND MATERIALS

Eight light-curing resin composites designated as A2 body shade were studied (Table 1).

After applying a drop of optical fluid (refractive index = 1.5) on a white or black background and laying a cover glass on the background, the resin composites were packed into a polytetrafluoroethylene mold (10 mm in diameter and 2-mm thick) on a cover glass. Optical fluid was placed between the cover glass and background so that an optical connection was achieved (Eldiwany & others, 1995). After packing the composite, another cover glass was pressed on top of the specimen and pressed with a 5-kg load for three minutes to produce a uniform thickness. Color and spectral reflectance measurements before curing were obtained with the two cover glasses attached. Five specimens were employed for each material.

The specimens were light cured for 40 seconds in three overlapping areas with a light-curing unit (Spectrum 800, Dentsply/Caulk, Milford, DE, USA) with an intensity setting of 400 mW/cm^2. The output of the curing light was checked with a radiometer (SDS/Kerr, Orange, CA, USA). "After curing" color measurements were made after storage in distilled water for 24 hours and blot drying. The specimens were then polished with 1,500-grit wet SiC paper on both sides. The final specimen thickness was 1.97 ± 0.10 mm. The color after polishing was measured after immersion in distilled water for 24 hours and blot

Table 1: *Materials Used in This Study*

Code	Brand Name	Composition	Batch #	Manufacturer
CHR	Charisma	BisGMA matrix, 65 vol% (76 wt%) barium aluminum fluoride glass filler of 0.02-2 μm, 5 vol% pyrogenic silicon dioxide filler of 0.02-0.07 μm	070066	Heraeus Kulzer, Wehrheim, Germany
CLF	Clearfil AP-X	BisGMA and TEGDMA matrix, barium glass and colloidal silica filler	759AA	Kuraray, Okayama, Japan
ESX	Esthet X	Micro matrix restorative, 60 vol% filler of 0.6-0.8 μm	0302132	Dentsply/Caulk, Milford, DE, USA
FSP	Filtek Supreme	58-60 vol% (78.5 wt%) combination of aggregated zirconia/silica cluster filler with primary particle size of 5-20 nm, and a non-agglomerated/non-aggregated 20 nm silica filler	3BK	3M ESPE, St Paul, MN, USA
PAE	Palfique Estelite	71 vol% (82 wt%) submicron zirconia spherical filler of 0.2 μm and composite filler	YE616233	Tokuyama Dental Corp, Tokyo, Japan
PO4	Point 4	57 vol% (75 wt%) inorganic filler of 0.04 μm	203498	SDS/Kerr, Orange, CA, USA
TEC	Tetric Ceram	60 vol% (79 wt%) filler of 0.04-3.0 μm (average 0.7 μm)	F56709	Ivoclar Vivadent, Liechtenstein
TPH	TPH Spectrum	57 vol% (77 wt %) bariumaluminoborosilicate glass and highly dispersed silicon dioxide of submicron filler (0.8 μm)	0301281	Dentsply/Caulk, Milford, DE, USA

drying. Thermocycling (TC) was then performed for 2,000 cycles between 5°C and 55°C in distilled water and the color was measured after blot drying.

The color was measured according to the CIELAB color scale relative to the standard illuminant D65 against a white background (*CIE L**=96.68, *a**=-0.18, and *b**=-0.22) and a black background (*CIE L**=1.15, *a**=-0.11, and *b**=-0.50) on a reflection spectrophotometer (CM-3500d, Minolta, Osaka, Japan) with specular component excluded (SCE) geometry. The illuminating and viewing configuration was CIE diffuse/10˚ geometry.

Color change (ΔE^*_{ab}) was calculated by the following equation, with the color coordinates over the white background; $\Delta E^*_{ab}=[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. The translucency parameter (TP) was obtained by calculating the color difference between the specimen over the white background and the specimen over the black background; TP= $[(L_B^*-L_W^*)^2 + (a_b^*-a_W^*)^2 + (b_B^*-b_W^*)^2]^{1/2}$, where subscript B refers to the color parameters over the black background and subscript W refers to those over the white background (Johnston, Ma & Kienle, 1995). The contrast ratio (CR) was calculated from the luminous reflectance (Y) of the specimens with a black (Yb) and white (Yw) backing to give Yb/Yw (0.0 = transparent, 1.0 = opaque) (Craig & Powers, 2001).

Differences in the values and changes in color, TP and CR by the composites and specimen conditions were analyzed by analysis of variance and Scheffe's multiple range tests (SPSS 11.0, SPSS, Chicago, IL, USA, p=0.05). Multiple regression analysis was used to determine the correlation among the color changes and *CIE L**, *a**, *b**, TP and CR values of specimens. Also, multiple regression analysis was used to determine the correlation among the color changes and changes in *CIE L**, *a**, *b**, TP and CR values. Regression analysis with forward data input was selected (p=0.01). When the tolerance between independent variables was lower than 0.3, the variable with the lower β value (= standardized correlation coefficient of each variable) was excluded from the regression (Norman & Streiner, 1994).

RESULTS

*CIE L** values before curing ranged from 69.2 to 77.5, and ranged from 65.6 to 73.7 after curing. The range of changes in *CIE L** value was from –4.2 to 1.8 after curing, from 1.3 to 4.4 after polishing and from –0.9 to –0.2 after TC. *CIE a** values before curing ranged from 3.3 to 5.8 and from 2.2 to 6.1 after curing. The range of changes in *CIE a** value was from –0.7 to 0.6 after curing, from –0.5 to 0.5 after polishing and from –0.4 to 0.5 after TC. *CIE b** values before curing ranged from 21.0 to 32.3 and ranged from 15.8 to 25.8 after curing. The range of changes in *CIE b** value ranged from –9.2 to –0.9 after curing, from –1.8 to 0.5 after polishing and from –1.0 to 0.3 after TC. Figure 1 presents the vectorial changes of *CIE a** and *b** values from before curing to after curing, and those before and after polishing are presented in Figure 2. Arrow tails indicate before curing or polishing values and arrow heads indicate after curing or polishing values. After curing, changes in *CIE a** varied by composite, however, *CIE b** decreased in all composites tested. After polishing, *CIE a** and *b** increased or decreased by composites. The magnitude of changes were higher after curing than after polishing.

ΔE^*_{ab} values after curing, polishing and TC are listed in Table 2. Changes in color varied by composite (p<0.05). Average ΔE^*_{ab} of the eight composites after curing was 6.4, and this value was significantly higher than those after polishing (3.3) or after TC (0.8). The range of ΔE^*_{ab} was 3.8 to 10.2 after curing, 1.9 to 4.5 after polishing and 0.4 to 1.3 after TC.

TP values before curing, after curing, after polishing and after TC are listed in Table 3. TP values varied by composite (p<0.05). The average TP value of eight composites before curing was 11.1, and this value was statistically significantly lower than those after curing,

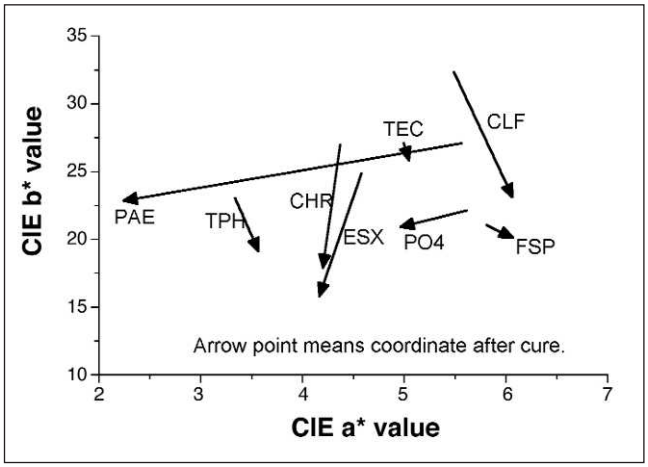

Figure 1. *Changes in color coordinates after curing.*

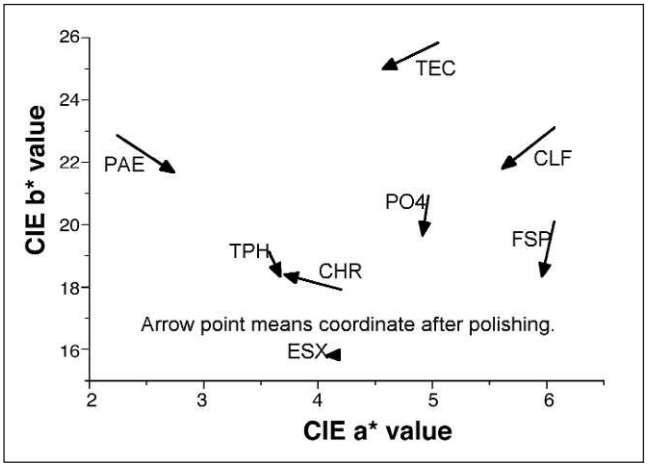

Figure 2. *Changes in color coordinates after polishing.*

after polishing and after TC (13.9, 14.5 and 14.4, respectively). The range of TP values before curing was 7.1 to 17.2, 11.5 to 15.8 after curing, 11.4 to 17.6 after polishing and 11.2 to 17.4 after TC. TP increased after curing except for ESX and PAE. Changes in TP after polishing varied by composite. TP generally decreased after TC.

CR values are listed in Table 4. CR values varied by composite ($p<0.05$). Changes in CR values by composite and specimen conditions were similar to the changes in translucency measured in TP. The correlation coefficient between TP and CR was -0.88 before curing, -0.79 after curing, -0.85 after polishing and -0.85 after TC.

The results of multiple regression analyses among ΔE^*_{ab} values and three-color coordinates (*CIE* L^*, a^* and b^*), TP and CR of before-and-after each corresponding condition (10 independent variables) are listed in Table 5. CR, *CIE* a^* and *CIE* b^* were significant predictors depending on the condition ($p<0.01$). Color change after curing was highly correlated with CR values.

The results of multiple regression analyses among ΔE^*_{ab} values and changes in three-color coordinates, TP and CR after each corresponding condition (five independent variables) are listed in Table 6. Δb^*, ΔL^*, ΔCR and Δa^* were significant predictors depending on the condition ($p<0.01$). And the multiple correlation coefficients (r) were as high as 0.94-0.99.

DISCUSSION

In this study, the color changes after curing varied among composites ($p<0.05$). FSP and PO4 showed relatively small color changes ($\Delta E^*_{ab}<4.0$); however, CHR, CLF and ESX showed greater color changes ($\Delta E^*_{ab}>9.1$). As the color change after curing was perceptible ($\Delta E^*_{ab}>3.8$) for all composites studied, the clinical practice of curing some composite on the tooth to select shades of composites before restorative procedures is prudent.

In previous studies, measurement with SCE geometry has resulted in accurate color determination (Lee & others, 2002). Therefore, SCE geometry was employed in this study considering the fact that the surface could be changed after polishing and TC. In this study, the range of three color coordinates was significant, though the shade designation was A2. The full range of *CIE* L^* value was about eight units, that of *CIE* a^* value was about 3 to 4 units and that of *CIE* b^* value was about 9 to 11 units (Figures 1 and 2).

Regression analysis was performed to determine which color parameter significantly influenced the color change (Table 5). The results after curing indicated that CR before curing (standardized correlation coefficient; β=-1.04) and CR after curing (β=0.60) significantly influenced curing color change. Thus, when translucency before curing was high (low CR value), the color change during curing procedure was high. Low translucency after curing (high CR value) was correlated with

Table 2: *Color Change (ΔE^*_{ab}) After Curing, Polishing and Thermocycling (n=5)*

Code	After Cure[a] (Gr 1)	After Polish[b] (Gr 2)	After TC[c] (Gr 3)	DG[d]
CHR	9.2 (0.2)[e4]	4.5 (1.0)[4]	0.9 (0.2)[3]	3<2<1
CLF	10.2 (0.3)[5]	3.0 (0.1)[23]	1.3 (0.1)[4]	3<2<1
ESX	9.1 (0.2)[4]	3.4 (0.4)[23]	0.9 (0.1)[23]	3<2<1
FSP	3.8 (0.4)[1]	3.5 (0.3)[23]	0.4 (0.1)[1]	3<2,1
PAE	5.7 (0.3)[3]	1.9 (0.3)[1]	0.6 (0.2)[12]	3<2<1
PO4	4.0 (0.2)[12]	3.6 (0.3)[34]	0.7 (0.1)[23]	3<2<1
TEC	4.4 (0.2)[2]	3.8 (0.7)[34]	0.4 (0.2)[1]	3<2<1
TPH	6.0 (0.2)[3]	2.6 (0.5)[12]	1.0 (0.2)[34]	3<2<1
AVG	6.4 (0.4)	3.3 (0.9)	0.8 (0.3)	3<2<1

[a]: *Between uncured and cured specimens. After curing, Mylar strip was removed and specimen immersed in DW for 24 hours and blot dried before measurement.*

[b]: *Between cured and polished specimens. Both specimens were in wet condition.*

[c]: *Between polished and thermocycled specimens. Both specimens were in wet condition.*

[d]: *DG = Different groups by the specimen condition. "<" means significantly different group marker (Scheffe test, p<0.05).*

[e]: *Standard deviations are in parentheses.*

Same superscript number means not-significantly different group.

Table 3: *Translucency Parameter (TP) After Curing, Polishing and Thermocycling (n=5)*

Code	Before Cure	After Cure (Gr 1)	After Polish (Gr 2)	After TC (Gr 3)	DG[a] (Gr 4)
CHR	11.2 (0.1)[b4]	15.8 (0.1)[5]	17.6 (0.8)[5]	17.4 (0.9)[5]	1<2<4,3
CLF	10.7 (0.3)[34]	14.7 (0.4)[4]	14.1 (0.2)[23]	13.5 (0.1)[23]	1<4<3<2
ESX	13.8 (0.3)[5]	12.5 (0.2)[2]	13.5 (0.6)[2]	13.3 (0.6)[2]	2<4.3,1
FSP	8.8 (0.5)[2]	11.5 (0.4)[1]	11.4 (0.4)[1]	11.2 (0.6)[1]	1<4.3,2
PAE	17.2 (0.7)[6]	15.1 (0.3)[4]	15.5 (0.6)[45]	15.5 (0.6)[4]	2.3.4<1
PO4	8.3 (0.4)[2]	13.4 (0.3)[3]	14.7 (0.3)[34]	14.5 (0.5)[34]	1<2<4,3
TEC	10.1 (0.4)[3]	15.3 (0.6)[45]	16.2 (0.9)[5]	16.9 (0.8)[5]	1<2<3,4
TPH	7.1 (0.1)[1]	13.0 (0.1)[23]	13.2 (0.3)[2]	12.9 (0.4)[2]	1<4.2,3
AVG	11.1 (3.1)	13.9 (1.5)	14.5 (1.9)	14.4 (2.1)	1<2.4,3

[a]: *DG = Different groups by the specimen condition. "<" means significantly different group marker (Scheffe test, p<0.05).*

[b]: *Standard deviations are in parentheses.*

Same superscript number means not-significantly different group in the same column.

limited color change during the curing procedure. Color changes after the curing and aging of resin composites of conventional and bleached shades were determined (Lee & Powers, 2001). As a result, higher *CIE a** and *b** values had a negative influence on the color change for both curing and aging. In this study, the results of similar regression analyses among ΔE^*_{ab} values after curing and color parameters indicated that CR values had significant influence on curing color change. This discrepancy in results reflects the fact that the difference in color coordinates of this study was relatively small compared to the previous study, because the color designation was the same as A2 and the change in translucency after curing was prominent.

Table 4: *Contrast Ratio (CR) After Curing, Polishing and Thermocycling (n=5)*

Code	Before Cure (Gr 1)	After Cure (Gr 2)	After Polish (Gr 3)	After TC (Gr 4)	DG[a]
CHR	0.82 (0.10)[2]	0.74 (0.14)[2]	0.72 (0.15)[1]	0.72 (0.15)[1]	4.3<2<1
CLF	0.85 (0.10)[23]	0.76 (0.13)[3]	0.77 (0.12)[34]	0.78 (0.12)[3]	2<3,4<1
ESX	0.78 (0.12)[1]	0.80 (0.13)[5]	0.79 (0.13)[4]	0.79 (0.13)[3]	1.4<2
FSP	0.85 (0.09)[23]	0.79 (0.11)[4]	0.79 (0.11)[4]	0.79 (0.11)[3]	2.3,4<1
PAE	0.76 (0.16)[1]	0.73 (0.12)[1]	0.72 (0.12)[1]	0.72 (0.12)[1]	3.4,2<1
PO4	0.89 (0.09)[4]	0.80 (0.13)[45]	0.78 (0.13)[34]	0.78 (0.13)[3]	4.3<2<1
TEC	0.87 (0.10)[34]	0.76 (0.11)[3]	0.75 (0.13)[2]	0.74 (0.14)[12]	4.3,2<1
TPH	0.87 (0.09)[34]	0.76 (0.11)[3]	0.76 (0.11)[23]	0.76 (0.11)[23]	3.4,2<1
AVG	0.84 (0.05)	0.77 (0.03)	0.76 (0.03)	0.76 (0.03)	4.3,2<1

[a]: *DG = Different groups by the specimen condition. "<" means significantly different group marker (Scheffe test, p<0.05).*

[b]: *Standard deviations are in parentheses.*

Same superscript number means not-significantly different group.

Table 5: *Influencing Color Parameters on the Color Change*

Color Change	Influencing Parameters[a] in Orders
ΔE^*_{ab} after curing	CR before curing (β^b=-1.04), CR after curing (β^b=0.60) r^c=1.00
ΔE^*_{ab} after polishing	*CIE a** (β^b=0.43) after curing r^c=0.43
ΔE^*_{ab} after thermocycling	*CIE b** (β^b=0.318) after thermocycling r^c=0.32

[a]: *Results from multiple regression among the color change and three color coordinates (*CIE L**, a* and b*), TP value and CR value of before- and after- each corresponding condition (10 independent variables).*

[b]: *Standardized correlation coefficient.*

[c]: *Multiple correlation coefficient.*

Table 6: *Influencing "Change of Color Parameters" on the Color Change*

Color change	Influencing Changes of Parameters[a] in Orders
ΔE^*_{ab} after curing	Δb^* after curing (β^b=-1.04), ΔL^* after curing (β^b=-0.15) r^c=0.99
ΔE^*_{ab} after polishing	ΔL^* after polishing (β^b=1.05), Δb^* (β^b=-0.11) after polishing r^c=0.98
ΔE^*_{ab} after thermocycling	ΔL^* after TC (β^b=-0.72), Δb^* (β^b=-0.73) after TC ΔCR after TC (β^b=0.23), Δa^* after TC (β^b=-0.15) r^c=0.94

[a]: *Results from multiple regression among the color change and changes in three-color coordinates, TP value and CR value of each corresponding condition (5 independent variables).*

[b]: *Standardized correlation coefficient.*

[c]: *Multiple correlation coefficient.*

Regression analysis was also performed to determine the degree of change to which color parameter influenced color change (Table 6). The results after curing indicated that Δb^* (β=-1.04) and ΔL^* (β=-0.15) significantly influenced color change. This result was in part coincidental with a previous study (Yap, Sim & Loganathan, 1999) where changes in *CIE L** value were significant after curing. In general, light-cured composites produce a characteristic chromatic shift toward the blue region of color space, which result in a perceived decrease in yellow chroma (Seghi & others, 1990). In this study, the range of changes in *CIE b** value after curing was –9.2 to –0.9, and six of nine composites showed changes of more than 3.3 units that could make color difference higher than the perceptible limit of 3.3 in ΔE^*_{ab} unit (Ruyter, Nilner & Moller, 1987).

Color and TP changes after the curing of resin composites have been evaluated. There was relatively high variability in both lightness and TP changes, with some products increasing and others decreasing (Johnston & Reisbick, 1997). In this study, changes in *CIE L** and *CIE b** values after curing ranged from –4.2 to 1.8 and –9.2 to –0.9, respectively. Lightness decreased in most materials and hue shifted due to the blue direction (higher *CIE b** value after curing). TP values increased after curing except for ESX and PAE (p<0.05). TP values increased or decreased after polishing and decreased after TC. Changes in translucency after long-term exposure to the oral cavity might contribute to shade mismatches. A greater TP value is indicative of greater translucency or less opacity. The TP is specific for a given thickness of material and for the backing used. The clinical meaning of TP would vary, depending on the application of the material. Currently, a TP functions only to specify the translucency of the material, and further research is indicated to determine the effects of changes in the TP on any changes in appear-

ance (Johnston & Reisbick, 1997). Nevertheless, six of the eight composites in this study showed a significant increase in TP after curing. Therefore, the change in translucency after curing was significant.

The change in color and TP values after the curing of composites in previous studies has been reported. ΔE^*_{ab} after curing varied from 3.7 to 12.0; whereas, the TP values of cured resin composites varied from 2.0 to 7.1 (Paravina, Ontiveros & Powers, 2002). In this study, although the filler type of seven composites was hybrid and only one (FSP) was nanofilled, the changes in color and TP value after curing were significant for all the materials. Curing-dependent color and TP changes indicate that dentists should use cured composite for matching shade and translucency.

The color stability of composites under various physico-chemical conditions improved when materials showed low water sorption, high filler-resin ratio, reduced particle size and hardness and an optimal filler-matrix coupling system. Optimal coupling meant direct grafting of the resin to the filler, thus providing the composite with lower water sorption rates (Dietschi & others, 1994). In this study, the color changes after TC were small, so although these values were statistically significantly different among the composites tested, none would reveal a clinically discernible color shift. These results may reflect differences in the properties of composites suggested in the Dietschi and others (1994) study.

Translucency of all-ceramic systems was measured by CR value (Heffernan & others, 2002a,b). In this study, CR values were 0.76 to 0.89 before curing, 0.73 to 0.80 after curing, 0.72 to 0.79 after polishing and 0.72 to 0.79 after TC, with a final specimen thickness of 1.97 mm. Translucency varied even though shade designation and thickness were the same. After polishing, CR values decreased for CHR and PO4 and increased for CLF ($p<0.05$). Therefore, the influence of polishing on the translucency of resin composites varied among the composites tested. Such variability could potentially influence their ability to match natural teeth.

Changes in the translucency of resin composites have been examined after immersion in 60°C water. Some composites demonstrated a significant decrease in translucency (6% to 7%) after water immersion (Nakamura & others, 2002). In this study, TP values changed slightly after TC, but in general, the values before and after TC were not different ($p>0.05$). This discrepancy may be the result of differences in the aging method. Opacity decrease has been reported to be a factor in discoloration for some tooth-colored restorative materials and might be caused by a refractive index change in the matrix phase of the materials (Inokoshi & others, 1996). In this study, the results of multiple regression analysis among ΔE^*_{ab} values after TC and changes in color parameters indicated that changes in CR significantly influenced color change ($\beta=0.23$, Table 6). Therefore, it was reconfirmed that changes in translucency significantly influenced the color change after TC, although other variables also influenced color change. Considering the fact that the infinite optical thickness at which the reflectance of a material with an ideal black background would attain 99.9% of its light reflectivity of resin composites ranged from 3.0 to 5.9 mm (Powers, Yeh & Miyagawa, 1983), changes in translucency could be a major contributing factor for color mismatch intraorally after long-term service.

CONCLUSIONS

Although shade designation of the composites used in this study was the same as A2, color coordinates (*CIE* L^*, a^* and b^*), TP and CR values and changes in color and translucency after curing, polishing and thermocycling varied significantly by brand of composite. Color changes after curing were perceptible in all composites ($\Delta E^*_{ab}>3.8$), and polishing caused perceptible color changes in five of the eight materials ($\Delta E^*_{ab}=1.9$-4.5). However, color change after thermocycling was negligible ($\Delta E^*_{ab}=0.4$-1.3).

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Degree of Remaining C=C Bonds, Polymerization Shrinkage and Stresses of Dual-cured Core Build-up Resin Composites

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Clinical Relevance

The polymerization behavior of dual-cured core build-up resin composites is strongly material-related. None of the materials can be efficiently polymerized in terms of the remaining C=C bonds in regions of restorations with no direct light accessibility.

SUMMARY

This study measured the degree of remaining C=C bonds (RDB), linear polymerization shrinkage (LPS) and polymerization stresses (PS) of dual-cured resin composite build-up materials using a variety of light exposure scenarios.

Four commercially available materials were used: Bis-Core, FluoroCore, Build-it! and Permalute. The RDB was measured using FTIR spectroscopy, and custom-made devices were used to measure LPS and PS values. Data were obtained using three different modes of photo-activation: NLC (No Light-Curing); ILC (Immediate Light-Curing, where 60 second light-curing was applied at the start of the observation period); and DLC (Delayed Light-Curing, where 60-second light-curing was applied 10 minutes from the start of the observation period). Statistical evaluation of the data at the end of the 13-minute observation period was performed with two-way analysis of variance (ANOVA), Tukey's Studentized Range (HSD) Test (p=0.05) and simple linear regression. Differences in the development of LPS and PS during the 13 minutes were studied using mathematical calculus.

Bis-Core presented the highest RDB and Permalute the lowest when ILC was applied, while no differences were found between Build-it! and FluoroCore and NLC and DLC increased RDB for FluoroCore and Permalute compared to ILC; whereas, no differences were noted for Build-it! and Bis-Core. Using DLC, a decrease in RDB was found only for Build-it!

Permalute and Bis-Core presented the highest and lowest LPS and PS after ILC, accordingly. Higher LPS and PS were recorded for Build-it! compared to Fluorocore in the ILC group.

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FluoroCore and Permalute exhibited a reduction in LPS and PS using NLC relative to ILC. No differences in LPS and PS values were detected for the materials Bis-Core and Build-it! when subjected to NLC or DLC, compared to ILC.

Simple linear regression showed that only the two polymerization shrinkage properties studied were highly correlated (LPS-PS r^2=0.85). The RDB rate was not correlated with either polymerization shrinkage properties (RDB-LPS r^2=0.40; RDB-PS r^2=0.57). A study of the evolution of the real-time curves of percentage values of LPS and PS showed that these properties evolved in a similar exponential mode and that, most often, there was a delay in the development of PS.

INTRODUCTION

Foundation restorations are often required in order to build damaged teeth to ideal anatomic form prior to their preparation. Despite amalgam, resin composites from the early days of chemically cured materials have also been used for foundation restorations, even though poor adhesion to dentin was provided with previous generations of bonding systems (Bowen, 1985; Munksgaard & Asmussen, 1984; Asmussen & Munksgaard, 1983). Lately, light-cured resin composites, in combination with improved bonding agents, were recommended for core build-ups. These materials offer ample working time and present mechanical properties comparable to amalgam (Bonilla, Mardirossian & Caputo, 2000). Light-cured core build-up materials are more convenient to use than amalgam, when mechanical retention is difficult to achieve and when easy placement is required without the use of elaborate matrices. Additionally, these materials expedite the clinical process, as the restored tooth may be prepared in the same appointment. Thus, light-cured resin composites are often the material of choice.

Nevertheless, there are some concerns associated with resin composites: polymerization shrinkage (Stavridakis, Kakaboura & Krejci, 2000; Stavridakis & others, 2003), delayed expansion due to water sorption (Pearson, 1979; Chai & others, 2004) and the high coefficient of thermal expansion (Asmussen & Jorgensen, 1978). As a result, poor adaptation of the material to preparation is common (Dietschi & others, 2002). Many strategies have been used to overcome limitations caused by polymerization shrinkage. Various layering techniques are suggested in direct resin composite restorations. Nevertheless, time limitations, when placing core build-ups, do not usually allow the clinician to use elaborate multi-layering techniques, as the ideal restoration is not the final goal. The use of photo-activated materials often poses the problem wherein it is difficult for light to reach the deepest parts of a restoration in order to provide maximal resin composite polymerization.

Dual-cured core build-up resin materials have been designed to allow the clinician to build extended foundation restorations quickly, in bulk, as the chemical mode of the polymerization process can initiate resin polymerization in deep resin layers. However, the incorporation of chemical and light curing modes in the same material has not been shown to ensure maximal curing of the material, as the different curing modes may counteract one another. Additionally, polymerization shrinkage of dual-cured resin composite build-up materials has not been assessed.

Thus, this study investigated the polymerization reaction of dual-cured resin composite build-up materials in terms of the degree of carbon-carbon double bond (C=C) remaining, linear polymerization shrinkage (LPS) and polymerization stresses (PS) developed under various light exposure scenarios.

METHODS AND MATERIALS

The dual-cured resin composite core build-up materials tested are listed in Table 1. Three different experimental groups for each material were prepared, corresponding to three different light-curing modes: no light-curing (NLC), the materials were not light cured and kept under dark conditions; immediate light curing (ILC), immediate light exposure of the materials for 60 seconds (Translux EC, Kulzer & Co GmbH, Wehrheim, Germany) with a relative power density of 500 mW/cm^2 (Curing Radiometer, Demetron Research, Danbury, CT, USA) and delayed light curing (DLC), specimens were dark-stored for 10 minutes, then light cured for 60 seconds, as described above. Within each experimental grouping, three different parameters were measured: remaining C=C bonds (RDB), linear polymerization shrinkage (LPS) and polymerization stresses (PS).

For evaluation of RDB, five rectangular specimens (3x2x0.5 mm) per polymerization mode were prepared between two transparent glass slides covered with transparent polystyrene strips. An additional group sample per material, consisting of unset pastes, was performed for the RDB measurements. Spectra of the unset pastes and the directly irradiated surfaces were

Table 1: *Dual-cured Resin Composite Core Build-up Materials Used*

Material	Manufacturer	Color	Batch #
Bis-Core	BISCO, Inc, Schaumburg, IL, USA	Natural	109268
FluoroCore	Caulk/Dentsply, Milford, DE, USA	Tooth shade	990211
Build-it!	Jeneric/Pentron Inc, Wallingford, CT, USA	A2	17564
Permalute	Ultradent Products, Inc, South Jordan, UT, USA	A2	34F8

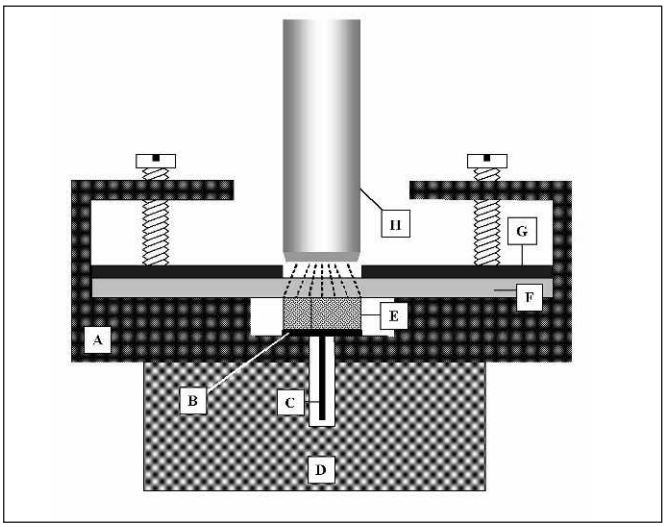

Figure 1: *Schematic presentation of the measuring device for linear polymerization shrinkage: (A) metal frame; (B) aluminum platelet; (C) diaphragm (8 x 8 mm); (D) infrared measuring sensor; (E) specimen; (F) glass plate (41 x 18 x 1 mm); (G) aluminum platen (13 x 20 mm); (H) light-curing tip.*

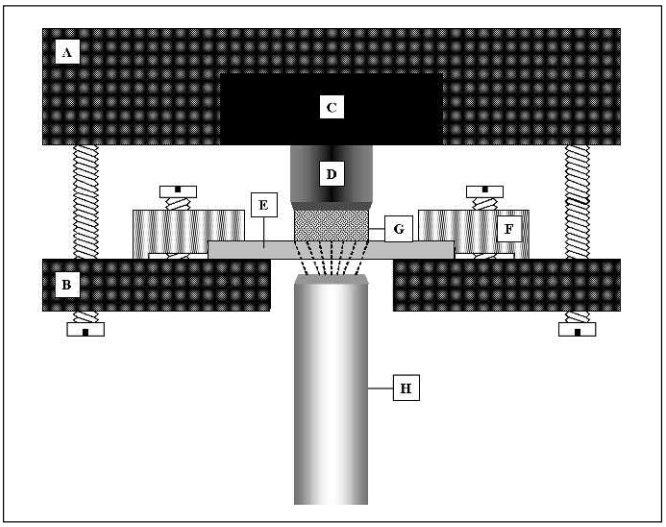

Figure 2: *Schematic presentation of the measuring device for polymerization stresses: (A) upper part of measuring device; (B) lower part of measuring device; (C) load cell; (D) metal cylinder; (E) glass plate; (F) holder of glass plate; (G) specimen; (H) light-curing tip.*

acquired employing a micro-MIR cell attached on a Fourier Transformation Infrared Spectroscopy (FTIR) spectrometer (Micro-MIR accessory and Spectrum GX FTIR spectrometer, Perkin-Elmer, Norwalk, CT, USA) under the following conditions: 4000-400 cm^{-1} range, 4 cm^{-1} resolution, 45° para edge KRS-5 mini-crystal of seven internal reflections and 40 co-added scans co-additions at 30 ± 1°C, which were kept constant with a temperature controller. The quantitative measurement of the % RDB was performed based on the two-frequency technique. The stretching vibrations of the methacrylate C=C bonds (1638 cm^{-1}) were used as an analytical frequency; whereas, the stretching vibrations of the aromatic bonds (1605 cm^{-1}) were used as a reference frequency for Bis-Core, Build-it! and Permalute, and the ester bonds (1712 cm^{-1}) for FluoroCore (Ruyter & Gyorosi, 1976; Rueggeberg, Hashinger & Fairhurst, 1990). The net peak absorbance areas of these peaks were used to quantify the extent of C=C remaining on the directly irradiated resin composite surfaces.

The measurement of LPS was performed with a custom-made measuring device (Figure 1) based on principles suggested by de Gee, Feilzer and Davidson (1993). A standardized amount of each dual-cured material was thoroughly mixed for 30 seconds, then placed on the aluminum platelet. The material was carefully flattened by means of a glass plate to a test height of 1.5 mm. Light-curing was performed from a distance of 1 mm through the glass plate. The vertical movement of the diaphragm caused by polymerization shrinkage of the tested material was detected by the infrared sensor for 13 minutes at a sampling frequency of 5 Hz to an accuracy of 100 nm. Using custom software, data were fed on-line by means of an A/D converter to a personal computer (Macintosh II fx, Apple Computer, Cupertino, CA, USA). Eight specimens for each material and polymerization mode were tested.

PS was measured using a custom-made device (Figure 2) developed following Feilzer, de Gee and Davidson (1987). The upper part consisted of a semi-rigid load cell (PM 11-K, Mettler, Greifensee, Switzerland) to which a metal cylinder was screwed, 8-mm in diameter. A standardized quantity of test material, the same as for the specimens used for measurement of the linear polymerization displacement, was placed on the cylinder and compressed onto a glass plate attached to the base of the device in order to produce a specimen 1.5-mm in height. To improve adhesion of the resinous core build-up material, the surfaces of the metal cylinder and glass plate were sandblasted with 50 mm Al_2O_3 (Microetcher, Danville Engineering, Danville, CA, USA) and silanized (Monobond S, Vivadent Ets, Schaan, Liechtenstein). Light curing was carried out via a recess in the lower frame, through the glass plate, at a distance 1-mm from the tested material. The polymerization stresses developed during the 13 minutes were measured with a load cell at a sampling frequency of 5 Hz. Using custom software, data were fed online by means of an A/D converter to a personal computer. Eight specimens for each material and polymerization mode were tested.

Statistical evaluation of the data at the end of the 13-minute observation period was performed by two-way analysis of variance (ANOVA). Following the ANOVA, Tukey's Studentized Range (HSD) Test (p=0.05) was used to identify pairwise differences. Simple linear

regressions were also performed to determine correlations among all the characteristics measured.

The difference in evolution of LPS and PS over time was also measured using the following equations:

$$AD_{PS\text{-}LPS(13\ min)} = \frac{(sum\%PS - sum\%LPS)}{sum\%LPS} \times 100\% \quad (1)$$

$$sum\%\ LPS = \sum_{i=0}^{780} \frac{Y_{LPS_1}}{Y_{LPS_{13min}}} \quad (2)$$

$$sum\%\ PS = \sum_{i=0}^{780} \frac{Y_{PS_1}}{Y_{PS_{13min}}} \quad (3)$$

where

$AD_{PS\text{-}LPS(13min)}$ is the proportional difference between the percentage of polymerization stresses (PS) and linear polymerization shrinkage (LPS) with respect to the maximal value observed at 13 minutes (780 seconds), which numerically expresses the area difference between PS and LPS real-time curves after 13 minutes.

Y_{LPS_i} is the percentage of maximal value at any specific time (i) during the measurement of LPS.

$Y_{LPS_{13min}}$ is the maximum value of LPS recorded at 13 minutes (780 seconds) and taken as 100%.

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This effort depicted the area difference derived between two curves: the real-time curve of percentage values of LPS and the real-time curve of percentage values of PS over time. Therefore, equation 1 was able to show the area that expresses the result of the deduction of the areas below the two aforementioned curves and expressed it in percentage points in relation to the larger curve (LPS)

RESULTS

The RDB values measured at the end of the observation period are reported in Table 2. Bis-Core presented statistically significant higher and Permalute statistically significant lower RDB when ILC was applied (Bis-Core: DLC=NLC<ILC, Permalute: ILC<DLC=NLC). FluoroCore and Build-it! exhibited statistically significant differences among all the different curing modes (FluoroCore: ILC<DLC<NLC, Build-it!: DLC<NLC< ILC). Statistically significant differences were observed among all the materials when DLC and NLC were applied (Build-it!<Permalute<Bis-Core<Fluorocore). Statistically significant differences were also detected among all the materials, except FluoroCore and Build-it!, in the ILC group (Permalute<Fluorocore=Build-it!<Bis-Core).

LPS values measured at the end of the observation period are reported in microns in Table 3. Bis-Core presented statistically significant lower LPS when NLC was applied (DLC=ILC>NLC). FluoroCore and Permalute exhibited statistically significant differences among all the different curing modes (FluoroCore: ILC>DLC>NLC, Permalute: DLC>ILC>NLC), while Build-it! demonstrated no differences (ILC=DLC= NLC). Statistically significant differences were found among all the materials, except Bis-Core and FluoroCore when DLC and NLC were applied (Permalute>Build-it!>Bis-Core=Fluorocore). ILC resulted in statistically significant differences among all the materials (Permalute>Build-it!> Fluorocore>Bis-Core).

PS values measured at the end of the observation period are reported in kilograms in Table 4. Bis-Core exhibited statistically significant differences among all the different curing modes (DLC>ILC>NLC). FluoroCore and Permalute presented statistically significant lower PS when NLC was applied

Table 2: *Remaining C=C Double Bonds (%) at 13 Minute Post-mix (mean±SD)*

	Method of Light-curing Application											
Material	**NLC**				**ILC**				**DLC**			
Bis-Core	48.6	±	1.4	C a	53.6	±	1.5	C b	45.7	±	1.7	C a
FluoroCore	78.7	±	2.3	D c	29.1	±	1.6	B a	68.5	±	2.6	D b
Build-it!	26.1	±	1.8	A b	31.3	±	2.8	B c	20.1	±	1.8	A a
Permalute	30.0	±	1.9	B b	21.5	±	0.5	A a	29.4	±	2.7	B b

NLC: No Light-Curing; ILC: Immediate Light-Curing; DLC: Delayed Light-Curing.

Within a column, values having similar upper case letters did not exhibit statistical difference (p>0.05); comparison of materials within the same method of light-curing application. Within a row, values having similar lower case letters did not exhibit statistical difference (p>0.05); comparison of methods of light-curing application within the same material.

Table 3: *Linear Polymerization Shrinkage (in microns) at 13 Minute Post-mix (mean±SD)*

	Method of Light-curing Application											
Material	**NLC**				**ILC**				**DLC**			
Bis-Core	24.3	±	1.3	A a	27.8	±	1.2	A b	28.9	±	0.8	A b
FluoroCore	21.6	±	1.8	A a	32.5	±	2.4	B c	29.7	±	0.9	A b
Build-it!	36.0	±	2.6	B a	38.6	±	2.8	C a	36.4	±	1.7	B a
Permalute	48.0	±	4.9	C a	62.4	±	4.4	D b	68.9	±	3.9	C b

NLC: No Light-Curing; ILC: Immediate Light-Curing; DLC: Delayed Light-Curing.

Within a column, values having similar upper case letters did not exhibit statistical difference (p>0.05); comparison of materials within the same method of light-curing application. Within a row, values having similar lower case letters did not exhibit statistical difference (p>0.05); comparison of methods of light-curing application within the same material.

Table 4: *Polymerization Stress (in kilograms) at 13 Minute Post-mix (mean±SD)*

	Method of Light-curing Application											
Material	**NLC**				**ILC**				**DLC**			
Bis-Core	2.36	±	0.12	B a	2.60	±	0.13	A b	2.77	±	0.09	A c
FluoroCore	1.26	±	0.13	A a	3.14	±	0.16	B b	3.09	±	0.17	A b
Build-it!	4.08	±	0.12	D a	4.29	±	0.21	C a	4.09	±	0.19	B a
Permalute	3.49	±	0.35	C a	6.08	±	0.47	D b	5.95	±	0.42	C b

NLC: No Light-Curing; ILC: Immediate Light-Curing; DLC: Delayed Light-Curing.

Within a column, values having similar upper case letters did not exhibit statistical difference (p>0.05); comparison of materials within the same method of light-curing application. Within a row, values having similar lower case letters did not exhibit statistical difference (p>0.05); comparison of methods of light-curing application within the same material.

(ILC=DLC>NLC). Build-it! exhibited no statistically significant differences among all the different curing modes (ILC=DLC=NLC). Statistically significant differences were observed among all the materials when ILC and NLC were applied (NLC: Build-it!>Permalute>Bis-Core>Fluorocore, ILC: Permalute>Build-it!>Fluorocore>Bis-Core). Finally, statistically significant differences were determined among all the materials except FluoroCore and Bis-Core in the DLC group (Permalute>Build-it!>Fluorocore= Bis-Core).

Linear regression proved high correlation between LPS and PS for all light-curing modes evaluated (r^2=0.85). RDB was not correlated with either LPS or PS (RDB-LPS r^2=0.40; RDB-PS r^2=0.57).

Table 5: *Results of Equation 1* That Numerically Express the Area Difference Between PS and LPS Real-time Curves After 13 Minutes*

Material	Light-curing Application	Equation 1
Bis-Core	NLC	-15.5%
Bis-Core	ILC	-3.4%
Bis-Core	DLC	-4.6%
FluoroCore	NLC	-29.4%
FluoroCore	ILC	-5.7%
FluoroCore	DLC	-27.1%
Build-it!	NLC	-3.9%
Build-it!	ILC	0.4%
Build-it!	DLC	3.1%
Permalute	NLC	-28.8%
Permalute	ILC	-2.9%
Permalute	DLC	-22.4%

NLC: No Light-Curing; ILC: Immediate Light-Curing; DLC: Delayed Light-Curing.

** The smaller the value of Equation 1 in absolute numbers, the more PS and LPD develop at similar rates.*

Table 5 shows the results of Equation 1. Figures 3 through 5 show representative curves of Permalute depicting the evolution of LPS and PS in different light-curing modes. The smaller negative values in Equation 1 indicate that LPS and PS develop in a more similar fashion, as there is less delay in the development of PS with respect to LPS. The results from Table 5 clearly show that ILC demonstrated the lowest negative values in all materials. On the other hand, NLC exhibited the highest negative values. DLC presented values which indicate that, in some materials (FluoroCore and Permalute), LPS and PS evolved in a similar fashion when ILC was applied, while in other materials (Bis-Core and Build-it!), the behavior resembled that of NLC.

DISCUSSION

The degree of remaining C=C bonds has been proven to influence several properties in resin composite materials (Peutzfeldt & Asmussen, 2000). Infrared spectroscopy is a well-established method for evaluating the conversion of methacrylate groups in light-cured resin composites (Silikas, Eliades & Watts, 2000; Park & Lee, 1996; Park, 1996; Chung & Greener, 1990; Papagiannoulis & Eliades, 1989; Eliades, Vougiouklakis & Caputo, 1987; Ferracane, 1985). This method has also been applied to study dual-cured resin cements (Braga, Cesar & Gonzaga, 2002a; Eliades & others, 2000) and resin-modified glass-ionomer materials (Braga, Condon & Ferracane, 2002b; Kakaboura, Eliades & Palaghias, 1996a,b).

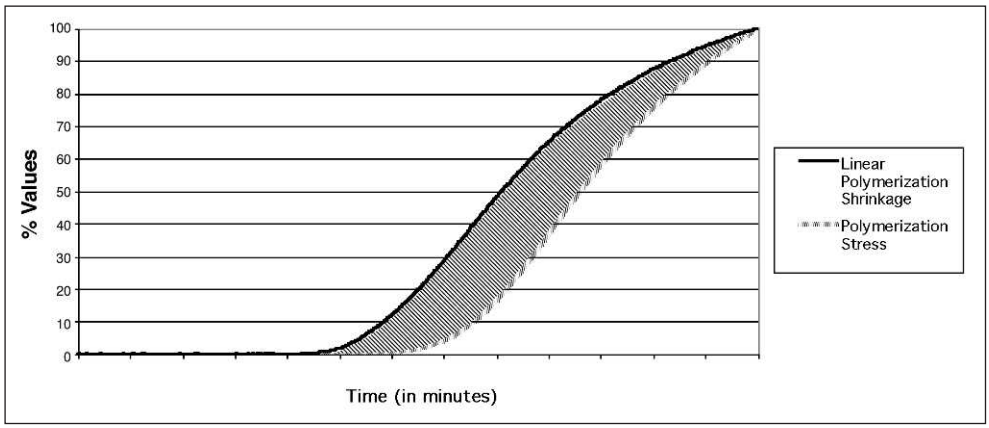

Figure 3: *Real-time curves of percentage values (13-minute value taken as 100%) of LPS and PS development using NLC for Permalute. The result of Equation 1, which numerically expresses the area difference (downward diagonal marked area) between PS and LPS real-time curves, was -28.8%.*

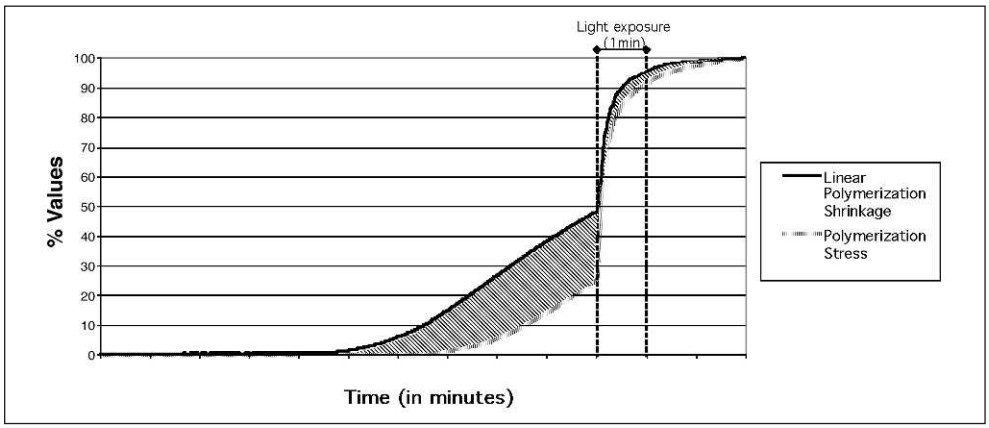

Figure 4: *Real-time curves of percentage values (13-minute value taken as 100%) of LPS and PS development using DLC for Permalute. The result of Equation 1, which numerically expresses the area difference (downward diagonal marked area) between PS and LPS real-time curves, was -22.4%.*

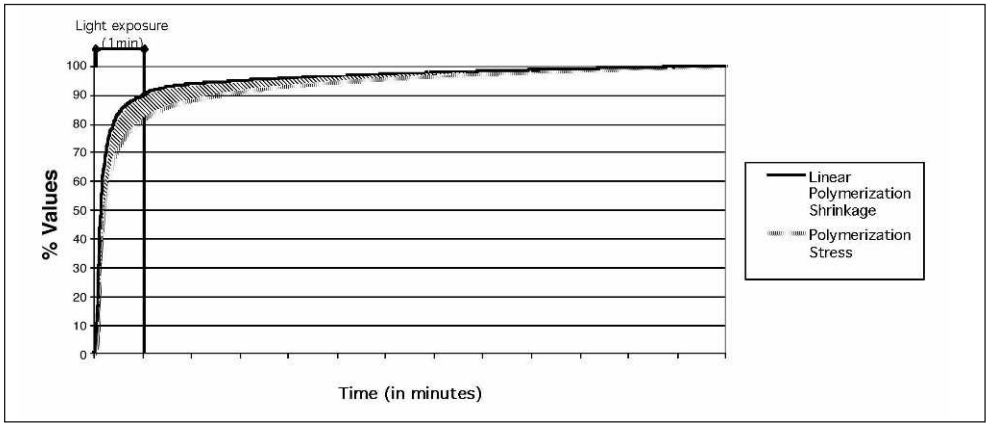

Figure 5: *Real-time curves of percentage values (13-minute value taken as 100%) of LPS and PS development using ILC for Permalute. The result of Equation 1, which numerically expresses the area difference (downward diagonal marked area) between PS and LPS real-time curves, was -2.9%.*

The RDB levels obtained after ILC simulate the values reached under common clinical practice. Also, as both mechanisms of polymerization, chemical and light, are induced within 13 minutes after ILC, it can be hypothesized that each material reached nearly its minimum RDB value. The wide range of RDB values observed under the ILC scenario (21.5% to 53.6%) were consistent with those reported for restorative resin composites (Chung & Greener, 1990; Park, 1996; Peutzfeldt & Asmussen, 2000; Silikas & others, 2000). The differences found in RDB values of the materials investigated may be attributed either to differences in monomer composition or to variations in catalyst systems. The results of this study suggest that ILC provides low levels of RDB on the direct irradiated surface of all materials except Bis-Core. However, an even lower RDB value was achieved in Build-it! when DLC was applied. This finding indicates that the immediate light curing of Build-it! may lead to increased steric hindrance, which decreases the segmental mobility of methacrylate groups and reduces reactivity of the chemical polymerization reaction.

A comparison of the three light-curing modes revealed interesting observations with respect to the dominant curing mode in each material. That was the reason why a clinically unacceptable 10-minute delay was chosen as a curing mode in this study. DLC provides ample time for the chemical curing mechanism to progress prior to light application. The dominant curing mode is a point of outmost clinical importance in order to differentiate which materials' chemical curing plays a substantial role compared to light polymerization. Thus, these mate-

rials may be used in areas where negligible light is applicable.

Differences in RDB between the surface layer of a restoration, expressed in this study using ILC, and the deep aspects, represented by the NLC group, may develop anisotropic properties in the core material under load. This difference, in turn, may stress the tooth-material interface and may cause a non-uniform distribution of stress within the material. According to the results of this study, such differences were found only for FluoroCore. This material's dominant curing mode was as a light-cured resin composite, because a very high RDB level (78.7%) was measured when NLC was applied. Even more, DLC resulted in high RDB, as well (68.5%). On the contrary, when this material underwent ILC, low RDB was recorded (29.1%). This result could be attributed to the fact that 60-second light-curing could not substantially increase the restricted mobility of the monomer chains embedded into the "poorly developed" polymer network, which resulted in a chemical cure within the first 10 minutes. Materials like Build-it! and Permalute exhibited high levels of chemical curing, as low RDB was found even when NLC was used. Nevertheless, when these materials were subjected to DLC, only Build-it! presented a decrease in RDB. This result could be due to the fact that the polymer network developed in Permalute by chemical cure did not allow any additional mobility of the polymer chains in order to improve the C=C conversion. Even though Build-it! and Permalute can be polymerized under dark conditions, the degree of C=C conversion under ILC surpassed that of NLC and light curing acted as the dominant mechanism. The curing mode had no impact on the RDB of Bis-Core, since high values were measured under the three curing modes applied. The latter finding may highlight the monomer type(s) and concentration as the main contributor factors for the insufficient polymerization of this material and not the curing mechanism.

Various experimental methods have been used to determine shrinkage and stress of resin composites during polymerization. Dilatometric methods are commonly used to measure polymerization shrinkage of chemically cured materials (Iga & others, 1991; Rees & Jacobsen, 1989; Hay & Shortall, 1988; Feilzer, de Gee & Davidson, 1988; Bullard, Leinfelder & Russell, 1988; Patel, Braden & Davy, 1987; Penn, 1986; Bandyopadhyay, 1982; de Gee, Davidson & Smith, 1981; Jacobsen, Whiting & Richardson, 1977; Hegdahl & Gjerdet, 1977; Jacobsen, 1975; Dennison & Craig, 1972; Lee, Swartz & Smith, 1969). These techniques present a significant number of problems related to the access of light to the light-cured material tested. Furthermore, when water is used, temperature changes occur due to thermal expansion/contraction of the surrounding fluid. Non-volume dilatometric methods have been used lately for measurement of polymerization shrinkage in light-cured resin-based restorative materials (Feilzer, de Gee & Davidson, 1993; de Gee, Feilzer & Davidson, 1993; Watts & Cash, 1991; Feilzer, de Gee & Davidson, 1989; Walls, McCabe & Murray, 1988; Bausch & others, 1982). The materials tested in this study were dual-cured, and both chemical and light-curing polymerization processes were assessed. Therefore, a non-volume dilatometric measurement technique, as described in the materials and methods section, was used to measure linear polymerization displacement. The LPS values of this study are reported in microns, as these were the recording units of the infrared sensor used for measuring the vertical movement of the diaphragm, which was caused by polymerization shrinkage of the test material. The technique used in this study permitted measurement of the evolution of linear polymerization displacement through time.

Methods commonly used for polymerization stress measurement differ in the configuration by which specimens are connected to the load cell (Feilzer & others, 1993; Bullard & others, 1988; Feilzer & others, 1987; Hegdahl & Gjerdet, 1977). Various techniques have been used to maintain the distance between the load-cell and the testing machine crosshead stable. The latter permits for the measurement of stress developing when shrinkage is restrained, simulating clinical conditions where resin composite is bonded to non-flexible preparation walls. The PS values of this study are reported in kilograms (actual measurements of load cell) and were not transformed in Megapascals (MPa) to avoid confusion with literature data with completely rigid experimental set-ups. In the experimental design of this study, axial specimen deformation was partially restricted, since the load cell was axially displaced 4 mm for each kilogram of measured force, resulting in a maximum of 28 mm deformation of the specimen. In this way, a semi-rigid configuration of a preparation having a C-factor of 2.67 was simulated. Several studies have demonstrated that cusps of molars and premolars deflect inward after placement of Class II resin composite restorations, with the amount of the reported contraction ranging from 18 to 45 μm (Smith & Caughman, 1989; Causton, Miller & Sefton, 1985; Lutz, Krejci & Barbakow, 1991), thus justifying the experimental set-up used in this research.

Most of the LPS and PS values encountered in this research, when ILC was applied, were consistent with those reported for restorative resin composites (Stavridakis & others, 2000; Stavridakis & others, 2003). However, in the case of Permalute, higher values were measured. Nevertheless, this result was expected, as Permalute is characterized by the manufacturer as a low-viscosity composite luting/restorative resin. The small filler fraction and high portion of monomer caused increased LPS and PS (Stavridakis & others, 2000). On the other hand, the high flow capacity of Permalute should compensate for the shrinkage effect during the

pre-gelation phase. However, using ILC, the material reaches the gel point rapidly. Therefore, viscous flow cannot reduce the shrinkage stress developed in Permalute. The low LPS and PS exhibited by Bis-Core cannot be characterized as an advantage, because they are attributed to the high RDB of the material.

The high correlation of LPS and PS noted in this study is in accordance with results from similar studies (Davidson & Feilzer, 1997; Stavridakis & others, 2000; Stavridakis & others, 2003). Generally, although the amount of shrinkage is related to the extent of C=C remaining, no high correlation was found between RDB and either LPS or PS. Thus, when ILC was applied, high RDB was associated with low values in LPS and PS. On the contrary, using NLC or DLC, neither LPS nor PS was related to RDB. For example, after DLC of FluoroCore, very high RDB resulted in unexpectedly high values of PS. This result may be explained by the kinetics of the polymerization process. It is likely that after 10 minutes of chemical cure, the material has reached a state where restricted mobility of free radicals in the polymer network resulted in the development of the polymer network. As a consequence, there was an increase in the modulus of elasticity that restricts the material's capacity for viscous flow and decreases its compliance, thus causing a dramatic increase in PS (Braga & Ferracane, 2002). One should always keep in mind that low polymerization stresses alone are not the only quest, as adequate C=C conversion is also a pre-requisite that was not obtained in some dual-cured materials. The evolution of PS was both material- and curing-mode dependent.

The inconsistencies noted between the development of LPS and PS may be more easily understood by observing the kinetics of the properties that were studied. In the chemical curing mode of Permalute (Figure 3), the delay in PS development is easily observed. Equation 1 expresses the differences in evolution of the two properties over time in a numerical manner. The application of Equation 1 in some cases resulted in high negative values, indicating that larger delays in development of PS, with respect to LPS, were evident due to the slow development of polymerization stresses. In other cases, the results of Equation 1 were again negative values but of a lower magnitude (Figures 4 and 5). In those cases, there was again a delay in the development of PS with respect to LPS, but of a lower magnitude, meaning that the two properties developed more at the same rate and at the same proportional level. In two instances, Equation 1 resulted in positive values, demonstrating that PS evolved faster than LPS.

From a clinical standpoint, materials with a low RDB, LPS and PS are preferable. Low RDB is necessary in order for the material to obtain maximal mechanical properties. This is why ILC is the polymerization mode of choice for FluoroCore, as the other two curing modes produce a low degree of RDB. The second aim is to use materials that produce low polymerization stresses. For this reason materials with low LPS are preferred, as this property is highly correlated with PS. Nevertheless, this research showed that there are other options available as a means of lowering PS, depending on the behavior of the dual-cured materials. In some situations, obtaining low polymerization stresses was acquired by using NLC when this curing mode provided low RDB (Bis-Core). In other cases, using DLC resulted in lower PS (Permalute and Build-it!).

CONCLUSIONS

1. Significant differences were manifested among the materials tested in terms of the remaining C=C bonds (RDB), linear polymerization shrinkage (LPS) and polymerization stresses (PS) under all different modes of photo-activation studied.
2. Bis-Core provided the highest percentage of RDB with the lowest LPS and PS; whereas, Permalute presented the lowest RDB along with the highest LPS and PS, irrespective of curing mode.
3. The RDB in Build-it! was equal, using either ILC or NLC conditions with no significant changes in either LPS and PS; DLC reduced RDB.
4. The polymerization of FluoroCore using NLC or DLC modes caused a dramatic increase in RDB.
5. No correlation was found between RDB and LPS or PS. On the contrary, a strong correlation was evident between LPS and PS.

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Effects of Dentin Disinfectants on the Shear Bond Strength of All-ceramics to Dentin

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Clinical Relevance

Both Consepsis and Tubulicid Red can be safely used to prevent the potential risk of complications resulting from bacterial activity without affecting the bond strength of IPS Empress 2 restorations clinical luting procedures.

SUMMARY

This study investigated the effect of dentin disinfectants on the shear bond strength of all-ceramic restorations luted with two different dual-polymerizing systems to dentin. Dentin disinfectants chlorhexidine gluconate-based Concepsis and benzalkonium chloride-based Tubulicid Red were applied in combination with Variolink 2 and Resilute. The buccal surfaces of non-carious extracted human premolars were flattened to expose dentin and subsequently polished with 600-grit wet silicon carbide paper. Each dual-polymerizing luting system had two test groups and a control group. Dentin from the test groups was first treated with dentin disinfectants, then the all-ceramic restorations were luted with dual polymerizing systems. The dentin without disinfectant application and ceramic restoration was used as a control. After the all-ceramic samples were luted to all treated surfaces, the specimens were thermocycled and tested for shear bond strength until failure. Analysis of the fractured dentin surfaces was performed using Optical Microscope-Nikon ECLIPSE ME 600 (Nikon Co, Tokyo, Japan) at between 10x and 1000x magnifications and the images were analyzed with Image Analyzer LUCIA 4.21 (Nikon Co). The data were analyzed with one-way ANOVA and Duncan test at a significance level of $p<0.05$. Application of the two dentin disinfectants increased the shear bond strength of both the Variolink 2 and Resilute systems, but this increase was statistically significant only in Tubulicid Red treated specimens ($p<0.05$). Surface analysis showed that all the specimens showed the adhesive failure mode between the dentin and composite luting agent interface.

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INTRODUCTION

Interest in all-ceramic restorations has increased in recent years. Ceramic inlays, onlays, veneers and complete-coverage crowns have gained popularity. These

restorations offer superior esthetics compared with metal-ceramic restorations (Christensen, 1999). The strength of all-ceramic crowns is influenced by the shape of the tooth preparation, pretreatment of the crown and abutment and the method of luting (Burke, 1995). The dentin-bonded crown can be defined as a complete coverage restoration in which the crown is bonded only to the underlying dentin using a luting composite. The bond is mediated by the use of a dentinal bonding system and chemomechanical adhesion to the ceramic surface (Strub & Beschnidt, 1998).

IPS Empress 2 (Ivoclar Vivadent, Schaan, Liechtenstein) is a lithium disilicate, heat-pressed all-ceramic material offering the advantages of increased biocompatibility, natural appearance and superior esthetics. A chemical bond between the etched porcelain and tooth structure is accomplished in the IPS Empress 2 system (Zawta, 2001).

One of the most important factors determining the success of a restoration is adhesion between the tooth structure and restorative material. Dentin bonding is designed to produce a hermetic seal between a composite restoration and the surrounding dentin (Oram & Pearson, 1994). The use of dual-polymerizing resin cement in combination with a dentin bonding agent has reduced the problem of loss of retention (Milleding, Ortengren & Karlsson, 1995). In spite of encouraging results, clinical performance of the dentin bond is impaired by composite polymerization shrinkage and stresses resulting from thermal dimensional changes (Van Meerbeek & others, 1998). The choice of restorative method has a critical impact on the behavior of the dentin-resin interface. In addition to the cementation variables, dentin is a heterogeneous substrate, and it is difficult to predict the overall behavior of the dentin-resin interface (Pashley & others, 1995).

If a proper adhesion cannot be achieved, microleakage, defined as the passage of bacteria, fluids, chemical substances, molecules and ions between the tooth and its restoration, can occur. This would threaten clinical performance and longevity of the restoration, contributing to staining, recurrent caries, adverse pulpal response and postoperative sensitivity (Bauer & Henson, 1984).

The problems associated with microleakage can be magnified by incomplete sterilization of the preparation from all infected enamel and dentin (Meiers & Kresin, 1996), especially if the carious lesion exists in the prepared tooth. Brännström (1987) has reported that multiple bacterial sources may be implicated in tooth preparation infection, including 1) invasion from the tooth surface via marginal gap formation between a tooth and restorative material; 2) bacteria present in the smear layer; 3) bacteria present in the dentinal tubules; 4) bacteria at the dentino-enamel junction and 5) bacteria recontaminating the surface of a tooth prior to placing a restoration. Even when the tooth preparation is sealed completely, bacteria existing in the smear layer can multiply and their toxins and degradation products can diffuse in the pulp, resulting in irritation and inflammation (Brännström, 1986). Numerous experiments have shown that infection is the main cause of pulpal damage; tissue fluids from pulp and organic substances within dentin and enamel provide sufficient substrate for microbial growth under restorations (Brännström, 1987). Brännström (1996) states that about 5% to 24% of crowns and fixed partial dentures may, in time, result in pulpal complications and periapical inflammatory lesions.

When the restoration walls are completely encased in enamel, excellent clinical service can be obtained since the seal would be perfect (Lacy & others, 1992). On the other hand, where dentin is exposed, the application of etchants will increase the permeability of dentin, increasing the possibility of pulpal irritation or damage and increase sensitivity (Kedici, Kalipcilar & Bilir, 1992). Since cut dentin is always bonded to the all-ceramic material, expectations of higher risks of sensitivity leading to these complications should be the concern of the restorative dentist practicing with all-ceramics.

To reduce the potential risk of pulpal inflammation resulting from bacterial activity, the use of antibacterial materials during restorative procedures has been recommended (Gultz & others, 1999; Palenik & Setcos, 1996; Meiers & Kresin, 1996). Currently, the application of a disinfectant after tooth preparation and before inserting the restoration is gaining wider acceptance (Gwinnett, 1992b; Meiers & Kresin, 1996; Meiers & Shook, 1996).

A potential problem in the use of a disinfectant with dentin bonding agents is the possibility of an adverse effect on the bond strength of the restorative material to dentin (Cao & others, 1995; Gurgan, Bolay & Kiremitçi, 1999; Meiers & Shook, 1996). Shear bond strength values to dentin between 18 and 20 MPa are considered to be optimal (Eick & others, 1993; Swift, Perdigão & Heymann, 1995). Dentin disinfectants applied to the dentin surface should not compromise the shear bond strength of the bonding systems. It has been suggested that disinfectants remoisten the cavity walls prior to placing an adhesive resin and actually enhance adhesion (Miller, 1995). Many studies have investigated the effect of cavity disinfectants on the bond strength of resin composites (Cao & others, 1995; Gurgan & others, 1999; Gwinnett, 1992a; Meiers & Shook, 1996; Perdigão, Denehy & Swift, 1994), reporting bond strengths as varying according to the active ingredients in cavity disinfectants and the brand of dentin adhesive system. However, there is a lack of literature on the effect of dentin disinfectants to the bonding performance of all-ceramic systems.

The null hypothesis of this study was that chlorhexidine gluconate-based disinfectant (Consepsis) and benzalko-nium chloride-based disinfectant (Tubulicid Red) would not adversely effect the shear bond strength of an all ceramic system bonded to dentin with the luting agents Variolink 2 and Resilute.

METHODS AND MATERIALS

Sixty non-carious, freshly extracted human premolars were used in this study. The teeth were scraped clean of any residual tissue tags, kept in 2.6% sodium hypochloride for 15 minutes and rinsed under running water for 15 minutes. They were then stored in distilled water at +4°C until use. The roots were removed from the crowns approximately 2 mm below the cemento-enamel junction using a slow speed diamond saw under copious water spray. The teeth were then mounted in a plastic holder filled with cold-cure acrylic resin. They were then placed with the buccal surfaces upward just above the surface of the embedding medium. Just prior to preparation of the test specimens, the underlying superficial dentin was exposed by mounting the buccal surfaces on a metallurgical polishing wheel and wet grinding them flush with the surface of the embedding medium using 180-grit, followed by 600-grit, silicon carbide paper. The exposed dentin surfaces were inspected with a dissecting microscope at 50x magnification to ensure that no enamel was left. The teeth were then randomly divided into two main groups according to the luting resin used, either Variolink 2 or Resilute. Each luting resin system was assigned to two disinfectant groups of 10 specimens each (Table 1).

The dentin disinfectants were applied with a sterile brush applicator after the etching step for each luting system and left in contact with the dentin surface for 60 seconds. The excess disinfectant was removed with five-second light air drying in order to prevent dessication. After dentin disinfection, a piece of adhesive tape, with a 4-mm diameter hole, was securely fastened to the center of the dentin surface to delimit the bonding surface. The specimens not treated with any of the disinfectants were used as controls for each luting system. All the luting steps were carried out as performed in the experimental groups.

Table 1: *Materials Used in the Study*

Material	Manufacturer	Batch #	Material Composition
Luting Systems			
Variolink-2	Ivoclar Vivadent, Schaan, Liechtenstein	Etchant: D 57027	37 % phosphoric acid
		Primer (Syntac pr.): E 34592	Tetraethylenglycol dimethacrylate, maleic acid, dimethylketone, water
		Adhesive (Syntac ad.): E 30794	Polyethyleneglycol dimethacrylate, glutaraldehyde, maleic acid, water
		Bond (Heliobond): E 51105	Bis-GMA, triethyleneglycol dimethacrylate
		Cement base: E 51946	Bis-GMA, UEDMA, TEGDMA, filler
		Cement low viscosity catalyst: E 52170	Bis-GMA, UEDMA, TEGDMA, filler
		Silane: E 26882	3-methacryloxy propyl-trimethoxysilane, water, ethanol
		Ceramic etchant: E 52923	5% hydrofluoric acid
		Oxygen inhibiting gel: D 50843	Glycerine, silica
ResiLute	Pulpdent Co, Watertown, MA, USA	Etchant: 020314	38% phosphoric acid
		Adhesive (DenTASTIC UNO): 020326	PMGDM, monomer resin, activators, acetone
		Dual-cure catalyst for adhesive (DenTASTIC DUO): 020419	Mg NTG-GMA, acetone
		Cement part 1: 020207	Monomer, filler
		Cement part 2: 020529	Monomer, filler
		Oxygen inhibiting gel: 020530	Glycerine, silica
		Ceramic etchant: 020606	9.6 % hydrofluoric acid
		Drying agent: 020606	Ethyl alcohol
		Silane: 020606	Ethyl alcohol, acetone, benzene
Dentin Disinfectants			
Consepsis	Ultradent Products, Inc, South Jordan, UT, USA	454D	2% chlorhexidine gluconate
Tubulicid Red	Dental Therapeutics Ab, Saltsjö-Boo, Sweden	311100	0.1% benzalkonium chloride, 0.2% EDTA, 1% sodium floride

A total of 60 IPS Empress 2 samples were fabricated by heat-pressing them into sprues and sectioning them into disc shapes, 2 mm in diameter and 3 mm in height, using diamond wheels at low speed. The samples were subjected to sandblasting with 50 μm Al_2O_3. Following ultrasonic cleaning for 10 minutes in distilled water, IPS Empress 2 discs were acid etched according to the etching procedures, then silanated as described in each luting system. Applying a load of 20 N, simulating finger pressure, the IPS Empress 2 samples were luted in accordance with the manufacturers' instructions using the compact material tester (EZTest, Shimadzu Corporation, Kyoto, Japan) on delimited dentin surfaces with the luting systems shown in Table 2.

Table 2: *Adhesive Luting Systems Used in the Study*

Material	Conditioning	Priming	Adhesive Resin	Cement
Variolink-2	Conditioner (15 seconds) wash and dry	Syntac primer (15 seconds)	Syntac adhesive (10 seconds)	Dual-cured cement
ResiLute	Conditioner (15 seconds) wash and dry	DenTASTIC UNO DUO		Dual-cured cement

The specimens were stored in distilled water at 37°C for 24 hours. They were then subjected to 500 thermal cycles between 5°C and 55°C water baths, with a dwell time of 30 seconds and a transfer time of 30 seconds.

The shear bond strength was measured with a Shimadzu Universal Testing Machine (Model AG-50kNG, Shimadzu Corporation, Kyoto, Japan). A knife-edge shearing rod with a crosshead speed of 0.5 mm/minute was used (Figure 1). The distance from the shearing rod to the dentin surface was maintained using a spacer of two celluloid matrices (Hawe Neos Dental, Gentilino, Switzerland). The load at failure was recorded by Labtech Notebook software version 6.3 (Labtech, Wilmington, MA, USA). The shear bond strengths of the specimens were calculated and expressed in MPa.

The analysis of the debonded dentin surfaces of all samples was performed over the entire bonding area using an Optical Microscope Nikon ECLIPSE ME 600 (Nikon Co, Tokyo, Japan) at between 10x and 1000x magnifications, and the images were analyzed with Image Analyzer LUCIA 4.21 (Nikon Co).

The shear bond strength data of the disinfectant groups in each luting resin group were analyzed by ANOVA (one-way analysis of variance) and Duncan test using SPSS 10.0 for Windows. For the surface analysis data of fracture sites, the Chi-Square test was used. All tests were performed at a 95% confidence level.

RESULTS

The shear bond strengths in MPa (Mean ± SD) for the six groups are shown in Table 3. Although these results show that application of the two dentin disinfectants increased the shear bond strengths of both Variolink 2 and Resilute luting systems, this increase was statistically significant only in the Tubulicid Red treated specimens ($p<0.05$).

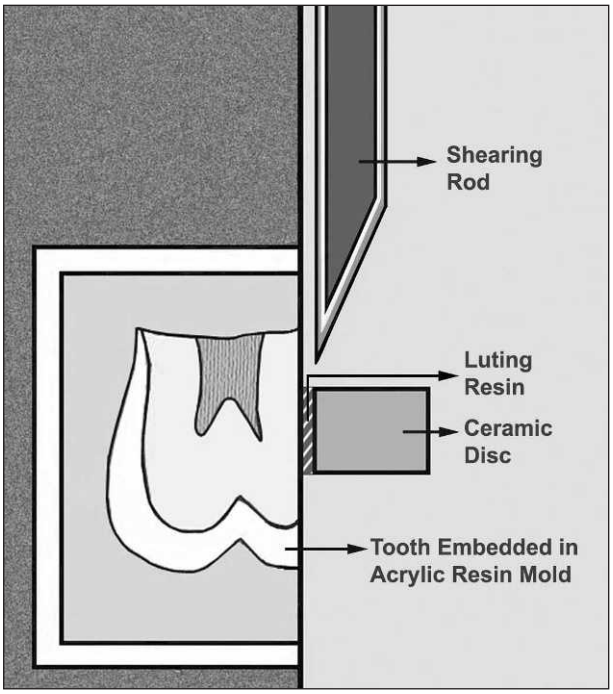

Figure 1: *Schematic diagram of the test assembly for determining shear bond strength.*

Light microscope analysis of the dentin surfaces indicated that all specimens showed the adhesive failure mode between the dentin and composite luting agent interface. However, adhesive resin remained on the dentin surfaces of nearly all specimens (Figure 2). Adhesive resin was completely debonded from the dentin surfaces of two specimens in Group 2 and Group 4 and one specimen in Group 5 (Figure 3). Adhesive resin was partly debonded from the dentin surface of one specimen in Group 4 and Group 6 (Figure 4). Pure dentin and pure ceramic cohesive failures were not observed. The Chi-Square test revealed no significant differences in failure mode frequency between the control group and the two disinfectant groups for either adhesive luting system ($p>0.05$).

DISCUSSION

The use of disinfectant solutions after tooth preparation has been recommended for disinfecting dentin and reducing post-operative sensitivity (Brännström, 1996, 1986; Gultz & others, 1999; Meiers & Kresin, 1996). However, any positive benefits are negated if the disinfectant decreases the shear bond strength of the all-ceramic material which is bonded with a resin composite by altering its sealing ability to dentin.

Table 3: *Treatment Groups and Mean Shear Bond Strength Values (MPa)*

Groups	Disinfectant	Dentin Bonding System	Luting Resin	Mean Std Deviation
1 Control	None	Syntac	Variolink 2	20.5700±5.0320[a*]
2	Consepsis	Syntac	Variolink 2	24.2430±3.0798[ab]
3	Tubulicid Red	Syntac	Variolink 2	26.9100±5.1387[b]
4 Control	None	DenTASTIC UNO DUO	ResiLute	15.0800±4.1386[c]
5	Consepsis	DenTASTIC UNO DUO	ResiLute	17.1640±2.5562[cd]
6	Tubulicid Red	DenTASTIC UNO DUO	ResiLute	20.3590±4.2260[d]

**Different letters indicate that there were statistically significant differences ($p<0.05$).*

This study found the chlorhexidine gluconate-based disinfectant (Consepsis) to not adversely affect the shear bond strength to dentin of IPS Empress 2 luted with Variolink 2 and Resilute. Previous studies tend to support the findings of this study, where treatment of the dentinal surface with chlorhexidine gluconate prior to application of dentin bonding agents did not have an effect on the bond strength of resin composite (Gwinnett, 1992b; Perdigão & others, 1994; Türkün, Türkün & Kalender, 2004; Say & others, 2004). Meiers and Shook (1996) reported that a chlorhexidine gluconate-based disinfectant, BISCO Cavity Cleanser, did not affect the bond strength of resin composite bonded with Tenure but decreased bonding with Syntac. However, Meiers and Kresin (1996) found that the same disinfectant did not significantly affect the sealing ability of Tenure and Syntac when compared to the control groups. In a similar study with resin modified glass-ionomer cements, Cunningham and Meiers (1997) found that chlorhexidine solution did not significantly affect the bond strengths of any of the cements tested. In addition, el-Housseiny and Jamjoum (2000) reported that these disinfectants do not adversely affect the bonding of resin composite to enamel or dentin before acid etching. On the other hand, Gurgan and others (1999) found that pre-applied chlorhexidine gluconate (Consepsis) reduced the bond strength of adhesive resin (Permagen). However, they observed no reduction in composite-to-dentin shear bond strength when the disinfectant was rinsed off prior to the bonding procedure. Nevertheless, washing chlorhexidine that contains a surfactant might only partially drive away the chlorhexidine molecules, and the bound molecules might serve as a co-surfactant on the conditioned dentin before the resin is applied (Schaeken, Keltjens & Van der Hoeven, 1991). Cao and others (1995) reported most commercial chlorhexidine gluconate-based disinfectants decreased the bond strengths of adhesive resins, to varying degrees. They found that Consepsis was the only disinfectant that did not significantly affect the bond strength of any of these adhesive resins.

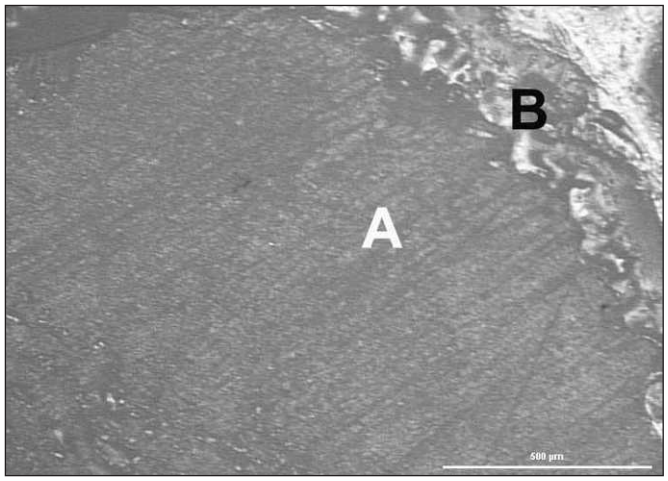

Figure 2: *Appearance of adhesive resin on the dentin surface.*

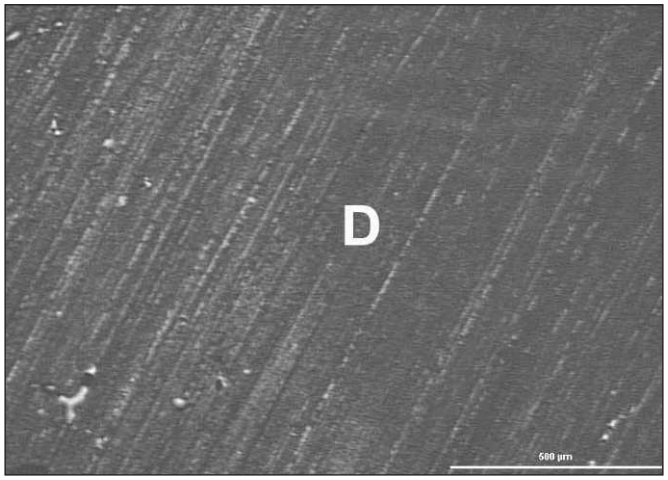

Figure 3: *Appearance of the dentin surface free from adhesive resin.*

The benzalkonium chloride-based dentin disinfectant (Tubulicid Red) did not alter the bond strength of IPS Empress 2 samples that were luted with Variolink 2 and Resilute. Surprisingly, it produced an increase in shear bond strength. Derhami, Coli and Brännström (1995) reported that treatment of the cavity walls with

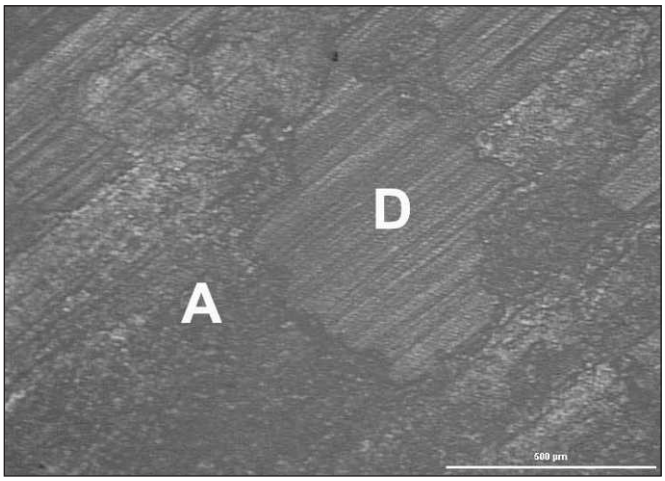

Figure 4: *Partly debonded adhesive resin from the dentin surface.*

Tubulicid Red, instead of the respective conditioners of the adhesive systems (Superbond D-liner and Clearfil Liner Bond), had no adverse effect on their sealing ability in Class II resin composite restorations. This may depend on the fact that benzalkonium chloride has been shown to link the collagen but does not impair hybridization (Gwinnett, 1992b). A previous study conducted by Türkün and others (2004) indicates that treatment of cavity preparations with Tubulicid Red before application of dentin bonding systems did not adversely affect both their sealing ability and bond strengths. Similarly, Say and others (2004) showed that the benzalkonium chloride-based disinfectant Ultracid F, after etching the dentin, did not have an adverse effect both on the shear and tensile bond strengths of dentin bonding systems. However, Cao and others (1995) showed that Tubulicid Red decreased the shear bond strength of All Bond 2 and Amalgambond Plus but did not adversely affect Permagen.

It has been shown that adhesive luting increases the fracture resistance of all-ceramic crowns (McCormick & others, 1993; Burke & Watts, 1994). Strub and Beschnidt (1998) tested five different all-ceramic crown systems, including the Empress system, for their fracture strengths. Their results revealed that the crowns of the Empress staining technique showed the fewest crown fractures. The probable reason for the fracture resistance of Empress crowns was attributed to their being pressed as full crowns. Their results indicate that the composite had a higher bond strength to ceramic material than to prepared dentin. The results of this study are in accordance with Strub and Beschnidt's study, since all the specimens showed the adhesive failure mode between the dentin and composite luting agent interface.

The application sequence of the disinfectant is also an important factor to be considered. While some clinicians prefer to apply disinfectants after tooth preparation, prior to the bonding procedure (Meiers & Shook, 1996; Gurgan & others, 1999; el-Housseiny & Jamjoum, 2000; Meiers & Kresin, 1996), others prefer to apply disinfectants after etching (Pilo & others, 2001; Cao & others, 1995; Schaeken & others, 1991; Perdigão & others, 1994; Say & others, 2004; Owens, Lim & Arheart, 2003). In this study, the effect of dentin disinfectants on the bond strength of all-ceramic luting systems was investigated after etching procedures. The manufacturer recommends the use of Consepsis and Tubulicid Red after etching. The use of dentin disinfectants would be more preferable after etching the dentin, as removal of the smear layer leads to the elimination of most microorganisms. Then, the use of disinfectants would be more beneficial for those microorganisms and their toxins that remain viable in the dentinal tubules (Say & others, 2004).

This study did not primarily intend to compare the shear bond strengths of the two luting systems; however, it is interesting to note that the adhesive performance of the luting systems was not the same in terms of shear bond strength. This may be due to the difference in the adhesive systems of the luting resins used. One adhesive system used in this study, Syntac, is a fourth-generation dentin bonding agent that works in three steps: etching, priming and bonding. The other adhesive system, DenTASTIC UNO DUO, is a fifth-generation one-bottle bonding system. One-bottle systems combine the primer and adhesives into one solution to be applied after the enamel and dentin are etched simultaneously (Kugel & Ferrari, 2000). In this system, the dentin surface should remain in a moist state to prevent collapse of the unsupported collagen and promote primer-resin infiltration (Gwinnett, 1992b; Kanca, 1992). In this study, Syntac showed more favorable results in the shear bond strength test than DenTASTIC UNO DUO. Studies that compare the shear bond strength of these adhesive systems are not available in the dental literature; therefore, comparison of the results of this study could not be performed.

Regarding the adhesive systems used in the study, Syntac adhesive system is water-based, while DenTASTIC UNO DUO is acetone-based. Reis and others (2003) have demonstrated that the degree of moisture of dentin is effective on the bond strength of adhesive systems. By monitoring the amount of water used to rewet the air-dried dentin surface, they have shown that total etch adhesive systems achieve optimal bond strengths at different moisture degrees, which is dependent on the solvent present in each system. Their data confirmed, in a quantitative manner, that water-based systems require a drier dentin surface, while acetone-based systems require a wetter dentin surface for improved bond strengths. In this study,

excess water (or disinfectant) was removed by light air drying instead of cotton pellet drying. The favorable results of Syntac in shear bond test may be attributed to this light air drying.

It should be noted that this study was performed under *in vitro* conditions. Simulation of oral conditions in the laboratory is quite difficult. However, laboratory studies that compare materials under similar conditions give us insight into probable clinical behavior and provide guidance for clinical trials.

CONCLUSIONS

Within the limitations of this *in vitro* study, the null hypothesis must not be rejected. The application of Consepsis and Tubulicid Red did not adversely affect the shear bond strengths of IPS Empress 2 all-ceramic samples, which were luted with Variolink 2 and Resilute dual polymerizing systems. Furthermore, Tubulicid Red caused an increase in the shear bond strength.

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Comparison of Linear Polymerization Shrinkage and Microhardness Between QTH-cured & LED-cured Composites

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Clinical Relevance

The second generation LED is an improvement over the first generation LED. Despite its reported efficiency in composite curing, the second generation LED performs as well as the QTH system for composite curing and does not produce more rapid composite polymerization compared to the QTH unit.

SUMMARY

This study evaluated the effectiveness of second generation light emitting diode (2ndLED) units in composite curing. In order to compare their effectiveness with that of conventional quartz tungsten halogen light curing units (QTH) and first generation LEDs (1stLED), the amount of linear polymerization shrinkage, polymerization speed and microhardness were measured. Linear polymerization shrinkage was measured every 0.5-0.55 seconds for 60 seconds when composite specimens (Z250, 3M ESPE Dental Products, St Paul, MN, USA) were light cured with five different light sources: XL 3000 (QTH, 3M ESPE Dental Products), Elipar FreeLight 2 (2ndLED, 3M ESPE Dental Products), Ultra-Lume LED2 (2ndLED, Ultradent Products, South Jordan, UT, USA), Elipar FreeLight (1stLED, 3M ESPE Dental Products) and experimental product X (1stLED, Biomedisys, Seoul, Korea). The amount of linear polymerization shrinkage in 60 seconds and the speed of polymerization shrinkage in the first 15 seconds were measured for the different lighting units. The amount of polymerization was compared with one-way ANOVA using Tukey at the 95% confidence level. In order to compare the speed of polymerization, the peak time (PT) showing the highest speed of polymerization and maximum speed of polymerization (S_{max}) were determined from the data and compared using one-way ANOVA with Tukey at the 95% confidence level for each material.

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For microhardness measurements, the microhardness of 2-mm composites, Z250, which had been light cured by XL 3000 (G1), FreeLight 2 (G2), Ultra-Lume LED2 (G3), FreeLight (G4) or experimental product X (G5) were compared on the upper and lower surface. The microhardness of each surface was compared between groups using two-way ANOVA with Tukey test at 95% levels of confidence.

The amount of polymerization shrinkage at 60 seconds was G1, G2, G3> G4, G5 ($p<0.05$). PT was G1, G3<G2<G4, G5. S_{max} was G1, G2 > G3 >G4, G5 ($p<0.05$).

On the upper composite surface, there was no difference in microhardness between groups ($p<0.05$).

On the lower surface, the microhardness was G1, G2> G3> G4, G5 ($p<0.05$).

There was no difference in microhardness between the upper and lower surface in G1 and G2; whereas, microhardness of the lower surface was lower in G3, G4 and G5.

It was concluded that 2nd LEDs and the conventional QTH unit cured composites more effectively than 1st LEDs.

INTRODUCTION

Adequate polymerization of a resin composite is considered a very important factor in obtaining adequate physical (Asmussen, 1982) and biological properties (Caughman & others, 1991). It is reported that the power density required for effective polymerization of a composite should be more than 280mW/cm^2 (Rueggeberg, Caughman, & Curtis, 1994). Quartz Tungsten Halogen (QTH) units have been most widely used for curing composites. However, the power density of a QTH system decreases as time passes, and halogen bulbs or filters should be replaced. The problem is that most dentists do not notice the decrease in light-emission if they fail to regularly check the power density of curing lights (Miyazaki & others, 1998; Martin, 1998).

Plasma Arc Curing (PAC) systems have a higher power density than conventional QTH units. They use a high-frequency electrical field to generate plasma energy, and matter is, thereby, transformed into a mixture of ions, electrons and molecules. The large amount of energy released during this process is used for curing photosensitive composites. Although manufacturers recommend that three seconds is sufficient for curing, 12 seconds of light curing is usually necessary for a shallow cavity not exceeding 2 mm (Park, Krejci & Lutz, 2002). In addition, as composites are cured faster in the PAC-cure than the QTH-cure, they may produce more strain in the cavosurface margin (Park & others, 2002). As the light spectrum emitted by the PAC system is narrower (450-470 nm) than the conventional QTH unit, some composites and bonding systems that do not use camphorquinone as a photo-initiator system do not react. The price of the PAC system is also much higher than the QTH unit.

An argon (Ar) laser, which has a more consistent light output over distance, has also been used to cure composites (Blankenau & others, 1991a,b; Kelsey & others, 1989). It emits specific bandwidths of light in ranges of 454 nm to 466 nm, 472 nm to 497 nm and 514 nm. Because it generates little infrared output, not much heat is produced. Even though 10 to 15 seconds is needed to cure composites with an Ar laser, the curing tip is so small that more time is generally needed to cure the restoration. Argon lasers also have narrow spectral outputs, they are expensive, inefficient and occupy too much space, and currently they are not popular (Burgess & others, 2002).

Light emitting diodes (LED) have recently been introduced to cure dental composites. LED curing lights use gallium nitride semiconductors that produce a blue light when subjected to an electrical current. An LED generates a narrower light-output of around 470 nm. As compared with the QTH unit, the LED has considerable merit from the clinician's viewpoint. Regarding its use as a semiconductor for light-emission, the power density of light does not decrease. Therefore, clinicians do not need to be concerned with loss of power density in the curing light. In addition, battery-powered LEDs have excellent battery life, because 1) the power requirements are significantly lower than QTH and PAC lights; 2) LEDs produce little heat during operation, which means that fans are not required to cool the units and 3) LEDs have no moving parts (Burgess & others, 2002). However, it has been reported that the power density of LEDs is so low that they do not sufficiently cure 2-mm composite specimens (Kurachi & others, 2001; Park & others, 2003). The recently introduced second generation LEDs (2nd LED) have a higher power density than first generation LEDs (1st LED).

A linear relationship between light intensity and polymerization contraction has been demonstrated (Sakaguchi & others, 1992). The contraction rate of light-cured composite is highest during the first 30 to 40 seconds of the polymerization reaction (Sakaguchi & others, 1992). This is clinically important, because the integrity of the tooth-composite interface is rapidly challenged during the early phase of polymerization, when the bond between hard tissue and the composite is still maturing. The speed and amount of polymerization shrinkage of a resin composite is easily and accurately measured by a specially designed linometer (de Gee, Feilzer & Davidson, 1993; Park, Krejci & Lutz, 1999, 2002).

In a resin composite, the physical properties are closely related to the degree of conversion and a hardness measurement is an effective way to evaluate the degree of cure (Rueggeberg & Craig, 1988).

This study evaluated the effectiveness of second generation LEDs (2nd LED) for composite curing. To compare its effectiveness with conventional LEDs (1st LED) and QTH units, the amount of linear polymerization shrinkage, speed of polymerization and microhardness of composites were compared.

METHODS AND MATERIALS

A. Measurement of Linear Polymerization Shrinkage

Z250 was used as the test composite. It was transferred to a Teflon mold to ensure that the same amount of composite was used for each linometer sample. The composite was then transferred to the disk in the custom-made linometer, which had been previously coated with a separating glycerin gel. The resin composite was then covered with a glass slide and loaded under constant pressure. The surface of the glass slide facing the composite had also been coated with separating gel. The composites were light cured with QTH, 1st LEDs or 2nd LEDs (Table 1). The power density of each curing unit was measured using a Coltolux Light Meter (Coltene, Altstätten, Switzerland) (Table 1). The tip of the curing light was positioned 2-mm above the slide glass and the specimens were light cured for 60 seconds. As the composite under the glass slide was cured, it shrank towards the light source and the aluminum disk under the composite was moved upward. The amount of disk displacement caused by linear shrinkage of the resin composite was measured using a Linear Variable Differential Transformer (LVDT) linometer (R&B Inc, Daejon, South Korea). The digital data was recorded on a computer for 60 seconds using the Microsoft Excel 2002 program. Park and others (1999, 2002) previously reported the design of the linometer. Fifteen measurements were made for each group and the amount of linear shrinkage occurring over 60 seconds was statistically compared using a one-way ANOVA test, and a Tukey's test was used as a post hoc test at the 95% confidence level.

B. Determination of Polymerization Speed

From the data in A, the peak time (PT) that showed the highest speed of polymerization and maximum speed of polymerization (S_{max}) in the first 15 seconds of curing were determined.

The speed of polymerization at time = t was calculated as follows;

$S_t = (L_{t+\Delta}t - L_{t-\Delta t})/(T_{t+\Delta t}-T_{t-\Delta t})$

S_t: Speed of polymerization shrinkage at time t

$L_{t+\Delta t}$: Amount of linear shrinkage at time $t = t + \Delta t$

$L_{t-\Delta t}$: Amount of linear shrinkage at time $t = t - \Delta t$

$T_{t+\Delta t}$: The time when $L_{t+0.5}$ was measured.

$T_{t-\Delta t}$: The time when $L_{t+0.5}$ was measured.

(Δt was 0.5-0.55s in this study)

PT and S_{max} were compared between groups with one way ANOVA test, using a Tukey's test as a post hoc test at the 95% confidence level.

C. Measurement of Microhardness

A 6-mm diameter hole was made in a 2-mm thick Teflon plate, and a glass slide was positioned along the lower side of the hole. Titanium-coated instruments (Composite Instrument, Coltene, Switzerland) were used to place the Z250 into the mold. The slide glass was placed on top of the composite and pressed flat. The specimens were light cured using QTH, 1st LEDs or 2nd LEDs (Table 1). Ten specimens were assigned to each group. After the composites were light cured, the specimens were removed from the mold. The upper surface (closer to the light source) and lower surface of the specimens were then marked with a pen. The specimens were stored in the dark in 100% humidity at 37°C for seven days. The microhardness of the upper and lower surfaces were then measured with a Vickers hardness-measuring instrument (Optidur, Göttfert Feinwerktechnik GmbH, Buchen, Germany). The microhardness was compared using a two-way ANOVA test and Tukey test to compute statistical significance at the 95% confidence level. In each group, the hardness ratio was also calculated.

Hardness ratio = (Microhardness of lower surface)/(Microhardness of upper surface)

Table 1: *Curing Lights Used in this Study*

Groups	Curing Light	Type	Curing Time (seconds)	Manufacturer	Power Density (mW/cm^2)
1	XL 3000	QTH	60	3M ESPE, St Paul, MN, USA	730
2	Elipar FreeLight2	2nd LED	60	3M ESPE, St Paul, MN, USA	980
3	Ultra Lume LED2	2nd LED	60	Ultradent Products, South Jordan, UT, USA	560
4	Elipar FreeLight	1st LED	60	3M ESPE, St Paul, MN, USA	330
5	X(Experimental Product)	1st LED	60	Biomedisis, Seoul, Korea	310

RESULTS

A. Polymerization Shrinkage

Change in the amount of linear polymerization shrinkage versus time is shown in Figure 1.

Table 2 lists the amount of linear polymerization shrinkage measured in 60 seconds.

The order of the amount of polymerization shrinkage in 60 seconds was G1, 2, 3 > G4, 5 ($p<0.05$).

B. Polymerization Speed

Changes in the speed of polymerization shrinkage versus time are shown in Figure 2.

The PT and S_{max} are listed in Table 3. The order of S_{max} was G1, 2 >G3 > G4, 5 ($p<0.05$). The order of PT was G1, G3 < G2 < G4, 5 ($p<0.05$).

C. Microhardness

In a two-way ANOVA, significant differences in microhardness were observed for curing lights (Group 1 to 5) ($p<0.05$) and observation surfaces (Upper surface or Lower surface) ($p<0.05$). A significant interaction existed between the curing lights and observed surface ($p<0.05$). There was no difference between groups in the microhardness of the upper surface. However, on the lower surface, the order of microhardness was G1, 2 > G3 > G4, 5 ($p<0.05$) (Table 4). There was no difference in microhardness between the upper and lower surface in G1 and G2; whereas, the microhardness of the upper surface was higher than the lower surface in G3, G4, G5 ($p<0.05$) (Table 4).

The hardness ratio was more than 0.8 in all groups (Table 4).

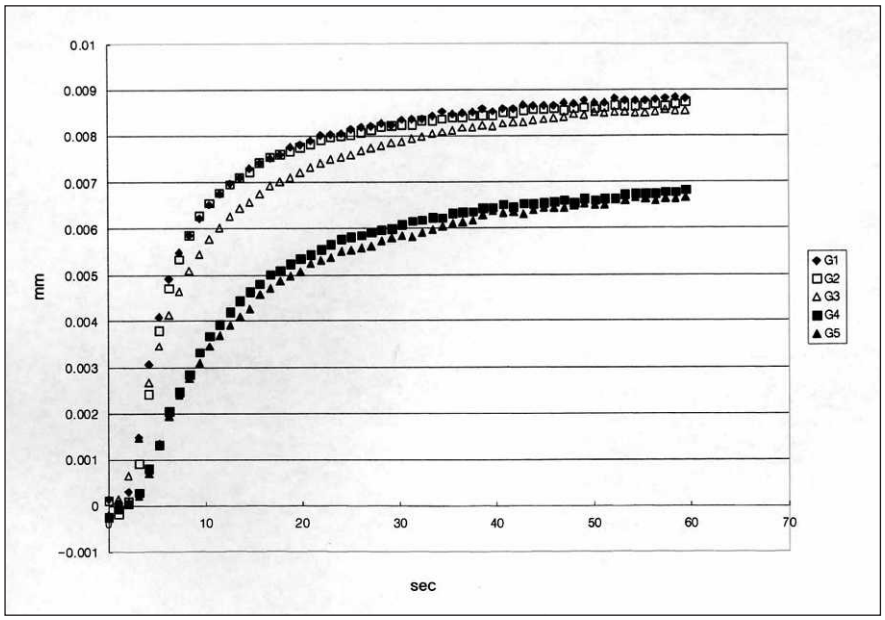

Figure 1: *Change in the amount of linear polymerization shrinkage versus time.*

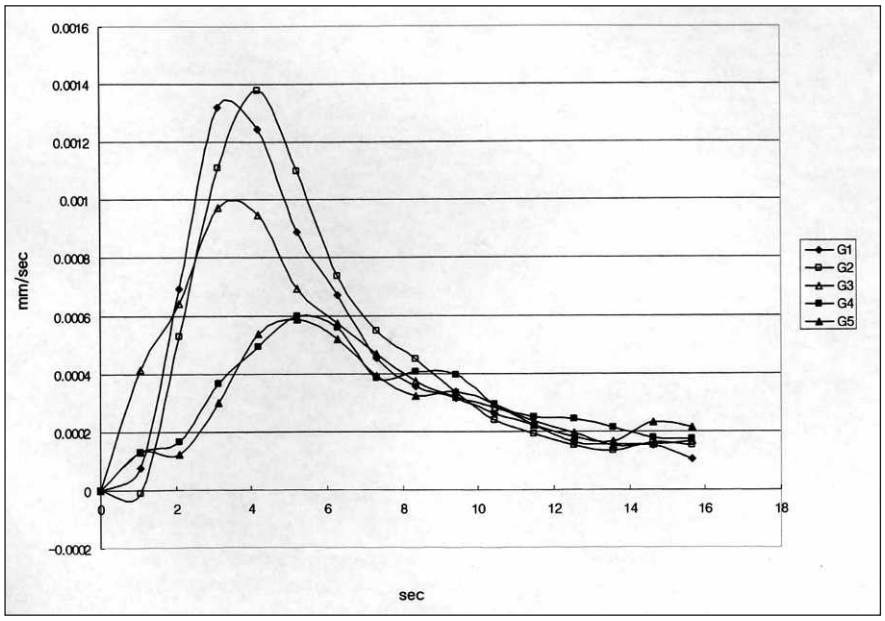

Figure 2: *Change in the speed of polymerization versus time.*

DISCUSSION

Results on the polymerization shrinkage, polymerization speed and microhardness showed that the 2nd LED was an improvement over the 1st LED and has similar performance to a QTH system.

LEDs were reported to be more effective than a QTH lamp in composite polymerization when power densities of both the LED and QTH lamp were adjusted to 300mW/cm^2, because LEDs produced a narrower spectrum of light that fell closely within the absorption range of the camphoroquinone (Mills, Jandt & Ashworth, 1999). When this study was first designed, it was assumed that the polymerization speed of G2 would be much faster than G1, because G2 was an LED and had a higher power density than G1. However, the shrinkage patterns of G1 and G2 were similar (Table 3, Figures 1 and 2). Some factors may be responsible for this discrepancy. First, the efficacy of the LED lamp may not be tied to the composite polymerization. Even though the efficiency of the LED lamp was 31% greater than the QTH lamp, the scrape-back lengths from the composites polymerized using the LED lamp were only 6% higher than those polymerized using the QTH lamp due to the exponential decay of light through the com-

posites (Halvorson, Erickson & Davidson, 2004). Second, the power density value may be incorrect, because a hand-held radiometer that has an opening with a set diameter was used. It has been reported that a radiometer with a fixed opening does not allow for an accurate measurement of power density (Leonard, Charlton & Hilton, 1999). Burgess and others (2002) reported that the power density of XL3000 (G1) and Elipar FreeLight2 (G2) was 750mW/cm^2 and 808 mW/cm^2, respectively, when they were measured using a radiometer that did not have a fixed opening and allowed for an accurate measurement. In this study, the power density was 730mW/cm^2 and 980mW/cm^2, respectively. Another possibility is that this might be a material-specific phenomenon. In Z250, a three-component initiation system (camphorquinone, tertiary amine and Iodonium salt) was introduced to initiate and activate the composite (US patent #5,545,676). Park and others (2002) reported that this initiation system was quite effective in composite polymerization kinetics and induced a more rapid cure with the QTH unit. However, the similarity in the amount of linear polymerization shrinkage and the similar shrinkage patterns between G1 & G2 is currently unclear, because there are many factors that can influence polymerization kinetics. Therefore, further research will be needed.

There was no difference in the amount of linear polymerization shrinkage between G1, G2 and G3 (Table 2, Figure 1). G3 showed a slower shrinkage pattern than G1 and G2 (Table 3, Figure 2), but it achieved the same shrinkage amount in 60 seconds. It has been reported that pre-polymerization at a low power density followed by a final cure at a high power density may allow for increased flow of material and a decrease in polymerization shrinkage stress in a restoration, which may produce a more favorable margin (Mehl, Hickel & Kunzelmann, 1997; Feilzer & others, 1995). G3 showed a slower cure than G1 and G2, but there was no difference in the amount of linear polymerization shrinkage in 60 seconds (Figures 1 and 2). Further research is needed to determine whether G3 provides better marginal adaptation than G1 and G2.

Table 2: *Amount of Linear Polymerization Shrinkage (μm) at 60 Seconds of Light Curing*

Groups	1	2	3	4	5
	8.8(1.1)a	8.7(0.6)a	8.5(0.7)a	6.8(0.8)b	6.6(0.9)b

Different letters indicate different amounts of linear shrinkage at p<0.05 level.
Figures in the parenthesis indicate standard deviations.

Table 3: *Peak Time (PT) That Showed the Highest Speed of Polymerization and Maximum Speed of Polymerization (S_{max})*

	G1	G2	G3	G4	G5
PT(sec)	3.65(0.05)c	4.20(0.03)b	3.65(0.05)c	5.25(0.05)a	5.25(0.03)a
S_{max} (μm/sec)	1.36(0.30)a	1.38(0.30)a	1.00(0.20)b	0.60(0.20)c	0.59(0.25)c

Different letters indicate different amounts of linear shrinkage at p<0.05 level.
Figures in the parenthesis indicate standard deviations.

Table 4: *Microhardness of Upper and Lower Surface*

	G1	G2	G3	G4	G5
Upper surface	81.7(1.8)a	81.3(1.5)a	82.0(1.8)a *	80.5(1.3)a *	80.7(2.7)a *
Lower surface	80.5(2.2)a	80.7(2.5)a	77.7(3.9)b	75.0(1.3)c	74.7(2.8)c
Hardness Ratio	0.98(0.02)	0.99(0.01)	0.95(0.02)	0.93(0.01)	0.93(0.03)

**Indicates different microhardness between upper and lower surface at 95% levels of confidence.*
Different letters indicate different microhardness on the upper or lower surface at the 95% levels of confidence.
Hardness Ratio: (Microhardness of lower surface)/(Microhardness of upper surface)

In this study, there were no differences in the microhardness of upper surfaces between groups. However, the order of microhardness of the lower surfaces was G1, G2 >G3 >G4, 5, and the microhardness of the upper surfaces was higher than the lower surfaces in G3, G4 and G5, whereas, there was no difference in G1 and G2. This is consistent with an article by Kurachi and others (2001). It has been reported that composite curing of a deep cavity layer is considered to be complete if the minimum hardness value is >80% of the maximum value measured on the specimen surface (Lutz, Krejci & Frischknecht, 1992; Breeding, Dixon & Caughman, 1991). Even though microhardness of the lower surface was lower than the upper surface in G3, G4 and G5, the hardness ratio was >0.8 in all groups. Therefore, it is possible that all LED systems used in this study can be used for a 2-mm cavity with 60 seconds of light curing. In a recent study, second generation LED was able to polymerize top and bottom composite surfaces equivalent to or greater than the 40 second-QTH control in only 10 seconds (Rueggeberg, Daronch & de Goes, 2004). Further research will be needed to determine the correct curing depth and curing time.

For Ultra-Lume LED 2 (G3), the power density was 560mW/cm^2. Due to its unique elliptical shape, exact power-density measuring was difficult. Burgess and others (2002) reported that the power density of Ultra-Lume LED2 was 500 mW/cm^2 when measured using a radiometer that did not have a fixed opening and allowed for accurate measurement. According to the manufacturer, the major emission of Ultra-Lume 2 lies

between 445 and 472, and the peak wavelength of the unit is shifted away from a conventional LED: from 468 nm to 459 nm. Such a shift may provide additional energy within the spectral region of some of the alternative photoinitiators on the market. In this study, the PT was G1, 3<G2<G4, 5 (Table 3). It is not yet clear whether the shift in peak wavelength in G3 induced such a fast PT.

As long as camphorquinone is used as the photoinitiator, LED curing lights have adequate cure. However, photoinitiators, such as 1- phenyl-1,2- propanedion (with a peak absorption of 410 nm), bisacylphosphine oxide or triacylphosphine oxide (with peak absorption of 320 nm to 390 nm) may fall outside this range. Since the wavelength of light that LEDs emit range from 440 nm to 490 nm, some bonding agents and composites that use other agents as photo initiators suffered curing problems (Palmer & others, 2004). Recently, a third generation LED, which has a bimodal emission spectrum and covers a broader spectral range, has been released.

The 1st LEDs, G4 & G5, aligned small LEDs of lower power output into arrays. Their power density was much lower than 2nd LEDs, G2 & G3. Ultra-Lume LED 2 has two LEDs that are not arranged in array, but they rely on AC power to produce higher output. Of the LEDs used in this study, FreeLight2 provided the highest power density. According to the manufacturer, the 5W Luxeon LED was introduced and the effectiveness of the emitted light is enhanced by the application of a special reflector that increases coupled light.

CONCLUSIONS

The results of this study show that second generation LED is an improvement over first generation LED and performs as well as a QTH system for composite curing.

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Shear Bond Strength of Tooth-colored Indirect Restorations Bonded to Coronal and Cervical Enamel

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MA Uzzaman • MF Burrow • J Tagami

Clinical Relevance

The cervical enamel region showed lower bond strengths of bonded indirect restoratives than the mid-coronal enamel region.

SUMMARY

This study evaluated the shear bond strength of resin inlays bonded with resin cement to cervical and mid-coronal enamel. Two regions of enamel, cervical and mid-coronal, were chosen from the buccal surface of extracted molars. Composite "inlays" (Estenia, Kuraray Medical Inc) were fabricated indirectly and cemented with a dual-cured resin cement (Panavia Fluoro Cement II, Kuraray Medical Inc). The resin cement was cured with or without light irradiation for 30 seconds. After 24-hours or one-week's storage in 37°C water, the bonded inlays were subjected to a microshear bond test, whereby a shear force was applied to the inlays at a crosshead speed of 1 mm/minute. The data were statistically analyzed using ANOVA and Fisher's PLSD test, with significance defined as $p<0.05$. Observations using confocal laser scanning microscopy were also performed after debonding the specimens. The light-cure method showed significantly higher bond strengths to both enamel regions compared with self-cure, especially at 24 hours ($p<0.05$). However, bond strength of the self-cured resin cement significantly improved after one week's storage ($p<0.05$; cervical enamel: $p=0.022$, mid-coronal enamel: $p=0.0024$). The cervical enamel showed significantly lower bonding than mid-coronal enamel ($p<0.05$), except for the self-cured specimens at 24 hours. Light curing of resin

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cement is a better choice than self-curing for luting of indirect restorations. The bond strength of indirect restorations to cervical enamel was lower than mid-coronal enamel.

INTRODUCTION

Advances in polymer chemistry have led to the development of an indirect adhesive procedure for esthetic restorations. Metal-free inlays, veneers, crowns, resin-bonded fixed prostheses, and even posts are now routinely bonded to tooth structure with resin cement (Burrow & others, 1996; el-Mowafy, Rubo & el-Badrawy, 1999; Christensen, 2000; Shimada, Yamaguchi & Tagami, 2002a; Foxton & others, 2002). On the other hand, evidence of insufficient marginal sealing of bonded restorations has also been shown in microleakage studies (Kubo & others, 2001; Belli & others, 2001; Jayasooriya & others, 2003). Especially, the cervical tooth region/composite interface has repeatedly been reported to be more vulnerable to microleakage than the occlusal or mid-coronal tooth region/composite interface *in vitro* (Kubo & others, 2001).

At the cervical enamel region, an irregular prism arrangement, lacking a normal keyhole appearance, has been frequently observed (Silverstone & others, 1975; Gaspersic, 1995; Shimada, Kikushima & Tagami, 2002b). The structural characteristics of cervical enamel are likely to have important clinical implications, as they may have some influence on the development of dental caries and, particularly, the efficacy of the bonding of restorative materials (Gaspersic, 1995; Shimada & others, 2002b).

Furthermore, although many studies have focused on the bonding of resin cement to enamel, dentin or indirect restorative materials, little is known about the bonding of indirect restorative materials to tooth structure with resin cement (Burrow & others, 1996; el-Mowafy & others, 1999; Christensen, 2000; Furukawa, Inai & Tagami, 2002; Shimada & others, 2002a,b; Foxton & others, 2002). This lack of information, especially onto enamel, is probably due to difficulties with fabricating samples and designing a simple method of testing two adhesive interfaces (one interface between the tooth and resin cement, and the other between the indirect restoration and resin cement).

Recently, a microshear bond test was developed that enabled measurement of the bonding of adhesive materials to a variety of tooth substrates; the method allows for ease of sample preparation and gives precise results with relatively small standard deviations (Shimada & others, 2002a,b).

This study evaluated the bonding of an adhesively luted resin inlay to coronal and cervical enamel. Bond strengths were assessed by means of a microshear bond test and failure mode was observed using a confocal laser scanning microscope (CLSM). CLSM has been widely used in biology for non-invasive and non-destructive imaging *in vivo* of many organ tissues and is convenient for detecting the failure mode of de-bonded surfaces, because an accurate 3-D image with no out-of focus blur can be quickly obtained by making and assembling a series of optical tomograms (Shimada & others, 1999; Jayasooriya & others, 2003; Foxton & others, 2002). Specimens examined by CLSM do not require any special preparation and are not subjected to distortion caused by dehydration, which results from procedures such as scanning electron microscopy. The null hypothesis was that there are no differences in the microshear bond strength of bonded indirect restorations between cervical enamel and mid-coronal enamel.

METHODS AND MATERIALS

Tooth Preparation

Bonding was performed on the buccal surfaces of enamel from extracted human molars stored at 4°C in saline. Forty teeth were used in this study. Before sectioning, two enamel regions obtained from the cervical and mid-coronal regions were chosen as substrates for the microshear bond test. The cervical region was 2 mm above the cemento-enamel junction. Each slice, approximately 1.0-mm thick, was obtained by cutting with a slow rotating diamond blade (Struers Minitom, Struers, Copenhagen, Denmark) under a flow of water. The enamel surfaces were then resurfaced with wet 600 grit SiC paper. Enamel depth was controlled for bond testing by obtaining a sample from near the center, between the dento-enamel junction (DEJ) and the surface.

The enamel surfaces were treated with ED Primer II (Kuraray Medical, Inc, Osaka, Japan, Lot # Liquid A: 000168; Liquid B: 00050) for 30 seconds, then an iris cut from micro bore tygon tubing (R-3603, Norton Performance Plastic Co, Cleveland, OH, USA), with an internal diameter and height approximately 0.7 mm and 0.5 mm, respectively (Figure 1), was mounted on the mid-coronal and cervical regions of the enamel slices to delineate the bonding area.

Preparation of Resin Composite "Inlay"

A cylinder from the same micro bore tygon tubing (R-3603) was cut and used as a mold for the resin composite "inlay" (Figure 1). A resin composite for indirect restorations (Estenia, Shade DA2, Kuraray Medical Inc, Lot #00209B) was placed into the tubing on a flat surface covered with a paper mixing pad; a clear plastic matrix strip was placed over the resin, gently pressed flat and irradiated for 60 seconds using a visible light curing unit for laboratory (α-Light II, J Morita Co,

Tokyo, Japan) prior to heat curing at 110°C for 15 minutes in air (KL 100, Kuraray Medical, Inc). Very small cylinders of resin inlay, approximately 0.7 mm in diameter and 0.5 mm high, were removed from the tygon tubing and silanized with application of a mixture of acidic primer (Clearfil SE Bond Primer, Kuraray Medical, Inc, Lot #00287B) and a silane agent (Clearfil porcelain bond activator, Kuraray Medical, Inc, Lot #00127A) for 20 seconds.

Bonding of Resin Composite Inlay

Forty enamel slices with tygon tubing were divided into two groups of 20 specimens each according to curing method:

Group 1 (self-cure): Dual-cured resin cement (Panavia Fluoro Cement II, Kuraray Medical, Inc, Lot # A paste: T030410, B paste: T030410) was mixed and injected into the tubing on the tooth surface. The small cylindrical resin "inlay" was then inserted into the tubing and air-inhibiting agent was applied to the resin cement borders and placed in darkness at room temperature (23°C).

Group 2 (light-cure): Dual-cured resin cement (Panavia Fluoro cement II) was mixed and injected into the tubing on the tooth surface. The small cylindrical resin "inlay" was inserted into the tubing and light cured (Curing Light XL3000, 3M ESPE, St Paul, MN, USA) for 20 seconds.

In this manner, the small cylindrical resin "inlays" were bonded to the enamel surface; 40 resin "inlays" were bonded to the mid-coronal and cervical regions, respectively. The specimens were stored at room temperature (23°C) for one hour prior to removing the tygon tubing. Each group was further divided into two sub-groups of 10 specimens each, including 10 mid-coronal resin inlays and 10 cervical resin inlays, according to storage periods of 24 hours or one week. All specimens were stored at 37°C.

Microshear Bond Test

The microshear bond test apparatus is shown in Figure 2. The enamel slice with cylindrical resin "inlay" was attached to the testing device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) with a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA), which, in turn, was placed in a Universal testing machine (EZ-test-500N, Shimadzu, Kyoto, Japan) for shear bond testing. A thin wire (diameter: 0.20 mm) was looped around the resin inlay, making contact with half of the cylinder base and the wire was held flush against the resin/tooth interface. A shear force was applied to each specimen at a crosshead speed of 1.0 mm/minute until failure occurred.

Ten specimens from each group were tested. The enamel regions (mid-coronal and cervical region), curing

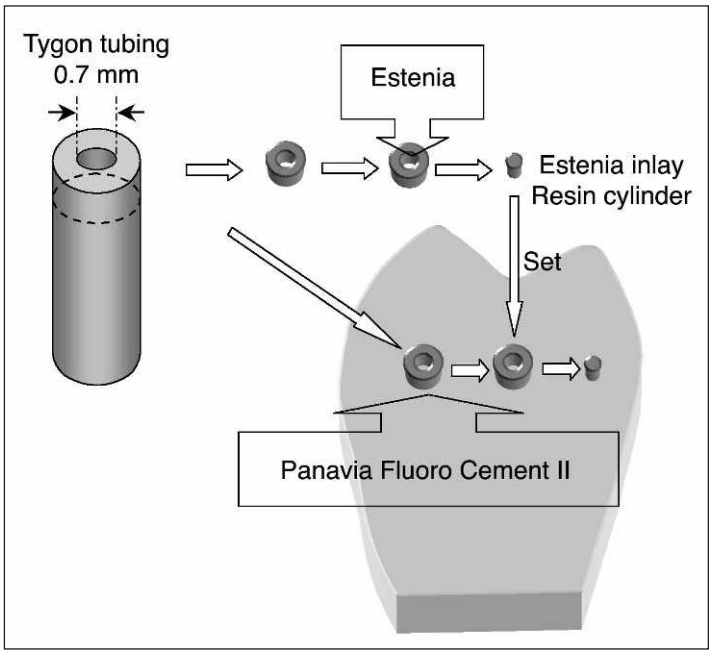

Figure 1. *Bonding procedure for Estenia inlay.*

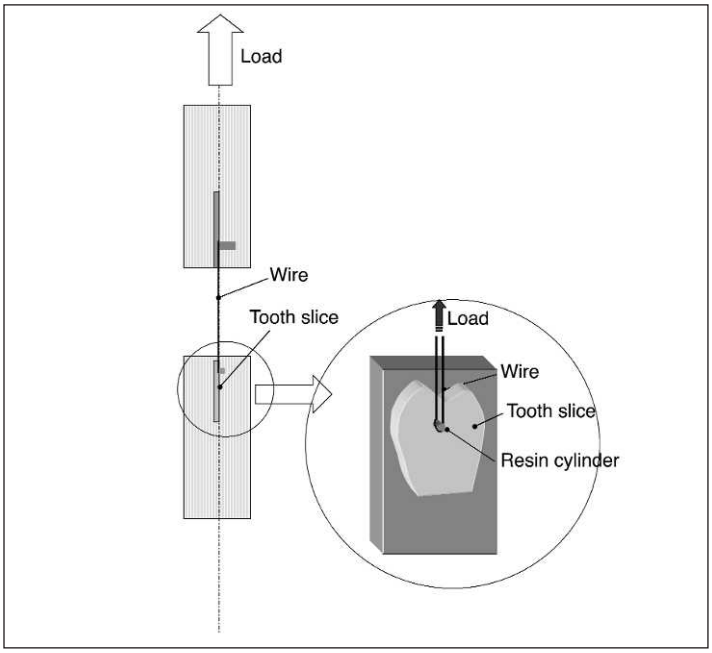

Figure 2. *Schematic diagram of the micro-shear bond test apparatus.*

method (self-cure and light-cure) and storage periods (24 hours and one week) were the three factors analyzed using three-way analysis of variance (ANOVA). After three-way ANOVA, one-way ANOVA and multiple comparisons were carried out using the Fisher's PLSD test. Statistical significance was defined as $p<0.05$.

All of the debonded tooth surfaces were examined with an optical microscope at 30x magnification and CLSM (1LM21H/W, Lasertec Co, Yokohama, Japan) to

determine the mode of failure. Failure modes were categorized into one of six types (Figure 3): T: 100% cohesive failure in the tooth substrate; A1: 100% adhesive failure between the tooth substrate or hybrid-like layer and resin cement; C: 100% cohesive failure in the resin cement; A2; 100% adhesive failure between the resin cement and resin inlay; I: 100% cohesive failure of the resin inlay; M: mixed failure with more than two failures of T, A1, C, A2 and I. The modes of failure were analyzed using the Kruskal-Wallis test, with significance defined as $p<0.05$.

RESULTS

The mean shear bond strength values (MPa), standard deviations and modes of failure are shown in Table 1. The ANOVA indicated that there were statistically significant interactions between the enamel regions and curing method (F=5.597, $p=0.0207$), as well as the curing method and storage periods (F=9.734, $p=0.0026$). However, no significant interaction was observed between the enamel regions or storage periods (F=0.028, $p=0.8685$). Fisher's PLSD test indicated that significant differences existed between the enamel regions ($p<0.0001$), curing method ($p<0.0001$) and storage period ($p<0.0001$).

The light-cured groups showed significantly higher bond strengths at one day than did the self-cured groups (Fisher's PLSD test, $p<0.05$). However, the bond strength of the self-cured groups was significantly improved after storage for one week. Mid-coronal enamel showed significantly higher bond strengths than cervical enamel if the resin cement was light-cured (Fisher's PLSD test, $p<0.05$), whereas, no difference was seen between the self-cured groups (Fisher's PLSD test, $p>0.05$).

CLSM observations revealed different failure patterns between the two curing methods but were not statistically significant (Kruskal-Wallis, $p=0.057$). If the resin cement was self-cured, adhesive failure generally occurred within the resin cement (type C failure) or between the resin cement and enamel (type M failure). The light-cured specimens also showed cohesive failure within the resin cement (type C failure) and adhesive or mixed failure between the resin cement and enamel surface (type A1 or M failure); however, some specimens showed cohesive failure within the enamel (type T or M failure). In both self-cured and light-cured specimens, failure never occurred within the "inlay" material (Table 2).

DISCUSSION

Previous research has shown that chemically initiated polymerization does not enable dual-cure resin cement to develop optimal mechanical properties (Burrow & others, 1996; el-Mowafy & others, 1999; Foxton & others, 2002; Furukawa & others, 2002). In this study, the bond strength of dual-cure resin cement was dependent upon the curing method; the shear bond strength of bonded resin inlay to enamel was high if the resin cement was light-cured. Meanwhile, the bond strength of indirect restoration bonded with self-cured resin cement significantly improved after one week, probably indicating continuation of the curing process of resin cement. In view of the adhesion results, it was interesting to note the mode of failure after the shear bond test. If the resin cement was self-cured, failure frequently occurred within the resin cement or the interface between the cement and enamel, possibly suggesting that the fracture toughness of self-cured cement was lower than enamel or resin inlay. However, light-cured specimens showed a more complex failure pattern, some of which also included enamel fracture within the debonded area. Probably, this complex failure pattern after light activation indicates an improvement in fracture toughness and good bonding to enamel. This change in failure pattern for curing strategies is similar to previous studies: the hardness and bonding of dual-cured resin cement have both been reported to be low under conditions of self-cure alone without light irradiation (el-Mowafy & others, 1999; Foxton & others, 2002).

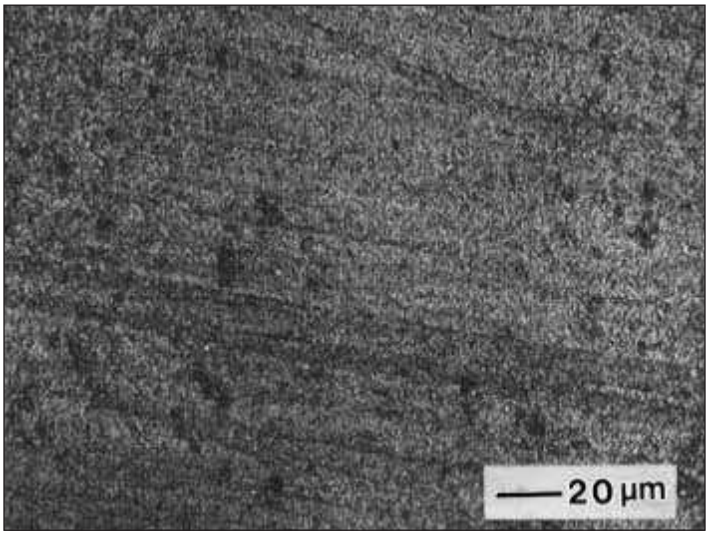

Figure 3. *CLSM image of a debonded surface. Mid-coronal enamel with light-cured cement. Adhesive failure below the bonding resin and enamel can be seen.*

Table 1: *Results of the Microshear Bond Test (MPa, Mean ± SD)*

	Cervical		Mid-coronal	
	Self-cure	**Light-cure**	**Self-cure**	**Light-cure**
1 day	19.6 ± 5.82^{c}	29.1 ± 6.80^{b}	22.6 ± 4.59^{c}	40.3 ± 7.79^{a}
1 week	26.0 ± 4.11^{b,c}	28.7 ± 3.14^{b}	31.2 ± 5.94^{b}	38.6 ± 8.75^{a}

Mean values designated with a different superscript letters were significantly different ($p<0.05$).

Table 2: *Frequency of Mode of Failure*

			n	Failure Mode					
				T	A1	C	A2	I	M
Cervical	Chemical-cure	1 day	10	0	2	3	0	0	5
		1 week	10	0	5	2	0	0	3
	Light-cure	1 day	10	0	7	0	0	0	3
		1 week	10	0	3	1	0	0	6
Mid-coronal	Chemical-cure	1 day	10	0	4	3	0	0	3
		1 week	10	0	3	3	1	0	3
	Light-cure	1 day	10	1	1	3	0	0	5
		1 week	10	0	0	0	0	0	10

Even though the shear bond strength values were still low, the one-week period clearly improved the bond strength of self-cured cement to a level similar to light-cured cement. In the cervical region, this improvement decreased the discrepancy of bonding between the self-cure and light-cure group ($p<0.05$). It is often probable that light-activation of resin cement in the cervical region is not sufficient clinically, especially if the cavity is located in the proximal region. Consequently, this improvement after one week would be expected to show a better clinical outcome of this resin cement in the cervical region. However, neither light curing nor self-curing could create a bond to cervical enamel to the same degree as that of mid-coronal enamel. Additionally, our previous laboratory study evaluating the bonding of directly bonded resin composite showed that both a self-etching adhesive and a total-etch one-bottle adhesive produced 36 to 37 MPa to the cervical enamel region (Shimada & others, 2002b). Clearly, these values were higher than that of adhesively luted indirect restorations obtained in this study. Further study on the regional effect of tooth structure on clinical success seems necessary for both direct and indirect bonded restorations.

In the case of light curing, the intensity of light penetrating through the indirect restoration has been shown to be reduced by thickness and shade (Strydom & others, 2002; Tashiro & others, 2004). Considering these factors, it becomes clear that a curing unit must be able to effectively transmit light through an indirect restoration in order to obtain sufficient hardness and bonding of the resin cement immediately after setting (Strydom, 2002; Foxton & others, 2002; Tashiro & others, 2004). Foxton and others (2002) showed that a 2-mm thick ceramic inlay did not adversely affect the bonding and hardness of light-cured resin cement. This was not a problem in the current study.

With regard to tooth-colored indirect restorations, factors affecting the overall restoration longevity may be related to luting and finishing procedures and may be dependent on the thickness and adhesion of the resin cement (Furukawa & others, 2002; Foxton & others, 2002). Furukawa and others (2002) reported that reinforcement by unification of the tooth substrate and indirect restoration was achieved if bonding of the resin cement to the tooth substrate was adequate. In the instance where the restoration was greater than 2-mm thick or included proximal regions, light activation of the resin cement may be difficult (Foxton & others, 2002). In such clinical cases, a review of the bonded indirect restoration, for instance after one week, may be recommended when bonding of a self-cured resin cement should be stable. Bonding of indirect restorations, especially at the cervical enamel region, was poorer than at the mid-coronal region even after light-curing. Further investigation is necessary to develop bonding resins and cements that produce high, uniform bond strength and/or gap-free margins for indirect restorations.

CONCLUSIONS

The results require that the null hypothesis should be rejected. The cervical enamel region showed poorer bond strengths of bonded indirect restorations than mid-coronal enamel. In spite of the limitations of this laboratory study, light curing of resin cement is the better choice than self-curing alone. Further investigations using clinical trials are necessary.

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Post-gel Polymerization Shrinkage Associated with Different Light Curing Regimens

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Clinical Relevance

The use of pulse delay and soft-start regimens significantly decreased post-gel polymerization shrinkage when compared to standard continuous cure.

SUMMARY

This study compared post-gel polymerization shrinkage associated with five different light curing regimens of similar light energy density. A light-cure unit (VIP, BISCO) that allowed for independent command over time and intensity was used. The five regimens investigated were pulse delay (PD), soft-start (SS); pulse cure (PC), turbo cure (TC) and standard continuous cure (C) [control]. With the exception of TC, the light energy density for all curing regimens was fixed at 16 J/cm². A strain-monitoring device and test configuration were used to measure the linear polymerization shrinkage of 2-mm thick composite specimens (Z100, 3M ESPE) during and post-light polymerization up to 60 minutes. Five samples were made for each curing mode. The results were analyzed using ANOVA/Scheffee's post-hoc test at significance level 0.05. Post-gel shrinkage ranged from 0.30% to 0.46 % at 60 minutes. The use of PD resulted in significantly lower shrinkage compared to PC, TC, SS and C. Shrinkage associated with SS was, in general, significantly lower than C. No significant difference in shrinkage was observed between PC, TC and C at all time intervals. The use of pulse delay and soft-start regimens decreased post-gel polymerization shrinkage.

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INTRODUCTION

Polymerization shrinkage has been a perennial problem with dental composites (Carvalho & others, 1996; Davidson & Feilzer, 1997; Yap & others, 2000; Sakaguchi & others, 1991). Modern composites undergo volumetric polymerization shrinkage of 1% to 5% (Davidson & Feilzer, 1997). Composite shrinkage can be subdivided into pre- and post-gel phases. During pre-gel polymerization, composite flow occurs and stress within the structure is relieved (Davidson & de Gee, 1984). Flow ceases after gelation and cannot compensate for shrinkage stresses. As a result, post-gel polymerization causes significant stresses in the surrounding tooth structure and affects the composite-tooth bond (Feilzer, de Gee & Davidson, 1987). Clinical problems such as post-operative pain, tooth fracture and marginal

debonding that can lead to microleakage and secondary caries may arise due to these stresses (Eick & Welch, 1986; Torstenson & Oden, 1989; Asmussen, 1975).

With the use of controlled polymerization, shrinkage stresses can be minimized by allowing flow to occur during setting. This can be achieved with the application of pulse activation (short pulses of energy) or soft start techniques (pre-polymerization at low intensity light followed by a final cure at high intensity). These curing modes have been shown to result in lower shrinkage, smaller marginal gap, increased marginal integrity and improved material properties (Hofmann & others, 2003; Kanca & Suh, 1999; Mehl, Hickel & Kunzelmann, 1997; Uno & Asmussen, 1991). Some studies have, however, found no significant difference in shrinkage when compared to continuous cure modes (Yap, Soh & Siow, 2002; Yap, Ng & Siow, 2001; Price, Rizkalla & Hall, 2000; Silikas, Eliades & Watts, 2000; Koran & Kürschner, 1998).

Table 1: *Technical Profiles of the Materials Evaluated*

Material/ (shade/Lot #)	Manufacturer	Recommended Curing Parameter	Composition
Z100 (A2/ 20021212)	3M-ESPE, St Paul, MN, USA	400 mW/cm² for 40 seconds	Resins: BIS-GMA, TEGDMA Fillers: Zirconia, silica Filler volume: 66%

BIS-GMA = Bisphenol A-glycidyl methacrylate
TEGDMA = Triethylene glycol dimethacrylate

Table 2: *Technical Profiles of the Materials Evaluated*

Light-curing Mode	Regimen	Light Energy Density
Pulse Delay PD	100 mW/cm² → Delay → 500 mW/cm² (10 seconds) (3 minutes) (30 seconds)	16 J/cm²
Soft Start (SS)	200 mW/cm² → 600 mW/cm² (20 seconds) (20 seconds)	16 J/cm²
Pulse Cure (PC)	400 mW/cm² → Delay → 400 mW/cm² (20 seconds) (20 seconds) (20 seconds)	16 J/cm²
Turbo Cure (TC)	600 mW/cm² (27 seconds)	16.2 J/cm²
Standard Continuous Cure (C)	400 mW/cm² (40 seconds)	16 J/cm²

The apparent discrepancies in findings may be due to differences in the composite material evaluated, curing regimens and testing methods used. In most studies, light energy density (intensity x time) of the curing regimens investigated was not controlled or standardized. Difficulty in controlling light energy density can be attributed to the use of manufacturer determined curing protocols and the fact that most commercial lights do not allow for operator variation of intensity and irradiation time. The polymerization process has been reported to be more dependent on light energy density than light intensity, alone (Yap & Seneviratne, 2001; Miyazaki & others, 1996). It is, therefore, crucial that the former is standardized when evaluating differences between curing regimens. This study compared the post-gel polymerization shrinkage associated with different light curing regimens of similar light energy density.

METHODS AND MATERIALS

A mini-filled resin composite (Z100, 3M-ESPE Dental Products, St Paul, MN, USA) of A2 shade and a commercial light-cure unit that allowed for independent command over time and intensity (Variable Intensity Polymerizer [VIP]; BISCO Inc, Schaumburg, IL, USA) were selected for this study. The technical profile of Z100 is shown in Table 1. VIP has an output wavelength range of 400-500 nm. It is programmed with pre-set exposure times of 2 through 5, 10 and 30 seconds, a continuous mode up to 225 seconds and an intensity setting of 100, 200, 300, 400, 500 and 600 mW/cm². The diameter of the exit window of the light cure tip is 11 mm. Light intensity for each light-curing mode was checked and verified with the in-built radiometer prior to use. Table 2 shows the details of the five light curing modes investigated. The control mode (C) involves light irradiation at 400mW/cm² for 40 seconds. Pulse delay (PD) uses an initial low energy dose (100 mW/cm² for 10 seconds), followed by a waiting time of three minutes and a final cure at high energy dose (500 mW/cm² for 30 seconds). For the soft start (SS) mode, an initial low energy dose (200 mW/cm² for 20 seconds) is followed immediately by a high energy dose (600 mW/cm² for 20 seconds). The pulse cure (PC) mode involves the use of two 400 mW/cm² 20 second pulses with 20 second intervals between them. The total light energy density of these four curing modes was 16 J/cm². Turbo cure (TC) involves irradiation at 600mW/cm² for 27 seconds, giving a total light energy density of 16.2 J/cm², which closely matched the standardized density of 16 J/cm². The sample size for all curing modes was five.

Post-gel polymerization shrinkage was measured using foil electrical resistance strain gauges (foil Strain Gauge, RS Components Ltd, Singapore). The gauges were 2 mm in length and had an electrical resistance of 120Ω and a gauge factor of 2.00. A dia-

grammatic representation of the test configuration for measuring polymerization shrinkage is shown in Figure 1. A glass slide served as the base of the set-up and a stiff, black delrin frame (inner length 7.0 mm, width 4.0 mm and height 2.0 mm) was used to circumscribe the composite sample with the exception of a window for the strain gauge leads. The strain gauges were attached onto the flat glass surface and resin composites were placed in the cavity of the delrin frame with the strain gauge in place. Care was taken to ensure complete filling of the frame, and the excess composite material was extruded by applying pressure through a second glass slide. This was subsequently removed with a plastic instrument. The surface tack of the composite was adequate to ensure adhesion between the strain gauge and the composite materials. The leads from the strain gauge were connected to a strain monitoring device (Strain Gauge Recorder, Cole Parmer Instruments, Vernon Hills, IL, USA) initially balanced at zero. The strain monitoring device consisted of a chart recorder that functions by rationing sense voltage to signal voltage and converting it to analog output. Dimensional changes were effectively transferred to the gauges and measured in terms of resistance.

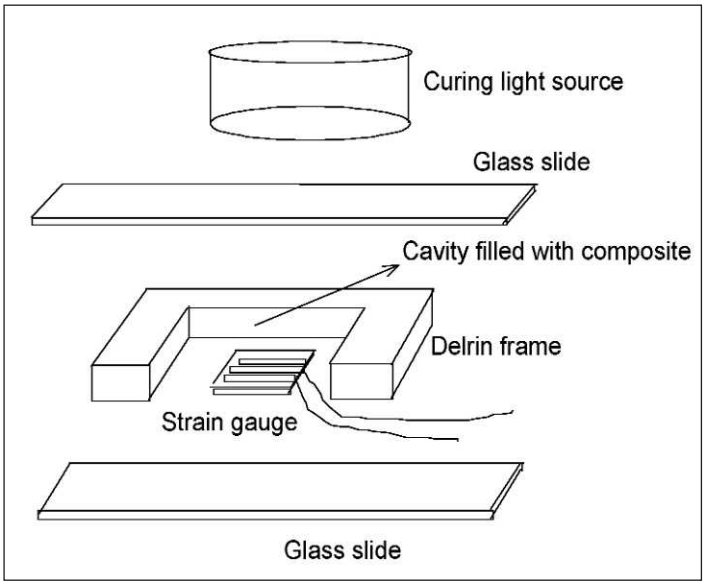

Figure 1. *Set up of the polymerization shrinkage experiment.*

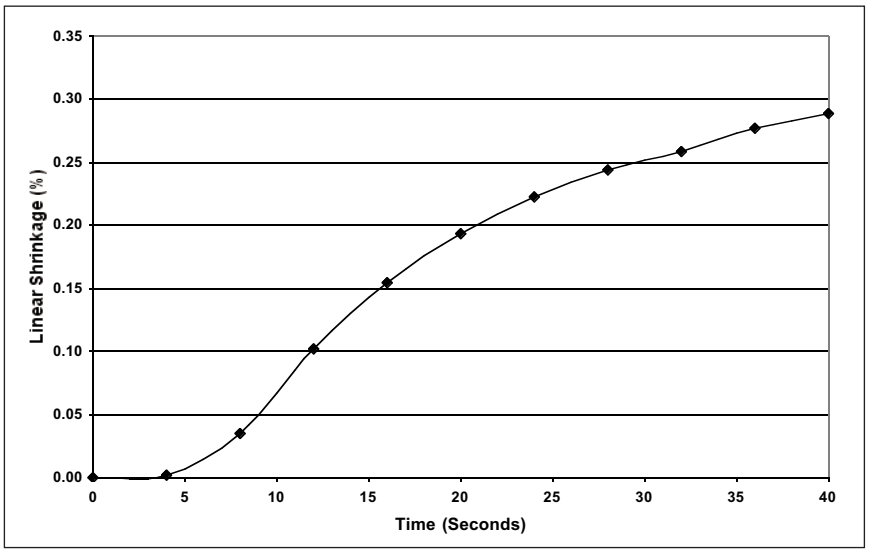

Figure 2. *Mean linear percent shrinkage during light polymerization for the control group.*

The composite specimens were light polymerized using the different cure modes, and the dimensional change during post-light polymerization was monitored in air at room temperature (25 ± 1°C). Post-light polymerization shrinkage measurements were taken at 0 (immediately after light polymerization), 1, 10, 30 and 60 minutes after removing the curing lights. The percentage linear shrinkage was derived from the following equation: Percentage linear shrinkage = ($\Delta L/L$ x 100) = ($\Delta R/R$)/K x 100 where ΔL = Change in length, L = Original length, ΔR = Change of resistance, R = Original resistance and K = Gauge factor (2). Data was subjected to one way ANOVA and Scheffe's post-hoc tests at significance level 0.05.

RESULTS

Figures 2 through 6 show the mean linear percent shrinkage during the light polymerization process. Table 3 and Figure 7 reflect the mean linear percent shrinkage post-light polymerization. The results of statistical analysis are shown in Table 4.

For the control (C) group (Figure 2), shrinkage began at four seconds and increased steeply from 0% to 0.16% between 4 and 16 seconds. This was followed by a more gradual and uniform increase in shrinkage between 16 seconds and 40 seconds. For pulse delay (PD) (Figure 3), no shrinkage was observed during the initial low energy cure and three minutes waiting time. Subsequently, light polymerization at higher energy intensity resulted in a low, gradual increase in shrinkage up from 0% to 0.02% between 132 seconds and 198 seconds. Between 198 seconds and 220 seconds, a steep increase in shrinkage was observed. The shrinkage profile of pulse cure (PC) (Figure 4) during light polymerization differed markedly from PD. Shrinkage associated with the initial cure resulted in progressive shrinkage even during the delay period. The rate of shrinkage increased steeply to 0.2% for the first 30 seconds. For the next 30 seconds, the rate of increase in shrinkage was lower and reached 0.28%. With soft-start (SS) (Figure 5), there was no shrinkage during the initial 12 seconds, but the shrinkage

increased gradually and uniformly to 0.18% during the remaining 28 seconds. For the turbo (TC) regime (Figure 6), there was a uniform increase in shrinkage from 0% to 0.24% between 2.7 seconds and 27 seconds.

Linear shrinkage immediately after light polymerization ranged from 0.12% to 0.29%. Regardless of curing modes, the composite continued to shrink after removal of the light source and linear shrinkage ranged from 0.30% to 0.46% at 60 minutes. At 0 minutes after light polymerization, the use of PD resulted in significantly lower shrinkage than with PC, TC, SS and C, and SS resulted in significantly lower shrinkage than PC and C. At one minute post-light polymerization, the shrinkage associated with PD was significantly less than PC, TC, SS and C. The results at 10, 30 and 60 minutes were identical. At 0, 10 and 60 minutes, SS had significantly lower shrinkage than C.

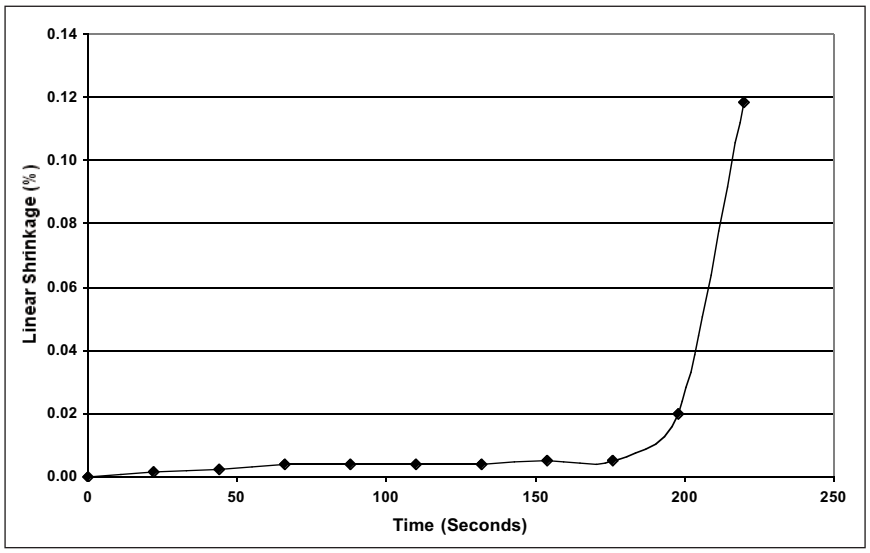

Figure 3. *Mean linear percent shrinkage during light polymerization with the pulse delay mode.*

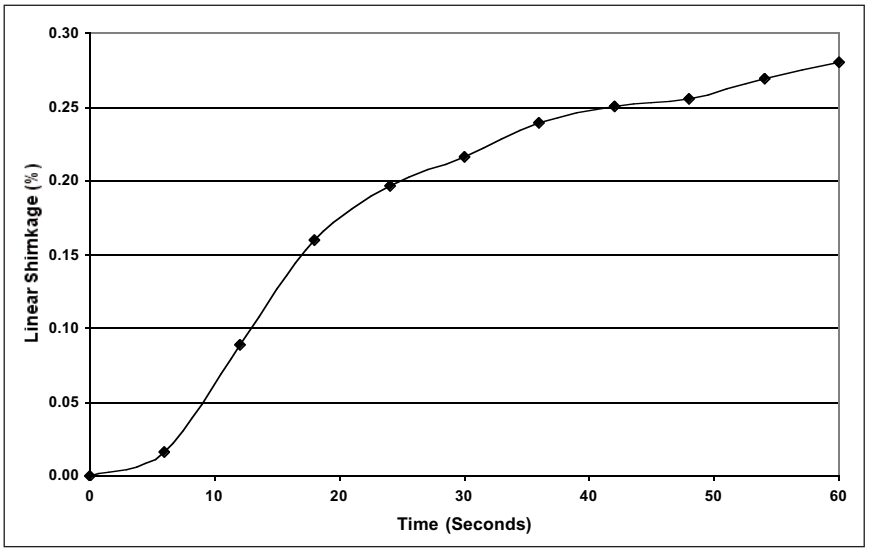

Figure 4. *Mean linear percent shrinkage during light polymerization with the pulse cure mode.*

DISCUSSION

Many methods and techniques have been employed to determine polymerization shrinkage. They range from dilatometer methods, specific gravity methods and deflecting disc systems to optical methods. The linear strain gauge method was selected, as it provides real-time measurement of the curing process and a means of studying the kinetics of polymerization (Sakaguchi & others, 1991). Z100 was chosen, as it exhibited large contraction stresses during post-gel shrinkage (Versluis & others, 1993). Shade A2 was used to reduce the effects of colorants on light polymerization (Bayne, Heymann & Swift, 1994), and 2-mm thick composite specimens were used to ensure uniform and maximum polymerization (Yap & others, 2000). BISCO VIP was selected, as its intensity and curing time can be easily manipulated to obtain similar light energy density for the different cure modes.

There are many factors influencing the transmission of light, including the thickness of the restorative material, the presence and size of filler particles, the shade of the restorative material and the distance of the light tip to the

Table 3: *Mean Linear Percent Shrinkage (SD) for the Different Light-curing Modes*

Time (minutes)	0	1	10	30	60
Control	0.29 (0.05)	0.36 (0.04)	0.41 (0.04)	0.43 (0.04)	0.46 (0.02)
Pulse Delay	0.12 (0.04)	0.21 (0.03)	0.26 (0.02)	0.28 (0.02)	0.30 (0.02)
Pulse Cure	0.28 (0.05)	0.33 (0.05)	0.38 (0.04)	0.40 (0.05)	0.41 (0.05)
Soft Start	0.18 (0.01)	0.30 (0.02)	0.34 (0.02)	0.37 (0.03)	0.39 (0.02)
Turbo	0.24 (0.03)	0.32 (0.02)	0.36 (0.02)	0.39 (0.02)	0.41 (0.03)

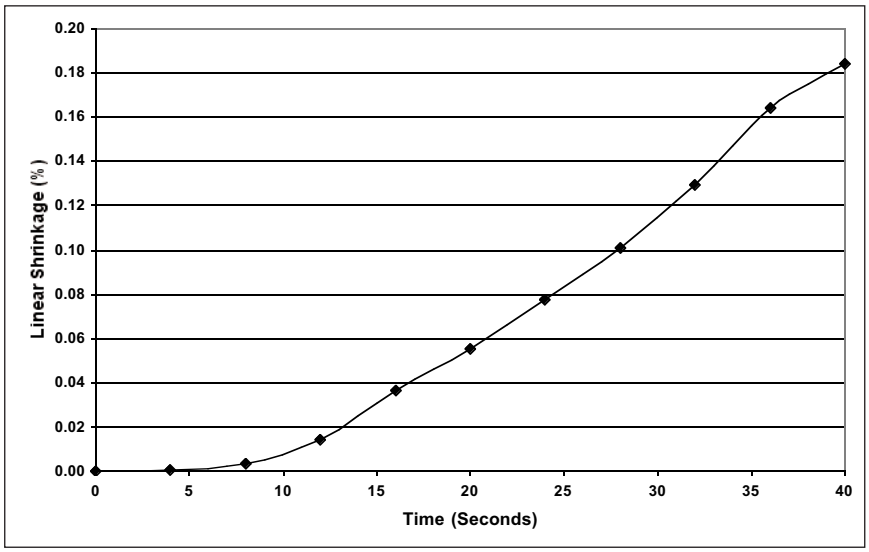

Figure 5. *Mean linear percent shrinkage during light polymerization with the soft start mode.*

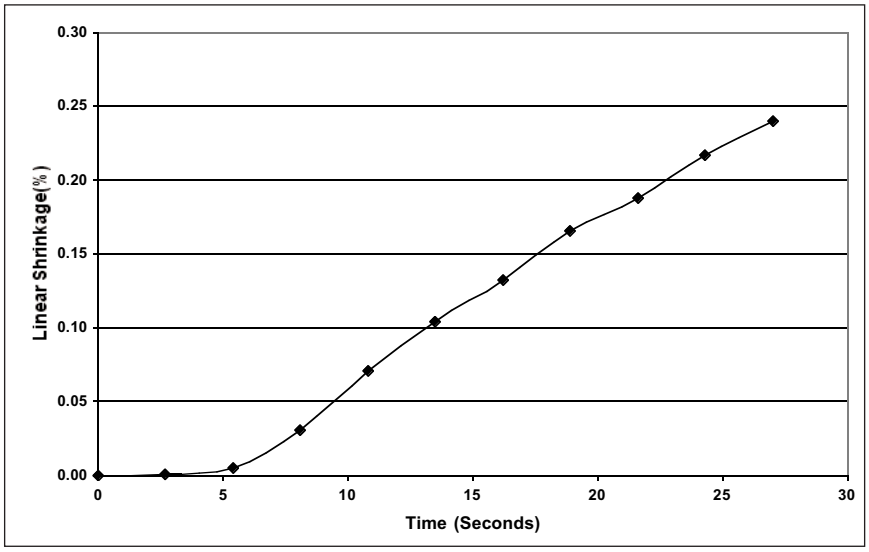

Figure 6. *Mean linear percent shrinkage during light polymerization with the turbo cure mode.*

Table 4: *Comparison of Mean Liner Percentage Shrinkage at the Various Time Intervals*

Time (minutes)	Significance
0	PD < PC, T, C SS < PC, C
1	PD < PC, SS, T, C
10	PD < PC, SS, T, C SS < C
30	PD < PC, SS, T, C
60	PD < PC, SS, T, C SS < C

< denotes statistically significant differences in mean linear percentage shrinkage (results of one way ANOVA/Scheffe's post-hoc test [p<0.05])

restoration surface (Tate, Porter & Dosch, 1999). Light energy density (intensity x time) indirectly affects polymerization shrinkage. Although greater light energy density results in a more effective cure, it also results in higher polymerization shrinkage (Sakaguchi, Douglas & Peter, 1992). As all these factors were standardized in this study, any reduction in polymerization shrinkage may be attributed to the light-curing regimen used. A minimum light intensity of 400mW/cm^2 was suggested for routine polymerization (Rueggeberg, Caughman & Curtis, 1994; Manga, Charlton & Wakefield, 1995). This light intensity, together with the manufacturer's recommended cure time of 40 seconds, was used as the control. With the exception of the turbo cure, the total light energy was kept at 16 J/cm^2. Light energy for the turbo cure was slightly greater at 16.2 J/cm^2. This was primarily due to technical constraints. Volumetric curing contraction determinations (Attin & others, 1995; Rueggeberg & Tamareselvy, 1995) are non-specific quantitative shrinkage measurements and constitute the total (pre- and post-gel) curing contractions. Dimensional changes in linear curing contraction determinations employed in this and other studies (de Gee, Feilzer & Davidson, 1993; Uno & Shimokobe, 1994) are, however, more or less "hindered" and should be regarded as a post-gel curing phenomenon. As displacement transducers (strain gauges) are activated by force, they can only monitor and measure post-gel polymerization shrinkage when the composite is sufficiently strong to exert forces (Davidson & Feilzer, 1997).

The rate of shrinkage during light polymerization in the control group was not uniform. Shrinkage was very rapid during the initial 16 seconds and decreased during the latter 24 seconds of the 40-second cycle. Soft-start polymerization, pulse activation or a combination of these curing modes could reduce the high initial shrinkage by allowing composite flow to occur during this period. The pulse-delay mode is a combination of pulse activation and soft-start polymerization. No linear shrinkage was detected during the initial low energy cure and the three-minute waiting period for pulse delay mode. This phenomenon may be accounted for by two possible hypotheses: either the light energy density (intensity x time) was insufficient to initiate polymerization or only pre-gel polymerization occurred. As stated earlier, the latter will not be

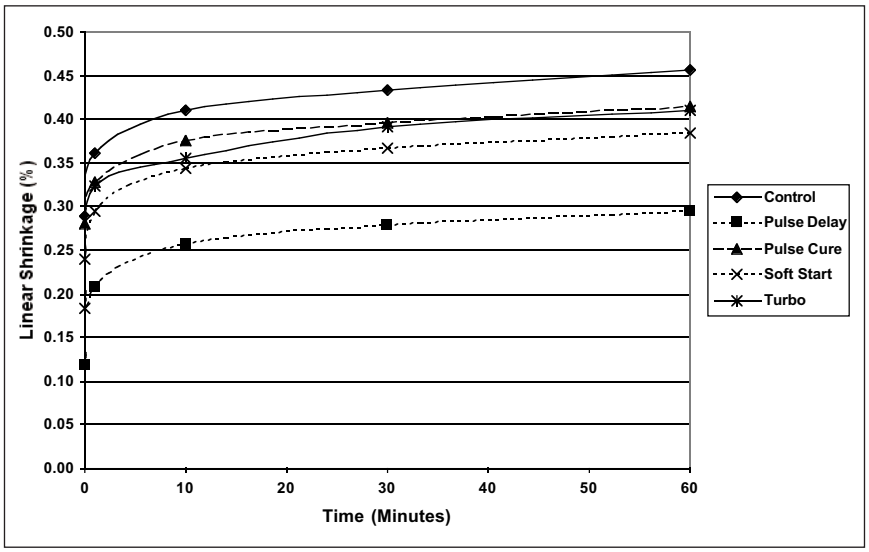

Figure 7. *Mean linear percent shrinkage after light curing for the various modes.*

detected by strain gauges. The initial use of low-light energy density must, however, be compensated for by a final cure at high light energy density (Yap & Seneviratne, 2001) to ensure optimal physico-mechanical properties and prevent clinical problems caused by the cytotoxicity of inadequately polymerized materials (Caughman & others, 1991). Post-gel shrinkage of pulse delay immediately after light polymerization was significantly lower than pulse cure, turbo and the control. As VIP only had a maximum intensity of 600 mW/cm^2, an initial 20-second cure of 200 mW/cm^2 and a final 20-second cure at 600mW/cm^2 were selected to characterize soft-start polymerization and to maintain the light energy density at 16 J/cm^2. Shrinkage immediately after light polymerization was significantly lower than pulse cure and control. Although soft-start had a higher shrinkage compared to pulse delay, the difference was statistically insignificant.

Resin composites continue to shrink after removal of the light source as a result of composite post-curing. Polymerization is approximately 75% complete at 10 minutes after light exposure and curing continues for at least 24 hours (O'Brien, 1997). Thermal contraction due to the loss of radiant heat can also contribute to the initial post-light polymerization shrinkage. At all time intervals, pulse delay was found to have significantly lower post-gel shrinkage compared to all other curing regimens. This can be explained by the use of both a low initial light energy density and a relatively long delay period that allowed for a prolonged period of the pre-gel state. Soft-start polymerization generally resulted in lower post-gel shrinkage than the control. Some studies found that soft-start polymerization significantly reduced post-gel shrinkage (Dennison & others, 2000; Sakaguchi & Berge, 1998). Others reported no significant difference in post-gel shrinkage between soft-start and continuous cure (Koran & Kürschner, 1998; Silikas & others, 2000; Yap & others, 2002). The difference can be explained by the fact that the use of initial lower light intensities was not compensated for by a final cure at higher intensities in the former studies. Commercially available soft-start polymerization light-cure units usually use a final cure of 500 mW/cm^2 or greater. The beneficial effect of the initial low intensity cure may therefore be annulled by the high intensity final cure. The results of this study have, however, shown that when light energy density is kept constant, the use of soft-start curing can reduce polymerization shrinkage.

CONCLUSIONS

Under the conditions of this *in vitro* study:

1. Post-gel shrinkage immediately after light polymerization ranged from 0.12% to 0.29% for pulse delay and continuous cure, respectively.
2. Composites continue to shrink after light curing, and shrinkage at 60 minutes post-polymerization ranged from 0.3% to 0.46% for pulse delay and continuous cure, respectively.
3. At all time intervals, the use of pulse delay resulted in significantly lower post-gel polymerization shrinkage compared to continuous, pulse and turbo cure.
4. Soft-start generally exhibited significantly lower post-gel shrinkage than continuous cure.

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Microscopic Evaluation of Dentin Interface Obtained with 10 Contemporary Self-etching Systems: Correlation with Their pH

G Grégoire • A Millas

Clinical Relevance

The morphology of the dentin/self-etch bonding system/composite interface enables a good seal to be obtained with most self-etching adhesives currently in use. Clinically, this may have an influence on the risk of post-operative sensitivity. Most acid primers did not systematically provide the best interfaces.

SUMMARY

This study investigated micromorphological differences in the hybridized complex formed using 10 commercially available self-etch bonding systems. In addition, the influence of the pH of the primer of these adhesives was evaluated. The self-etching systems tested were AdheSE, Adper Prompt L-Pop, Clearfil SE Bond, Etch&Prime 3.0 (Degussa, Germany), Prime & Bond NT Non Rinse Conditioner (Dentsply, Konstanz, Germany), One-Up Bond F, OptiBond Solo Plus Self Etch, Prompt L-Pop and Xeno III.

One hundred non-carious human third molars were used. The teeth were divided into two groups of 50 and prepared for evaluation by optical microscopy or scanning electron microscopy. The specimens in each group were further divided into 10 subgroups of five specimens each to evaluate the 10 bonding systems. The pH of the primers of the bonding systems was measured. The results demonstrated morphological differences at the interface, depending on adhesive composition. The differences mainly concerned thickness of the hybrid layer, the absence or presence of microscopic voids at the adhesive-composite interface and whether the dentinal tubuli were completely sealed. The pH was not the determining factor conditioning the action of the self-etching adhesives.

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INTRODUCTION

Most early bonding systems were hydrophobic, which did not allow them to adapt to dentin properly. The adhesive must be able to diffuse and penetrate in an

aqueous environment and, therefore, be hydrophilic or amphiphilic. Amphiphilic monomers contain bifunctional, hydrophilic and hydrophobic groups with a great affinity for both dentin and restoration material (Van Dijken & Horstedt, 1998). The non-rinsing self-etch adhesives, designed to bond with dentin covered with a smear layer, contain higher concentrations of acid monomers, often mixed with water, to make the adhesive systems sufficiently acid to cross the smear layer and form a bond with the underlying dentin. They incorporate the smear layer in the hybrid layer (Tay & Pashley, 2001; Van Meerbeek & others, 2003). This intended utilization assumes that the acidic resin monomers will penetrate beyond the smear layer into mineralized dentin (Watanabe, Nakabayashi & Pashley, 1994).

The self-etching adhesives vary in their acidity by virtue of the composition and concentration of polymerizable acids and acidic resin monomers in these systems (Pashley & Tay, 2001). Water is also an essential component, as it provides hydrogen ions that are indispensable for demineralization of the smear layer and hard tissues. Acidic resin monomers are used as both the etchant and priming resins, thus reducing the likelihood of incomplete resin infiltration within the partially demineralized dentin (Kwong & others, 2002; Kiyomura, 1987).

The component parts of self-etch bonding systems are not all new: phosphonates have already been used as enamel or dentin adhesives (Anbar & Farley, 1974). However, the aim at that time was for to the resins to chelate with the Ca. The acids were therefore neutralized to increase their chelating power and thus reduced their etching capacity (Tay & Pashley, 2001). The new adhesives include polymerizable acid monomers with phosphoric acid functional groups such as MDP or phenyl-P, and the concentration of these monomers is high (Tani & others, 1993; Hayakawa, Kikutake & Nemoto, 1998). According to Watanabe and others (1994), the etching potential of self-etch bonding systems induces the formation of a hybridized complex comprising a surface zone made up of the hybridized smear layer with a true hybrid layer underneath.

This research used optical and scanning electron microscopy to investigate the interface obtained between 10 self-etch bonding systems and dentin. The objectives of this microscopy study were to examine how effective the self-etching systems were in penetrating the dentin smear layer and to produce hybrid layers within the subsurface dentin; that is, to form a hybridized complex. The primary null hypothesis tested was that the pH of self-etch adhesives plays a major role in the formation of the hybrid layer and resin tags and influences the morphology of the dentin-adhesive-composite interfaces. The secondary null hypothesis was that the topography of the interface would be practically identical for all the self-etch bonding systems.

METHODS AND MATERIALS

One hundred non-carious human third molars were used within one month following extraction. They were stored at 4°C in a 1% chloramine T solution. The teeth were equally divided into two groups and prepared for evaluation with optical microscopy (Grégoire, Akon & Millas, 2002) (n=50) or with scanning electron microscopy (n=50) (Perdigão & others, 1996). The specimens in each group were further divided into 10 subgroups of five specimens each to evaluate 10 different adhesives (Table 1). Dentin disks were cut from crown segments parallel to the occlusal surface at the top of the pulp chamber (Tagami, Tao & Pashley, 1990; Camps & others, 1997).

Using a low-speed saw (Isomet 2000, Buehler Ltd, Evanston, IL, USA) equipped with a rotating diamond-impregnated copper disk (11-4244-15 HC, Buehler Ltd) under water spray, a fine smear layer was obtained which was reproducible on all specimens. The dentin specimens were considered to be deep dentin, an area where the number and diameter of the tubuli increase (Fogel, Marshall & Pashley, 1988). Deep dentin was chosen to favor diffusion of the dentin bonding system in demineralized dentin, thus providing the best conditions for the resin-dentin interaction mechanism.

The adhesive was applied to each group following the manufacturer's instructions (Table 2). The same composite, Esthet X (Vivadent), was applied to each specimen and photopolymerized for 20 seconds.

The pH of each adhesive was measured four times using a Bench pH Meters 210 microchip (Hanna Instruments, Woonsoket, RI, USA) with a Hanna Instruments HI 1083 B electrode. The mean and standard deviation were calculated. (Table 3).

Specimen Preparation for Optical Microscopy

Five dentin disks from each group were prepared for optical microscopy. They were fixed in 10% formaldehyde (Rhône-Poulenc Ltd, Manchester, England) for two days, demineralized with 5% trichloracetic acid (Merck, Darmstadt, Germany) for five days, then rinsed in water for two hours. The specimens were dehydrated in ascending grades of alcohol and subsequently cleared in toluene for 30 minutes. The specimens were impregnated with paraffin (Merck) for two days, then embedded in paraffin and 5 µm sections were cut with a microtome. The sections were stained with crystal violet and picric acid.

Observations were made by two blind study operators who worked independently using the same optical microscope (020-507-010 Wild Leitz GmbH, Heerbrugg, Switzerland). When in disagreement, the investigators

Table 1: *Bonding Products Tested*

Products Tested	Manufacturer	Groups	Classification	Flacon 1: Batch #	Flacon 2: Batch #	Composition
AdheSE	Vivadent Schaan, Liechtenstein	I	Two-step self-etch	E 70925 F 39363		Primer: Phosphoric acid acrylate, Bis-acrylic acid amine, water, initiators, stabilizers Bonding: HEMA, highly dispersed silicone dioxide, initiators, stabilizers
Adper Prompt L-Pop	3M-ESPE Seefeld, Germany	II	One-step self-etch	20021002 158821		Mono and di HEMA phosphates, Bis-GMA, Camphor quinone, Substituted aromatic amine, substituted, aminobenzoate, phosphine oxide, stabilizer
Clearfil SE Bond	Kuraray Osaka, Japan	III	Two-step self-etch	00112B 00114B 0051A 0053A		Primer: HEMA, MDP, Hydrophilic dimethacrylate, water, Ethanol, dl-camphorquinone, N, N-Diethanol-p-toluidine Adhesive: HEMA, MDP, Bis-GMA, Hydrophilic dimethacrylate, dl-camphorquinone, N,N-Diethanol-p-toluidine, Silanated colloidal silica
Etch & PrimeE 3.0	Degussa-Hüls AG Hanau, Germany	IV	One-step self-etch	30000155	30000155	Universal: HEMA, ethanol, distilled water, stabilizers Catalyst: HEMA, Pyro-phosphate, initiators, stabilizers
I Bond	Heraeus Kulzer, NY, USA	V	One-step self-etch	010024		UDMA, 4-META, acetone, water, Glutaraldehyde, camphorquinone
Non Rinse Conditioner/ Prime&Bond NT	Dentsply Konstanz, Germany	VI	Two-step self-etch	9901000169 0101100058	0007324 0005216	NRC: Itaconic acid, maleic acid, water NT: Di- and trimethacrylate resin, Functional silica, PENTA , Cetylamine hydrofluoride, acetone, stabilizers, initiators, silica nanofiller
One-Up Bond F	Tokuyama Corp, Tokyo, Japan	VII	One-step self-etch	N482461E		Bonding agent A: Phosphoric monomer Propanedioic acid, 2-methyl-1-oxo-2-propenyl oxy decyl, Bis-GMA, multi functional methacrylic monomers co-initiator Bonding agent B: HEMA monofunctional monomers, silicate filler, dye-sensitizer, borate derivative, water
Optibond Solo Plus Self-Etch	SDS Kerr Glendora, CA, USA	VIII	Two-step self-etch	2051872	21168	HEMA, Bis GMA, GDM, TS 530, OX , Barium borosilicate, sodium hexafluorosilicate, ethanol
Prompt L-Pop	3M-ESPE, Seefeld, Germany	IX	One-step self-etch	FW0056206 L666136		Liquid 1: Methacrylic phosphates, initiators, stabilizers Liquid 2: Fluorine complex, stabilizers, water

Table 1: *Bonding Products Tested (cont.)*

Products Tested	Manufacturer	Groups	Classification	Flacon 1: Batch #	Flacon 2: Batch #	Composition
Xeno III	Dentsply Konstanz, Germany	X	One-step self-etch	360-02 0206001237	360-05	Universal: HEMA, purified water, ethanol, 2,6-di-tert-butyl-p-cresol Catalyst: Tetra-methacryl-ethyl-pyrophosphate, Penta-methacryl-oxy-ethyl-cyclo-phosphazen-mono-fluoride, 1,6 dimethacryl-ethyl-oxy-carbonyl amino-trimethyl hexan, 2,6-di-tert-butyl-p-cresol, 2,3-bornanedione, P-dimethyl amine ethyl benzoate

HEMA: Hydroxy ethyl methacrylate
PENTA: Dipentaerythritol penta acrylate monophosphate
GDM: Glycidyl dimethacrylate
MDP: 10-methacryloyloxydecyl dihydrogen phosphate
Bis-GMA: Bis-phenol A diglycidylemethacrylate

Table 2: *Application Techniques of the Self-etch Adhesives Tested*

Products Tested	Application Procedure
AdheSE Vivadent, Schaan, Liechtenstein	Apply primer for 10 seconds, massaging gently, briefly air-dry. The total reaction time should not be shorter than 30 seconds Apply bonding and allow to act for 10 seconds, briefly air-dry and light activate for 10 seconds
Adper Prompt L-Pop 3M-ESPE St Paul, MN, USA	Activate blister pack by emptying the liquid out of the red blister into the yellow blister Brush the adhesive onto the entire dentin surface, massage it in for 15 seconds, apply pressure, this time cannot be shortened Use a gentle stream of air to thoroughly dry the adhesive to a thin film Harden the adhesive with a halogen light for 10 seconds
Clearfil SE Bond Kuraray, Japan	Apply the Primer and allow to act for 20 seconds Briefly air-dry Apply the bond, spread with a light air blow and light activate for 10 seconds
Etch&Prime 3.0 Degussa, Germany	Mix equal amounts of the universal and catalyst Apply to tooth substrates and allow to act for 30 seconds, with no rinsing, Briefly air-dry, light-activate for 10 seconds Re-apply adhesive mixture, briefly air-dry and light-activate for 10 seconds
I Bond Heraeus-Kulzer, USA	Apply a copious amount. Apply two additional coats. Wait for 30 seconds. Use a gentle stream of air. Light activate for 20 seconds
Non Rinse Conditioner Prime & Bond NT Dentsply, Konstanz, Germany	Apply NRC on slightly moist dentin 20 seconds Briefly air-dry while keeping slightly moist Apply NT for 30 seconds, no rinsing, briefly air-dry, light activate for 20 seconds
One-Up Bond F Tokuyama, Japan	Mix equal droplets of the bonding agents A (clear liquid) and B (bright yellow liquid) until a pink, homogenous liquid mixture is obtained Apply to tooth substrates and leave undisturbed for 20 seconds, no rinsing—without further air-drying, light-activate for 10 seconds or 15 seconds
OptiBond Solo Kerr, USA	Apply self etch primer to the enamel/dentin surface with brushing motion for 15 seconds Air thin for 3 seconds Shake OptiBond Solo Plus bottle briefly. Apply OptiBond Solo Plus to the dentin surface with light brushing motion for 15 seconds Air thin for 3 seconds Reapply OptiBond Solo Plus with light brushing motion for 15 seconds Air thin for 3 seconds Light cure for 20 seconds
Prompt L-Pop 3M-ESPE, St Paul, MN, USA	Activate blister pack by emptying the liquid out of the red blister into the yellow blister Apply activated mixture to tooth substrates with shaking for 15 seconds, no rinsing Briefly air-dry, light-activate for 10 seconds, using only halogen light-curing unit
Xeno III Dentsply, Konstanz, Germany	Mix equal amounts of the universal and catalyst Apply to tooth substrates and allow to act for at least 20 seconds, no rinsing Briefly air-dry, light-activate for 10 seconds

Table 3: *pH Values of Adhesives*

Products Tested	Groups	Classification	pH Primer
AdheSE Vivadent, Schaan, Liechtenstein	I	Two-step self-etch	1.69 ± 0.10
Adper Prompt L-Pop 3M-ESPE, Seefeld Germany	II	One-step self-etch	0.35 ± 0.10
Clearfil SE Bond Kuraray, Osaka, Japan	III	Two-step self-etch	1.90 ± 0.10
Etch & Prime 3.0 Degussa-Huls AG, Hanau, Germany	IV	One-step self-etch	0.76 ± 0.10
I Bond Heraeus, Kulzer, NY, USA	V	One-step self-etch	1.77 ± 0.02
Non Rinse Conditioner Prime & Bond NT Dentsply, Konstanz, Germany	VI	Two-step self-etch	1.15 ± 0.08
One-Up Bond F Tokuyama Corp, Tokyo, Japan	VII	One-step self-etch	1.09 ± 0.10
Opti Bond Solo Plus Self-Etch SDS Kerr, Glendora, CA, USA	VIII	Two-step self-etch	1.10 ± 0.07
Prompt L-Pop 3M-ESPE, Seefeld, Germany	IX	One-step self-etch	0.70 ± 0.10
Xeno III Dentsply, Konstanz, Germany	X	One-step self-etch	0.98 ± 0.1

Values are mean ± SD

examined the specimens together and came to a consensus.

For each specimen in the 10 groups, observations and microphotographs of the hybrid layer, resin tags and adhesive lateral branches were made at intervals to illustrate the representative features. To standardize the observations, microphotographs of the adhesive system were taken at the original magnification (320x).

Specimen Preparation for Scanning Electron Microscopy

Five dentin disks from each group were prepared for SEM. They were split in two, decalcified for 20 seconds in 36% phosphoric acid and deproteinized for 60 seconds in 2% sodium hypochlorite.

The specimens were fixed in a solution of sodium cacodylate containing 2.5% glutaraldehyde. The fixative was rinsed and the specimens dehydrated in ascending grades of alcohol, up to 100%. The specimens were dried by immersion in pure hexamethyldisilazane (HMDS) for 20 minutes. This treatment was preferred over critical point drying, because it protects the collagen network better (Perdigão & others, 1996). The HMDS was allowed to evaporate for 15 minutes in air before the specimens were sputter-coated in gold palladium.

The specimens were examined with a SEM (S-450, Hitachi, Tokyo, Japan) using an acceleration voltage of 20 KV. The structures analyzed were the architecture of the interface, the thickness of the hybrid layer and the morphology of the resin tags. The specimens were evaluated double blind by two calibrated operators. For each specimen in the 10 groups, observations and microphotographs of the hybrid layer, resin tags and adhesive lateral branches were made at intervals to illustrate representative aspects. To standardize the microscopic observations, microphotographs of the interface between the adhesive resin and dentin substrate were taken at magnifications of 2000x, 2500x and 3000x.

RESULTS

Table 3 shows the pH values of the adhesives, including the means and standard deviations.

The adhesives can be divided into three groups for classification purposes: those with a pH <1 (Adper Prompt L-Pop [3M-ESPE, St Paul, MN, USA], Prompt L-Pop, Xeno III [Dentsply, Konstanz, Germany] and Etch and Prime), those with a pH between 1 and 1.50 (OptiBond Solo Plus [Kerr Corporation, Orange, CA, USA], One-Up Bond F [Tokuyama Corp, Tokyo, Japan] and Prime and Bond NT) and those with a pH between 1.50 and 2 (AdheSE, Clearfil SE Bond [Kuraray, Japan] and I Bond [Heraeus-Kulzer, Armonk, NY, USA]).

The structure observed varied not only from specimen to specimen, but also from one area to another within a given specimen. An objective description of all observations was recorded.

Interface Structure Observed by Scanning Electron Microscopy

When reporting the SEM results, the term "hybridized complex" will be used, rather than referring to a resin-impregnated smear layer and true hybrid layer. It is difficult, if not impossible, to see the difference with the SEM as there is no clear boundary between the two. In a bonding system with a phosphoric acid pre-etch, the demineralizing etchant is rinsed away, thus

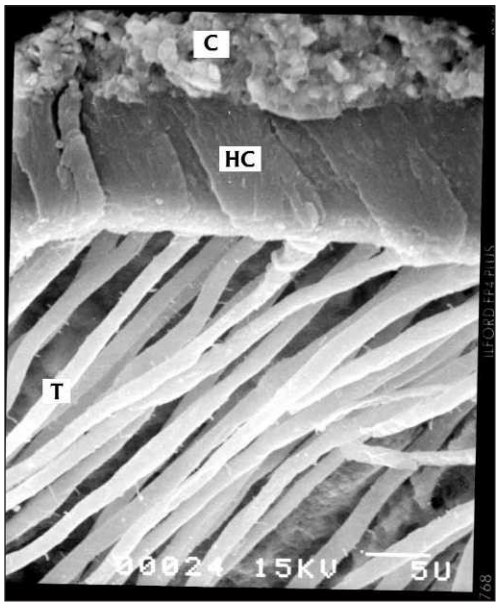

Figure 1. *SEM micrograph showing the adhesive-dentin interface with AdheSE after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl. A conical swelling can be observed. Note the thick, uniform hybridized complex and the numerous tags with conical swellings at their junction with the hybrid layer (original magnification 2500x).*

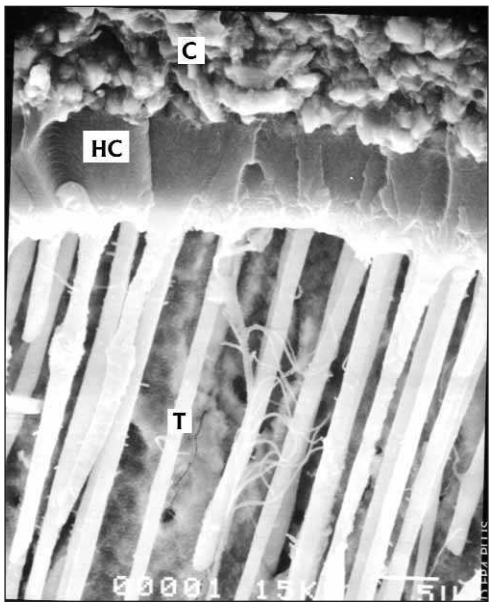

Figure 2. *SEM micrograph showing the adhesive-dentin interface with Adper Prompt L-Pop after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl. A thick hybridized complex with numerous tags can be seen (original magnification 2500x).*

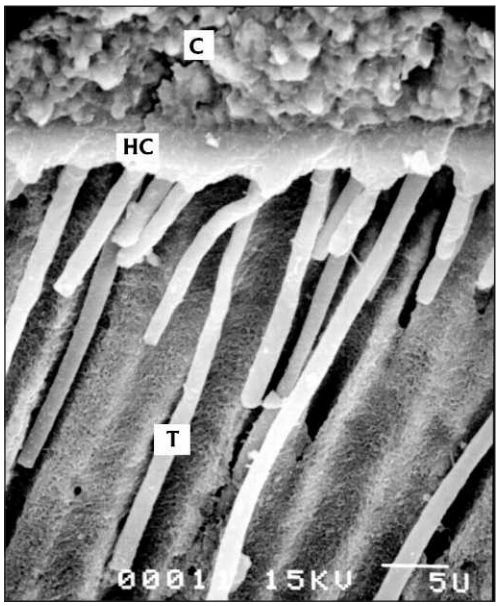

Figure 3. *SEM micrograph showing the adhesive-dentin interface with Clearfil SE Bond after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl (original magnification 2500x).*

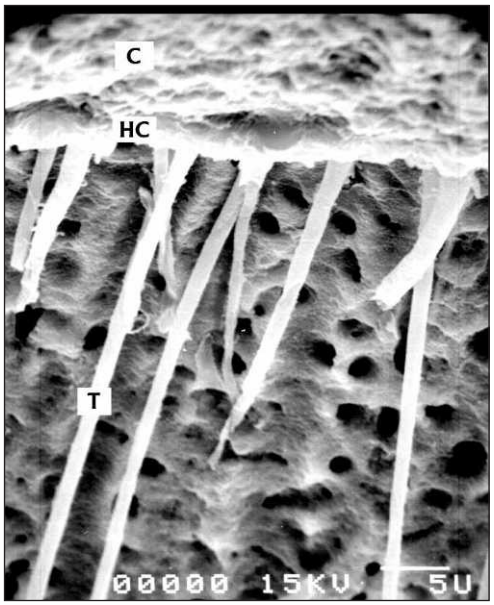

Figure 4. *SEM micrograph showing the adhesive-dentin interface with Etch & Prime 3.0 after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl. A blister can be observed between the adhesive and the composite (original magnification 2500x).*

eliminating the calcium phosphates. The self-etch systems are not rinsed and the dissolved compounds remain where they are. From the depth of the dentin to the outside, there is a gradient in the concentration of demineralized products and pH. The hybridized complex is made up of calcium salts, phosphates, collagen, water and adhesive resin. The ions released by the acid of the primer are trapped in a polymer structure. Thus, there are no distinct areas; instead, there is a complex zone characteristic of each system.

The interface obtained in Group I had a thick hybridized complex (1 to 11 µm thick), very long tags (up to 100 µm) with small side branches and a conical swelling at the base (Figure 1). For Group II, the hybridized complex was about 6 µm thick. Tags were numerous and fine, some reaching 80 µm long. Conical swellings could be distinguished at the base of the hybridized complex (Figure 2). The interface in Group III showed a hybridized complex varying between 2 and 3 µm in thickness with tags of 5 to 30 µm. They were thicker at their base and, thus, formed plugs (Figure 3). In Group IV, the hybridized complex was about 1 µm thick. At the interface between the hybridized complex and the restoration composite it was possible to see fairly frequent voids, which Tay and others (2002) refer to as blisters (Figure 4). The interface obtained in Group V showed a thin, irregular hybridized complex, having a thickness of about 1 µm. The fine tags, often widely spaced and

leaving numerous tubuli empty, were 60 to 70 µm long (Figure 5). For Group VI, the hybridized complex formed was about 3 µm thick. All the tags were attached to the hybridized complex by large conical swellings. A few side branches could also be seen. The tags were 60 to 70 µm long (Figure 6). In Group VII, a hybridized complex, 2 to 3 µm thick, was observed, with a few fine tags up to 40 µm long, often broken (Figure 7). The Group VIII interface showed a regular hybridized complex about 9 µm thick. The numerous tags, which were about 40 µm long, had side branches (Figure 8). In Group IX, the hybridized complex was continuous and about 3.5 µm thick. The long, thin tags reached up to 60 µm (Figure 9). In Group X, a regular, continuous hybridized complex 5 to 8 µm thick was observed. The tags were numerous and dense, sometimes reaching 50 to 70 µm in length. Side branches were seen. Contact between the hybrid layer and tags was very good and formed a kind of swelling. Such conical shapes of the upper part of the tags ensure a good seal as the hybrid layer extends into the walls of the dentin tubuli, leading to hybridization of the walls (Figure 10). This structure was also noted with NRC NT and AdheSE (Vivadent, Schaan, Liechtenstein). Van Meerbeek and others (2001) consider this interpenetration as more important than the length of the tags.

Interface Structure Observed Under Optical Microscopy

The stains used in this experiment colored the resin bright pink and the dentin

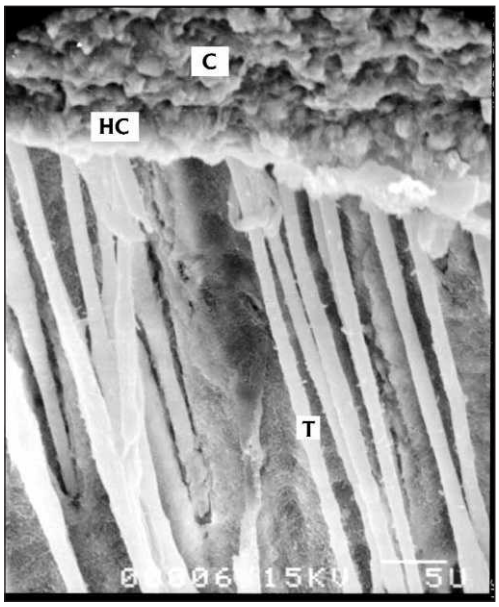

Figure 5. *SEM micrograph showing the adhesive-dentin interface with I Bond after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl. The hybridized complex is thin (original magnification 2500x).*

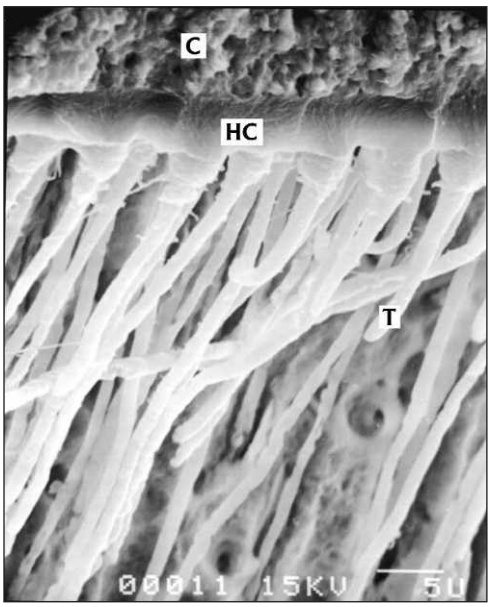

Figure 6. *SEM micrograph showing the adhesive-dentin interface with Non Rinse Conditioner Prime&Bond NT after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl. Tags are numerous and systematically show marked conical swellings at their bases, which is the sign of a good seal (original magnification 2500x).*

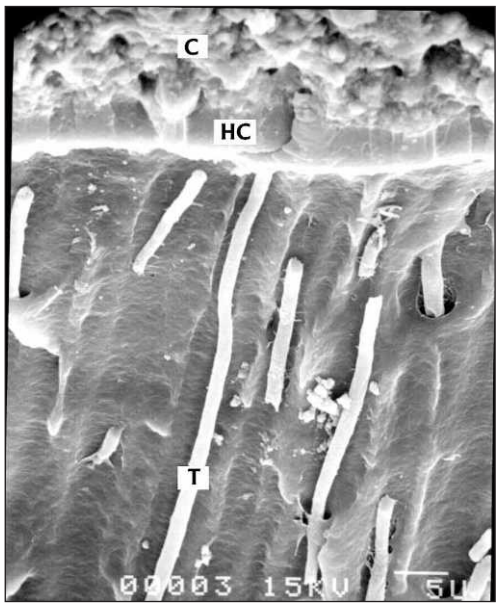

Figure 7. *SEM micrograph showing the adhesive-dentin interface with One-Up Bond F after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl. Particularly noteworthy is the small number of tags that often seem not to be well attached to the hybrid layer (original magnification 2000x).*

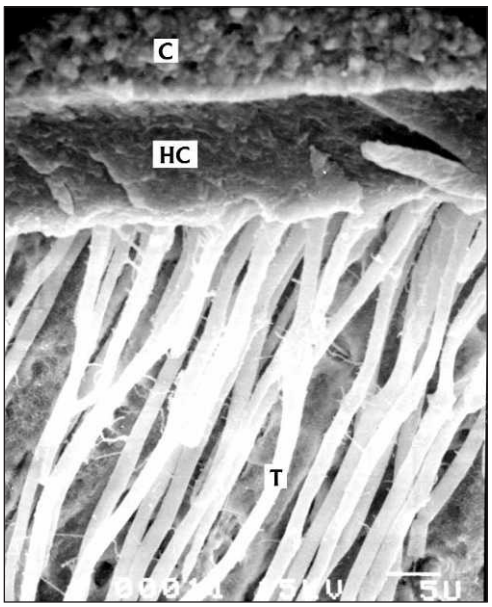

Figure 8. *SEM micrograph showing the adhesive-dentin interface with Opti Bond Solo Plus after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl. Note the thick hybridized complex and very numerous tags (original magnification 2000x).*

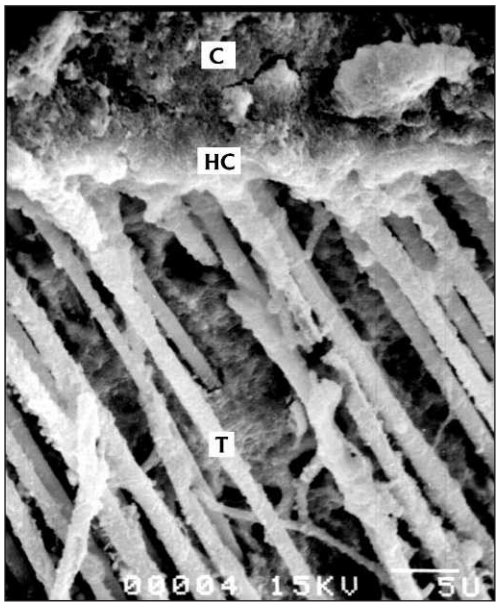

Figure 9. *SEM micrograph showing the adhesive-dentin interface with Prompt L-Pop after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl. Hybridized complex 3.5 to 4 mm thick. Tags are fairly numerous (original magnification 2500x).*

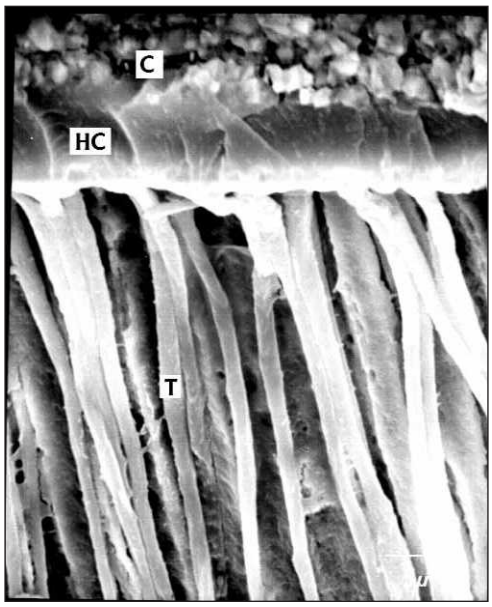

Figure 10. *SEM micrograph showing the adhesive-dentin interface with Xeno III after demineralization with 36% H_3PO_4 and deproteinization with 2% Na OCl. The hybridized complex is thick and dense. Numerous tags (original magnification 3000x).*

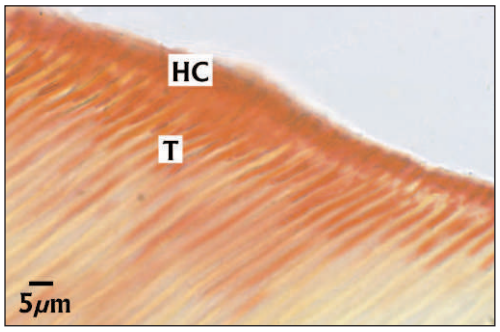

Figure 11. *Optical micrograph showing the adhesive-dentin interface with AdheSE. The hybridized complex is thick with numerous tags. V-shaped plugs are visible at the tubuli mouths (original magnification 320x).*

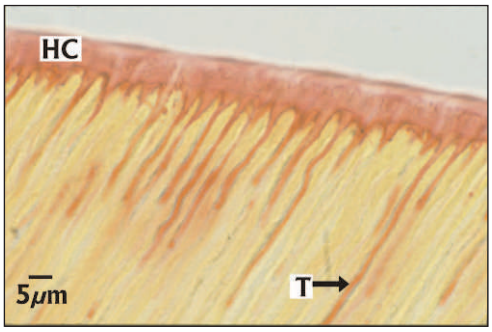

Figure 12. *Optical micrograph showing the adhesive-dentin interface with Adper Prompt L-Pop. V-shaped tags are visible (original magnification 320x).*

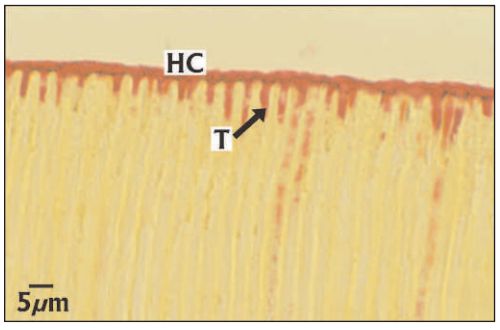

Figure 13. *Optical micrograph showing the adhesive-dentin interface with Clearfil SE Bond. Irregular tags are visible. Some longer ones are not homogeneous and do not seem to be in continuity with the hybridized complex (original magnification 320x).*

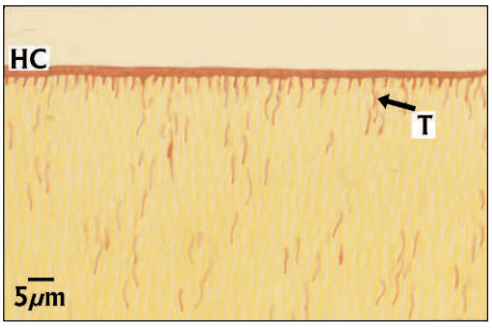

Figure 14. *Optical micrograph showing the adhesive-dentin interface with Etch & Prime 3.0. The resin sometimes penetrates fairly deeply into the tubuli but is not in contact with the hybridized complex (original magnification 320x).*

yellow, thus, allowing the boundaries between the two to be determined accurately. The contrast was such that differences were clear, even in black and white photographs. The composite could not be observed, because it fell out during the technical preparations for optical microscopy.

With Group I, the hybridized complex was regular, and numerous tags blocked the tubuli mouths with V-shaped plugs (Figure 11). Group II formed a continuous hybridized complex. The numerous V-shaped tags sealed the tubuli mouths well (Figure 12). Group III had a continuous hybrid complex. The tags were often short and conical (Figure 13). The hybridized complex of Group IV varied in thickness. Fine tags were observed, sometimes broken, which did not block the tubuli (Figure 14). In Group V, the hybridized complex was not uniform. Tag formation was irregular and the tubuli did not seem to be perfectly sealed, as gaps could be seen between the tags and the tubuli walls (Figure 15). Group VI had a continuous hybridized complex, with V-shaped tags blocking the tubuli mouths (Figure 16). In Group VII, the hybridized complex was regular but the tags were not in contact with the walls of the tubuli (Figure 17). Group VIII gave a regular hybridized complex, with tags that were not in contact with the tubuli walls (Figure 18).

For Group IX, the hybridized complex was continuous and the tags long and conical, apparently blocking off the tubuli (Figure 19). Finally, the

hybridized complex of Group X was regular. All the tubuli were filled with long, fine tags (Figure 20).

DISCUSSION

The two observational methods used, optical and scanning electron microscopy, proved to be complementary. The optical microscope gave a very clear image of the hybrid layer and resin tags with respect to dentin structures not impregnated with resin. The variation in color intensity allowed the homogeneity of the hybrid layer to be assessed. Since the technique required complete demineralization of hard dental tissues, there was no possibility of composite examination, which prevented any study of the hybrid layer/composite junction. With the scanning electron microscope, advantages of the staining disappeared, but the structures and their relationships could be viewed on a different scale.

It was easy to compare the morphological differences (hybridization and tags) produced by using 10 different bonding systems.

The pH values of the adhesive systems measured in this study enabled three groups to be distinguished among the adhesives: those with a pH well below 1, adhesives having a pH close to 1 and those for which the pH was between 1.5 and 2. These differences had been noted by Tay and Pashley (2001) and Van Meerbeek and others (2003), with few variants. Adper Prompt L-Pop, Etch and Prime 3.0 and Prompt L-Pop have a pH lower than 1 (see Table 3). Great differences were noticeable in the thickness of the hybridized complex and in the density of the tags. Adper Prompt L-Pop gave a thicker, uniform hybridized complex, corresponding to deeper demineralization. Numerous tags joined the hybrid layer at a conical swelling, showing good penetration of resin into the tubuli walls. With Etch and Prime 3.0, many ovoid zones of separation between the adhesive and composite were observed. These separation zones, resembling blisters, were found systematically on all the Etch and Prime 3.0 specimens but did not exist for any other adhesive. The same technique was

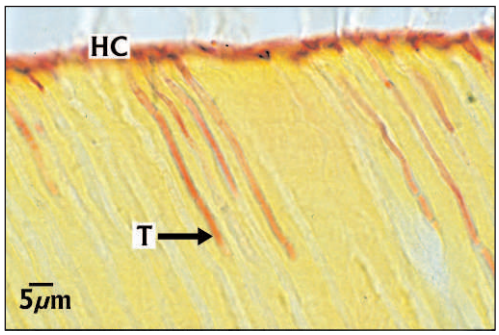

Figure 15. *Optical micrograph showing the adhesive-dentin interface with I Bond. Note the non-uniform hybridized complex (original magnification 320x).*

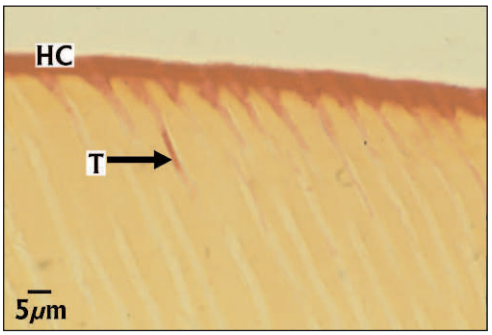

Figure 16. *Optical micrograph showing the adhesive-dentin interface with Non Rinse Conditioner Prime&Bond NT. V-shaped plugs are visible (original magnification 320x).*

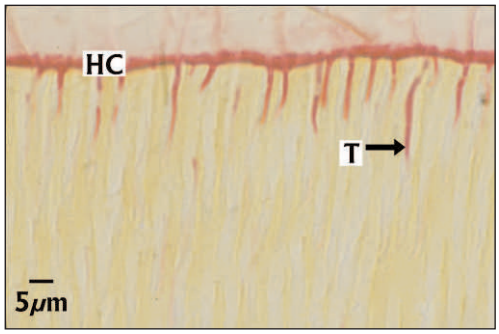

Figure 17. *Optical micrograph showing the adhesive-dentin interface with One-Up Bond F. The tags are not very numerous and most do not adhere to the tubuli walls (original magnification 320x).*

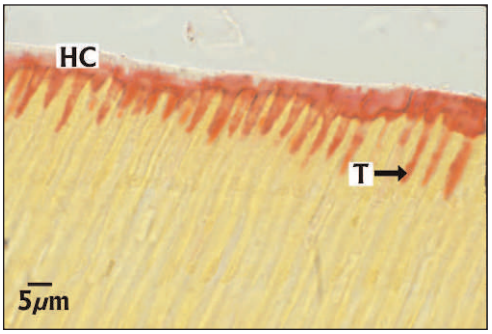

Figure 18. *Optical micrograph showing the adhesive-dentin interface with Opti Bond Solo Plus. Note the numerous V-shaped tags (original magnification 320x).*

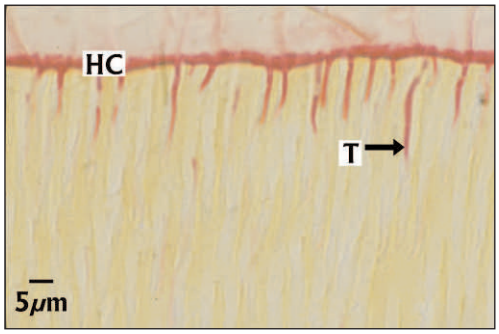

Figure 19. *Optical micrograph showing the adhesive-dentin interface with Prompt L-Pop. Numerous fine tags are visible (original magnification 320x).*

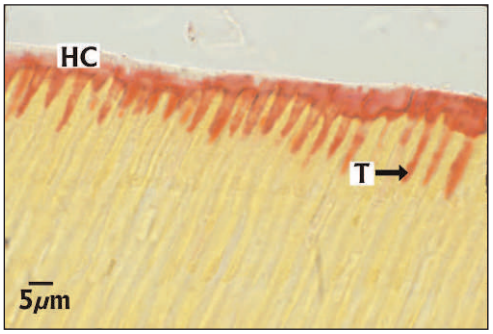

Figure 20. *Optical micrograph showing the adhesive-dentin interface with Xeno III. The hybridized complex is thick and continuous. Tags are numerous and thick, with V-shaped tag—hybridized complex junction (original magnification 320x).*

used for all specimens, so that the hypothesis that light-cured composite may not have been activated immediately, thus leading to formation of blisters, can be set aside (Tay, Pashley & Peters, 2003). Tay and Pashley (2001) studied the interface of Prompt L-Pop using transmission electron microscopy and demonstrated the existence of a true hybrid layer having a thickness of 3.5 to 4.0 mm. The same value was also found in the current study. The optical microscope revealed the regularity of this layer, proving that demineralization and penetration had been homogeneous. Non Rinse Conditioner (NRC), OneUp Bond F, OptiBond Solo Plus Self-etch and Xeno III have a pH close to 1. Xeno III is a one-step, while the other three are two-step systems. NRC, OptiBond Solo Plus Self-etch and Xeno III gave hybridized complexes of different thicknesses (the thinnest obtained with NRC), but which appeared to be homogeneous. The tags were numerous and had marked conical swellings at their bases, which is the sign of a good seal. The acid monomers penetrated the fine smear layer that was formed using the diamond-impregnated disk and demineralized the dentinal sub-surface and the mouths of the dentin tubuli. The results of this demineralization were the obtention of resin funnels making a good junction between the tag and the hybrid layer. With One Up Bond F, demineralization was not obtained down to the dentinal sub-surface and tubuli mouths. The junction between the hybrid layer and tags was very thin, and sometimes the bond was absent. The other three bonding systems tested, AdheSE, Clearfil SE Bond and I Bond had a pH between 1.7 and 1.9. Van Meerbeek and others (2003) classes Xeno III and I Bond as "Intermediary strong" self-etch adhesives and states that their resultant interfacial interaction is consequently expected to be similar. In this study, I Bond gave a hybridized complex that was much thinner than Xeno III and was thinner than AdheSE, which has a similar pH. In the three bonding systems that have a pH between 1.7 and 1.9, AdheSE provided a thick, uniform hybridized complex and numerous thick tags with swellings at their junction with the hybrid layer, proving that the zone had been correctly demineralized.

Under the conditions of this study, the pH grouping of the 10 systems tested did not correspond to groups with similar bonding. Different interface morphologies occurred with the same pH, leading to rejection of the first null hypothesis.

Thus, pH, alone, cannot explain the differences in action. Demineralizing power depends on several factors: pH, the dissociation constant (pKa), the structure of the compounds, which may be more or less chelating, solubility of the salts formed and the application time. Action depends on the acid monomers and their concentration.

Xeno III contains a phosphoester (a substituted phosphoric acid with two acid functions). This phosphoester is a strong acid, which explains the product's low pH. In addition to the acid monomer, its composition includes a pyrophosphate function (tetra-methacryl-ethyl-pyrophosphate), a hydrophobic chain (trimethyl-hexane) and a prepolymer (tetra-methacryl). It is a very amphiphilic product. Pyrophosphate is a chelating phosphate for calcium, which increases the demineralizing power of the product. Adding a hydrophobic chain to the acid components is intended to keep the calcium in the organic phase after it is dissolved. The incorporation of calcium will then crosslink the photopolymerized polymer by an ion bond. Adper Prompt L-Pop also contains a very acid phosphoester. However, it is one of the rare products not having a hydrophobic chain. So, it dissolves the calcium but does not incorporate it in the organic phase. Its action is therefore close to that of phosphoric acid, which dissolves calcium but does not retain it. As an exception, AdheSE contains a phosphonate with less acid than a phosphoester (hence a higher pH), but the absence of hydrophobic function brings its action closer to Adper Promt L-Pop. The presence of a phosphoric acid acrylate and a Bis acrylic acid amine completes its acidic action. However, not all differences can be explained by the specific esters contained in the self-etch primers. The second null hypothesis must be rejected, since the interface morphology was seen to vary from one self-etch bonding system to another.

CONCLUSIONS

Within the limitations imposed by the experimental conditions, the authors draw the following conclusions:

1. The two observational methods used, optical and scanning electron microscopy, proved to be complementary. The optical microscope gave a clear image of the hybrid layer and resin tags, thus making it possible to check the layer's uniformity and continuity over the preparation as a whole. The scanning electron microscope allowed details of the dentin/adhesive interface to be better analyzed.
2. There are morphological differences at the interface, which depend on the self-etching adhesive composition.
3. The pH is not the determining factor conditioning the action of self-etching adhesives.

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Enamel Bond Strengths of Pairs of Adhesives From the Same Manufacturer

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S Duarte Jr • MM Lopes

Clinical Relevance

Some total-etch adhesives bond more effectively to enamel than the corresponding self-etch adhesives from the same manufacturer. For self-etch adhesives, roughening enamel with a diamond bur resulted in a tendency toward higher bond strengths.

SUMMARY

Most manufacturers of dental adhesives have both a total-etch adhesive and a simplified self-etching adhesive available on the market. This study measured the enamel microtensile bond strengths of five pairs of enamel adhesives as a function of enamel roughness. The proximal surfaces of 25 extracted mandibular molars were sectioned with a diamond saw to obtain 50 enamel rectangles with an area of 8x4 mm². The enamel rectangles were divided in two equal parts via a groove to obtain 4x4 mm² squared bonding surfaces. One half was roughened with a coarse diamond bur under water for five seconds, while the other half was kept intact. The enamel surfaces were randomly assigned to 10 enamel adhesives grouped into five pairs. Each pair included one self-etch adhesive and one total-etch adhesive from the same manufacturer: Adper Prompt and Adper Single Bond (PLP and SB, 3M ESPE); AdheSE and Excite (ADH and EXC, Ivoclar Vivadent); OptiBond Solo Plus SE and OptiBond Solo Plus (OPTSE and OPT, Kerr); Tyrian SPE/One-Step Plus and One-Step (TYR and OST, BISCO, Inc); Xeno III and Prime&Bond NT (XEN and PBNT, Dentsply). The adhesives were applied according to the manufacturers' instructions. Buildups were constructed with Filtek Z250 (3M ESPE). The specimens were sectioned in sticks with a cross section of 0.8±0.2 mm² and tested to failure in tension at a crosshead speed of 1 mm/minute. Two-way ANOVA followed by Duncan's *post-hoc* test at $p<0.05$ was computed. The highest mean bond strengths were obtained with total-etch adhesives. For "roughened enamel," three pairs of materials had statistically different means in which the total-etch adhesive resulted in statistically higher bond strengths

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(MPa) than the corresponding self-etch adhesive: EXC (36.6) > ADH (23.0) at $p<0.026$; OPT (34.5) > OPTSE (25.3) at $p<0.028$; PBNT (36.6) > XEN (19.5) at $p<0.0001$. For "intact enamel," four pairs of materials resulted in statistically different means: SB (31.7) > PLP (20.9) at $p<0.049$; EXC (37.9) > ADH (16.3) at $p<0.0001$; OST (30.1) > TYR (18.0); PBNT (43.8) > XEN (16.0) at $p<0.0001$. When the same adhesive was compared on intact vs roughened enamel, all the self-etch materials resulted in lower bond strengths on intact enamel, but this difference was only significant for TYR ($p<0.042$) and ADH ($p<0.050$). For total-etch materials, only OPT resulted in statistically lower bond strengths when applied on intact enamel ($p<0.011$).

INTRODUCTION

In the last 10 years, the rate of development of new dentin/enamel adhesives has been too rapid to allow for clinical evaluation of all materials. Laboratory tests have therefore been used to screen new adhesives for their ability to bond to enamel and dentin. The introduction of enamel acid etching with phosphoric acid in 1955 (Buonocore, 1955) has changed the concepts of restorative and preventive dentistry. Enamel bonding is now considered a durable, reliable clinical procedure. More recently, the application of acids to dentin has also been proposed and widely used to improve dentin bond strengths (Buonocore, Wileman & Brudevold, 1956; Fusayama & others, 1979; Kanca, 1992). Current adhesive systems utilize one of two approaches to interact with dental substrate—the "total-etch technique" or the "self-etch technique" (Perdigão, 2002).

While total-etch (TE) adhesives include a phosphoric acid gel that demineralizes dentin and enamel simultaneously, self-etch (SE) adhesives do not require a separate acid-etch step. SE adhesives condition and prime enamel and dentin simultaneously without rinsing, relying on their ability to partially dissolve hydroxyapatite to generate a resin-infiltrated zone with minerals incorporated (Perdigão, 2002). SE adhesives are composed of aqueous mixtures of acidic functional monomers, generally phosphoric acid esters or carboxylates, with a pH higher than that of phosphoric acid gels (Pashley & Tay, 2001; Oliveira & others, 2003). Water is a very important component of SE adhesives, as it participates in the chemical reaction associated with the interaction of SE with enamel and dentin. Water is needed for acidic monomers to ionize and trigger the demineralization of hard dental tissues. SE adhesives are classified in three categories according to their acidity: mild, moderate and aggressive (Tay & others, 2000b). Because of their higher pH, SE adhesives result in shallow enamel demineralization compared to phosphoric acid (Pashley & Tay, 2001).

As SE adhesives have become popular among clinicians (CRA Newsletter, 2001), most dental adhesive manufacturers have both a TE adhesive and a simplified SE adhesive available on the market. SE adhesives have been reported to bond well to normal dentin and ground enamel *in vitro* (Blunck & Roulet, 1999; Hannig, Reinhardt & Bott, 1999; Pashley & Tay, 2001; Oliveira & others, 2003; Castro & others, 2003; Perdigão & Geraldeli, 2003). Conversely, SE materials may not bond as well to sclerotic dentin or intact enamel (Kanemura, Sano & Tagami, 1999; Tay & others, 2000a; Perdigão & Geraldeli, 2003).

Therefore, this study compared the enamel adhesion strength of SE adhesives with the corresponding TE adhesives from the same manufacturer. The null hypothesis tested is two-fold: 1) There is no difference in enamel bond strengths between each SE adhesive and TE adhesive made by the same manufacturer; 2) There is no difference in enamel bond strengths for the same adhesive applied to roughened enamel versus intact enamel.

METHODS AND MATERIALS

Twenty-five extracted sound molars stored in 0.5% chloramine solution for up to one month were used in this study. The enamel was evaluated under a stereomicroscope to observe the presence of possible cracks or fissures. If defects were detected, the tooth was discarded. Care was taken to choose teeth with similar proximal flat surfaces in order to eliminate intervening variables. Each tooth was cleaned with pumice using a prophy cup under low speed for 10 seconds. The selected teeth were attached to a phenolic ring (Buehler Ltd, Lake Bluff, IL, USA) with sticky wax and sectioned in a precision low-speed diamond saw (Isomet 1000, Buehler Ltd) to obtain an enamel specimen of 8.0 x 4.0 mm^2 from each proximal surface. The two proximal surfaces were divided into two halves 4.0m x 4.0 mm^2, separated by a groove prepared with the diamond saw. One half was roughened with a coarse diamond bur (Two Striper 770.8C, Premier Products Co, Plymouth Meeting, PA, USA) for five seconds under water, while the other half was left intact, resulting in a total of 50 roughened and 50 intact enamel surfaces. The enamel specimens were then randomly assigned to each of 10 experimental groups (Table 1).

This study included five self-etch adhesives and five total etch-adhesives. Composite buildups 6-mm high were constructed in three increments on the bonded surfaces with Filtek Z250 shade A2 (3M ESPE, St Paul, MN, USA). Light curing was carried out with an Astralis 10 (Ivoclar Vivadent, Amherst, NY, USA) curing device in the adhesion mode. The intensity of the light was consistently above 600mW/cm^2 as measured with a radiometer (Kerr Dental, Orange, CA, USA).

Table 1: *Materials Used*

Manufacturer	Material	Composition	Manufacturers' Instructions
3M ESPE St Paul, MN, USA	Adper Prompt L-Pop Self-Etch Adhesive Lot #132149	HEMA (2-hydroxy ethyl methacrylate) phosphates provide the acidic component, with HEMA, BisGMA, and a modified polyalkenoic acid providing the resin components. Water based.	1. Apply adhesive to the entire surface of the cavity, rubbing in the solution with moderate finger pressure for 15 seconds. 2. Gentle air stream of air to dry (surface smooth and glossy). 3. Repeat as necessary. 4. Light cure 10 seconds (3-second exposure with a plasma arc lamp).
	35% H_3PO_4 silica-thickened gel + Adper Single Bond Lot #20030607	Water, ethanol, HEMA, BisGMA, dimethacrylates, a photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids.	1. Apply H_3PO_4 for 15 seconds. 2. Apply two consecutive coats of bond adhesive to enamel and dentin. 3. Dry gently for 2 to 5 seconds. 4. Light-cure for 10 seconds.
Ivoclar Vivadent Schaan, Liechenstein	AdheSE Primer Lot #F15093 Bonding Lot #F15094	*Primer*: Phosphonic acid acrylate, Bis-acrylic acid amide, Water, Initiators and stabilizers *Bonding Component*: Dimethacrylate, HEMA, Highly dispersed silicon dioxide, Initiators and stabilizers.	1. Apply self-etch primer. Once the cavity is thoroughly coated, brush for 15 seconds. Total reaction time should not be shorter than 30 seconds. 2. Dry with mild airflow. 3. Apply self-etch bonding. 4. Dry with mild airflow. 5. Light cure for 10 seconds.
	37% H_3PO_4 silica-thickened gel + Excite Lot #E48521	Dimethacrylates, alcohol, phosphonic acid acrylate, HEMA, SiO_2, initiators and stabilizers.	1. Apply H_3PO_4 on enamel 15 seconds and dentin 10 seconds. 2. Rinse and dry (moist). 3. Apply and brush for 10 seconds. 4. Evaporate 3 seconds. 5. Light cure for 20 seconds.
Dentsply Caulk, Milford, DE, USA	Xeno III Lot #0301001041	*Liquid A*: HEMA, Purified water, Ethanol, 2, 6-Di-tert-butyl-p hydroxy toluene (BHT), Nanofiller *Liquid B*: Tetramethacryloxyethyl pyrophosphate (Pyro-EMA), Pentamethacryloxyethyl cyclophosphazen mono fluoride (PEM-F), Urethane dimethacrylate (UDMA), 2,6-Di-tert-butyl-p-hydroxy toluene (BHT), Camphorquinone, p-Dimethylamino ethyl benzoate (EPD).	1. Dispense equal amounts of A and B. 2. Mix for 5 seconds. 3. Apply generous amounts of adhesive. Leave on for at least 20 seconds. 4. Uniformly spread adhesive by a gentle stream of air until there is no flow (at least 2 seconds). 5. Light-cure 10 seconds.
	34% H_3PO_4 silica-thickened gel + Prime & Bond NT Lot #020509	PENTA, UDMA + T-resin (cross-linking agent) + D-resin (small hydrophilic molecule), butylated hydroxitoluene, 4-ethyl dimethyl aminobenzoate, cetilamine hydrofluoride, acetone, silica nanofiller.	1. Apply H_3PO_4 for 15 seconds. 2. Brush generous amounts of Prime & Bond NT to thoroughly wet the cavity surface. This surface should remain wet for 20 seconds, which may necessitate additional Prime & Bond NT. 3. Air thin for 5 seconds. Surface should have a uniform glossy appearance. If not, repeat steps 1 and 2. 4. Light cure for 10 seconds.
BISCO Inc Schaumburg, IL, USA	Tyrian SPE Uni-Dose/One-Step Plus Tyrian SPE Lot #0200002694 One Step Plus Lot #0200004974	*Part A*: Ethanol *Part B*: 2-Acrylamido-2-methyl propanesuflonic acid, Bis (2-(methacryloyloxy)ethyl) phosphate, ethanol *One Step Plus*: BPDM (Biphenyl dimethacrylate), HEMA, acetone, glass frit.	1. Shake Tyrian SPE unit-dose for 3 to 5 seconds. 2. Slowly press the chambers together, noting color change to Tyrian (purple). 3. Stir, using a foam pellet or an applicator. 4. Dry the preparation for at least 5 seconds. Make sure no moist spots remain on the tooth. 5. Apply 1 to 2 coats. Agitate slightly for 20 seconds. 6. Using a foam pellet (new or original blotted dry), blot excess from preparation, until Tyrian color disappears. 7. Shake One-Step Plus bottle for 3 to 5 seconds. 8. Apply 2 coats of One-Step Plus. 9. Air dry for a minimum of 10 seconds. 10. Light-cure for 10 seconds per surface.

Table 1: *Materials Used (cont.)*

Manufacturer	Material	Composition	Manufacturers' Instructions
	32% H_3PO_4 polymer-thickened gel + One-Step Lot #0200003632	BPDM (Biphenyl dimethacrylate), HEMA, acetone, photoiniators.	1. Apply H_3PO_4 for 15 seconds. 2. Rinse thoroughly and dry (moist). 3. Apply for 10 seconds. 4. Dry with mild airflow. 5. Light cure for 20 seconds.
Kerr Dental Orange, CA, USA	OptiBond Solo Plus SE Lot #205187	Ethyl alcohol, water, alkyl dimethacrylate resins, Stabilizers, Activators.	1. Apply SE Primer for 15 seconds using light brushing motion. 2. Air thin for 3 seconds. 3. Apply OptiBond Solo Plus for 15 seconds using light brushing motion. 4. Air thin for 3 seconds. 5. Reapply adhesive for 15 seconds using light brushing motion. 6. Air thin for 3 seconds. 7. Light cure for 20 seconds.
	37.5% H_3PO_4 silica-thickened gel + OptiBond Solo Plus Lot #203D20	Ethyl alcohol, alkyl dimethacrylate resins, barium aluminoborosilicate glass, fumed silica (silicon dioxide), sodium hexafluorosilicate.	1. Apply H_3PO_4 for 15 seconds. 2. Rinse thoroughly and dry (moist). 3. Apply for 15 seconds, using light brushing motion. 4. Air thin for 3 seconds. 5. Reapply for 15 seconds, using light brushing motion. 6. Air thin for 3 seconds. 7. Light cure for 20 seconds.

Table 2: *Two-way ANOVA*

Source	Type III Sum of Squares	df	Mean Square	F	Sig
Adhesive	29324.653	9	3258.295	9.918	**.000**
Enamel Surface	1938.890	1	1938.890	5.902	**.015**
Adhesive* Enamel Surface	4195.188	9	466.132	1.419	**.176**
Error	184294.107	561	328.510		
Corrected Total	219667.829	58			

The bonded specimens were attached to a phenolic ring with cyanoacrylate glue (Zapit, Dental Ventures of America, Inc, Corona, CA, USA) and sectioned in X and Y directions to obtain bonded sticks with a cross-sectional area of 0.8±0.2 mm^2. The sticks were tested individually by setting and attaching them on the Geraldeli's jig (Perdigão & Geraldeli, 2003) using cyanoacrylate glue (Zapit). The sticks were then submitted to a tension load using an Instron 4204 testing machine (Instron Co, Canton, MA, USA) at a 1 mm/minute crosshead speed. A Mitutoyo absolute digital caliper (Mitutoyo Corp, Kanogawa, Japan) with an accuracy of 0.001 mm was used to measure the sides of the bonding interface and calculate the bonding area in mm^2. The load (in Kg) and the bonding surface area of the specimen were registered on a worksheet and the microtensile bond strengths calculated in MPa.

A two-way ANOVA followed by Duncan's *post-hoc* test was computed. The significance level was set at 95%. Statistical analysis was carried out with the SPSS 10 for Windows (SPSS, Inc, Chicago, IL, USA) software package.

RESULTS

Two-way ANOVA (Table 2) found statistical differences between pairs of means at $p<0.0001$ for the variable "adhesive" and $p<0.015$ for the variable "enamel substrate." The interactions were not significant ($p>0.176$). The Duncan's test grouped the adhesives into five subsets. PBNT, EXC and SB were grouped in the highest subset, followed by SB, OST and OPT in the second subset.

For "*roughened enamel*" (Table 3), three pairs of materials resulted in statistically different means: EXC > ADH at $p<0.026$; OPT > OPTSE at $p<0.028$; PBNT > XEN at $p<0.0001$. For "*intact enamel*," four pairs of materials resulted in statistically different means: SB > PLP at $p<0.049$; EXC > ADH at $p<0.0001$; OST > TYR at $p<0.005$; PBNT > XEN at $p<0.0001$. When the same adhesive was compared on intact vs roughened enamel (Table 4), all self-etch materials resulted in lower bond strengths on intact enamel, but this difference was only significant for TYR ($p<0.042$) and ADH ($p<0.050$). For total-etch adhesives, OPT resulted in statistically higher bond strengths to "*roughened enamel*" than to "*intact enamel*." All other

Table 3: *Comparison of Pairs of Adhesives from the Same Manufacturer*

Intact Enamel				Roughened Enamel		
Significance	**Mean ± SD**	**N**	**Enamel Adhesive**	**N**	**Mean ± SD**	**Significance**
$p<0.049$	20.9±9.9	28	Adper Prompt L-Pop	31	26.8±9.5	NS
	31.7±12.9	25	Adper Single Bond	31	35.8±10.0	
$p<0.0001$	16.3±7.2	30	AdheSE	32	23.0±10.3	
	37.9±13.5	29	Excite	24	36.6±14.2	$p<0.026$
$p<0.0001$	16.0±7.3	31	XENO III	30	19.5±7.8	
	43.8±12.7	27	Prime&Bond NT	31	36.6±9.0	$p<0.0001$
$p<0.005$	18.0±8.8	26	Tyrian SPE/One Step Plus	24	28.0±11.2	NS
	30.1±10.6	29	One Step	32	29.3±6.3	
NS	20.9±7.5	29	OptiBond Solo Plus SE	32	25.3±8.0	
	21.9±9.7	29	OptiBond Solo Plus	31	34.5±11.3	$p<0.028$

Since the group sizes are unequal, the harmonic mean of the group sizes was used
NS = Not significantly different

Table 4: *Mean bond Strengths for Each Adhesive in Function of the Enamel Substrate*

Self-etch			
Adhesive	**Means**		**Significance**
	Roughened Enamel	**Intact Enamel**	
AdheSE	23.0	16.3	$p<0.050$
Adper Prompt L-Pop	26.8	20.9	NS
OptiBond Solo Plus SE	25.3	20.9	NS
Tyrian SPE/One Step Plus	28.0	18.0	$p<0.042$
XENO III	19.5	16.0	NS
Total-etch			
Adhesive	**Means**		**Significance**
	Roughened Enamel	**Intact Enamel**	
Adper Single Bond	35.8	31.7	NS
Excite	36.6	37.9	NS
One-Step	29.3	30.1	NS
OptiBond SOLO Plus	34.5	21.9	$p<0.011$
Prime&Bond NT	36.6	43.8	NS

NS- Not significantly different

total-etch adhesives resulted in statistically similar bond strengths regardless of the degree of enamel roughness.

DISCUSSION

As bonding to enamel has been successful and reliable since the introduction of the acid-etch technique, bonding to dentin has been considered the weakest link of bonded restorations and, consequently, the subject of numerous research reports (Pashley & others, 1995; Swift, Perdigão & Heymann, 1995; Eick & others, 1997; Sano & others, 1999; Hashimoto & others, 2000). With the introduction of SE adhesives, phosphoric acid has been replaced with acidic monomers that are not rinsed off and, therefore, are incorporated in the adhesive joint. As SE adhesives do not rely on a separate acid-etching step, research interest has shifted back from dentin bonding to enamel bonding (Ferrari & others, 1997; Perdigão & others, 1997; Gordan, Vargas & Denehy, 1998; Shinchi, Soma & Nakabayashi, 2000; Shimada & others, 2002).

Phosphoric acid etching of enamel creates microporosities on enamel surfaces (Buonocore, Matsui & Gwinnett, 1968). Low viscosity enamel bonding agents flow into the irregularities of etched enamel (Miyazaki, Sato & Onose, 2000) to provide reliable enamel bond strengths, prevent microleakage and reinforce residual tooth structure (Buonocore & others, 1968; Swift & others, 1995; Perdigão & Geraldeli, 2003).

The enamel etching capability of SE adhesives has been studied profusely (Perdigão & others, 1997; Ferrari & others, 1997; Kanemura & others, 1999; Miyazaki & others, 2000; Miyazaki & others, 2002; Perdigão & Geraldeli, 2003; Shimada & Tagami, 2003). One of the shortfalls of SE adhesives is that they may not etch enamel to the same depth achieved with phosphoric acid (Miguez & others, 2003; Perdigão & Geraldeli, 2003). Other studies, however, have demonstrated that enamel bonding with SE adhesives is of the same magnitude as enamel bonding after phosphoric acid etching (Hannig & others, 1999; Shimada & others, 2002). In this study, SE adhesives resulted in lower bond strengths on intact enamel than on roughened enamel, which is in agreement with other studies

that reported SE adhesives as being only effective on ground enamel but less effective on intact enamel (Pashley & Tay, 2001; Perdigão & Geraldeli, 2003). The low bond strengths of SE adhesives to intact enamel, present in the cervical aspect of some restorations, raise clinical concerns (Opdam & others, 1998). Optimal bonding to intact enamel is important to accomplish good clinical performance and prevent microleakage (Swift & others, 1995). A recent clinical study showed that enamel etching prior to the application of an SE adhesive is crucial for the clinical performance of composite restorations, especially those corresponding to Class IV and V lesions (Matsuya & others, 2003). Another study reported the 10-year clinical performance of an SE adhesive. Forty out of 44 restorations were rated as Bravo for marginal integrity, while 39 had some degree of marginal discoloration (Akimoto & Takamizu, 2004).

The lower adhesion strength of SE adhesives to intact enamel may be a result of the less pronounced enamel etching pattern obtained with SE adhesives on intact enamel than the pattern obtained with phosphoric acid (Shimada & others, 2002; Perdigão & Geraldeli, 2003). Intact enamel is less accessible to SE adhesives, because it is hypermineralized and may contain more fluoride than instrumented enamel (Kanemura & others, 1999). Additionally, changes occur in the outermost enamel layer after eruption (Brudevold, 1948), and a prismless enamel layer may be present (Gwinnett, 1967), preventing further penetration of the SE adhesives. Longer application times may actually compensate for the higher pH of SE adhesives compared to phosphoric acid etchants (Itou & others, 2001). In fact, when a self-etching primer was applied for 60 seconds instead of the recommended 30 seconds, Ferrari and others (1997) demonstrated that an adequate seal is provided in Class V cavities both *in vitro* and *in vivo*.

When the acid etch technique was a relatively new clinical procedure, formation of tag-like resin extensions into enamel microporosities was considered the mechanism of bonding of resin to phosphoric acid-etched enamel (Gwinnett & Matsui, 1967; Myers, Rossi & Cartz, 1974). Resin tag penetration into roughened enamel treated with SE adhesives has been deemed comparable to phosphoric acid etched enamel (Gordan & others, 1998). Other studies reported that the etching pattern of SE adhesives was not as well defined as TE adhesives on roughened enamel but resulted in similar enamel bond strengths (Perdigão & others, 1997; Shimada & others, 2002). This similarity in bond strengths for SE adhesives in spite of their shallower etching pattern compared to TE adhesives may seem conflicting. However, the length of the tags created on phosphoric acid-etched enamel may contribute little to bond strength. Adhesive strength may be a result of the ability of resin to penetrate between enamel crystallites and rods (Shinchi & others, 2000). Additionally, an ideal etch pattern is not essential in order to produce a strong bond (Hobson & McCabe, 2002).

OPT and OPTSE were the only pair of adhesives that resulted in statistically similar bond strengths on *intact* enamel. This similarity was not a result of increased bond strengths for the SE adhesive OptiBond Solo Plus Self-Etch (Kerr Dental), but was due to the relatively low bond strengths associated with TE adhesive OptiBond Solo Plus (Kerr Dental). When used on roughened enamel, the enamel bond strengths of OPT reached the same magnitude as the other TE adhesives, which is corroborated by Yost and others (2004).

PLP and TYR resulted in similar bond strengths compared to their corresponding TE adhesives, while the remaining three SE adhesives, ADH, OPTSE and XEN, still resulted in significantly lower enamel bond strengths to roughened enamel than the corresponding TE adhesives. This distinct behavior of different SE adhesives might be related to variations in pH. According to the respective manufacturers, PLP has a pH of 1.1, while the pH associated with TYR is 1.2. The inability of some SE adhesives to bond adequately to roughened enamel raises an important clinical question as to whether dentists are currently considering the ease of application of the new simplified adhesives to be more important than the longevity of the bonded restorations, ultimately, compromising the dental health of our patients.

The null hypotheses tested in this study are rejected. Although the enamel bond strengths of each SE adhesive were generally lower than the corresponding enamel bond strengths of the TE adhesive made by the same manufacturer, for some pairs of materials, the difference was not statistically different. Additionally, some adhesives resulted in similar bond strengths to roughened enamel than to intact enamel.

CONCLUSIONS

1. SE materials resulted in lower bond strengths on intact enamel than on roughened enamel, but this difference was only significant for Tyrian/One Step Plus and for AdheSE.
2. For the same manufacturer, most TE adhesives bonded better to enamel than their corresponding SE adhesive.

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Microtensile Bond Strength of Light- and Self-cured Adhesive Systems to Intraradicular Dentin Using a Translucent Fiber Post

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LF Valandro • P Mathias • A Muench

Clinical Relevance

The use of fiber posts for the restoration of endodontically treated teeth has increased tremendously over the last few years. However, the choice of an adhesive system that provides a reliable, long-lasting bonding mechanism to root canal dentin is still unclear.

SUMMARY

This study evaluated the bond strength of a light- and self-cured adhesive system to different intraradicular dentin areas (cervical, middle and apical thirds). Twenty single-rooted teeth were instrumented and their roots were prepared to receive a #2 translucent fiber post (Light Post). The root canals were irrigated with 0.5% sodium hypochlorite for one minute, rinsed with water and dried using paper tips. The teeth were divided into two groups (n=10): Single Bond [SB] (light-cured) and Scotchbond Multi-Purpose Plus [SBMP] (self-cured). To avoid polymerization of the materials through the root lateral walls, the teeth were placed in a silicone mold and the adhesives applied with a thin microbrush according to manufacturer's instructions. The resin cement, Rely X ARC, was inserted into the root canals using Lentulo burs. The post was then placed and the light-curing procedure was carried out for 40 seconds (±500 mW/cm²). The roots were kept in a 100% relative moisture environment for 24 hours and stored in distilled water for an additional 24 hours. Each root was perpendicularly sectioned into 1-mm thick sections, resulting in approximately four slices per region. Dumbbell-shaped slices were obtained by trimming the proximal surfaces of each slice using a diamond bur until it touched the post. The bonded area was calculated, slices were attached to a special device and submitted to microtensile testing at 1 mm/minute crosshead speed. Data were analyzed using ANOVA and Tukey's test. The

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mean bond strength values (MPa) were: SBMP: cervical=10.8[a], middle=7.9[bc], apical=7.1[bc]; SB: cervical=8.1[b], middle=6.0[c], apical=6.9[bc]. Significant differences were found between adhesive systems only for the cervical third. The cervical region showed higher mean bond strength values than the middle and apical regions ($p<0.0001$).

INTRODUCTION

Prefabricated posts have been used for some time with the aim of improving the retention of core build-up materials in endodontically-treated teeth when the remaining crown dentin is scarce. Some researchers and practitioners have advocated the use of endodontic posts, which have mechanical properties that more closely resemble dentin, such as elastic modulus, thus achieving better stress distribution (Sidoli, King & Setchell, 1997).

Carbon fiber posts were the first used for this purpose (Duret, Reynaud & Duret, 1990), followed by glass fiber posts, carbon fiber posts coated with quartz and quartz fiber posts, all presenting elastic modulus similar to dentin.

One of the main concerns regarding prefabricated endodontic posts is the bonding mechanism of composite materials both to posts and root dentin. The bonding effectiveness among dentin, resin cement and post can be evaluated by means of microleakage (Mannocci, Ferrari & Watson, 2001a), bond strength tests (Gaston & others, 2001) and using microscopic analysis (Ferrari & others, 2000b).

Traditionally, dual-cured adhesive systems in association with self- or dual-cured resin cements are used for cementation of endodontic posts. Some manufacturers indicate only light-cured adhesives and dual-cured resin cements for this procedure. Some studies, employing microtensile bond strength tests, have been performed in order to investigate the bond strength of such materials in specific areas of the root. However, the external surface of the root was used as the bonding substrate in these experiments (Yoshiyama & others, 1996a,b; 1998).

By means of a microtensile test, Gaston and others (2001) evaluated the regional bond strength of two resin cements to intraradicular dentin. However, no post was inserted into the root canal. Ferrari, Vichi and Grandini (2001) described a uniform bonding mechanism along the entire root canal when a light-cured single bottle adhesive system was employed for cementation of posts, using scanning electron microscopy (SEM). However, few investigations regarding the bond strength of light-cured adhesives have been associated with resin cements and fiber posts in different root canal regions (Bouillaguet & others, 2003). Hence, some doubts about the bonding of light-cured materials to intraradicular dentin still remain, especially in areas of difficult light access, such as the middle and apical thirds of the root canal.

This *in vitro* study evaluated the regional (cervical, middle and apical thirds) bond strength of light- and self-cured adhesive systems to intraradicular dentin employed for the cementation of a translucent quartz fiber post.

METHODS AND MATERIALS

Twenty single-rooted teeth previously stored in 0.5% chloramine solution were used for this study. The occlusal surfaces were transversely sectioned at 1 mm above the cementoenamel junction using a low speed diamond saw mounted in a Labcut 1010 cutting machine (Extec Corp, Enfield, CT, USA) under constant water cooling.

The root canals were manually instrumented along the entire working length (WL) using #15 to 40 K-file series (Maillefer, Ballalgues, Switzerland) and, subsequently, were enlarged using #2, 3 and 4 Largo drills (Maillefer). Irrigation using distilled water was carried out after each file or drill size change throughout the shaping process. Teeth presenting some obliteration along the root canal or with a working length of less than 14 mm were discarded from the sample. The root canals were not obturated, because the purpose of this study was to evaluate bond strength in the entire root canal extension, including the deepest regions, such as the apical third.

The root apices were externally sealed using the Single Bond adhesive system (3M ESPE, St Paul, MN, USA) and Filtek Z250 resin composite (3M ESPE) in order to avoid extravasation of luting materials through the apex. The root canals were prepared to receive a #2 translucent quartz fiber post (Light Post—

Table 1: *Luting Materials Used in this Experiment*

Adhesive Systems (Manufacturer)	**Mode of Activation**	**Resin Cement** (Manufacturer)
Scotchbond Multi-Purpose Plus—SBMP [Activator, Primer, Catalyst—3 bottles] (3M-ESPE)	Self-cured	Rely X ARC (3M-ESPE)
Single Bond—SB [1 bottle] (3M-ESPE)	Light-cured	Rely X ARC (3M-ESPE)

Figure 1: *Perpendicular sectioning of root-post sets into approximately 1-mm thick sections.*

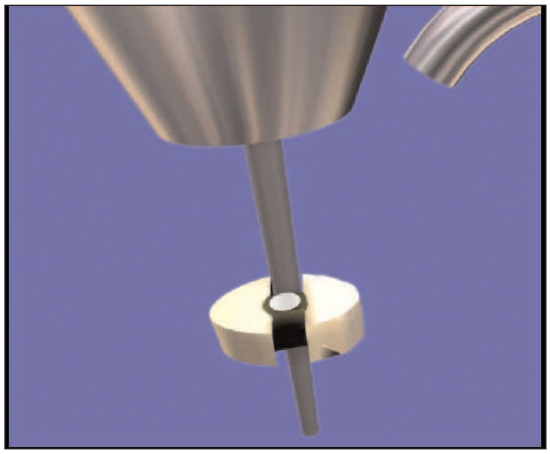

Figure 2: *Proximal slice trimming using tapered diamond bur.*

BISCO, Schaumburg, IL, USA) using the burs supplied by the manufacturer. The prepared roots were randomly divided into two groups (n=10) according to the adhesive systems tested.

The roots were molded with heavy-body addition silicone impression material (Simply Perfect—Discus Dental, Culver City, CA, USA) to avoid light propagation through external surfaces of the roots during polymerization of the adhesive systems and resin cement.

Prior to application of the resin cement systems (Table 1), the root canals were irrigated with 0.5% sodium hypochlorite solution for one minute, rinsed with distilled water and dried using paper tips. The canals were etched with 35% phosphoric acid (3M ESPE) for 30 seconds, rinsed with distilled water and thoroughly dried until no visible moisture could be observed. The fiber posts were etched with 35% phosphoric acid (3M ESPE) for 60 seconds, rinsed with distilled water and thoroughly air dried, as recommended by the manufacturer.

With the roots placed in silicone molds, the adhesive systems were applied strictly according to the manufacturer's recommendations and the resin cement was inserted into the root canal with the aid of a Lentulo bur (Maillefer). The posts were positioned into the root canals immediately after insertion of the resin cement, and light curing was performed through the cervical portion of the root for 40 seconds. Before testing, the root-post sets were kept in a 100% relative moisture environment for 24 hours, then stored in distilled water for an additional 24 hours, always at 37°C.

The roots were affixed with sticky wax into a special device adapted to the Labcut 1010 cutting machine (Extec Corp) and perpendicularly sectioned into approximately 1-mm thick sections using a low speed diamond saw under constant water cooling (Figure 1). This procedure resulted in 12 serial sections or slices

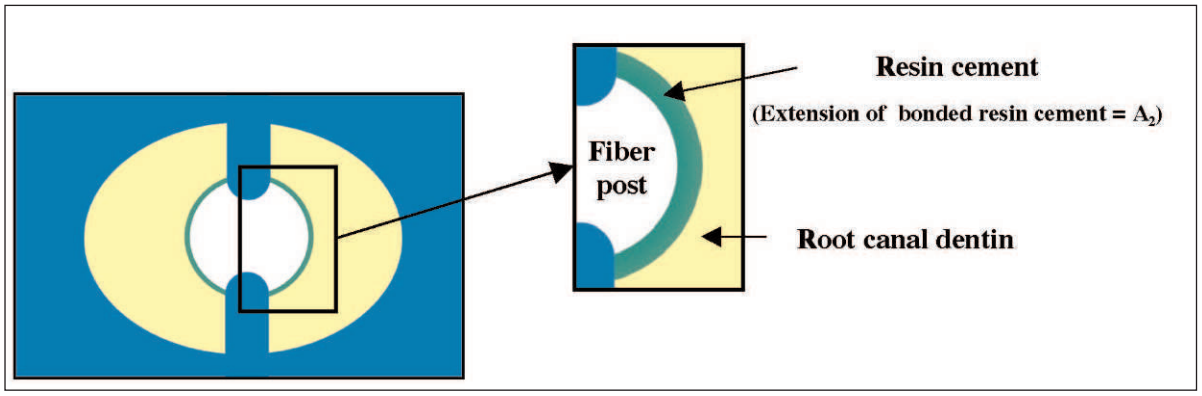

Figure 3: *A dumbbell-shaped slice. A close view showing the extension of resin cement bonded to root dentin (A_2).*

per root, identified as cervical 1, 2, 3, 4; middle 1, 2, 3, 4 and apical 1, 2, 3, 4.

With the aid of a 4x magnifying glass, sections were held with finger pressure and trimmed using a tapered diamond bur (ref 3195—KG Sorensen, São Paulo, Brazil) from the mesial surface until the bur touched the post. The same procedure was carried out from the distal surface, so that a dumbbell-shaped section could be obtained, as demonstrated in Figure 2. All slices were examined under a stereomicroscope (Bausch & Lomb, Bern, Switzerland) at 25x magnification to ensure that the diamond bur really touched the post during trimming procedures (Figure 3).

An electronic caliper with 0.01-mm precision was used to measure the thickness (A_1) of each section. The resin cement extension bonded to the root canal dentin in one side of each dumbbell-shaped slice (Figure 3) was obtained by applying the following equation: A_2 = (PP÷2) - DBWD, where PP is the post perimeter and DBWD is the diamond bur working diameter (0.6 mm). The post diameter was 1.8 mm in the cervical and middle thirds and 1.2 mm in the apical third. The working diameter was 0.6 mm. The PP was calculated using the formula: PP=2πr, where the π value is 3.14 and r is post radius. Hence, the following PP values were obtained for the cervical and middle thirds: PP_{CM} = 2 x 3.14 x 0.9 = 5.6 mm, and for the apical third: PP_A = 2 x 3.14 x 0.6 = 3.8 mm. Therefore, the extension of resin cement bonded to dentin (A_2) in the cervical and middle thirds was: A_{2CM} = (5.6÷2) - 0.6 = 2.2 mm, and in the apical third was: A_{2A}= (3.8÷2) - 0.6 = 1.3 mm. The following equation was applied to calculate the total bonded area (TBA): TBA = A_1 x A_2. Dumbbell-shaped sections were attached to a special device designed for the microtensile bond strength test using cyanoacrylate glue (Super Bonder Gel—Loctite, São Paulo, Brazil) (Figure 4). Each slice was submitted to microtensile bond strength testing in a Universal Testing Machine (Kratos Industry, São Paulo, Brazil) at a 1 mm/minute crosshead speed until failure occurred. After each test, fractured sections were examined under a stereomicroscope at 25x magnification (Bausch & Lomb) to determine the mode of fracture. Failure modes were classified as: a) adhesive between resin cement and post (RC - P), b) adhesive between resin cement and root dentin (RC - RD), c) mixed failure, if the fracture was partially at the resin cement-post interface and partially at the resin cement-root dentin interface or d) cohesive within the resin cement, post or root dentin.

Statistical Analysis

The load at failure divided by the total bonded area was used to calculate the bond strength in MPa for each slice or serial section. During preparation of the serial sections, some bonds were lost before they could be tested and, hence, they were scored as zero bonds for statistical purposes. For each tooth, the sum of bond strength values of each region (cervical, middle and apical) was divided by four, resulting in bond strength values that were considered a statistic unit or specimen (n=10) during statistical analysis. Considering the variables tested, that is, two adhesive systems and three root canal regions, six mean bond strength values were obtained. Working with 10 replications (n=10) resulted in 60 mean bond strength values that were submitted to two-way ANOVA with region as the linked factor. Post-hoc comparisons were made using Tukey's test at a significance level of 5% (p<0.05).

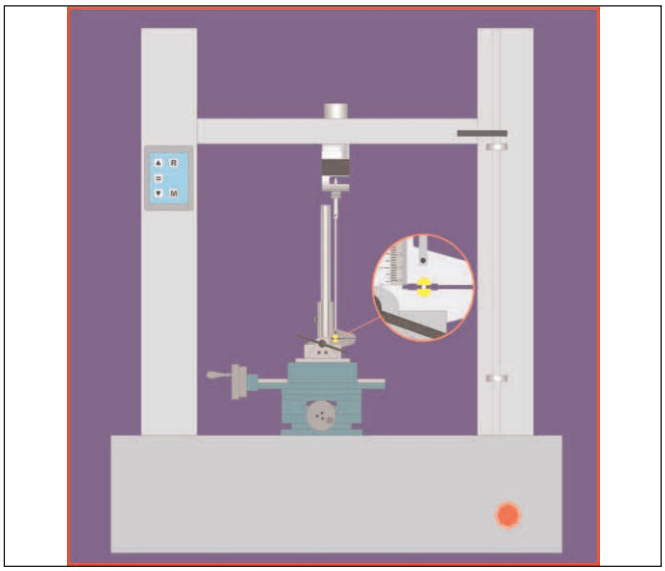

Figure 4: *Microtensile bond strength test device.*

RESULTS

Table 2 describes the total number of slices, mode of fracture distribution and number of slices lost during preparation for each experimental condition. The mean bond strength values (MPa), standard deviations (SD), number of tested slices for each experimental condition and respective statistical interpretation are shown in Table 3. Significant differences in bond strength values were found among regions (p<0.0001). For the SBMP adhesive system, the mean bond strength values in the cervical region were demonstrated to be higher than in the middle and apical thirds, and for the SB adhesive system, bond strength values in the cervical region were higher than in the middle third (p<0.05). There was no statistically significant difference between the middle and apical thirds. Regarding adhesive systems, significant differences were only observed for the cervical third when Scotchbond Multi Purpose Plus (SBMP) was used and led to the highest mean bond strength values for this region. Such behavior demonstrated that the self-cured adhesive system (SBMP) presented good results.

Table 2: *Total Number of Slices (sl), Mode of Fracture Distribution (%) and the Number of Slices Lost During Preparation*

			Mode of Fracture			
Materials	**Regions**	**Total # of sl**	**Adhesive (RC-P)***	**Adhesive (RC-RD)****	**Mixed**	**Number of sl Lost in Preparation**
Light Post + SBMP/RelyX	Cervical	38	34 (89.5)	1 (2.6)	3 (7.9)	0
	Middle	39	36 (92.3)	3 (7.7)	0	0
	Apical	40	37 (92.5)	2 (5.0)	0	1 (2.5)
Light Post + SB/RelyX	Cervical	37	14 (37.9)	9 (24.3)	11 (29.7)	3 (8.1)
	Middle	35	14 (40.0)	8 (22.9)	11 (31.4)	2 (5.7)
	Apical	39	18 (46.2)	5 (12.8)	8 (20.5)	8 (20.5)

**RC - P: Resin cement - post **RC-RD: Resin cement-root dentin*

Table 3: *Mean (n=10) Bond Strength Values (MPa), Standard Deviations (SD), Number of Tested Slices (sl) and Respective Statistical Interpretation for Each Experimental Condition*

Regions / Adhesives	Cervical	Middle	Apical
SBMP	10.8 ± 2.60 (38)[a]	7.9 ± 1.57 (39)[b,c]	7.1 ± 1.60 (40)[b,c]
SB	8.1 ± 4.10 (37)[b]	6.0 ± 3.67 (35)[c]	7.0 ± 4.11 (39)[b,c]

Values are mean ± SD (number of tested slices). Groups identified by different superscript letters are statistically different ($p<0.05$).

DISCUSSION

The good performance of adhesive systems when bonded to enamel and coronal dentin is well documented in Restorative Dentistry. However, some aspects related to intraradicular dentin are still uncertain, especially when adhesive systems are used for endodontic post cementation, once many failures have been clinically observed (Ferrari & others, 2000a).

Some experiments evaluating the microtensile bond strength of resin materials to intraradicular dentin were carried out by only applying adhesive systems and resin cements into root canals without any post insertion (Gaston & others, 2001; Ngoh & others, 2001; Mannocci & others, 2001b; Kanno & others, 2002). The current study verified that it is possible to perform microtensile bond strength tests to evaluate intraradicular dentin bonding using non-rigid prefabricated endodontic posts, as was also observed by Bouillaguet and others (2003), who used resin composite prefabricated posts (Z100) luted with different resin cement systems.

One of the critical aspects of bonding to root canal dentin is the use of adhesive systems that depend on light curing. Ferrari and Mannocci (2000) observed by means of scanning electron microscopy that hybrid layers were present in intraradicular dentin when a carbon fiber post was luted *in vivo* using a light-cured adhesive system combined with a self-cured resin cement. Other studies (Ferrari & others, 2001; Vichi & others, 2002) also demonstrated the presence of an inter-diffusion resin-dentin zone (IRDZ) in root canal dentin when endodontic posts were luted using light- and self-cured adhesives. Vichi and others (2002) described a more effective micromechanical bonding mechanism in the apical third with self-cured adhesives instead of light-cured systems. Comparing SEM observations reported by Vichi and others (2002) with the bond strength values observed in this study, it was not possible to establish any concordance between the findings of both experiments once the bond strength values obtained for the self-cured adhesive—Scotchbond Multi Purpose Plus—in the apical third of root dentin did not differ from that demonstrated for the light-cured system—Single Bond. One of the reasons why the light-cured adhesive was polymerized, especially in areas of difficult light access, such as the middle and apical thirds, may be due to halogen light propagation through the translucent fiber post used in this study. In SEM observations, Pest and others (2002) also reported good results with the combination of translucent posts and light-cured resin cements.

In this study, the mean bond strength values for both adhesive systems (Single Bond: 7.0 MPa and Scotchbond Multi Purpose Plus: 8.6 MPa) were much lower than those observed by Mallmann and others (2001) when they performed a microtensile bond strength test employing the same materials used in this experiment, but using coronal dentin as the bonding substrate. They reported mean bond strength values of 40.97 MPa for Single Bond and 41.00 MPa for Scotchbond Multi Purpose Plus. Besides the different characteristics between substrates, coronal dentin and root canal dentin, it is believed that polymerization contraction of the resin cement might be the factor that most influenced bond strength values. This phenomenon may affect the dentin-adhesive interface at different levels, depending on the configuration factor (C-factor) and adhesive system used, as described by

Mallmann and others (2003). When endodontic posts are cemented into root canals, the C-factor may exceed 200 (ratio of bonded and unbonded surfaces), as recently suggested by Bouillaguet and others (2003). This is because there is a large area of resin cement bonded to the dental substrate and endodontic post and almost no free area to compensate for polymerization contraction. Using a similar methodology to this study, Bouillaguet and others (2003) also observed that the combination of Single Bond adhesive system with Rely X ARC resin cement produced lower bond strength values (5.3 MPa). However, when teeth were sectioned longitudinally and the endodontic post was cemented into the root canal, the mean bond strength values were much higher (23.2 MPa). This increase in bond strength was attributed to a low stress generation on the adhesive systems during resin cement polymerization contraction, regardless of whether in the dentin-adhesive or post-adhesive interface, due to a small configuration factor.

Among the variables tested in this study, the region was the factor that demonstrated the highest significant differences ($p<0.00001$). This study's findings regarding bond strength values to different thirds of root canals are in agreement with SEM observations reported by Ferrari and Mannocci (2000), which showed higher resin tag density in the cervical third compared to the middle and apical thirds. On the other hand, Gaston and others (2001) verified higher mean bond strength values in the apical third of root dentin when compared to the middle and cervical regions, and reported far superior values (23 MPa) to those observed in this experiment. However, although both were microtensile bond strength tests using root canal dentin as bonding substrate, Gaston and others (2001) did not actually insert any endodontic posts into the root canal. Moreover, the roots were sectioned longitudinally and the resin cement was applied directly through the open surface of the root canal, thus reducing polymerization contraction, which may be a crucial factor when using resin cements for root canal dentin bonding.

Several factors, such as materials used for root canal obturation, dentin and/or post surface treatments to obtain an effective bonding, resin cement polymerization contraction and even the compatibility between adhesive systems and resin cements may influence cementation of the endodontic posts into root canals. Hence, some doubts regarding these points still remain and must be solved in future experiments.

CONCLUSIONS

No significant difference was found between the light-cured (Single Bond) and self-cured Scotchbond Multi-Purpose Plus system. Higher mean bond strength values in the cervical region, when compared to the middle and apical thirds of root dentin, could be observed only with the self-cured adhesive system.

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Surface Changes and Acid Dissolution of Enamel After Carbamide Peroxide Bleach Treatment

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Clinical Relevance

Bleaching treatment should be performed with caution in patients who are at high risk for enamel erosion.

SUMMARY

Objectives: To evaluate the effects of home bleaching (carbamide peroxide) on enamel surface morphology and the degree of acid dissolution.

Methods: Buccal surfaces of 15 caries-free human premolars were used in the study. The 15 teeth were cut in half in a buccal-lingual direction at midline; in total, 15 pairs of specimens were obtained. Group A consisted of five pairs that studied surface morphology change and Group B consisted of 10 pairs that studied the susceptibility of bleached enamel to acid dissolution. Tooth halves were prepared following a fluoride-free prophylaxis paste cleaning. One half of the tooth was untreated (control), and the other half was bleached (experimental) for eight hours daily for 10 days using 10% carbamide peroxide. Tooth samples were then stored in distilled water for seven days, after which scanning electron microscopy (SEM) was performed on Group A. Only halves from the same tooth were compared to rule out natural variations between teeth. Group B was etched with 37% phosphoric acid before being examined by SEM. The severity of the acid attack was graded from I to V, and the grades of each pair were compared.

Results: Results of the SEM observation showed that surface porosity slightly increased after bleaching, and more surface dissolution by phosphoric acid was seen with bleached, compared to unbleached, enamel.

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INTRODUCTION

With the desire for a whiter, more-attractive smile, tooth bleaching in the dentist's office or at home is rapidly gaining in popularity. The most popular method is home bleaching, also referred to as "nightguard vital bleaching," "matrix bleaching" and "dentist-prescribed/home-applied bleaching." The home technique involves wearing a custom-made, vacuum-formed tray

containing a mild bleaching agent, usually 10% carbamide peroxide, which is suspended in a viscous gel and applied for a period of time during the day or at night (Christensen, 1997; Haywood, 1997). Because of its simplicity, ease, relative effectiveness and low cost, there has been rapid acceptance of this technique by dentists.

Enamel is more than 96% inorganic mineral (hydroxyapatite), with the remaining 4% being water and organic material, namely, the protein enamelin (ten Cate, 1998). Carbamide peroxide contains about 3% hydrogen peroxide and 7% urea, both of which are protein denaturants. Urea also deproteinizes enamel by dissociating hydrogen bonds in protein molecules (Goldberg & others, 1983; Arends & others, 1984; Hegedus & others, 1999). It is possible that hydrogen peroxide and urea penetrate into the enamel subsurface and change the organic matrix during the bleaching procedure. Thus, the enamel's physical and chemical properties might differ from those of unbleached enamel.

Dental erosion is the loss of hard tissue from the tooth's surface by a chemical, not a bacterial, process. It is a common condition that is increasing in prevalence. For example, in a Swiss study, 16% of 391 subjects had at least one tooth showing signs of erosion (Lussi, 1996). The frequent consumption of citrus fruits and soft drinks and recurrent vomiting were predisposing factors. The progression of erosion results in tooth structure loss or total destruction of the teeth. Some factors such as enamel type, temperature and exposure time influence the development of dental erosion (Amaechi, Higham & Edgar, 1999). It is possible that tooth whitening also plays a role. Rotstein and others (1996) reported a higher solubility of enamel after bleaching. Cimilli and Pameijer (2001) observed a chemical change in surface enamel from hydroxyapatite to primary calcium orthophosphate after carbamide peroxide bleach treatment. They also observed dissolution of calcium from enamel. Attin and others (2003) used a hardness test and pointed out that carbamide peroxide gel rendered enamel more susceptible to demineralization. Although there is little direct evidence, it is possible that bleached enamel may be more prone to acid dissolution.

The phosphoric acid etching of unbleached, intact surface enamel was poor, exposing only some prismatic enamel. This poorer etching effect of the buccal surface of human premolars was seen in studies (Oliver, 1987; Gardner & Hobson, 2001; Hobson, Rugg-Gunn & Booth, 2002), but not in studies by Bhad and Hazarey (1995). Bhad and Hazarey (1995) etched ground enamel, instead of intact enamel surfaces, and found mainly typical etching (honeycombed and fish scale appearance). Nevertheless, the structure of surface enamel was prismless and hypermineralized, contained more inorganic materials and was more acid-resistant than ground enamel (Kanemura, Sano & Tagami, 1999). Oliver (1987) stated that when grinding was not undertaken, etching lacked uniformity, and increased amounts of poor or/no etching were evident. Gardner and Hobson (2001) pointed out that individual teeth react differently to phosphoric acid, and some etch poorly, even with 60 seconds of acid application. Hobson and others (2002) stated that poor etching was common on the middle third of the buccal surface of teeth when intact enamel was etched with 37% phosphoric acid for 30 seconds. It is still not clear whether bleached enamel is more prone to acid dissolution than unbleached enamel. This study compared the degree of acid dissolution of intact surface enamel in both bleached and unbleached cases.

Many studies have concentrated on the surface alterations of bleached enamel. However, the results are controversial (Table 1) (Haywood & others, 1990; Bitter, 1992; Mcgukin, Babin & Mayer, 1992; Shannon & others, 1993; Ben-Amar & others, 1995; Ernst, Marroquin & Willershausen-Zonnchen, 1996; Zalkind

Table 1: *SEM Studies of Carbamide Peroxide-treated Enamel Surface Changes*

	Tooth Samples	Treatment Time	Surface Change
Haywood and others, 1990	32 premolars	245 hours	No change
Bitter, 1992	14 teeth	30 hours	Increased porosity
McGuckin and others, 1992	14 anterior teeth	30 days	Increased porosity
Shannon and others, 1993	72 unerupted teeth	4 weeks	Superficial erosion
Ben-Amar and others, 1995	32 anterior teeth	21 days	Slight change
Flaitz and Hicks, 1996	10 molars	?	Increased porosity
Ernst and others, 1996	10 molars	6 hours	No or slight change
Josey and others, 1996	24 teeth	10 hours, 7 days	Shallow depression
Zalkind and others, 1996	18 premolars	7 days	Various changes, + and -
Bitter, 1998	14 teeth	14 days	Enamel prism exposure
Potocnik and others, 2000	6 teeth	336 hours	Prism exposure
Akal and others, 2001	40 anterior teeth	4 weeks	Surface dissolution

& others, 1996; Josey & others, 1996; Flaitz & Hicks, 1996; Bitter, 1998; Akal & others, 2001; Potocnik, Kosec & Gaspersic, 2000), because the pH and composition of commercial bleaching agents, dosage, treatment duration and frequency, and origin of tooth samples differed from study to study. In particular, natural variations in enamel morphology may be very significant. Enamel is a herterogeneous material, both from a structural and chemical viewpoint; it varies in mineral content, amount of organic matrix and chemical construction (Goldberg & others, 1983). Variation occurs between individuals and teeth, and age also changes enamel surface characteristics, permeability and color (ten Cate, 1998).

In this study, changes in the surface morphology of enamel samples after carbamide peroxide bleaching were evaluated, along with the relative susceptibility of bleached and unbleached enamel to acid dissolution. Paired enamel samples from premolars were compared to exclude natural variations between samples. The hypotheses to be tested were: 1) surface porosity increased after bleaching and 2) there was a higher acid dissolution of bleached enamel.

METHODS AND MATERIALS

Fifteen freshly extracted, non-carious premolars, free from enamel defects and extracted from patients ranging in age from 12 to 30 years, were selected for this study and stored in a 0.5% sodium azide solution until used. Five teeth were used to study bleach-induced surface changes. The other 10 were used to determine the dissolution susceptibility of bleached relative to unbleached surface enamel. Each tooth was cut in half in the buccal-lingual direction at midline with a low-speed saw. One-half was used as a test specimen and the other as the control. In total, 30 parts from 15 teeth were obtained. The halves were embedded in acrylic resin and the labial surface was exposed. The surfaces of the samples were cleaned using a pumice slurry and slow rotating handpiece with an attached rubber cup. Bleaching gel was applied to the test group of teeth for optimal whitening, as recommended by the manufacturer.

For the 15 paired specimens (n=30), half of each tooth was treated, while the other half served as the control. The sample pairs were assigned to one of four treatment groups. Group A consisted of five pairs that studied the surface morphology change of bleached enamel and Group B consisted of 10 pairs that studied the susceptibility of bleached enamel to acid dissolution. The specific groups were designated AC (bleaching control group: no bleaching, no acid etching); AT (bleaching treatment group: 10% carbamide peroxide home bleaching [Opalescence, Ultradent Products, Inc, South Jordan, UT, USA] without acid etching); BC (no bleaching, only acid etching with 37% phosphoric acid for one minute and irrigation with air/water syringe for 30 seconds) and BT (10% carbamide peroxide home bleaching, then acid etching).

Bleaching Procedure

One-millimeter-thick bleaching gel (10% carbamide peroxide, Opalescence, Ultradent Products, Inc) was applied to the halves of 15 teeth and left for eight hours per day. A Mayer strip covered the gel to ensure intimate contact between the gel and the tooth sample. Test samples were then stored in a 100% humidity container. At the end of the procedure, the bleaching gel was removed by rinsing with tap water for 30 seconds and each tooth half was brushed for 30 strokes and rinsed with tap water. For the remaining time of that day, the samples were stored in distilled water. The procedure was continued for 10 days. Control samples were treated the same except that a drop of water was used instead of bleaching gel.

After bleaching treatment, the samples were stored in distilled water for one week, then washed with tap water for one minute and cleaned ultrasonically for 10 minutes. Group AC and Group AT teeth were examined by SEM. Group BC and Group BT teeth were etched for 60 seconds with 37% phosphoric acid and washed for 30 seconds with an air/water syringe before SEM examination (Figure 1).

All samples were vacuum-desiccated and gold sputter-coated prior to examination of the surfaces using SEM (Hitachi S-3500N) at an accelerating voltage of 15.0 kV. Photographs were taken of the control and experimental specimens and surface changes were compared.

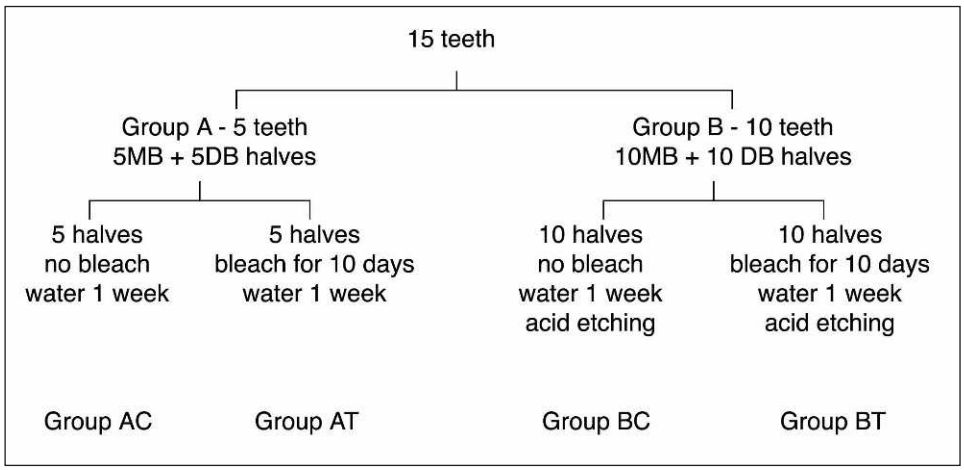

Figure 1. *Summary of procedure.*

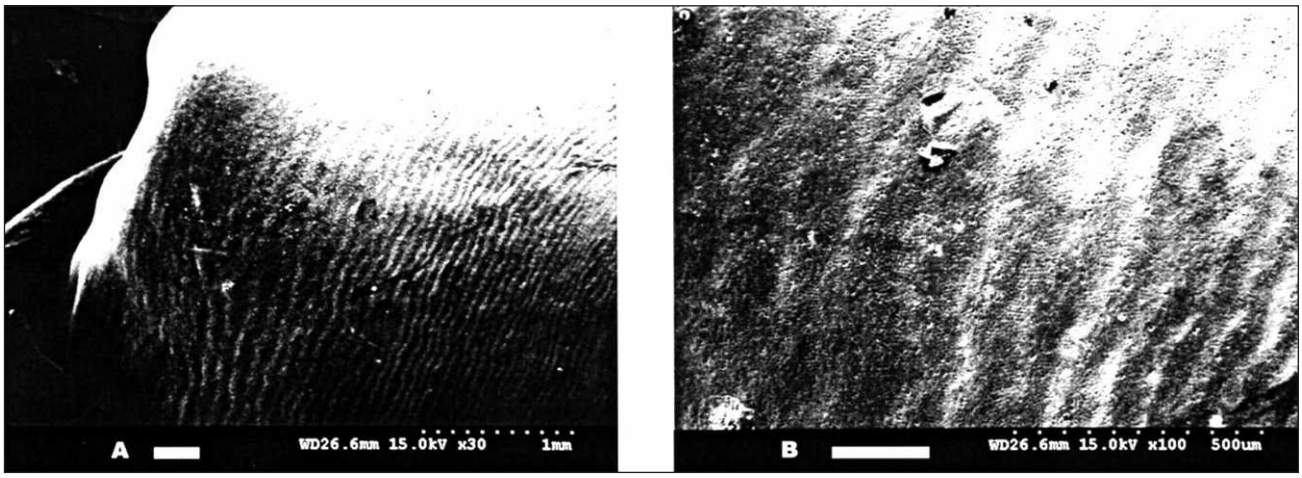

Figure 2. *Scanning electron micrograph of labial surface of premolar, showing perikymata. The surface shows undulating patterns with grooves and a convex ridge. There is some surface pitting. A, 30x; B, 100x, bar=200 µm.*

Table 2: *Results of the Degree of Acid Dissolution of Bleached and Unbleached Enamel*

	Control	Treatment	Degree of Change
Sample A	I	III	+2
Sample B	I	II	+1
Sample C	III	III	0
Sample D	II	IV	+2
Sample E	II	II	+2
Sample F	II	V	+3
Sample G	II	IV	+2
Sample H	II	IV	+2
Sample I	II	IV	+2
Sample J	III	IV	+1

$t=6.32$, $t_{99.5}(9)=3.2$, $p<0.05$

The study examined the middle third of the buccal surface of sample pairs. Only sample pairs of the same tooth were compared and recorded. To provide a representation of events over the entire surface, this study used magnifications of 50x, 200x, 500x and 1000x to compare surface changes. Attention was focused on the degree of acid dissolution over a large area of the enamel surface rather than to specific sites. In Group A, no statistical analysis was performed, because the observation of SEM was designed to be qualitative. In Group B, paired *t*-test was performed to compare the degree of acid dissolution.

RESULTS

Enamel Surface Characteristics With and Without Bleaching Treatment

In both Groups AC and AT samples, surface perikymata, the surface manifestations of the striae of Retzius, were predominant (Figure 2). The gross structure showed undulating and overlapping patterns with grooves and convex ridges (at both 30x, and 100x).

In Group AC samples, an alternating structure of rows of numerous shallow and deeper prism-end pits were combined with a smooth band (at 200x and 500x). Pits or pit rows were usually located in grooves (at 500x and 1000x). Scratch lines were common on the surface of all samples (Figure 3A).

The surface perikymata of Group AT samples looked well defined at lower magnification (200x). At high magnification (500x and 1000x), pits in grooves increased and some prisms were exposed, but the smooth band areas were less affected (Figure 3B). The experimental group showed greater density of surface pitting than the control.

Acid Dissolution Patterns of Bleached and Unbleached Enamel

Because surface enamel is prismless and hypermineralized, Hobson and others (2002) used a four-point scale to assess the quality of etching on certain surface areas from a flat enamel surface, a pitted enamel surface, a discernible etched patterns, to ideal etching. To record and compare SEM results, this study modified Hobson's observation and assigned degrees I–V to indicate progressively greater acid dissolution of surface enamel as follows (Figure 4): Degree I: little change, only micropitting; Degree II: prismatic enamel structure visible and sparsely distributed; Degree III: prismatic enamel structure obvious; Degree IV: complete loss of the aprismatic surface structure and prismatic enamel observed throughout the lesion and Degree V: typical etched patterns (honeycomb or fish scale appearance) over the entire surface.

The extent of acid dissolution in Group BC samples varied, and the change in grade ranged from Degree I to III (Table 2). On the less-affected samples, only sur-

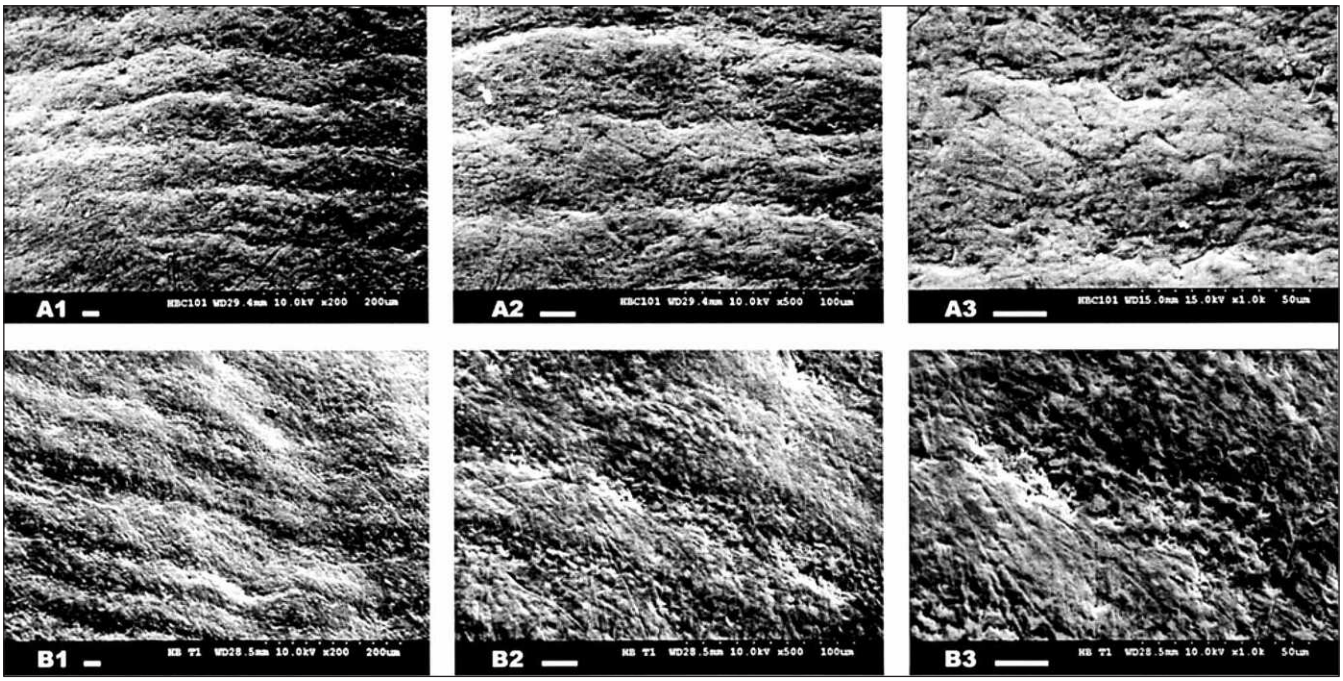

Figure 3. *Scanning electron micrograph of paired unbleached and bleached enamel surfaces. A. Unbleached surface has some scratches and pitting, and pitting is in lines of Retzius and appears to run parallel to the furrow (A1, 200x; A2, 500x; A3, 1000x). B. On the bleached surface, the density of pitting is increased (B1, 200x; B2, 500x; B3, 1000x). Bar=20 µm.*

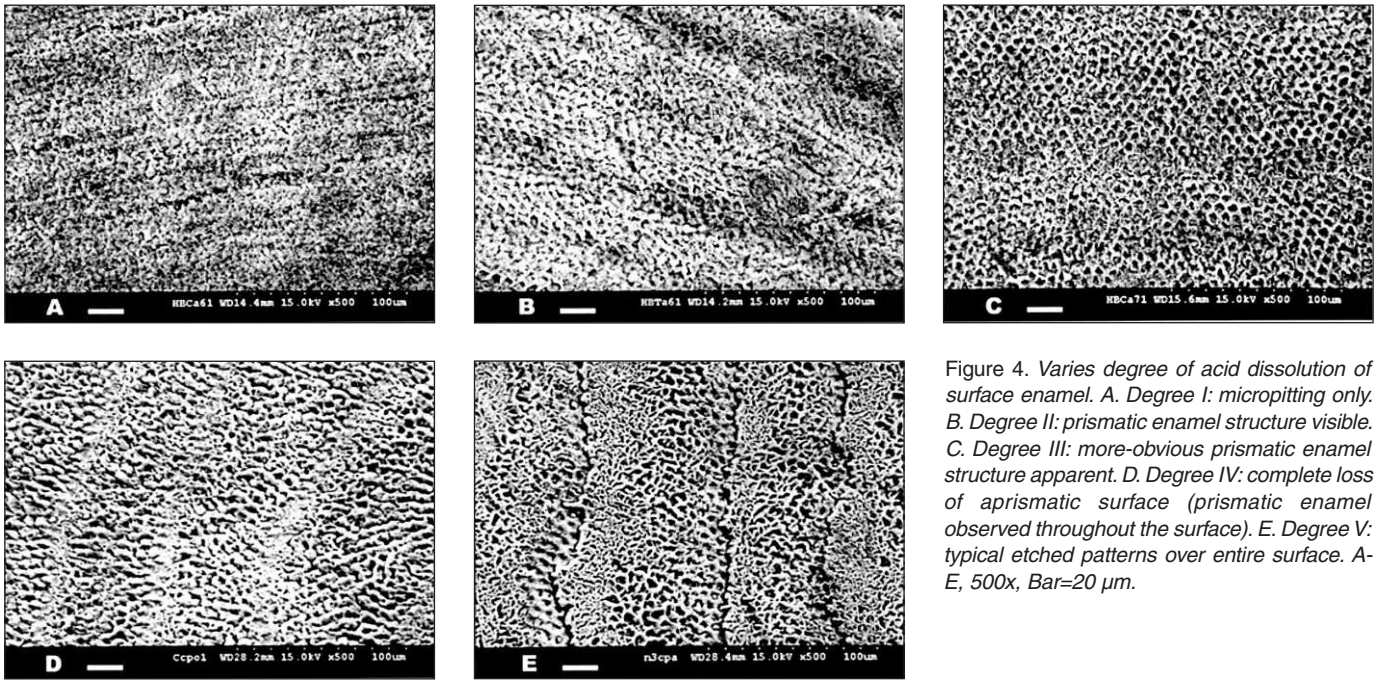

Figure 4. *Varies degree of acid dissolution of surface enamel. A. Degree I: micropitting only. B. Degree II: prismatic enamel structure visible. C. Degree III: more-obvious prismatic enamel structure apparent. D. Degree IV: complete loss of aprismatic surface (prismatic enamel observed throughout the surface). E. Degree V: typical etched patterns over entire surface. A-E, 500x, Bar=20 µm.*

face pitting was found, while others showed sparsely or obviously distributed prismatic enamel. Sparsely distributed prismatic enamel (Degree II) was the most common pattern (Figure 5A).

Acid dissolution patterns in Group BT ranged from Degree II to V. Degree III (prismatic enamel clearly evident) was the most common pattern (Figure 5B).

Paired *t*-test was used to compare paired samples of bleached and unbleached surface changes after acid exposure. The results revealed that acid attack seems

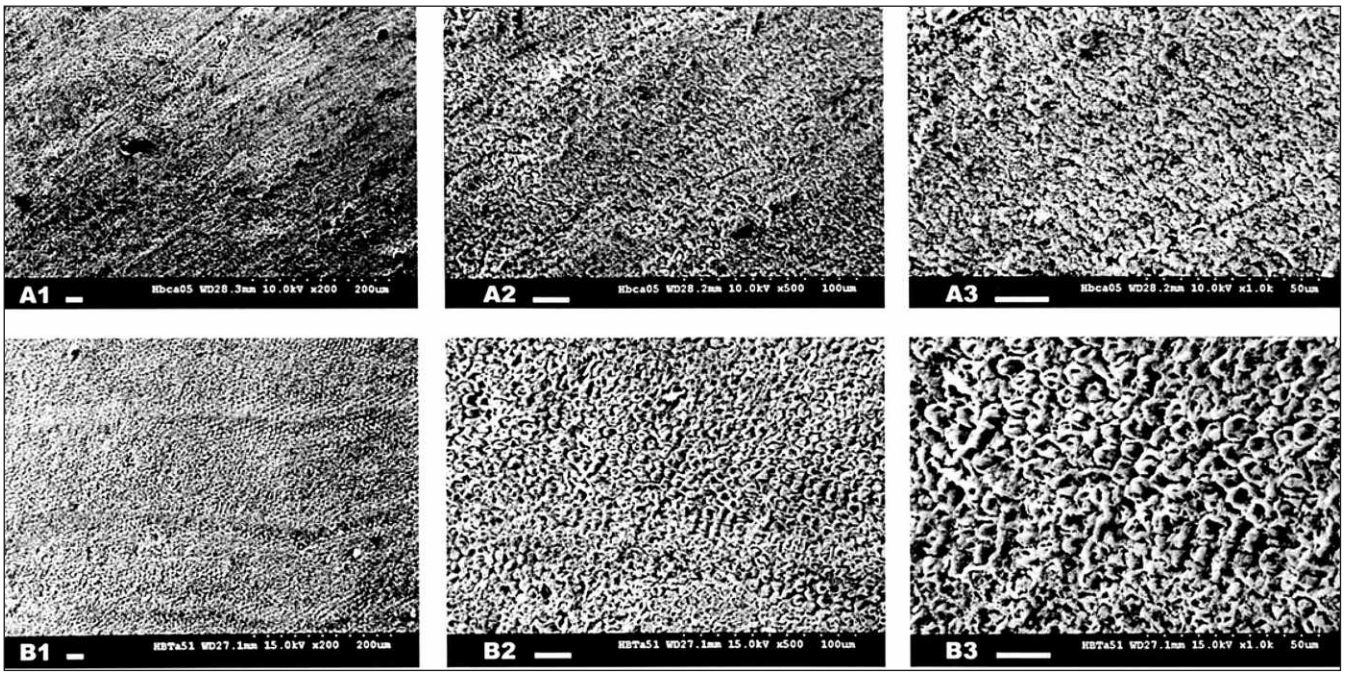

Figure 5. *Scanning electron micrograph showing acid etching of paired unbleached and bleached enamel surfaces. A. Unbleached and acid etched, degree II acid dissolution (A1, 200x; A2, 500x; A3, 1000x). B. Bleached and acid etched, degree III acid dissolution (B1, 200x; B2, 500x; B3, 1000x). Bar=20 µm.*

more severe on bleached than on unbleached surfaces (Table 2) ($p<0.05$).

DISCUSSION

This study assessed surface changes caused by the bleaching of premolars. Surface perikymata were predominant in all samples, while in a study by Josey and others (1996), enamel surfaces were amorphous. The samples in this study were from younger individuals, compared to the samples used in the latter study and, so, the surface was mainly covered by perikymata, which are wavelike features normally found on the enamel surface (Kodaka & others, 1990). Perikymata presents itself as parallel, alternating ridges and grooves (Risnes, 1984). The grooves are considered to be a more porous, less mineralized area (Huang & others, 1998). These structures are known to undergo gradual attrition, mechanical abrasion or chemical erosion over time (Smith, 1991). In the unbleached treatment group, more or less porosity was found in grooves. After bleaching treatment, the effect of bleach on enamel was not uniform. This study found that the pits in the grooves increased, while the smooth bands remained relatively smooth after bleach treatment. The unevenness of the chemical attack on enamel surfaces has been discussed in other reports. Klinger, Rudolph and Gabriel (1978) showed that the solubility of enamel, when treated with lactic acid, varied along the lines of Retzius. The results of this study showed that the smooth ridge area is less affected than the porous groove. The possible explanation may be that the grooves of the perikymata act as a large diffusion pathway (Huang & others, 1998). It seems that bleaching agents tend to easily attack porous and hypomineralized areas. The use of premolars from young individuals was proposed to eliminate natural variations in surface enamel between samples, because, with aging, enamel characteristics change, including loss of permeability, the wearing down of surfaces and increased fluoride content (ten Cate, 1998). Aged and well-mineralized enamel surfaces might be less affected by bleaching. The discrepancy between the results of this study and others (Table 1) can partially be explained by differences in the sample age and surface texture. The first hypothesis, that bleach treatment increases surface porosity, was confirmed. On the basis of these results, caution is recommended before deciding to bleach the teeth of a young age group.

Because the structure of surface enamel is prismless and hypermineralized, that is, it contains more inorganic materials and is more acid-resistant than ground enamel (Kanemura & others, 1999), its structure is regarded as different from that of subsurface enamel. Hobson and others (2002) described how, when intact surface enamel is etched, the greatest area of the etched enamel surface consists of pitting and only a small percentage of the enamel surface shows an ideal etching pattern. This could be due to non-uniform etched pattern distribution and the fact that the discernible etched pattern could be limited to either small or large areas. This study compared the acid dissolu-

tion properties of enamel surfaces of bleached and unbleached sample pairs through a comparison of sample pairs from the same tooth; a higher degree of acid dissolution was seen in samples of the bleached group compared to the unbleached group (Table 2). It seems that the solubility of the enamel surface by 37% phosphoric acid increased after bleaching treatment. Attin and others (2003) evaluated the effects of carbamide peroxide gel on the formation of erosively induced demineralization of enamel and found that carbamide peroxide gel renders enamel more susceptible to demineralization. The results of the current study were in agreement with that of Attin and others (2003).

The second hypotheses, that bleach treatment increases in acid dissolution of bleached enamel, has also been confirmed. The increase in acid dissolution by bleaching may be secondary to bleach-induced changes in the composition and microstructure of the enamel. Bleaching with hydrogen peroxide and urea (both of which are protein denaturants) may enhance the chemical destruction of the protein matrix around the enamel crystallite. During deproteinization, any mineral elements associated with enamel proteins are also removed (Goldberg & others, 1983; Arends & others, 1984; Hegedus & others, 1999). Several studies have shown microchemical variations after bleaching. Josey and others (1996), using polarized light microscopy, observed blurring of the striae of Retzius in enamel sections and suggested a loss of subsurface minerals. McCracken and Haywood (1996) reported a loss of calcium from enamel after six hours exposure to 10% carbamide peroxide, and the amount of calcium loss was similar to that of teeth exposed to cola for 2.5 minutes. Cimilli and Pameijer (2001) demonstrated that some home bleaching agents caused the dissolution of calcium from enamel through the conversion of hydroxyapatite to primary calcium orthophosphate. It may be due to the fact that the solubility factors of hydroxyapatite and primary calcium orthophosphate are 1.6×10^{-58} and 1.0×10^{-3}, respectively, so that calcium orthophosphate is more soluble than hydroxyapatite. The clinical implications of the above observations are still unknown. The results of this study indicated that bleached enamel may be more prone to acid dissolution (Table 2). The bleach-induced loss of chemical molecules did not cause much change in surface morphology, only surface pitting increased in a localized area, but it did increase susceptibility to acid dissolution.

Most bleach treatment studies have used *in vitro* samples, and the results showed little surface change or only pitting (Table 2). However, Shannon and others (1993) used enamel slabs that were attached to removable appliances worn by volunteers and found superficial erosion of the enamel surface. In another *in vivo* study by Bitter (1998), where teeth were bleached for 14 days, maintained in the oral cavity for 21–90 days, then extracted and examined by SEM, the results indicated exposure of the enamel prismatic layer, frequently to the depth of the enamel rods and possibly the dentin, and concluded that long-term bleach-induced changes do occur (Hobson & others, 2002). Both of those *in vivo* studies found more severe surface changes than did the *in vitro* studies. The results of this study might explain the difference between *in vivo* and *in vitro* studies. Acid in the oral cavity, together with an increase in acid dissolution of bleached enamel, probably results in erosive lesions *in vivo*; whereas, *in vitro*, only artificial saliva or distilled water was used as the storage medium, which is neutral (not acidic), thus, permitting only pitting-type surface damage. In this study, the acid resistance of bleached enamel was assessed by phosphoric etching for one minute; clinical acid challenge through food, fruits and soft drinks, although milder, is more prolonged and frequent. It is still unknown whether the effects of bleaching are long-term and whether dietary components augment bleach-induced enamel erosion. More research is needed, especially *in vivo*, and the dynamic interaction of saliva and bleaching agent should be considered.

Natural variations in tooth enamel surfaces make comparison of changes in bleached and unbleached surface morphology difficult. Sample pairs from the same tooth were used to evaluate surface changes and it was found that pitting increased in localized areas. However, after acid etching of the bleached enamel, the amount of dissolution appeared to be higher. This increase may be attributable to infiltration of the enamel's crystallite protein matrix by hydrogen peroxide and urea. This change may increase the enamel's solubility in acid. Considering the erosive effects of dietary components on the dentition, the change in the amount of acid dissolution may be of great importance. It must be emphasized that this study was performed *in vitro* with intact enamel stored in distilled water. In the oral cavity, the remineralization property of saliva must be considered. Many clinical procedures, as well as contact with food, damage surface enamel; therefore, the risks/benefits of bleaching must be considered. The results of this study indicate that surface porosity was slightly increased and more surface dissolution by acid occurred after bleaching.

The possible adverse effects of bleached enamel found in this study were mild surface pitting at localized areas and higher surface dissolution by acid. To avoid the possible adverse effects of bleaching, the topical application of fluoride or a surface glaze is recommended after bleaching. Topical fluoride application may improve remineralization of bleached enamel and protect the teeth from erosive challenges (Attin & others, 1997; Imfeld, 1996). Dental structure coated with a protective coating (for example, dental adhesive) was shown to increase acid resistance (Hachiya & others,

1995; Kuhar & others, 1999). Because the results of this *in vitro* study cannot necessarily be extrapolated to the clinical situation, it is suggested that bleaching be performed with caution in young patients and those at high risk of dental erosion. More research is needed to evaluate the altered acid dissolution properties after bleaching and their clinical relevance

CONCLUSIONS

Within the limits of this study, it was concluded that surface porosity was slightly increased and more surface dissolution by acid occurred after bleaching.

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Temperature Change and Hardness with Different Resin Composites and Photo-activation Methods

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Clinical Relevance

Temperature increase is affected by the material, shade and light source; however, light source is the most important factor that produces different temperature changes during the photo-activation of resin composite.

SUMMARY

This study verifies whether there is any temperature change during photoactivation of two resin composites (Filtek Z250 and Filtek Flow) with three different light curing methods (conventional halogen light curing unit, light emitting diodes curing unit and xenon plasma arc curing unit) and the relationship of temperature change with resin composite hardness. A type-K thermocouple registered the temperature rise peak in an elastomer mold during photoactivation. After photoactivation, the specimens were submitted to Knoop hardness test performed by an indenter (HMV-2000) under a load of 50g for 15 seconds. Both the temperature change data and results of the Knoop hardness test were submitted to ANOVA and Tukey's test at the 5% significance level. No statistical differences in temperature rise were recorded for the different composites following processing by light curing unit ($p>0.05$). The conventional halogen source produced statistically higher temperatures ($p<0.05$) than the other units. The plasma arc source promoted statistically lower ($p<0.05$) Knoop hardness values and temperature changes than the other light curing units.

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INTRODUCTION

Tooth-colored restorative materials, based on dimethacrylate monomers, have increasingly been used to replace missing tooth structures and to modify tooth esthetics. Currently, light cured composites set via the additional polymerization and exposure to light of a requisite wavelength and intensity initiate the generation of free radicals that propagate polymerization, which leads to material hardening (Rueggeberg, 1999; Deb & Sehmi, 2003). However, polymerization of light-activated resin composites causes temperature increase by an exothermic reaction process and by energy absorbed during irradiation (McCabe, 1985; Shortall & Harrington, 1998).

The most widely used light curing source for resin composites photoactivation is the conventional halogen light curing unit (Hofmann, Hugo & Klaiber, 2002). The main radiant output from a conventional halogen source is infrared energy, which is absorbed by resin composites and results in an increased molecular vibration and heat generation. Thus, conventional halogen light curing units require heat-absorbing filters to reduce the passage of infrared energy from the source to the tooth. However, unfiltered infrared energy can result in heat generation at the pulp chamber (Rueggeberg, 1999).

The plasma arc curing units are designed for high-speed curing of composites in direct restorations. However, the increased power of commercially available dental-curing units has also increased the potential for generating unacceptable temperatures in pulp tissue (Hansen & Asmussen, 1993; Hannig & Bott, 1999; Loney & Price, 2001).

Relatively new light-emitting diode (LED) technology promises comparable curing abilities to conventional halogen light curing units at lower polymerization temperatures (Uhl, Mills & Jandt, 2003). These blue light-emitting diodes curing units emit a narrow light wavelength (455 nm-486 nm) that correlates with the spectral absorbance range of camphorquinone (Parr & Rueggeberg, 2002). The output of first generation LED light curing units is limited; however, newer LED sources have substituted conventional LED elements with small chips that contain very large surface-emitting LED chips (Caughman & Rueggeberg, 2002).

External heat applied to teeth can cause pulpal trauma, because an increase in the intrapulpal temperature can result in irreversible damage to the pulp tissue (Lisanti & Zander, 1952; Zach & Cohen, 1965). This thermal trauma may be induced by cavity preparation or by the exothermic setting reaction of cavity linings and restorative materials (McCabe & Wilson, 1980).

Therefore, this study investigated the correlation between temperature rise and curing effectiveness of three light curing units and two resin composites with different viscosities.

METHODS AND MATERIALS

Two restorative resin composites with different viscosities were used in this study: medium viscosity Filtek Z250 (3M ESPE Dental Products, St Paul, MN, USA) and low viscosity Filtek Flow (3M ESPE Dental Products). The restorative resin composite compositions are shown in Table 1.

The three light curing units used include a conventional halogen light-curing unit (XL 2500, 3M ESPE), a plasma arc curing unit (Apollo 95E, DMD, Westlake Village, CA, USA) and a light-emitting diodes curing unit (Ultrablue Is, DMC Equipamentos LTDA, São Carlos, São Paulo–SP, Brazil).

Temperature Test

Temperature change was recorded using a type-K thermocouple connected to a digital thermometer (Iopetherm 46, IOPE, São Paulo–SP, Brazil). The resin composites were applied in a circular elastomer mold (3 mm in inner diameter and 2 mm in height) with the thermocouple positioned in the center of the mold. Between the thermocouple and the composite, a 0.5-mm dentin disc was positioned to simulate the leftover dentin (Figure 1). The resin composites were then covered with a Mylar strip and digitally pressed. Thirty specimens were prepared for each composite (10 for each light source). For photoactivation, the curing tips were positioned close to the elastomer mold/restorative composite. With the conventional halogen LCU and LED LCU, photoactivation was performed for 20 seconds. For the PAC LCU, three seconds was used according to the manufacturer's instructions. The light intensities were verified with a digital radiometer Hilux Dental Curing Light Meter (Benlioglu Dental Inc, Ankara, Turkey). The groups are shown in Table 2.

All measurements were taken in a temperature/humidity-controlled room, with a constant temperature of 20°C ± 1°C and 30% relative humidity.

For temperature measurements, the initial temperature was recorded following temperature stabilization;

Table 1: . *Composition of Resin Composites*

Composite	Matrix	Filler
Filtek Flow (A3) Batch #: 3XU (1370A2)	Bis-GMA TEGDMA Patented dimetacrilate polymer	Zircon/silica: 47% in volume
Filtek Z250 (A3) Batch #: 1BA (1400A2)	Bis-GMA UDMA Bis-EMA	Zircon/silica: 60% in volume

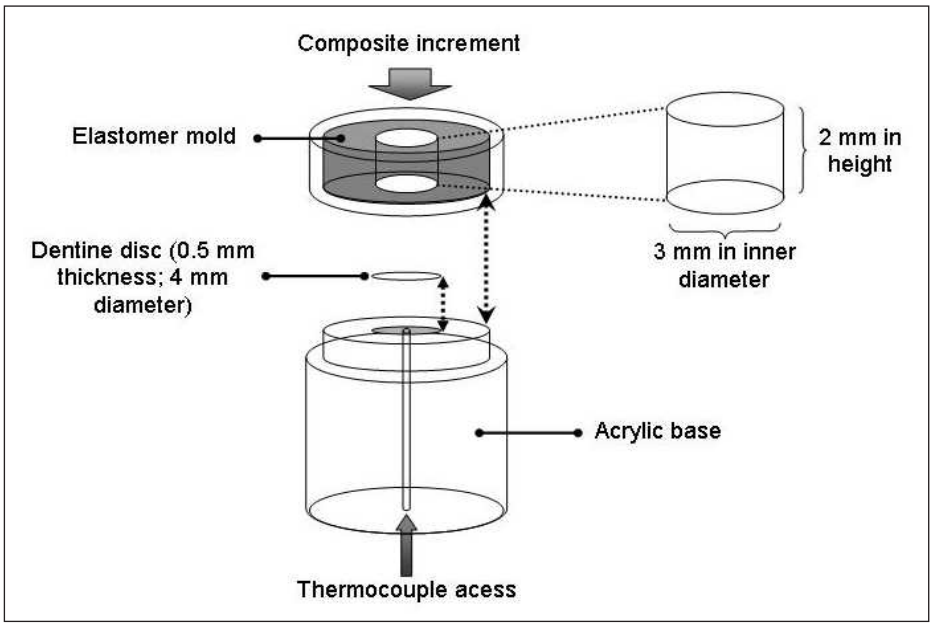

Figure 1. *Apparatus for measuring temperature changes.*

the composite was then light cured and the temperature peak was registered. The initial temperature was deducted from the final temperature.

The temperature change data were submitted to two-way ANOVA and the means were compared by Tukey's test (5% of significance level).

Knoop Hardness Test

After the photoactivation procedure, the specimens were placed in 3/4-inch diameter PVC rings filled with self-curing acrylic resin (JET, Artigos Odontológicos Clássico, São Paulo–SP, Brazil) to keep them fixed. After 24 hours, each group was flattened with carbide sandpaper of decreasing grit (320, 400, 600 and 1200) on an automatic polisher APL-4 (Arotec Ind Com, Cotia–SP, Brazil) to obtain flattened surfaces. The Knoop hardness readings were performed with an indenter (HMV-2000, Shimadzu, Tokyo, Japan) under a load of 50g for 15 seconds.

Three indentations were made on three different regions: surface, 1-mm and 2-mm depths, for a total of nine indentations per specimen. The values obtained in micrometers were converted to Knoop Hardness Number (KHN) by indenter software.

The results of the Knoop hardness test were submitted to two-way ANOVA and the means were compared by Tukey's test (5% of significance level).

RESULTS

Table 3 shows the mean values of Knoop hardness in each appraised depth for all the photoactivation methods with Filtek Z250. It was noted that, generally, there was no statistical decrease in hardness as depth increased. Only PAC LCU had a significant decrease in hardness values at the 2-mm depth ($p<0.05$). Comparing the methods, at the surface and 1-mm depth, there were no statistical differences ($p>0.05$). At the 2-mm depth, PAC LCU was statistically lower ($p<0.05$) than the others.

Table 4 shows the mean values of Knoop hardness in each appraised depth for all the photoactivation methods with Filtek Flow. It was also verified that, similar to Z250, there generally was no decrease in hardness as depth increased, and only PAC had a significant decrease in hardness values at the 2-mm depth ($p<0.05$). Comparing the methods at all depths, there were no statistical differences ($p>0.05$) between the conventional halogen LCU and LED LCU. However, PAC LCU was statistically lower ($p<0.05$) than the others.

Table 5 shows the mean values of the temperatures recorded (Celsius degree). No statistical difference was observed for the different composites with any of the photoactivation methods ($p>0.05$). However, the light curing unit factor demonstrated that the conventional halogen LCU produced statistically higher temperatures ($p<0.05$) than the other units. It was also observed that the LED LCU produced statistically higher temperatures than PAC LCU ($p<0.05$).

DISCUSSION

Thermal transfer is affected by dentin thickness (Goodis & others, 1989). According to Loney and Price (2001), thicker dentin specimens reduce temperature changes. While dentin has a relatively low thermal conductivity, the potential for pulpal damage is greater in deep cavities when the residual dentin thickness is small and the tubular surface area increases (Shortall & Harrington, 1998). Thus, the 0.5-mm dentin disc was used to standardize the leftover dentin and provide a more realistic value of thermal transfer.

Resin composite shade is another factor that could affect temperature change (Uhl & others, 2003). With conventional halogen light curing units, darker shades produce higher temperatures than lighter shades. However, for LED light curing units, there is a tendency for composites that have a lighter shade to produce higher temperatures (Uhl & others, 2003). This can be explained by their generally higher light transmittance for light at a wavelength of 470 nm, where there is the

Table 2: *Characteristics of the Groups*

LCU	Intensity of Light	Equipment	Exposure	Z250 (n)	Flow (n)
Halogen	600 mW/cm²	XL 2500	20 seconds	10	10
LED	400 mW/cm²	Ultrablue 1S	20 seconds	10	10
PAC	1450 mW/cm²	Apollo 95E	3 seconds	10	10

Table 3: *Mean Values of Knoop Hardness (KHN) in Each Appraised Depth for All the Photoactivation Methods with Filtek Z250*

	Halogen	LED	PAC
Surface	80.89 (8.53)[a,A]	76.44 (7.77)[a,A]	74.85 (6.53)[a,A]
1 mm	76.92 (4.81)[a,A]	75.08 (4.65)[a,A]	73.89 (8.23)[a,A]
2 mm	74.12 (5.38)[a,A]	73.48 (4.89)[a,A]	65.72 (12.58)[b,B]

Mean values followed by different small letters in the column differ statistically among themselves for the Tukey´s Test at the 5% level. Mean values followed by different capital letters in the line differ statistically for the photoactivation methods by the Tukey´s Test at the 5% level (Standard Deviation).

Table 4: *Mean Values of Knoop Hardness (KHN) in Each Appraised Depth for All the Photoactivation Methods with Filtek Flow*

	Halogen	LED	PAC
Surface	38.18 (7.35)[a,A]	36.54 (3.55)[a,A]	23.94 (7.77)[a,B]
1 mm	41.32 (9.16)[a,A]	39.50 (3.70)[a,A]	27.19 (4.91)[a,B]
2 mm	36.53 (7.45)[a,A]	35.01 (5.59)[a,A]	17.17 (7.91)[b,B]

Mean values followed by different small letters in the column differ statistically among themselves for the Tukey´s Test at the 5% level. Mean values followed by different capital letters in the line differ statistically for the photoactivation methods by the Tukey´s Test at the 5% level (Standard Deviation).

Table 5: *Mean Values of Temperature Rise (Celsius Degree)*

	Mean Values of Temperature Rise in Celsius Degree		
	Halogen	LED	PAC
Filtek Z250	1.36 (0.13)[a,A]	1.08 (0.20)[a,B]	0.83 (0.25)[a,C]
Filtek Flow	1.60 (0.19)[a,A]	1.15 (0.27)[a,B]	0.85 (0.30)[a,C]

Mean values followed by different small letters in the column differ statistically among themselves for the Tukey´s Test at the 5% level. Mean values followed by different capital letters in the line differ statistically for the photoactivation methods by the Tukey´s Test at the 5% level (Standard Deviation).

maximum absorption of camphorquinone (Uhl & others, 2003). For this reason, only one shade was used in this study.

According to McCabe (1985) and Shortall and Harrington (1998), the degree of temperature rise in the tooth is caused by the exothermic setting reaction of the composite during the polymerization process and the energy input of the light curing unit. The rate of the exothermic setting reaction is a function of the irradiance of the light curing unit, the composite chemical composition and the light transmission properties of the resin composite (Shortall & Harrington, 1998). As light passes through the composite, it is absorbed and scattered, attenuating the intensity and reducing the effectiveness of the light for resin polymerization as the depth increases (Rueggeberg, 1999; Knezevic & others, 2001). Since the resin composite should be cured in increments of 2-mm thicknesses to assure maximal polymerization of the material, this measurement was used in this study.

As Knoop Hardness provides a good parameter to estimate monomer conversion in a light-cured composite (Ferracane, 1985; DeWald & Ferracane, 1987; Rueggeberg & Craig, 1988), this was used as an indirect measure of the degree of cure in this study.

The temperature rise with the two composites used is higher in the case of photoactivation with a conventional halogen light curing unit than with LED and plasma arc sources (Table 5). With conventional halogen light curing units, the emitted radiation at the light curing tip is mainly visible light and infrared energy, which does not contribute to activation of photoinitiators (Hofmann & others, 2002; Deb & Sehmi, 2003). This could explain why the higher temperature produced by conventional halogen light curing units does not correlate with Knoop Hardness, when compared to LED source. The temperature rise during photoactivation was smaller with the LED light curing unit than with the conventional halogen source, but the mean values of Knoop Hardness were not statically different ($p>0.05$). Thus, the LED source promoted a similar degree of cure with lower temperatures since it emits a narrow wavelength of light (455 nm-486 nm) that correlates with the spectral absorbance range of camphoquinone (Parr & Rueggeberg, 2002).

Plasma arc curing sources have higher intensities of light emitted over a narrow range of wavelengths and, due to the high irradiance, offer a much shorter curing time in comparison to conventional halogen light units (Deb & Sehmi, 2003). However, the high irradiance delivered by the plasma arc source over a few seconds is not enough to produce optimum properties in resin composites and has a great influence on the degree of polymerization (Peutzfeldt, Sahafi & Asmussen, 2000). Thus, it can be suggested that plasma arc light curing units led to inefficient curing (Deb & Sehmi, 2003). This confirms the current study, where a plasma arc curing unit promoted lower temperatures, but also caused lower mean values of Knoop Hardness. Thus, the lower temperature during photoactivation cannot be regarded as an advantage, but has to be attributed

to incomplete activation of the materials by the plasma arc curing unit, which results in lower hardness values.

The cure temperature rise of a photoactivated resin composite is based on the total energy delivered to it (Rueggeberg, 1999; Yap & Seneviratne, 2001). The conventional halogen light curing unit produces 12.0 J/cm^2 (600 mW/cm^2 for 20 seconds), the LED produces 8.0 J/cm^2 (400 mW/cm^2 for 20 seconds) and the plasma arc produces 4.35 J/cm^2 (1450 mW/cm^2 for 3 seconds). The difference in energy produced by these units probably accounts for differences in the observed temperature changes.

With regard to resin composite composition, Shortall and Harrington (1998) concluded that temperature rise also relates to the light transmission characteristics of the resin composite. Masutami and others (1988) suggested that the resin exothermic reaction has a greater influence on temperature rise during curing than the light source. However, Lloyd, Joshi and Mcglynn (1986) and Strang and others (1988) reported that the major factor for a temperature increase during light-activated polymerization of resin composites is the energy absorbed during irradiation; whereas, the exothermic composite polymerization process is of secondary importance for the temperature increase. These findings support this study, where material composition does not affect the temperature rise observed with all light curing units (Table 5).

CONCLUSIONS

With Filtek Z250 and Filtek Flow resin composites, it was noted that, generally, there was no decrease in Knoop hardness as depth increased, with all photoactivation methods.

Comparing the photoactivation methods at all depths, there were no statistical differences ($p>0.05$) in Knoop Hardness values between conventional halogen and light-emitting diodes curing units.

Plasma arc curing units promoted statistically lower ($p<0.05$) Knoop Hardness values and temperature changes compared to other units.

The composite factor does not show any statistical difference in temperature rises for any light curing unit ($p>0.05$).

The conventional halogen light curing unit produced statistically higher temperatures ($p<0.05$) compared to other units.

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Fluoride Release and Neutralizing Effect by Resin-based Materials

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Clinical Relevance

The fluoride-releasing and neutralizing ability of resin-based materials are affected by the nature of fluoride incorporated into materials.

SUMMARY

This study evaluated the fluoride-releasing and neutralizing abilities of resin-based materials containing fluoride in water and aqueous lactic acid. Two composites, containing a low-solubility fluoride component (Heliomolar) and a fluoro-alumino-silicate glass (UniFil S), and two giomers, containing surface reaction type pre-reacted glass-ionomer filler (Beautifil) and full reaction type glass-ionomer filler (Reactmer paste), were used. Resin-modified glass-ionomer cement (Fuji II LC) was used as a control. The fluoride release and pH value in storage medium, after immersion in each material, was measured for 10 weeks. For UniFil S and Beautifil, the amount of fluoride released in acid solution markedly increased compared to storage in water ($p<0.05$). Although all materials, except Heliomolar, neutralized the storage media, the neutralizing ability of these resin-based materials in acid solution sharply decreased with aging of the specimens, except for Fuji II LC. These results suggest that the nature of fluoride incorporated into resin-based materials affect the fluoride-releasing and neutralizing ability of materials in water and aqueous lactic acid.

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INTRODUCTION

For evaluation of the effect of fluoride-releasing restorative materials on the potential for secondary caries inhibition, determining the amount of fluoride released from the materials in distilled/deionized water is generally investigated. However, the fluoride-releasing ability of a material is affected by the nature of the storage medium, particularly its pH value. In acid solution, the amount of fluoride released is reported to be significantly greater than in distilled/deionized water (Forss, 1993; Attin & others, 1999; Itota & others, 1999;

Karantakis & others, 2000; Behrend & Geurtsen, 2001; Sales & others, 2003). Since secondary caries occurs in a low pH environment around restorations, determining the fluoride released from materials in acidic conditions may be important for evaluating caries inhibition by materials.

On the other hand, it has been reported that glass-ionomer cements (GICs) and certain resin-modified GICs neutralize acid solutions (Nicholson, Czarnecka & Limanowska-Shaw, 1999a; Nicholson & others; 1999b; Czarnecka, Limanowska-Shaw & Nicholson, 2002). This effect is thought to be produced by the formation of a salt between the cement and the acid solution and the dissolution of soluble products from materials. The neutralizing ability of these materials, which changes the pH of the surrounding aqueous media, may contribute to the inhibition of secondary caries arising from the formation of lactic acid by bacteria.

Recently, numerous resin-based materials containing a fluoride-releasing source, normally a fluoride-rich glass or pre-reacted glass-ionomer filler, have been developed. Although these materials can be expected to prevent secondary caries by fluoride release (Itota & others, 2002; Yap & others, 2002; Han & others, 2002), it is not known whether they produce a greater fluoride release in acidic conditions and whether this also produces a marked neutralizing effect. As the nature of the fluoride incorporated into resin-based materials differs in different products, this may affect the fluoride-releasing and neutralizing ability of materials.

This study evaluated the fluoride-releasing and neutralizing ability of resin-based materials containing fluoride in distilled/deionized water and in lactic acid solution in order to determine whether the nature of fluoride affects these properties. The weight changes of the materials after immersion in storage media were also evaluated in order to examine whether the fluoride incorporated into the materials is disintegrated by acid solution.

METHODS AND MATERIALS

Test Specimens

Two resin composites containing an inorganic fluoride (Heliomolar, Ivoclar Vivadent AG, Schaan, Liechtenstein) and a fluoro-alumino-silicate glass (UniFil S, GC Co, Tokyo, Japan), two giomers containing surface reaction type pre-reacted glass-ionomer filler (Beautifil, Shofu Inc, Kyoto, Japan) and full reaction type pre-reacted glass-ionomer filler (Reactmer paste, Shofu Inc) were used. A resin-modified GIC (Fuji II LC Capsule, GC Co) was used as a control. The composition of these materials is given in Table 1.

Ten disk specimens of each material were prepared by a single operator. Resin-based material was placed in a Teflon mold (diameter of 10 mm, depth of 1.0 mm) mounted on a polymethylmethacrylate plate covered with a Melinex film (Toray, Tokyo, Japan) and compressed using a second plate covered with film, then light-cured for 40 seconds from the top and bottom sides using a visible light-curing unit (Visilux II, 3M, St Paul, MN, USA). Fuji II LC was mixed mechanically for 10 seconds in a Silamat device (Vivadent), placed in a mold, then light-cured for 40 seconds from the top and bottom sides. After setting, all specimens were removed from the molds and stored at 37°C and 100% relative humidity for 24 hours. The specimens were then ground by hand with a dry 1200-grit silicone carbide paper and their dimensions (diameter and thickness) and weight (W_0) were measured.

Measurement of Specimen Weight, pH Value and Fluoride Content of the Storage Medium

Five specimens of each material were immersed in individual plastic containers with 5 ml distilled/deionized water and another five specimens were individually immersed in 5 ml lactic acid solution which was adjusted to pH 4.0. After storage at 37°C for one week, the specimens were removed from their containers, wiped with a clean dry cloth, waved in the air for 15 seconds and weighed 60 seconds after removal from

Table 1. *Materials Used in This Study*

Material	Type	Compositions	Manufacturer
Heliomolar	Resin composite	Ytterbium trifluoride, silica filler, Bis-GMA, UDMA, decandiol dimethacrylate, catalyst	Ivoclar Vivadent AG, Schaan, Liechtenstein
UniFil S	Resin composite	Fluoro-alumino-silicate glass, silica filler, UDMA, dimethacrylate, catalyst	GC Co, Tokyo, Japan
Beautifil	Giomer	S-PRG, fluoro-boroalumino-silicate glass, Bis-GMA, TEGDMA, catalyst	Shofu Inc, Kyoto, Japan
Reactmer paste	Giomer	F-PRG, glass filler, UDMA, HEMA, catalyst	Shofu Inc, Kyoto, Japan
Fuji II LC Capsule	Resin-modified GIC	Fluoro-alumino-silicate glass, polyacrylic acid, HEMA, TEGDMA, UDMA, water, catalyst	GC Co, Tokyo, Japan

S-PRG=surface reaction type pre-reacted glass-ionomer
F-PRG=full reaction type pre-reacted glass-ionomer
Bis-GMA=2,2-bis [4-(2'-hydroxy-3'-methacryloyloxy-propoxy) phenyl] propane
HEMA=2-hydroxyethyl methacrylate
TEGDMA=triethylene glycol dimethacrylate
UDMA=urethane dimethacrylate

the solution (W_n). After measuring the weight, each specimen was placed in a new container with a fresh 5-ml aliquot of distilled/deionized water or lactic acid solution and storage was continued.

The pH value of the storage medium after immersion of the specimen was measured using an ion meter (660 pH Meter, Russell pH Ltd, Auchtermuchty, UK). The pH values of the distilled/deionized water and lactic acid solution without a specimen were also measured as controls.

After measurement of the pH value, 5 ml of each storage solution and 0.5 ml of acetic buffer solution (TISAB III, Orion Research Inc, Beverly, MA, USA) were mixed and fluoride concentrations were determined using a calibrated fluoride-specific electrode (Combination Electrode 96-09BN, Orion Research Inc) attached to an ion meter (model 720A, Orion Research Inc).

Measurements of the weight, pH and fluoride release were made each week for 10 weeks. Each phase of the experiment involved replacing the storage medium on a weekly basis as the specimen was aged and soaked. After 10 weeks, the specimens were removed from the storage media and reconditioned in a desiccator at room temperature until a constant desiccated weight was obtained (W_e). The weight changes by water sorption (W_{sor}) and water solubility (W_{sol}) were calculated and converted to a percentage of the initial desiccated weight (W_0) according to the following equations: $W_{sor} = 100 \times (W_n - W_0)/W_0$ and $W_{sol} = 100 \times (W_0 - W_e)/W_0$, where W_n is the sample weight after each period tested.

Statistical Analysis

Data were analyzed by ANOVA and Scheffe's test at a significance level of 0.05. Three-way ANOVA was used to investigate the effects of material, storage medium and aging on both fluoride release and weight change of the specimen by sorption. Two-way ANOVA was used to investigate the effect of material and storage medium on weight change of the specimen by solubility and pH value.

RESULTS

The cumulative amount of fluoride released from each material in distilled/deionized water and lactic acid solution is given in Figure 1 and Table 2. For clarity, the data are replotted for all materials except Fuji II LC and Heliomolar in Figure 2. All materials except Heliomolar showed a significantly greater amount of

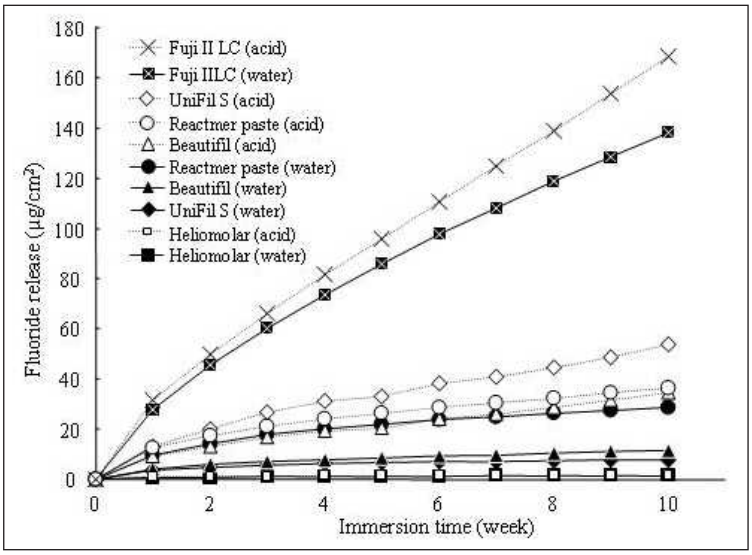

Figure 1. *The plots of the amount of fluoride release from all materials in distilled/deionized water and lactic acid solution. Note: The storage medium (water or lactic acid solution) was replaced weekly.*

Table 2. *Cumulative Amount of Fluoride Release, Percentage Weight Changes by Water Sorption (W_{sor}) and Water Solubility (W_{sol}) of Each Material in Distilled/Deionized Water and Lactic Acid Solution*

		Fluoride release (μg/cm²)		W_{sor} (%)		W_{sol} (%)
		1 Week	10 Weeks	1 Week	10 Weeks	
Heliomolar	Water	0.62±0.14[4]	1.34±0.20[5]	0.70±0.05[3]	0.76±0.07[3]	0.35±0.07[2]
	Acid	1.03±0.31[D]	1.49±0.40[D]	0.68±0.17[C]	0.60±0.04[C]	0.52±0.04[B]
UniFil S	Water	3.61±0.28[3]	7.95±0.74[4]	0.45±0.04[4]	0.55±0.02[4]	0.18±0.02[3]
	Acid	12.87±0.72[B]	53.95±2.73[B]	0.45±0.08[C]	0.20±0.05[D]	0.58±0.03[B]
Beautifil	Water	4.25±0.21[3]	11.69±0.49[3]	0.65±0.06[3]	0.77±0.01[3]	0.17±0.03[3]
	Acid	9.41±0.91[C]	34.54±0.91[C]	0.53±0.07[C]	0.34±0.06[D]	0.62±0.08[B]
Reactmer paste	Water	9.70±0.57[2]	28.48±2.09[2]	3.22±0.14[2]	4.97±0.09[1]	-1.23±0.08[4]
	Acid	12.50±0.54[B]	36.37±1.61[C]	3.18±0.11[B]	4.80±0.13[A]	-0.94±0.06[C]
Fuji II LC	Water	27.95±1.07[1]	138.04±2.68[1]	3.62±0.10[1]	3.35±0.11[2]	2.46±0.09[1]
	Acid	31.64±2.17[A]	168.36±6.71[A]	3.47±0.16[A]	3.28±0.11[B]	2.64±0.09[A]

Mean values in the same column identified by the same superscript are not significantly different (p>0.05). Pairs of means linked by horizontal lines indicate no significant difference between 1 week and 10 weeks values. Pairs of means linked by vertical lines indicate no significant difference between water storage and lactic acid storage.

fluoride release in lactic acid solution than in distilled/deionized water ($p<0.05$). The amount of fluoride release for UniFil S and Beautifil markedly increased in acid solution compared to water. Fuji II LC gave the greatest amount of fluoride release in both media, while Heliomolar gave the lowest ($p<0.05$).

The percentage of weight changes caused by water sorption and water solubility of each material in distilled/deionized water and in lactic acid solutions is shown in Figure 3 and Table 2. For clarity, the data are replotted for all materials except Fuji II LC and Reactmer paste in Figure 4. For the four resin-based materials, the weight change by water sorption of the specimens in acid solution was less than that in water after 10 weeks ($p<0.05$). The weight changes for Heliomolar, UniFil S and Beautifil in both media were below 1% over 10 weeks, while the Reactmer paste was greater than 3% after one week and showed a marked increase in weight over 10 weeks of storage. There was no significant difference in weight change for Fuji II LC stored in both media. For the weight change caused by solubility, the values for all materials in acid solution (except Reactmer paste) were significantly greater than in water ($p<0.05$). For Reactmer paste, the weight of the specimens after immersion in both media showed a small net gain even after desiccation.

Figures 5 and 6 and Table 3 show the pH of the distilled/deionized water and lactic acid solutions after immersion of test material over a one-week period at each period tested up to 10 weeks. In water, all materials except Heliomolar gave significantly higher pH values than the control after one week ($p<0.05$), but UniFil S and Reactmer paste showed no significant difference from the control at 10 weeks ($p>0.05$). Heliomolar produced a significantly lower pH value than the control for 10 weeks ($p<0.05$). In acid solution, all materials gave a significantly higher pH value than the control after one week ($p<0.05$). However, all materials except Fuji II LC showed a tendency for the pH to decrease with aging of the specimen and there was no significant difference between Heliomolar and the control after 10 weeks. When the storage medium pH value for each material was compared between the two media, only Fuji II LC at one week showed no significant difference ($p>0.05$).

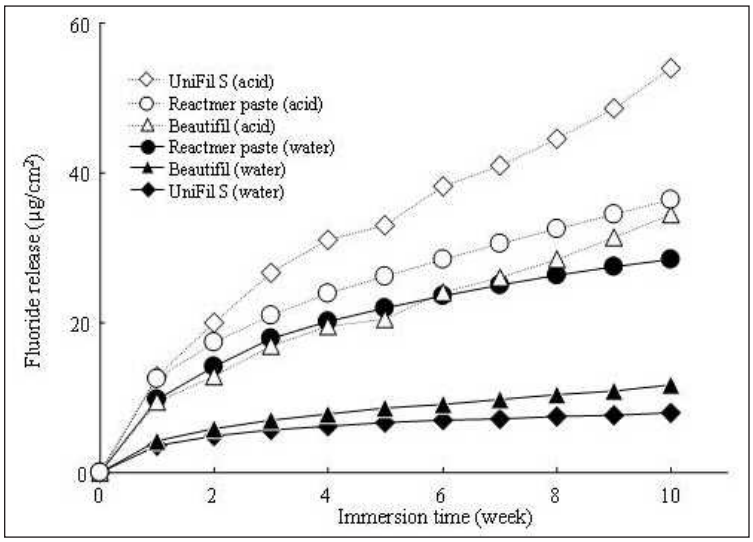

Figure 2. *The plots of the amount of fluoride release from the three resin-based materials in distilled/deionized water and lactic acid solution.*

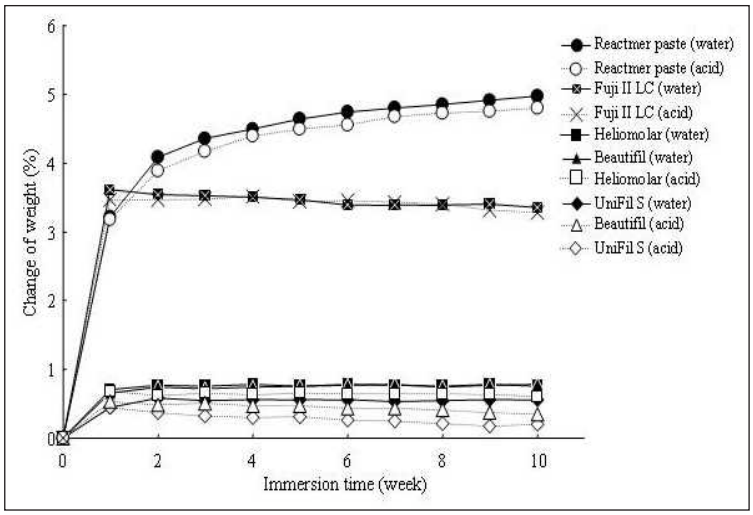

Figure 3. *The plots of the percentage of weight changes of the specimen by water sorption for each material group in distilled/deionized water and lactic acid solution. Note: The storage medium (water or lactic acid solution) was replaced weekly.*

DISCUSSION

The amount of fluoride released from all materials except Heliomolar was greater in lactic acid solution than in distilled/deionized water, and the amount of fluoride released from Beautifil and UniFil S very markedly increased in lactic acid solution compared to water. These observations may be explained by the nature of the different fluoride sources incorporated into these resin-based materials. Heliomolar produced the lowest fluoride release, because this material contains ytterbium trifluoride, which has extremely low solubility (Vermeersch, Leloup & Vreven, 2001). On the other hand, UniFil S contains fluoro-alumino-silicate glass filler, Reactmer paste contains pre-reacted glass-ionomer (PRG) filler and Beautifil contains both of these types of fillers as fluoride source. However, the PRG within Beautifil is similar to fluoro-almino-silicate glass, because the acid base reaction for this material is limited to the surface of the glass. It is likely that the glass used in UniFil S and Beautifil was readily disintegrated by lactic acid, because these kinds of glass have little or no glass-ionomer matrix phase, which

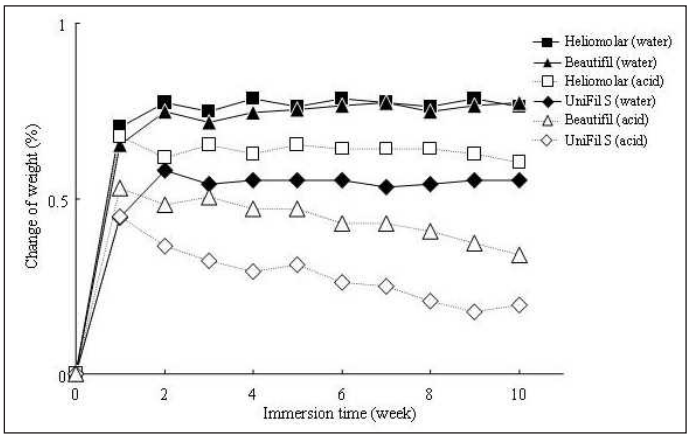

Figure 4. *The plots of the percentage of weight changes of the specimen by water sorption for the three resin-based material groups in distilled/deionized water and lactic acid solution.*

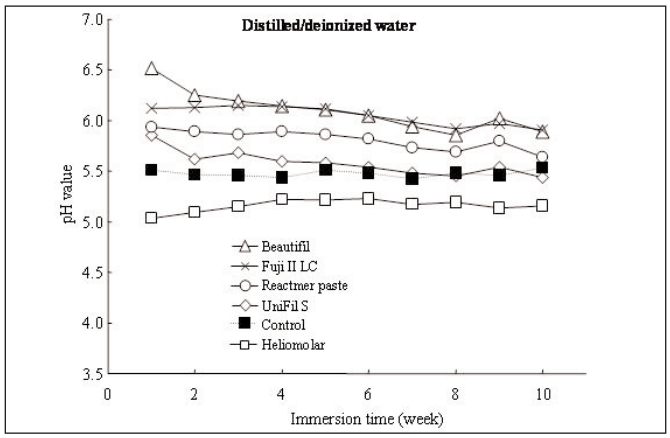

Figure 5. *The plots of the pH value of each material group in distilled/deionized water for 10 weeks. Note: Distilled/deionized water was replaced weekly.*

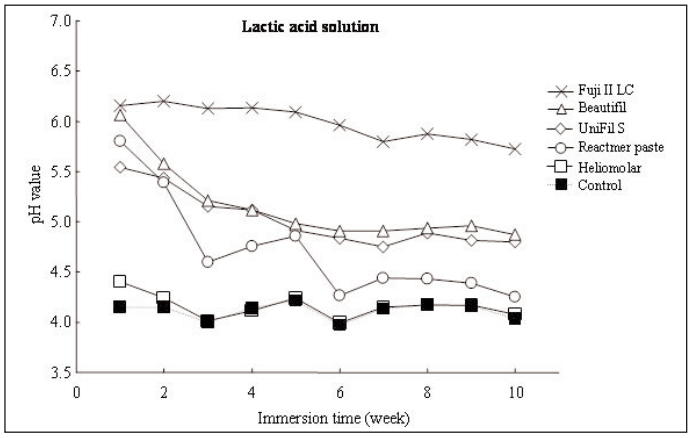

Figure 6. *The plots of the pH value of each material group in lactic acid solution for 10 weeks. Note: Lactic acid solution was replaced weekly.*

could account for the high fluoride release in acid conditions. The lower water sorption and higher water solubility of UniFil S and Beautifil in acid solution compared to that in water suggests that the weight loss was due to erosion and disintegration of the glass filler. In addition, a glass-ionomer matrix, which is easily penetrated by water, may be formed on the surfaces of the glass particles by reaction of the glass with lactic acid, and this may result in a significantly greater fluoride release from these materials. On the other hand, the PRG filler within Reactmer paste, in which the glass is fully consumed and converted to a hydrogel matrix, might be more resistant to acid attack and can suppress the surface reaction in lactic acid solution (Sales & others, 2003). The weight of Reactmer paste in this study showed a small net gain even after desiccation. This finding agrees with a previous report that Reactmer paste showed greater expansion and a gain in weight after desiccation for two weeks (McCabe & Rusby, 2004). As it is not likely that the water absorbed in Reactmer paste is tightly bound to the resin matrix, this gain might be caused by an insufficient desiccation of the specimen of Reactmer paste.

As for the neutralizing ability of these materials, far from neutralizing the storage medium, Heliomolar decreased the pH value of distilled/deionized water. The unpolymerized monomer released from Heliomolar may be respon-

Table 3. *Value of pH of the Distilled/Deionized Water and Lactic Acid Solution After Immersion of Each Material*

		pH value	
		1 week	**10 weeks**
Heliomolar	Water	5.03±0.14^{5}	5.16±0.10^{4}
	Acid	4.40±0.16^{D}	4.08±0.09^{D}
UniFil S	Water	5.86±0.05^{3}	5.44±0.03^{3}
	Acid	5.54±0.04^{C}	4.80±0.04^{B}
Beautifil	Water	6.52±0.04^{1}	5.89±0.03^{1}
	Acid	6.06±0.03^{A}	4.87±0.02^{B}
Reactmer paste	Water	5.93±0.09^{2,3}	5.64±0.04^{2}
	Acid	5.80±0.04^{B}	4.25±0.02^{C}
Fuji II LC	Water	6.12±0.10^{2}	5.90±0.04^{1}
	Acid	6.16±0.03^{A}	5.72±0.02^{A}
Control	Water	5.51±0.08^{4}	5.53±0.05^{2,3}
	Acid	4.15±0.04^{E}	4.03±0.05^{D}

Mean values in the same column identified by the same superscript are not significantly different (p>0.05). Pairs of means linked by vertical lines indicate no significant difference between water storage and lactic acid storage.

sible for decreasing the pH value of water, because it is known that the dissolution of ytterbium trifluoride causes no reduction in pH value of the storage medium (Young & others, 1996). Although all materials except Heliomolar, showed a greater neutralizing effect in acid solution at the initial period, the neutralizing ability of materials, except Fuji II LC, became weaker with aging of the specimens. Fuji II LC was the only material that retained a pH value of greater than 5.5 in acid solution after 10 weeks. The neutralizing effect in acid solution is an important property of the materials for secondary caries inhibition, because the organic acid which is produced by oral bacteria may be neutralized (Nicholson & others, 1999b). Stephan (1940) concluded that caries results from prolonged exposure to a pH below the critical value of 5.5. These results may suggest that glass-ionomer cements provide a caries-inhibitory effect not only by fluoride release but also through an acid neutralizing ability. Although some resin-based materials produced a neutralizing effect in acidic storage media, it is desirable that these materials should promote an even more rapid neutralization of the acid solution for caries inhibition.

CONCLUSIONS

In conclusion, resin-based materials containing fluoridated glass filler showed a greater fluoride release and more marked neutralizing effect in lactic acid solution compared to a material containing ytterbium trifluoride. However, a resin-modified GIC showed significantly greater fluoride release and neutralizing effect in acid solution compared to resin-based materials containing fluoride. Further investigations will be necessary to clarify the effect of fluoride incorporated into resin-based materials on secondary caries inhibition.

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Effect of Handpiece Maintenance Method on Bond Strength

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Clinical Relevance

Handpieces, which do not require the user to lubricate them, are available. Neither these handpieces, nor the use of more traditional lubricated handpieces, adversely affect the bond strength of current types of bonding agents.

SUMMARY

This study evaluated the effect of dental handpiece lubricant on the shear bond strength of three bonding agents to dentin. A lubrication-free handpiece (one that does not require the user to lubricate it) and a handpiece requiring routine lubrication were used in the study. In addition, two different handpiece lubrication methods (automated versus manual application) were also investigated. One hundred and eighty extracted human teeth were ground to expose flat dentin surfaces that were then finished with wet silicon carbide paper. The teeth were randomly divided into 18 groups (n=10). The dentin surface of each specimen was exposed for 30 seconds to water spray from either a lubrication-free handpiece or a lubricated handpiece. Prior to exposure, various lubrication regimens were used on the handpieces that required lubrication. The dentin surfaces were then treated with total-etch, two-step; a self-etch, two-step or a self-etch, one-step bonding agent. Resin composite cylinders were bonded to dentin, the specimens were then thermocycled and tested to failure in shear at seven days. Mean bond strength data were analyzed using Dunnett's multiple comparison test at an 0.05 level of significance. Results indicated that within each of the bonding agents, there were no significant differences in bond strength between the control group and the treatment groups regardless of the type of handpiece or use of routine lubrication.

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INTRODUCTION

Dentin bonding has undergone many changes in recent years and continues to evolve. New products and, perhaps more importantly, new techniques for their clinical application, are routinely introduced to the profession. Despite these changes, bonding remains relatively technique-sensitive, and bond strength can be

affected by many factors, including improper technique (Chan & others, 2003; Peutzfeldt & Asmussen, 2002; Van Meerbeek & others, 2003), inadequate preparation or contamination of the bonding surface substrate (Johnson & others, 1994).

The effect on bond strength of dentin contamination has been previously investigated (el-Kalla & García-Godoy, 1997; Hiraishi & others, 2003; Hitmi, Attal & Degrange, 1999; Johnson & others, 1994; Kaneshima & others, 2000; Sung & others, 2002). Contaminants that have been studied include saliva, blood, plasma, dental cements, caries-detecting dyes and irrigating solutions. The effects have varied depending on the contaminant and when the contamination occurred during the bonding process.

It has been anecdotally suggested that contamination with lubrication oils used for maintaining dental air-turbine handpieces may adversely affect bond strength (Christensen, 1999). Residual lubricants retained in the handpiece can be displaced from the handpiece onto the tooth surface during operative procedures. It is these contaminants that some researchers believe may reduce bond strength if not properly removed. Lubrication-free air-turbine handpieces (those that do not require the user to lubricate them) are commercially available. However, these handpieces should not be interpreted as being devoid of lubrication, for their bearings are lubricated at the factory and bearing lubricant is released over time. Therefore, lubrication-free handpieces still have the potential to release some lubrication oils during use.

A number of studies have examined the effect of contamination from handpiece lubrication oils on enamel and/or dentin bond strengths (Knight, Draughn & Evans, 1999; Powers, Finger & Xie, 1995; Rosa & others, 2000; Xie, Powers & McGuckin, 1993), but the results have been equivocal. None of these studies used the most recently developed bonding agents. This investigation determined the effect of handpiece lubricant and the method of applying it to handpieces on dentin shear bond strength when using a total-etch, two-step; a self-etch, two-step and a self-etch, one-step dentin bonding system.

METHODS AND MATERIALS

One hundred and eighty non-carious recently extracted human third molars stored in a Chloramine-T solution were used in this study. The occlusal surfaces of the teeth were removed by sectioning with a water-cooled diamond saw (Buehler, Lake Bluff, IL, USA) to prepare flat dentin surfaces. Each tooth was mounted in autopolymerizing acrylic resin (Tray Resin, Dentsply/Caulk, Milford, DE, USA) using cylindrical polytetrafluoroethylene molds, placing the prepared dentin surface approximately 2 mm above the end of the resin cylinder. After the resin had polymerized, the dentin surface was hand finished with wet 400- and 600-grit silicon carbide abrasive papers and examined at 8x magnification using a stereomicroscope (Zeiss Stemi SR, Carl Zeiss Inc, Thornwood, NY, USA) to ensure that all enamel had been removed from the bonding area. The teeth were randomly divided into 18 treatment groups of 10 teeth each. The prepared teeth were stored at room temperature in distilled water prior to bonding.

Two dental air-turbine handpieces with different maintenance requirements were used. The Star 430 SWL (DentalEz Group, Lancaster, PA, USA) does not require routine lubrication by the end-user, because it has factory pre-lubricated bearings. The W&H 98L (W&H DentalWerk, Bürmoos, Austria) requires lubrication after each use; the lubricant can either be applied manually or by using an automatic-processing station. Only one handpiece of each type was used to help standardize treatments of the various groups. Both handpieces were maintained and cleaned following manufacturer's recommendations. The W&H handpiece was manually lubricated with the manufacturer's aerosol cleaner/lubricant precisely as recommended. Automatic lubrication of the W&H handpiece was accomplished using the Assistina Plus 301 Cleaning and Lubricating Unit (W&H DentalWerk). Because clinicians have anecdotally reported that air-turbine handpieces have greater longevity if they are relubri-

Figure 1. *Specimen exposure to handpiece spray.*

Table 1: *Bonding Systems Used*

Material	Manufacturer	Type of DBA
Single Bond	3M ESPE	Total-etch, two-step
Clearfil SE Bond	Kuraray	Self-etch, two-step
One-Up Bond F	Tokuyama	Self-etch, one-step

Table 2: *Description of Dentin Treatment Prior to Bonding*

Group	Lubrication Regimen
Control	No exposure of dentin to handpiece
Star	Exposure of dentin to Star handpiece
W&H auto pre- (manufacturer recommended)	Exposure of dentin to W&H handpiece: automated lubrication of handpiece before autoclaving
W&H man pre- (manufacturer recommended)	Exposure of dentin to W&H handpiece: manual lubrication of handpiece before autoclaving
W&H auto pre-/post	Exposure of dentin to W&H handpiece: automated lubrication of handpiece before and after autoclaving
W&H man pre-/post	Exposure of dentin to W&H handpiece: manual lubrication of handpiece before and after autoclaving

cated after exposure to the rigors of steam sterilization, two additional test groups were added for the W&H handpiece. In these groups, in addition to the normal pre-sterilization lubrication, the handpiece was also lubricated following sterilization. In one group, this was done manually, while in the other group, automatic lubrication was used. Between each use, the handpieces were sterilized using a steam autoclave (model 2540EK, Tuttnauer, Ronkonkoma, NY, USA). After lubrication and prior to use, the handpieces were run for 30 seconds at maximum speed with distilled water coolant spray to purge the handpiece of potential gross amounts of lubricant (Knight & others, 1999). Prepared dentin surfaces were then exposed to 30 seconds of water coolant spray from the handpiece operating at full speed approximately 10 mm from the dentin surface. This distance was determined by inserting a #56 bur fully into the handpiece chuck, as would be done clinically (Figure 1). After exposure to the treatment method, the dentin surface was rinsed and blotted with cotton gauze, leaving the dentin slightly moist. Table 1 lists the bonding products used in this study.

The manufacturer's directions for applying each dentin bonding agent (DBA) were followed. For photo-polymerization, a halogen-based Optilux 501 visible-light curing unit (Sybron Dental Specialties, Orange, CA, USA) was used. The adequacy of its irradiance level was measured using a laboratory-grade laser power meter (Molectron Power Max 500D with PM10 probe, Coherent Molectron, Portland, OR, USA) and was found to be approximately 1000 mW/cm^2. After application of the bonding agent, Z100 resin composite (3M ESPE, St Paul, MN, USA) was placed onto the treated dentin surface using a 4-mm high split polytetrafluoroethylene mold held in place by a positioning ring. The resin was placed in 2-mm increments and each increment was photo-polymerized for 40 seconds. After polymerization of the final increment of resin composite, the positioning ring and mold were removed and the specimens stored in 37°C distilled water. Forty-eight hours after preparation, the specimens were thermocycled for 500 cycles between 5°C and 55°C in distilled-water baths with a dwell time of 30 seconds for each bath and a transfer time of 10 seconds. Seven days after bonding, the specimens were tested to failure in shear at a crosshead speed of 0.5 mm/minute using a perforated steel ring attached by a chain to a universal testing machine (Series 1000, Tinius Olsen, Willow Grove, PA, USA). The dentin treatment groups to which each of the bonding agents was applied are listed in Table 2. The primary scientific issue of interest was the evaluation of differences between the control group and each of the treatment groups for each of the three bonding agent classes. These planned (*a priori*) contrasts were evaluated using Dunnett's tests at an 0.05 level of significance.

The debonded surfaces were examined using a stereomicroscope at 8x to determine the failure mode. Failures were classified as Adhesive, Cohesive or Mixed and were defined as follows: Adhesive failures were those that occurred at the resin composite/dentin interface; Cohesive failures occurred within the resin composite and/or dentin interface and Mixed failures were a combination of Adhesive and Cohesive ones.

RESULTS

Mean bond strength results for the bonding agents are shown in Tables 3, 4 and 5. For the total-etch, two-step bonding agent (Single Bond), there were no significant differences in bond strength between the control and the treatment groups. For a majority of the groups, most failures were cohesive in nature. Similarly, no significant differences in bond strength were noted for the self-etch, two-step product (Clearfil SE Bond). A majority of the failures in most groups, however, were mixed. For the self-etch, one-step bonding agent (One-Up Bond F), although there were more adhesive failures and a trend towards lower bond strengths when manual lubrication was used, none of the groups' mean bond strength values differed significantly from the control group.

DISCUSSION

Demands by the dental professional for dentin bonding agents that are easier to use and faster to apply have

resulted in the development of self-etching and self-priming products. These products usually involve fewer application steps and are, therefore, simpler to use and require less chair time than traditional total-etch three-step bonding agents. Total-etch three-step and some of the simplified bonding agents have been shown to provide good clinical service (Van Meerbeek & others, 2003). The most recently marketed bonding products use an even simpler application technique than earlier ones. The procedure involves application of a single solution that etches, primes and bonds in one step. These newer bonding agents, however, do not appear to perform as well in laboratory bond strength tests (Van Meerbeek & others, 2003).

Table 3: *Mean Shear Bond Strengths for Single Bond (MPa)*

Group	Mean ± st dev	Failure Modes A	C	M
Control	26.5 ± 5.7	0	9	1
Star	26.0 ± 3.1	0	9	1
W&H auto pre- (manufacturer recommended)	24.8 ± 3.9	0	10	0
W&H man pre- (manufacturer recommended)	26.9 ± 6.0	0	6	4
W&H auto pre-/post	25.5 ± 3.6	0	5	5
W&H man pre-/post	27.9 ± 5.0	0	5	5

n = 10
A = Adhesive failure at the resin composite/dentin interface
C = Cohesive failure within the resin composite and/or dentin
M = Combination of adhesive and cohesive failures
No significant differences were found between the control group and each individual experimental group.

Table 4: *Mean Shear Bond Strengths for Clearfil SE Bond (MPa)*

Group	Mean ± st dev	Failure Modes A	C	M
Control	25.0 ± 5.3	0	1	9
Star	24.0 ± 5.6	1	3	6
W&H auto pre- (manufacturer recommended)	20.5 ± 7.0	3	0	7
W&H man pre- (manufacturer recommended)	21.3 ± 4.8	3	1	6
W&H auto pre-/post	25.6 ± 4.1	0	8	2
W&H man pre-/post	23.8 ± 4.6	1	1	8

n = 10
A = Adhesive failure at the resin composite/dentin interface
C = Cohesive failure within the resin composite and/or dentin
M = Combination of adhesive and cohesive failures
No significant differences were found between the control group and each individual experimental group.

Table 5: *Mean Shear Bond Strengths for One-Up Bond F (MPa)*

Group	Mean ± st dev	Failure Modes A	C	M
Control	14.5 ± 4.0	8	1	1
Star	17.0 ± 4.4	0	0	10
W&H auto pre- (manufacturer recommended)	19.3 ± 5.9	3	0	7
W&H man pre- (manufacturer recommended)	14.0 ± 4.9	5	0	5
W&H auto pre-/post	19.6 ± 3.8	1	3	6
W&H man pre-/post	10.0 ± 5.2	8	0	2

n = 10
A = Adhesive failure at the resin composite/dentin interface
C = Cohesive failure within the resin composite and/or dentin
M = Combination of adhesive and cohesive failures
No significant differences were found between the control group and each individual experimental group.

The effect on the bond strength of these new products to dentin that has been contaminated with handpiece lubrication oils has not been evaluated. Theoretically, total-etch procedures should remove any minute amounts of handpiece lubricant from the tooth substrate surfaces. Self-etching bonding agents should also possess the inherent acidity required to degrade lubrication contaminants. However, in contrast to total-etch products, where the etchant is rinsed from the tooth surface following use, self-etching products employ an etchant that is not removed by rinsing following use. As a result, there is the potential for lubricants to remain on the dentin surface despite application of the etchant. It is possible that the contaminants will interfere with the basic process of mechanical interlocking between bonding agent and dentin that is responsible for adhesion.

Previous studies that have examined the effect of handpiece oil contamination on shear bond strength to dentin have produced equivocal results. In one study, contamination resulted in a significant decrease for one

bonding agent but a slight increase for another (Xie & others, 1993). In a second study, contamination caused a significant decrease in bond strength (Powers & others, 1995). The current study found that none of the tested bonding agents exhibited a significant difference in bond strength between the control and the experimental groups. For the self-etch, one-step bonding agent (One-Up Bond F), there were more adhesive failures and a trend toward lower bond strength values when manual lubrication was used, but again the differences were not significant. These results should be reassuring for clinicians, because it indicates that they need not be concerned about the influence of residual handpiece lubricant on dentin bond strength. However, because bonding remains a technique-sensitive procedure, proper isolation of the treated teeth and meticulous adherence to manufacturers' instructions should be followed. Future research should investigate the effect of oil contamination on the dentin bond strength of a larger number of product brands within each bonding agent class.

CONCLUSIONS

Under the conditions of this study, the handpiece maintenance method did not produce a significant difference in bond strength when using a total-etch, two-step; a self-etch, two-step or a self-etch one-step dentin bonding agent.

Disclaimer

The opinions expressed in this paper are those of the authors and do not represent the official opinion of the United States Air Force, the United States Navy, the Department of Defense or the United States Government. The use of specific products in this study does not imply their endorsement.

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The Influence of Sodium Hypochlorite and Root Canal Sealers on Post Retention in Different Dentin Regions

L Muniz • P Mathias

Clinical Relevance

The irrigant solution and endodontic cement used for root canal treatment can significantly affect post retention when the posts are luted with dual-cured composite cement.

SUMMARY

This study evaluated the influence of 5.25% NaOCl irrigant and root canal sealers on post retention in different dentin regions. Seventy-two human incisors were decoronated at the cemento-enamel junction and randomly divided into six groups (n=12) according to irrigant and sealer technique: G1-Distilled water (DW) without sealer; G2-DW + AH Plus (Dentsply/Maillefer); G3-DW + Endofill (Dentsply/Maillefer); G4-5.25%NaOCl without sealer; G5-5.25% NaOCl + AH Plus; G6-5.25% NaOCl + Endofill. Specimens were stored in a humid environment for 30 days at 37°C and were prepared with FRC Postec's drills for post insertion. The posts were cemented with Excite DSC/Variolink II (Ivoclar/ Vivadent). The specimens were sectioned through their long axis into three dental slices approximately 2.5 mm each, representing the cervical (C), middle (M) and apical (A) thirds of the root preparation. After calculating the adhered area of the specimens, they were submitted to the push-out test in a universal testing machine. The data were submitted to an analysis of variance (ANOVA) at a 5% significance level and to the Tukey test ($p<0.05$). The mean values (MPa) obtained for cervical, middle and apical areas of the root preparation, respectively, were: G_1=8.6; 12.5 and 14.3, G_2=13.5; 15.4 and 16.9; G_3=6.9; 10.0 and 12.1; G_4=13.0; 14.9 and 15.4; G_5=11.3; 13.5 and 18.0; and G_6=11.0; 11.8 and 11.5. Based on the results, the eugenol-based sealer (Endofill) resulted in significantly lower mean retention strength values compared with the resin-based sealer (AH Plus). The apical region showed the greatest retention. The lowest resistance to dislodgment was found in the cervical region, mainly in the groups that used distilled water for irrigating the root canal.

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INTRODUCTION

The use of pre-fabricated fiber posts adhesively cemented can potentially reduce the incidence of root fractures (Ferrari & others, 2000c). This is mainly because the elasticity modulus of fiber posts is closer to dentin than

that of metal posts (Ferrari, Vichi & Godoy, 2000b; Heidecke, Butz & Strub, 2001).

In spite of the advantages, the indication of fiber posts for teeth with a remaining coronal structure smaller than 2 mm was limited (Ferrari & others, 2000c), mainly due to the difficulty of adhesion to intracanal dentin (Patierno & others, 1996; Manocci & others, 2001). This difficulty may be related to modification of the adhesive substrate after endodontic procedures (Goldman, De Vitre & Pier, 1984; Schwartz, Murchison & Walker, 1998; Nikaido & others, 1999; Morris & others, 2001).

Endodontic treatment includes chemical and mechanical preparation of the pericanal dentin, which is the substrate for application of the adhesive system. Sodium hypochlorite is the substance most used in root canal preparation due to its antimicrobial properties. It acts by removing organic tissues and denaturing protein, including the collagen fibers in dentin (Estrela & others, 2002). It is important to note that collagen fibers are the fundamental element for hybrid layer formation (Nakabayashi, Kojima & Masuhara, 1982). With regard to filling the root canal, the majority of the endodontic sealers used contain eugenol. These products might adversely affect the adhesion process, because residual eugenol impregnated in the root canal walls might interfere with the polymerization of resin-based materials (Tjan & Nemetz, 1992; Hagge, Wong & Lindemuth, 2002). However, this effect of eugenol has been questioned (Schwartz & others, 1998; Burns & others, 2000).

Because it is difficult to directly visualize the sealer during the adhesive procedure, especially in the middle and apical root thirds and, because of the different regional characteristics of intracanal dentin (Ferrari & others, 2000a), it becomes necessary to assess the behavior of adhesive systems at the various levels of the root. It is possible to test adhesive strength with the use of mechanical tests that analyze specimens with reduced areas (Yoshiyama & others, 1998; Gaston & others, 2001; Ngoh & others, 2001).

This study assessed the influence 5.25% NaOCl irrigant solution and different endodontic sealers on the retention of fiber posts luted with a resin-based cement.

METHODS AND MATERIALS

Seventy-two human incisors with root sizes ranging between 14 mm and 16 mm were selected for this study. The teeth were stored in a 0.5% chloramine T solution until they were used.

The specimens were cleaned and the crowns sectioned at the cemento-enamel junction (Patierno & others, 1996) using double-faced diamond disks (#7020, KG Sorensen Ind, Barueri-SP, Brazil) at low speed under cooling and then discarded. After the apical foramen was enlarged using K-files #15 to 30, the root canal was reamed of the initial thirds with a #12 Batt drill. During these procedures, the root canal was irrigated with 10 mL of distilled water.

Table 1: *Division of the Experimental and Control Groups According to the Factors Under Study (n=12)*

Irrigating Solution	Endodontic Sealer	Regions
Distilled Water (36 teeth)	None	Cervical Middle Apical
	AH Plus	Cervical Middle Apical
	Endofill	Cervical Middle Apical
5.25% NaOCl (36 teeth)	None	Cervical Middle Apical
	AH Plus	Cervical Middle Apical
	Endofill	Cervical Middle Apical

The specimens were randomly divided into groups according to the factors under study (Table 1).

Endodontic Treatment

The working length was determined by viewing the tip of a #30 file and allowing a 1-mm withdrawal. All the teeth were instrumented with #35, 40, 45, 50 and 55 K-files, while 5 mL of the chemical substance under study was used to irrigate the preparation after each instrument (distilled water or 5.25% NaOCl). After apical preparation, instrumentation of the canal was done with #60, 70 and 80 K-files, also using 5 mL of the tested chemical substance at each change of instrument. Each file was used for three minutes, totaling 24 minutes of chemical-mechanical preparation for each tooth. Final irrigation was done with 5 mL of distilled water (Morris & others, 2001) for all groups.

The prepared canals were blotted with #55 (Dentsply/Maillefer, Ballagues, Switzerland, lot: 2080) paper points and obturated filled with #55 gutta-percha cones (Dentsply/Maillefer, lot: 5710) with either (1) Endofill (eugenol-based endodontic sealer), (2) AH Plus (resin-based endodontic sealer) or (3) no sealer (control condition), utilizing the lateral condensation technique. After cutting away the excess gutta-percha, the coronal access was sealed with temporary filling cement (Cotosol–Vigodent S/A Ind, Rio de Janeiro–RJ, Brazil) and stored in a humid environment at 37°C for 30 days.

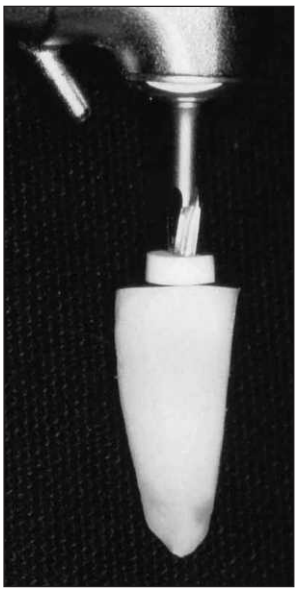

Figure 1. *Modeling of the canal for the pre-fabricated post.*

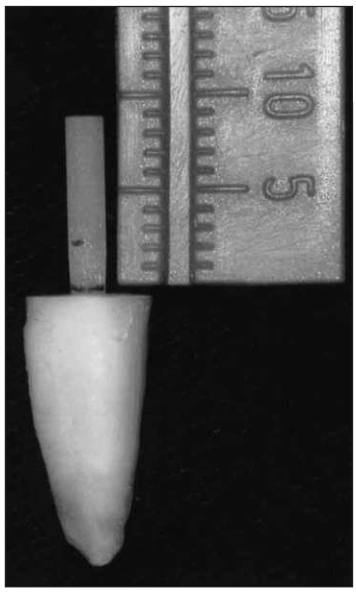

Figure 2. *Testing the post inside the root canal and the 2-mm measurement of the post for cutting.*

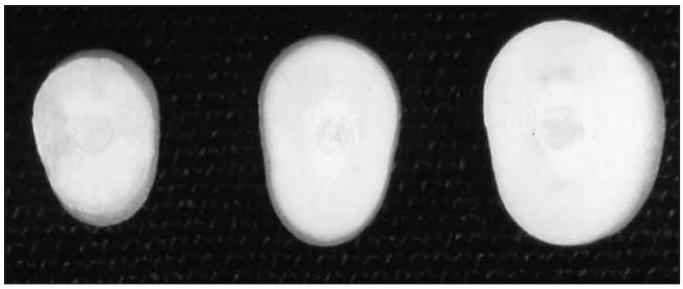

Figure 3. *Root slabs representing the cervical, medium and apical thirds of the cemented post.*

Root Canal Preparation for Posts and Adhesive Cementing

With #3 Gates Glidden drills, 11-mm to 12-mm of obturation material was removed from inside the root canal, maintaining 3-mm of gutta-percha in the apical third of the specimen. Post preparation was done with a #3 pilot reamer and a #3 reamer (Ivoclar/Vivadent, Schaan/ Liechtenstein) (Figure 1), using 5 mL of distilled water to irrigate the canal during and after modeling.

The fiberglass posts (FRC Postec–No 3, Ivoclar/Vivadent, lot: E94033) were tested inside the canal (Figure 2) and sectioned with a double-faced diamond disk (#7020, KG Sorensen Ind) 2 mm above the specimens' coronal margin. The posts were cleaned with 70% isopropyl alcohol, silanated (Monobond-S-Ivoclar/Vivadent, lot: e10084) and stored for 60 seconds in a closed receptacle. The surplus silane was removed with a short blast of air.

The resin-based system Variolink II (Ivoclar/Vivadent) was used to cement the posts. The root canal was etched with phosphoric acid at 37% (TotalEtch, Ivoclar/Vivadent, lot: E44932) for 15 seconds, washed for 10 seconds and dried with paper points. A dual-cured adhesive agent (Excite DSC, Ivoclar/Vivadent, lot: E27904) was applied with an extra-fine microbrush (Ferrari, Vichi & Grandini, 2001), and after 20 seconds, the surplus was removed with absorbent paper tips with a short blast of air to evaporate the solvent. The prepared resin-based cement Variolink II (Ivoclar/Vivadent, lot: E14434) was placed inside the root canal with lentulo drills (#40). The post was introduced into the root canal, the surplus cement was removed from the coronal area and the exposed margins were photopolymerized (Optilight 600, Gnatus Equip Med Odont Ltda, calibrated to ±500mW/cm^2) for 60 seconds. The specimens were stored for 24 hours in an environment with 100% humidity and then immersed in distilled water at 37°C for 24 hours (Patierno & others, 1996).

Obtaining the Test Specimens and Push-out Test

The specimens were sectioned perpendicular to the long axis with a diamond disk (Extec 1010 Extec Corp, Enfield, CT, USA) into approximately 2.5-mm slices under cooling (Frankenberger & others, 2000) (Figure 3).

With a digital caliper and 4x magnifying glass, the height of the slices and their internal diameters were measured, greatest base and smallest base, to calculate the adhered area of each test specimen.

The studies that use the push-out methodology tried to obtain tapered specimens to favor extrusion of the posts during mechanical testing and to reduce the difficulty of calculation of the area from mathematical formulas for regular geometrical figures (Wakefield & others, 1998; Frankenberger & others, 2000). It is not always possible to obtain these figures in the root canal and, although an attempt was made to standardize the shape of the canal preparation, many specimens in this study presented one or two elliptic-shaped bases, making it difficult to calculate the adhered area of each specimen. The lateral areas of these solids were calculated integrating complex mathematical functions used to generate the figure in the tri-dimensional space. Calculation of the double integrations was done using the computer program Maple, version 5.1 (Maple, Waterloo Inc, Waterloo, Canada).

The resultant area of the solid with circular base of ray R and elliptic top of axes E_1 and e_1 (Figure 4A) can be expressed as:

$$\text{Lateral area} = \int_0^{2\pi}\int_0^{1} f(t,s)\,ds\,dt \quad \text{where:}$$

$f(t,s)=[R\cos(t),R\sin(t),0]+s[((e_1/2)-R)\cos(t),((E_1/2)-R)\sin(t),h]$

$=\{[R+s((e_1/2)-R)]\cos(t),[R+s((E_1/2)-R)]\sin(t),sh\} s\in(0,1), t\in(0,2\pi)$.

The lateral area of the solids with elliptic base and top of axes e_1 and E_1 and e_2 and E_2, respectively (Figure 4B), can be expressed as:

Lateral area = $\int_0^{2\pi}\int_0^1 g(t,s)dsdt$ where:

$g(t,s)=[(e_1/2)\cos(t),(E_1/2)\sin(t),0] + s[((e_2/2)-(e_1/2))\cos(t), ((E_2/2) - (E_1/2))\sin(t), h]$

$s \in (0,1), t \in (0, 2\pi)$.

The test specimens were fixed in a device fitted to the Universal Testing Machine (EMIC/Equipamento e Sistemas de ensaio Ltda, Curitiba, Brazil). A tip 0.8-mm in diameter (Figure 5) was placed over the smallest base of the test specimen and compression force was applied in an apical-coronal direction (load cell 50 Kgf) at a speed of 0.5 mm/minute until the post piece was dislocated (Patierno & others, 1996).

Statistical Methods

The experimental design used was a factorial Split Plot arrangement, distributing the factors irrigation solution (two conditions) and sealer (three conditions) in the main plots; as sub-plots, the factor region was considered (three levels).

Statistical analysis of the data was performed using the analysis of variance (ANOVA) and Tukey Test at the 5% level.

RESULTS

Statistically significant differences were found for the factors' irrigation solution, sealer and region. The results are presented in Tables 2, 3 and 4.

The analysis of the irrigation substance (Table 2) shows lower mean retention values for distilled water in comparison with 5.25% NaOCl in the cervical third.

With regard to the endodontic sealers (Table 3), AH Plus presented greater mean retention values than Endofill in all thirds when the irrigation substance was distilled water, and in the apical third when NaOCl was used. There was no statistical difference among the groups that used the eugenol-based sealer and those that did not use sealer (control condition).

Analysis of the regions considering the different treatments (Table 4) demonstrated significant differences between the apical and cervical thirds, with better behavior of the apical third, except when NaOCl was used with Endofill or with the control condition. The comparison between the middle and cervical thirds shows greater mean values of retention in the middle third when distilled water was used with Endofill or with the control condition (without sealer). When compared to the middle third, the apical third presented greater retention mean values only in association with NaOCl at 5.25% and AH Plus.

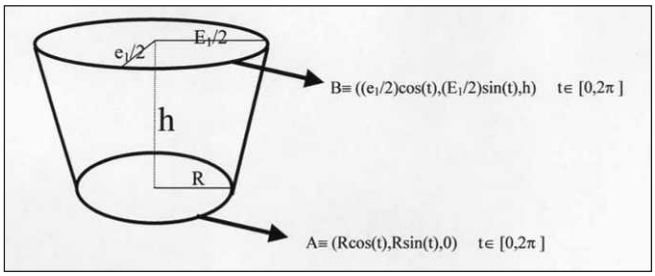

Figure 4A. *Solid with circular base and elliptic top.*

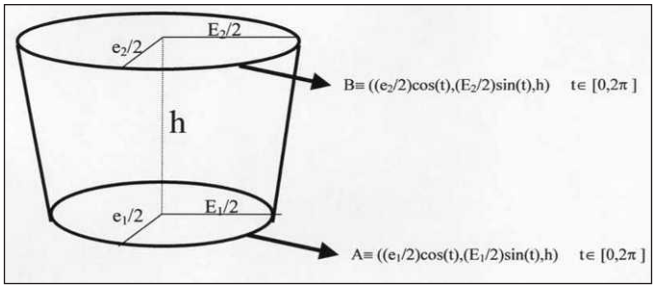

Figure 4B. *Solid with elliptic base and top.*

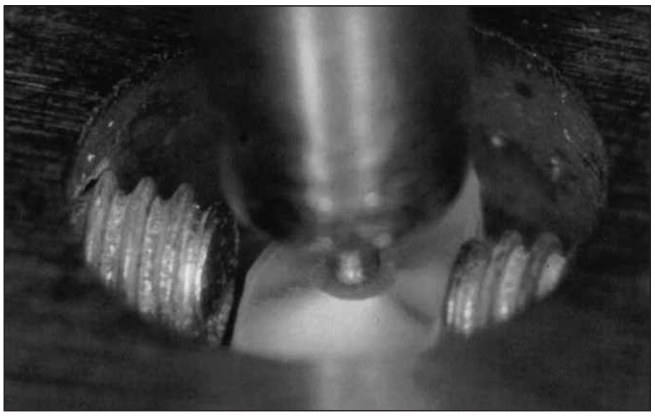

Figure 5. *Application of load on a piece of post.*

DISCUSSION

In this study, the endodontic procedure and the protocol for the adhesive cementation of fiberglass posts are performed in the clinical situations, except for the use of distilled water as an irrigating solution or for filling the root canal without endodontic sealer (control conditions). Contrary to this study, some studies did not use posts to assess post retention (Gaston & others, 2001; Ngoh & others, 2001), while others used longitudinal cuts of the specimens (Burns & others, 2000). The specimen cutting methodology avoids important

factors that make intracanal cementing difficult, as it favors visualization of the space prepared for placement of the post and removal of filling sealer residues. This methodology also allows for a greater reach of light during photopolymerization and significantly reduces the cavitary factor associated with polymerization of the resin-based luting materials (Bouillaguet & others, 2003). In this study, the posts were cemented into the root canal, enabling the reproduction of polymerization shrinkage as a cavitary factor compatible with clinical procedures.

An analysis of Table 2 allows one to observe differences between 5.25% NaOCl and distilled water only in the cervical third, which may be explained by the greater dentin permeability of this region (Ferrari & others, 2000a), making it more susceptible to action of the chemical substances used in preparing the root canal. Although the substance/filling sealers interaction was not assessed in this study, analysis of the irrigation substance on the adhesive substrate should be assessed in the control group for the sealer since the filling sealer comes into contact with the dentin after action of the irrigating substances.

The lower retention values observed for distilled water in the cervical third of the tooth may be justified by the need for a mechanical preparation of the root canal, which determines the formation of scrapings of dentin which are deposited into the dentin tubules (Becker & Woollard, 2001; Estrela & others, 2002). During the experimental phase, there was great difficulty in maintaining the patency of the canal, as distilled water is not capable of dissolving organic matter, compared to 5.25% NaOCl (Guerisolli, Souza Neto & Pécora, 1998; Estrela & others, 2002). Organic tissue remaining on the teeth favors the formation of a smear layer rich in organic components, making it difficult for acid substances to act (Yamada & others, 1983). Thus, the action of 5.25% NaOCl on the organic tissue may have favored better conditioning of the dentin substrate with phosphoric acid during adhesive procedures.

Distilled water is not used as an irrigating solution in endodontic treatment. It was used in this study to establish a control, enabling assessment of the effects of the 5.25% NaOCl treatment during endodontic procedures. However, further studies should be conducted to analyze other chemical substances used in endodontic treatment, such as Chlorexidine, EDTA and NaOCl, itself, in other concentrations.

The analysis of the factor sealer (Table 3) did not show statistically significant differences between the control (without sealer) and Endofill. However, the retention means in the groups in which the sealer was not used have been shown to be greater, being close to the critical value of significance for the middle and apical thirds, when 5.25% NaOCl was used as the irrigating solution. The canal filling without sealer does not represent a clinical reality but was used to establish a control for endodontic seal-

Table 2: *Means (SD) (in MPa) of Retention Values of Posts Considering the Effect of Chemical Substances in the Dentin Regions and in the Control Condition for the Factor Sealer*

Substance	Without Sealer		
	Cervical	Middle	Apical
Distilled Water	8.6 (± 6.2) a	12.5 (± 6.7) a	14.3 (± 4.8) a
5.25% NaOCl	13 (± 6.1) b	14.9 (± 4.6) a	15.4 (± 3) a

The comparisons are made per column, vertically. Different letters identify statistically significant differences (p<0.05/Tukey LSD=3.82).

Table 3: *Means (SD) (in MPa) of the Post Retention Values Considering the Interaction of Endodontic Sealers with Chemical Substances and with the Dentin Regions*

Sealer	Distilled Water			5.25% NaOCl		
	Cervical	Middle	Apical	Cervical	Middle	Apical
With no sealer	8.6 (± 6.2) a	12.5 (± 6.7) ab	14.3 (± 4.8) ab	13 (± 6.1) a	14.9 (± 4.6) a	15.4 (± 3) ab
AH Plus	13.5 (± 2.8) b	15.4 (± 3.2) b	16.9 (± 5.8) b	11.3 (± 3) a	13.5 (± 3.4) a	18 (± 3.8) b
Endofill	6.9 (± 3.5) a	10 (± 4.1) a	12.1 (± 4.4) a	11 (± 5.8) a	11.8 (± 4.8) a	11.5 (± 5.6) a

The comparisons are made per column, vertically. Different letters identify statistically significant differences (p<0.05/Tukey LSD=4.58).

Table 4: *Means (SD) (in MPa) of the Posts Retention Values Considering the Interaction of Dentin Regions with Chemical Substances and with Endodontic Sealers*

Region	Distilled Water			5.25% NaOCl		
	Without Sealer	AH Plus	Endofill	Without Sealer	AH Plus	Endofill
Cervical	8.6 (± 6.2) a	13.5 (±2.8) a	6.9 (± 3.5) a	13.0 (± 6.1) a	11.3 (± 3) a	11 (± 5.8) a
Middle	12.5 (± 6.7) b	15.4 (± 3.2) ab	10 (± 4.1) b	14.9 (± 4.6) a	13.5 (± 3.4) a	11.8 (± 4.8) a
Apical	14.3 (± 4.8) b	16.9 (± 5.8) b	12.1 (± 4.4) b	15.4 (± 3) a	18 (± 3.8) b	11.5 (± 5.6) a

The comparisons are made per column, vertically. Different letters identify statistically significant differences (p<0.05/Tukey LSD=2.83).

ers, as in the work of Hagge and others (2002), who observed statistically lower retention mean values for eugenol-based sealer.

With regard to the comparison of endodontic sealers, the use of AH Plus resulted in greater retention mean values. The resin-based endodontic cement allowed for greater compatibility with the adhesive system used for post cementation, as it was not always possible to completely remove the sealer from the canal walls (Boone & others, 2001).

Considering the controversy in the literature (Tjan & Nemetz, 1992; Schwartz & others, 1998; Ngoh & others, 2001; Hagge & others 2002) and the results of this study, eugenolic sealer should not be considered the best option for filling root canals where adhesive procedures are performed.

With regards to tooth regions (Table 4), greater retention means are observed for the apical third, followed by the middle third and the cervical third. In contrast to this study, Yoshiyama and others (1998) observed less bond strength in the apical region. It is worth emphasizing that the study by Yoshiyama and others uses dentin from the outside surface of the tooth and not intracanal dentin. Furthermore, the apical third studied was the apical third of the root, not the preparation for the post. As with the current study, Gaston and others (2001) divided the roots into three parts with reference to the preparation destined for cementing the post, also observing greater bond values in the apical third.

The difficulty of visualization and access to performing adhesive procedures inside the root canal increases the possibility of sealer or gutta-percha residue on the dentin canal walls, especially in the apical third, reducing the surface available for adhesion (Scotti & Ferrari, 2003). Furthermore, the uncertainty of complete polymerization of the adhesive system and contact of the post with the filling material in the apical region makes adhesion difficult in this third. Thus, how does one explain the greater retention values in the apical third found in the current study?

During endodontic treatment, it is necessary to widen the canal in its initial thirds to facilitate penetration of the irrigating solution, determining a conical shape for the canal (Becker & Woollard, 2001). Thus, post preparation will be performed with reamers that will wear the apical thirds more intensely. Therefore, the cervical region is more open to the influence of chemical substances and endodontic sealers, as greater wear of the apical third eliminates portions of the dentin that were in contact with the chemical substances, or which may still present cement residues (Boone & others, 2001).

In this study, after un-obstructing the canal with the Gates-Glidden drill, canal modeling was done with two canal reamers. This allows for more intimate contact of the post with the dentin canal walls, mainly in the apical third, forming locking areas. Thus, retention of the post in this region will occur by adhesive bonding and mechanical overlapping, which is also observed in the clinic situation. The push-out test allows for an assessment of the mechanical overlapping. However, assessment of the bond strength only, as occurs in microtensile bond strength tests, is not possible. This makes it difficult to compare the results of studies that use different mechanical test methodologies (Yoshiyama & others, 1998; Ferrari & others, 2001).

As opposed to the apical third, the cervical regions require a greater volume of cementing material, increasing stress at the adhesive interface during polymerization shrinkage (Patierno & others, 1996; Frankenberger & others, 2000). This increased stress may be critical for adhesion, taking into consideration the high cavitary factor of the root canal (Morris & others, 2001; Bouillaguet & others, 2003; Scotti & Ferrari, 2003).

Based on the retention values found in the apical third, it is probable that the adhesive has been effectively polymerized, despite the distance from the light source. The post used contains glass fibers that, according to the manufacturer, conduct light energy (Ivoclar/Vivadent), which may have helped in polymerizing the adhesive system in its most apical portions (Lui, 1994). In spite of this possibility, some authors question the capability of light conduction through the posts and polymerization of the cement and adhesive, should this light conduction occur (Ferrari & others, 2001; Morris & others, 2001; Scotti & Ferrari, 2003). It is worth emphasizing that the resin-based cement (Variolink) and adhesive (Excite DSC) used present dual cure; thus, areas where the light has not reached may have been polymerized chemically.

In spite of the advantage of the fiber posts, the cementing procedures require knowledge of the root canal and adhesive techniques. Thus, different specialties, such as Endodontics, Prosthesis and Dentistry should operate in an integrated manner in order to increase the longevity of the restorations. Endodontic treatment, according to the results of this study, seems to interfere in post retention. Thus, further studies should be carried out to establish a clinical protocol for improved retention of the posts, considering the techniques, adhesive materials and endodontic procedures.

CONCLUSIONS

NaOCl at 5.25% favored retention in the cervical third when compared to distilled water.

Eugenol-based sealer (Endofill) determined lower retention values than resin-based cement (AH Plus).

The retention of root canal posts was influenced by the dentin region of the preparation, the apical third being the most retentive.

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Effect of Curing Time and Light Curing Systems on the Surface Hardness of Compomers

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Clinical Relevance

The LED light curing units used in this study can be expected to effectively polymerize 2-mm thick compomers in 20 seconds.

SUMMARY

This study compared the Vickers hardness of the top and bottom surfaces of two compomers (Compoglass F and Dyract AP) polymerized for 20 and 40 seconds with two different light curing systems. Five samples for each group were prepared using Teflon molds (9x2 mm) and were light-cured either with a conventional halogen lamp (Optilux 501) or LED light (LEDemetron I) for 20 or 40 seconds. After curing, all the samples were stored in distilled water for 24 hours at 37°C. The Vickers hardness measurements were obtained from the top and bottom surfaces of each sample. ANOVA, Scheffé and *t*-test were used to evaluate the statistical significance of the results. For the top and bottom surfaces, the light curing systems and curing times tested showed no statistical difference, except for Optilux 501, which used 20 seconds for both compomers ($p<0.05$). There was no significant difference in the microhardness of both surfaces of Compoglass F and Dyract AP cured for either 20 or 40 seconds using LEDemetron I. With Optilux 501, the microhardness of samples cured for 40 seconds was significantly higher than 20 seconds ($p<0.05$).

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INTRODUCTION

Polyacid-modified resin-based composites (compomers) have been widely used since their introduction to the market. These resin-based composites combine glass polyalkenoate components, such as fluoro-silicate-glasses, with polymerizable resin composite, such as dimethacrylates (Geurtsen, Spahl & Leyhausen, 1998; Geurtsen, Leyhausen & Garcia-Godoy, 1999; Kwon &

others, 2002) and have the advantages of fluoride release, esthetics, improved physical properties compared to glass-ionomer cements and ease of handling properties (El-Kalla & Garcia-Godoy, 1999; Kwon & others, 2002; Millar, Abiden & Nicholson, 1998). They are recommended for use in the restoration of primary teeth and non-stress bearing cavities in permanent teeth (Abdalla, Alhadainy & Garcia-Godoy, 1997; Huth & others, 2003; Yap, Low & Ong, 2000).

Compomers are light polymerizable. Visible light-curing units are used to polymerize light sensitive restorative materials. Halogen light-curing units are the most commonly used units in dentistry. However, despite the popularity of halogen lights, they have some disadvantages. The halogen bulb generates high heat, which degrades the bulb's components over time (Dunn & Bush, 2002; Hammesfahr, O'Connor & Wang, 2002; Leonard & others, 2002; Mills, Jandt & Ashworth, 1999), so they have a limited effective lifetime of 40 to 100 hours (Soh, Yap & Siow, 2003).

More recently, blue LED (light-emitting diodes) light curing units have been introduced as an alternative to halogen lights. LED light curing units may have a potential for use in dental practice, because their performance does not significantly reduce with time as do conventional halogen light curing units (Mills & others, 1999). LEDs produce light within a narrow spectral range and are therefore highly efficient light sources (Garcia-Godoy, Garcia-Godoy & Garcia-Godoy, 2003; Hofmann, Hugo & Klaiber, 2002; Leonard & others, 2002). LEDs have a lifetime of more than 10,000 hours and undergo little degradation of light output over time (Hammesfahr & others, 2002; Soh & others, 2003). These lights have the potential to be powered with rechargeable batteries. Another advantage is that they produced less heat, so there may be lower potential for gingival and pulpal irritation (Leonard & others, 2002).

Adequate polymerization of light curing materials depends on the light source intensity, wavelength, exposure duration (Leonard & others, 2002) and size, location and orientation of the tip of the source, and shade, thickness and composition of the material (Mc Cabe & Carrick, 1989).

With the new technologies, it is thought that adequate resin polymerization and improved mechanical properties may be obtained in a shorter time when using high power light curing units. Shorter curing times can reduce chair time, especially in children and when incremental techniques are applied. The recommended curing time for halogen lights is 40 seconds for 2-mm thick resin. Optimal cure times for LEDs and their ability to cure all resins are not yet clear, although one study showed similar bottom:top curing ratios than halogen light (Garcia-Godoy, Garcia-Godoy & Garcia-Godoy, 2004).

Surface hardness is an indirect measure of the degree of conversion, and important information can be obtained by comparing hardness values at the top and bottom surfaces (Dietschi, Marret & Krejci, 2003; Hofmann & others, 2002; Jandt & others, 2000; Mills & others, 1999). Many investigators have reported the top and bottom surface hardness of resin composites polymerized with different types of light curing units (Hofmann & others, 2002; Jandt & others, 2000; Leonard & others, 2002; Mc Cabe & Carrick, 1989; Mills & others, 1999). However, there is one study (Oberholzer & others, 2003) that compares the top and bottom surface hardness of compomers polymerized with halogen light or LED.

This study evaluated the top and bottom surface microhardness of two compomers using a conventional visible light or an LED light-curing unit at 20 or 40 seconds of polymerization time.

METHODS AND MATERIALS

The two tested compomers in this study were Compoglass F (Ivoclar Vivadent, Schaan, Liechtenstein) and Dyract AP (Dentsply Caulk, Milford, DE, USA). Four different groups of five specimens each were prepared for each material. A total of 40 specimens were prepared using a Teflon mold (2x9 mm). A conventional halogen light-curing unit (Optilux 501, Kerr Corporation, Danbury, CT, USA) and an LED light-curing unit (LEDemetron, Kerr Corporation) were used to polymerize the compomers. The study groups were designed as:

Group 1: Compoglass F, polymerized with LEDemetron for 20 seconds.

Group 2: Compoglass F, polymerized with LEDemetron for 40 seconds.

Group 3: Compoglass F, polymerized with Optilux 501 for 20 seconds.

Group 4: Compoglass F, polymerized with Optilux 501 for 40 seconds.

Group 5: Dyract AP, polymerized with LEDemetron for 20 seconds.

Group 6: Dyract AP, polymerized with LEDemetron for 40 seconds.

Group 7: Dyract AP, polymerized with Optilux 501 for 20 seconds.

Group 8: Dyract AP, polymerized with Optilux 501 for 40 seconds.

The materials were handled according to the manufacturers' instructions. The mold with the specimen material was held between two glass slides, covered with a transparent Mylar strip and gently pressed together to remove excess compomer. The samples were polymerized from the top surface for 20 or 40 sec-

onds by using a conventional halogen light curing unit (Optilux 501) or an LED light curing unit (LEDemetron I) with an 8-mm light guide. The distance between the light source and sample was standardized by using a 1-mm glass slide. The end of the light guide was in contact with the cover glass during the light-polymerization process. The light output for Optilux 501 was 600 mW/cm^2 and for LEDemetron 1, it was 800 mW/cm^2, measured with a curing radiometer (Demetron). The measurement was done after every five specimens. After curing, all the samples were stored in distilled water for 24 hours at 37°C to assure complete resin curing. Microhardness measurements were obtained 24 hours after polymerization by using a Vickers Hardness Testing Machine (Buehler, Lake Bluff, IL, USA). Vickers hardness readings were undertaken using a 50gr load for 20 seconds. Three indentations were made at random on each specimen, and a mean value and hardness ratio and depth of cure percentage were calculated. The hardness ratio was calculated by dividing the bottom hardness value by the top hardness value and multiplying this with 100 depth of cure percentage was calculated.

ANOVA, Scheffé and *t*-test were used to evaluate the statistical significance of the results at 0.05 significance level.

RESULTS

Table 1 shows the mean (±SD) microhardness values and hardness

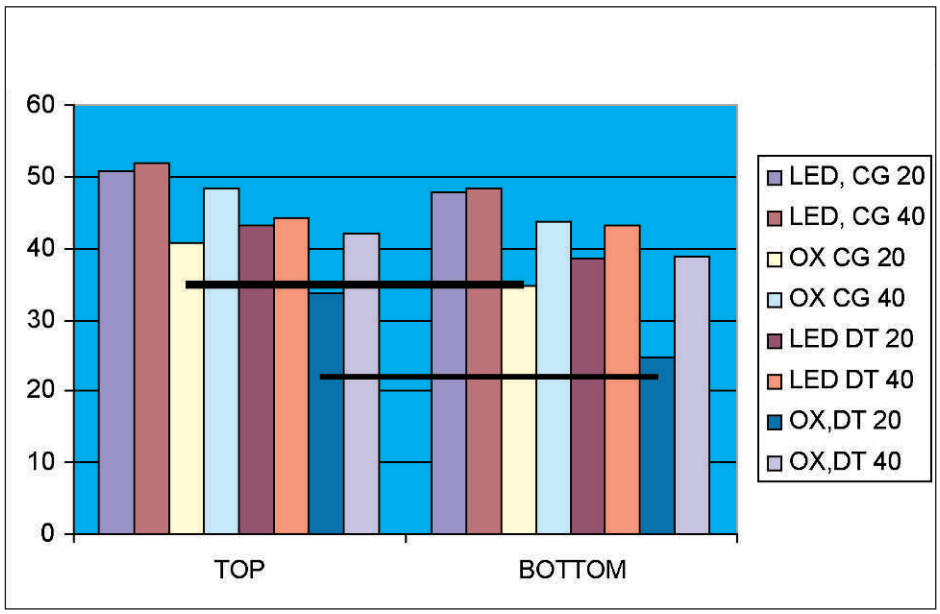

Figure 1. *Bars show that top surface hardness was significantly higher than the bottom surface hardness (p<0.05). (LED: LEDemetron I; OX: Optilux 501; CG: Compoglass F; DT: Dyract AP; 20: 20 seconds; 40:40 seconds).*

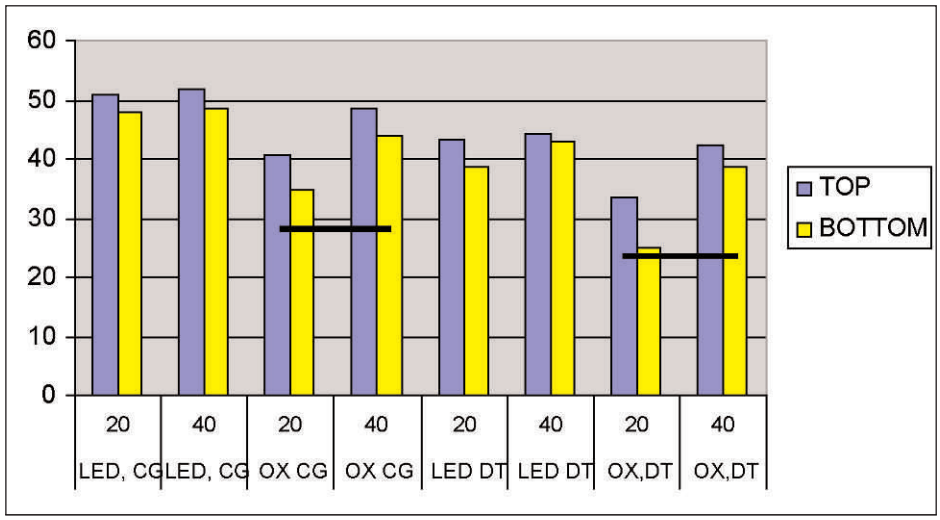

Figure 2. *Bars show that significant difference in Optilux 501 groups (p<0.05) where the microhardness was significantly higher (p<0.05) in the 40 seconds group than that of 20 seconds on both compomers. (LED: LEDemetron I; OX: Optilux 501; CG: Compoglass F; DT: Dyract AP; 20: 20 seconds; 40:40 seconds).*

Table 1: *Mean Microhardness (Std Dev) and Hardness Ratio and Depth of Cure of Compomers*

		Compoglass F				Dyract AP			
		Top	Bottom	Hardness Ratio	Depth of Cure (%)	Top	Bottom	Hardness Ratio	Depth of Cure (%)
LED	20	50.8 (3.5)	47.8 (2.3)	0.94	94	43.2 (0.8)	38.5 (4.6)	0.89	89
	40	51.8 (2.6)	48.4 (3.1)	0.93	93	44.2 (1.9)	43.0 (5.0)	0.97	97
OPTILUX 501	20	40.7 (1.7)	34.7 (1.8)	0.85	85	33.6 (1.9)	24.8 (2.2)	0.73	73
	40	48.5 (4.6)	43.8 (5.5)	0.90	90	42.1 (1.8)	38.8 (4.3)	0.92	92

ratio for each group. Microhardness ranged from 24.8 VHN for Dyract AP, using Optilux 501 for 20 seconds at the bottom surface, to 51.8 VHN for Compoglass, using LED for 40 seconds at the top surface. Independent of the applied light curing unit and polymerization time, it was found that Compoglass F hardness was significantly higher than Dyract AP ($p<0.05$), and the hardness values of both compomers polymerized using LEDemetron I were significantly higher than the values obtained with Optilux 501 ($p<0.05$).

For the top and bottom surfaces, the light curing systems and curing times tested showed no statistically significant differences except the Optilux 501 20 second groups; for both compomers, the top surface hardness was significantly higher than the bottom surface hardness ($p<0.05$) (Figure 1).

No significant difference was found between the microhardness of both surfaces of Compoglass F and Dyract AP cured for either 20 or 40 seconds using LEDemetron I; whereas, there was a significant difference in the Optilux 501 groups, where the microhardness was significantly higher ($p<0.05$) in the 40 second group than that of the 20 second group on both compomers (Figure 2).

When LEDemetron I and Optilux 501 were compared, no significant difference was found between the 40 second curing groups; however, the microhardness values of samples cured for 20 seconds with LEDemetron I were significantly higher than the Optilux 501 20 second group (Figure 3).

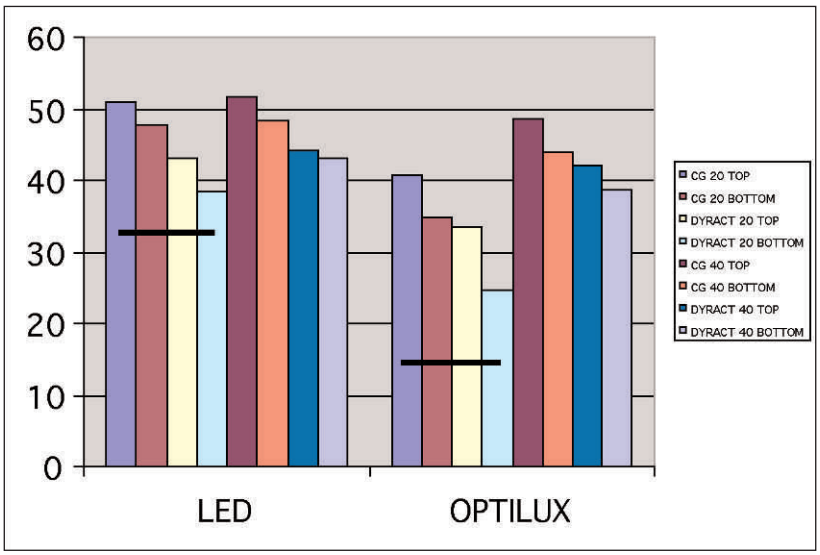

Figure 3. *The microhardness values of samples cured for 20 seconds with LEDemetron were significantly higher than Optilux 501 20 second group (LED: LEDemetron I; OX: Optilux 501; CG: Compoglass F; DT: Dyract AP; 20: 20 seconds; 40:40 seconds).*

DISCUSSION

The hardness of light-polymerized dental materials is dependent on polymerization conditions, light intensity and polymerization time. Hardness is defined as the resistance to permanent indentation or penetration. It is a property that is used to predict the wear resistance of a material and its ability to abrade or be abraded by opposing dental structures and materials (Anusavice, 1996) and it has been shown to be a good indicator of conversion of double bonds (Ferracane, 1985; Rueggeberg & Craig, 1988). Therefore, in this study, hardness was used as an indirect measurement of the degree of cure by conventional halogen light and LED light.

To assess the effectiveness of cure by different curing light units, top and bottom surface hardness values are compared. The results of some studies have showed that top surface hardness was significantly higher than bottom surface hardness (Dunn & Bush, 2002; Cavalcante & others, 2003; Sharkey & others, 2001; Soh & others, 2003). One possible explanation for this difference is that, as light passes through the bulk of the composite, light intensity is reduced due to light scattering by filler particles and the resin matrix (Ruyter & Øysæd, 1982).

Sharkey and others (2001) stated that the small difference between the top and bottom surfaces might be due to effective light transmission through the material. In this study, two polymerization times and light curing systems were tested. Although the mean hardness values at the top of each compomer was greater than the hardness at the bottom, the difference was not significant except for the Optilux 501 20 second group. The non-significant differences between the top and the bottom hardness of the compomer samples indicate that sufficient energy penetrates through the material to polymerize top and bottom surfaces to a similar extent (Rueggeberg & others, 1993).

There are many variables affecting the amount of light energy received at the top and bottom of a restoration, such as design of the light guide, power density, exposure duration, shade and opacity, thickness and composition of the material (Mc Cabe & Carrick, 1989; Rueggeberg & others, 1993).

Exposure duration is the factor that may be considered the most important clinical variable, because it is the only parameter that is directly influenced by the dentist's selection (Dietschi & others, 2003). In clinical conditions, the time needed for proper light curing of composite with a halogen lamp is quite extensive (Rueggeberg, Caughman & Curtis, 1994).

The microhardness at the top and bottom surface of both compomers cured with the LEDemetron I for 20 and 40 seconds, respectively, was not found to be signif-

icantly different. However, there were significant differences in the Optilux 501 groups, where the 40 second cure group was significantly harder than the 20 second cure group. This indicates that the hardness, when cured for 20 second exposure time, is similar to the 40 second exposure duration with LEDemetron I. However, a 20 second curing duration with Optilux 501 was not enough to polymerize the compomers to the same extent as a 40 second cure. Due to the reduction in bottom surface hardness and the conversion of conventional resin composites with increasing thickness, it has been recommended that composites should not be irradiated in increments greater than 2.0-mm thick. Manufacturers of halogen light curing units recommend 40 seconds of curing for 2.0-mm thickness. In this study, 40 seconds of exposure duration was used as a reference (Rueggeberg & others, 1994).

Contrary to the results of this study, Dunn and Bush (2002) found that halogen light curing units produced significantly harder top and bottom surfaces than LED light-curing units, but they used a first generation LED light. Leonard and others (2002), also using a first generation LED light, reported that the time needed to polymerize composites with LED was between 83 and 131 seconds; whereas, halogen lights required only 21 and 42 seconds to cure hybrid and microfill resin composites. Garcia-Godoy and others (2004) found that the two LED systems (Freelight 2 and LEDemetron) were not significantly different from each other and produced equivalent hardness on the composites tested. In this study, when LEDemetron was compared with Optilux 501, no significant differences were found between the 40 second curing samples. However, the top and bottom surface hardness of compomers cured with LEDemetron for 20 seconds was significantly higher than with the Optilux 501 20 second group. The differences between these studies may be due to the LED light curing unit's properties and material type. According to the manufacturers, the LEDemetron curing system produces the same or greater intensity than Optilux 501 for 10 seconds cures.

With light cured resins, the physical and mechanical properties are determined by size, distribution and content per volume of the filler particles in the matrix. Smaller particles and a higher filler content can increase the hardness of the material. Microfill resin composites have been shown to require more power than hybrid resin composites for adequate polymerization (Ruyter & Øysæd, 1982), as their small filler particles cause light scattering which decreases the effectiveness of the curing light (Leonard & others, 2002).

Ideally, the bottom/top hardness percentage would be 100%; however, previous studies have used 80% as the minimum value required to indicate adequate polymerization (Breeding, Dixon & Caughman, 1991; Watts, Amer & Combe, 1984). The hardness ratio should be 1 if polymerization is completely effective, as the hardness of the bottom surface should be the same as the top surface (Yap, Wong & Siow, 2003). Using this criteria, all the groups in this study achieved adequate cure, as the hardness ratio obtained was found to be greater than 80% with the exception of the Dyract Optilux 501, which was the 20 second curing group.

The results of this *in vitro* study indicated that the LED light curing unit used in this study can effectively polymerize compomers in 20 seconds of curing time. Reducing polymerization time is an important point, especially in young children, so that the LEDemetron could be a good alternative when polymerizing compomers in children or in large cavities where incremental techniques are required. Further research is needed regarding the effectiveness of short polymerization times with LED curing units as they evolve.

CONCLUSIONS

Under the conditions of this *in vitro* study:

- The LED systems evaluated were not significantly different from each other and produced equivalent hardness on composites.
- The LED light curing unit used in this study can be expected to effectively polymerize 2-mm thick compomers in 20 seconds.

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Clinical Technique/Case Report

Divergent Cast Post and Core Technique for Severely Compromised Molars with Potential for Corrosion Fracture Considered

D Newitter

Clinical Relevance

Endodontically treated molars with almost total coronal destruction may be salvageable by using multiple posts in divergent canals. In order to avoid possible corrosion stress fracture, it is best to fabricate all the parts of a metal multiple post and core assembly from the same metal alloy. This article describes an easy method for making a multi-piece metal post and core assembly from the same homogeneous casting alloy.

INTRODUCTION AND PURPOSE

Near total loss of the clinical crown of molars (Figure 1) presents a challenge for restoration. It is often most desirable to avoid periodontal crown lengthening for these cases due to the risk of iatrogenic furca involvement as a compromising factor in prognosis. Therefore, if the tooth is to be retained and restored, a method for building a secure core for supporting a crown is needed.

Conventional approaches for securing resistance and retention from the coronal portion of the tooth do not apply for these severely damaged teeth because of the lack of supporting coronal tooth substance. Stable matrixing for direct restoration is difficult or impossible to achieve. A core with a single post may not offer adequate retention for the core and resistance to dislodgment of the core, whether by the direct or indirect method. Passive placement of a flowable core material, such as resin composite or glass ionomer, for extensive coronal replacement with or without a matrix, is less suitable than metal materials for long-term service due to physical properties that can lead to failure (Lloyd & Adamson, 1987; Oliva & Lowe, 1987; Huysmans & others, 1992; Kovarik, Breeding & Caughman, 1992; Yaman & Thorsteinsson, 1992; Gateau, Sabek & Dailey, 1999).

In this difficult situation, the dentist is caught between the conflicting needs to restore the tooth to function by harnessing additional tooth structure and the maintenance of periodontal integrity. A practical approach to this dilemma is to harness the divergent geometry of multiple molar canals by creating a multiple-piece cast assembly that has a core with multiple divergent (non-parallel) posts (Wearn, 1974; Michnick & Raskin, 1978; Weiner, 1981; Mora & Firtell, 1984; Chiche & Mikhail, 1985; Boberick & Rickert, 1999).

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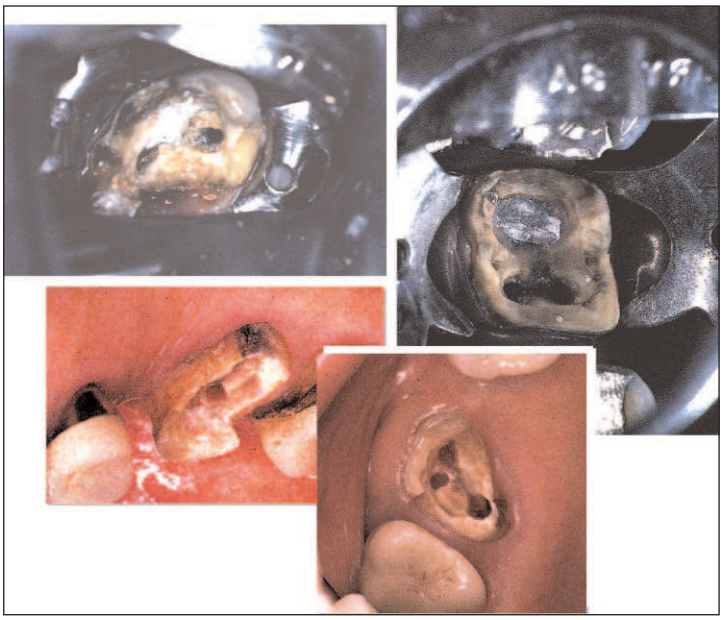

Figure 1. *Severely damaged teeth.*

Retention, by this design, relies upon the geometric divergent relationship of the posts from each other, that is, "mechanical lock" (Wearn, 1974). The cement and goodness of post adaptation to canal walls are not critical factors. In fact, with the use of cylindrical posts, Wearn (1974) recommended adapting the core pattern to the occlusal portion of flared canals, rather than sacrificing root wall thickness for adapting a larger size cylindrical post only for the sake of gaining a good fit to the canal walls. Resistance is borne by the roots and any remaining vertical components of the pulp chamber and clinical crown. The union of core to roots is secure and depends upon the strength of the roots for resisting lateral forces.

Some reported methods for fabricating a cast divergent post and core assembly were complex procedures (Michnick & Raskin, 1978; Mora & Firtell, 1984; Chiche & Mikhail, 1985). Some authors (Wearn, 1974; Michnick & Raskin, 1978; Weiner, 1981; Mora & Firtell, 1984; Chiche & Mikhail, 1985; Boberick & Rickert, 1999) advocated the use of a combination of metals for the parts of the assembly. Those parts were gold alloy from multiple sources and types, stainless steel cylindrical posts and the casting of dental gold to prefabricated metal posts.

A post and core assembly with unlike metals poses a potential for corrosion due to generation of a galvanic current (Pameijer, Glantz & Mobasherat, 1983; Ravnholt, 1988; Fernandes & Dessai, 2001). Non-noble metals, such as stainless steel, can promote corrosion or be subject to corrosion in non-biologic (Pickering, Beck & Fontana, 1962; McNally, 2004) and in biologic environments such as in the mouth (Silness, Gustavsen & Hunsbeth, 1979; Pameijer & others, 1983; Beague, 1992; Luu & Walker, 1992; Angell, 1999; Beech & Gaylarde, 1999; Kusy & others, 2002). Corrosion mechanisms are complex and can be related to factors such as biofilm, low oxygen tension and electrical potentials (Wilson & others, 1995; Breech & Gaylarde, 1999), conditions that exist in the oral environment. Corrosion has been implicated, although not proven, as a factor in tooth root fracture (Angmar-Månsson, Omnell & Rud, 1969; Rud & Omnell, 1970; Petersen, 1971; Silness & others, 1979); science based documentation of corrosion expansion stress was not provided. Considerable physical damage has been attributed to expansion forces in confined spaces generated by the corrosion of metal (Pickering & others, 1962; Hoke, Chama & Rosengarth, 1983; NTSB, 1984; Hahin & South, 1992; Virmani & Clemena, 1998), and it has been postulated that these forces may cause fracture of the roots when posts corrode (Silness & others, 1979). The research by Pickering and others (1962) and Hoke and others (1983) demonstrated stress forces and/or the wedging effect of metal corrosion in confined spaces. To avoid encouraging corrosion, it has been recommended that metals of differing electrochemical potential not be mixed, and that noble metal posts and cores made of the same metal be used (Angmar-Månsson & others, 1969; Arvidson & Wróblewski, 1978; Ravnholt, 1988; Luu & Walker, 1992).

This article presents an easy method for fabricating a divergent post and core assembly from the same homogeneous metal for all of the parts.

TECHNIQUE

Overview

Figure 2 shows the product resulting from the tooth-conserving fabrication of a cast post and core assembly pattern directly in the mouth. The assembly in Figure 2 consists of a cast core and three cylindrical posts, one of which is attached to the core. The core can have one attached post, plus one or two removable posts. The attached post can be cylindrical (parallel sided) or custom designed to fit the anatomy of a tapered

Figure 2. *Example of cast divergent post & core assembly with three cylindrical posts.*

canal. After the core and attached post are cemented, the removable posts are immediately cemented into their respective root canals through holes in the core that are aligned with the root canals. The removable cylindrical posts were cast from plastic patterns invested in the same mold used for the core. Prefabricated metal posts were not used. The result was a very secure post/core assembly with minimal removal of the remaining tooth structure, with particular emphasis on maintaining root wall thickness. The stability of the united assembly does not depend on a cement or luting material; the geometry provides stability. Fabrication of all parts from the same molten alloy solution presents a more favorable environment for avoiding electro-corrosive potentials.

Figure 3. *Example of cast divergent post & core assembly with a fixed custom post and two cylindrical accessory posts. These cylindrical posts had to be recast because they were shortened by error prior to trial seating.*

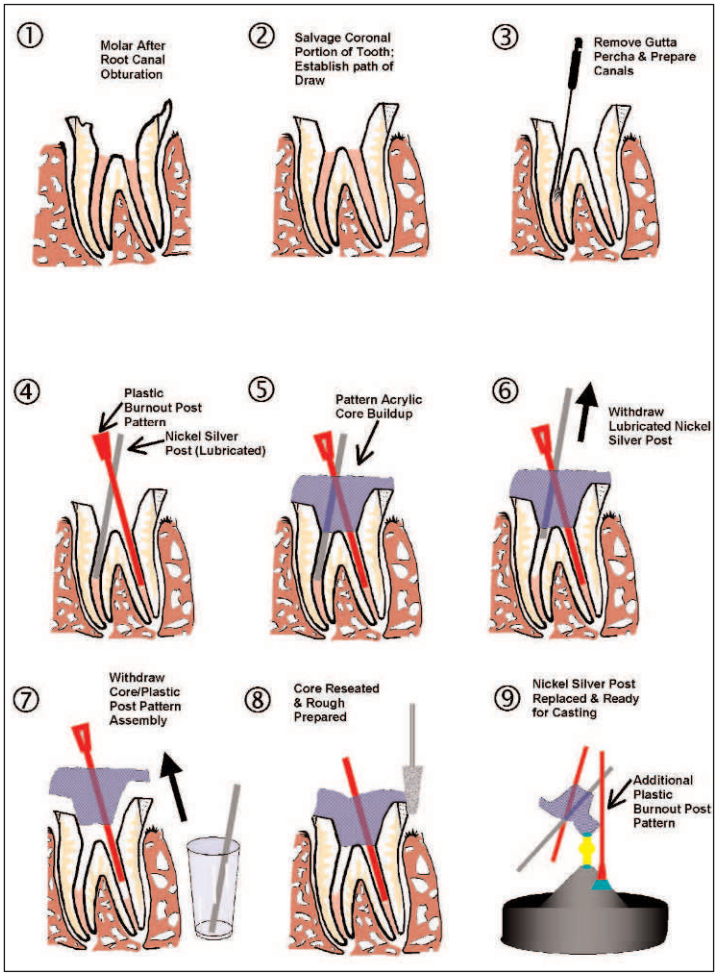

Figure 4. *Basic illustrations of technique for divergent post & core method with all parts cast in the same casting mold.*

Considerations for Selection of Canals and Posts

Depending upon the number, size, path and configuration of the root canals, two or three canals are selected to receive posts. Preservation of tooth structure is of primary consideration. That translates to minimal reduction in thickness of canal walls and to avoidance of perforation to the outer root surface.

Note that Figure 3 shows a post and core assembly with two cylindrical removable (accessory) posts and one fixed custom fitted post. The custom fitted post is most useful for a flared canal. For this method, as presented here, the fitted post must be cast attached to the core. An attached (fixed) post can be either custom fitted or cylindrical and must have a path of draw that is complementary to the draw of the core. There are more complicated methods for incorporating accessory, removable posts that are custom fitted, but that is not the topic of this article (Michnick & Raskin, 1978; Mora & Firtell, 1984). The focus here is on practicality, simplicity and ease of use.

Supplies and Instruments

1. Non-end-cutting rotary instruments for canal preparation: Gates Glidden Drills (sizes 1–6) or Peeso Reamers (sizes 1–6) (Pulpdent Corporation, Watertown, MA, USA).

2. Endodontic spreader or plugger for heating in a flame to remove gutta percha.

3. Selection of matched sizes (3–7) of cylindrical plastic post patterns and nickel silver posts (Parapost system, Coltene Whaledent, Cuyahoga Falls, Ohio, USA): Sizes 3 and 4 are most often used.

4. Twist drills sized matched (1–7) to cylindrical post diameters for reaming post hole in cast core (Parapost system, Coltene Whaledent).

5. Petroleum jelly (Vaseline, Unilever, New York, NY, USA).

6. Pattern resin (Duralay, Reliance Dental Mfg Co, Worth, IL or GC Pattern Resin, GC America Inc, Alsip, IL, USA).

7. Nitric acid concentrated (Baker Analyzed Reagent 69.0-71.0%, JT Baker Inc, Phillipsburg, NJ, USA).

8. Lentulo spiral (Pulpdent Corporation).

9. Porcelain Casserole (William Dixon Co, Div of Grobet, USA, Carlstadt, NJ, USA).

Technique

Figure 4 is a series of illustrations showing the first nine steps for fabrication of a core with divergent posts:

- Steps 1 and 2: After obturation of the root canals of a mandibular molar, undercuts were eliminated from the coronal and pulp chamber portions of the tooth by removing tooth structure and/or block out with a material such as glass ionomer cement.
- Step 3: Removal of gutta percha from the canals can be accomplished with a heated endodontic spreader or plugger in addition to using a Gates Gliddon drill. Gates Gliddon drills or Peeso reamers, rather than twist drills, are preferred for shaping and/or enlarging the canals. Twist drills are comparatively aggressive end cutting instruments that are more difficult to control.
- Step 4: A plastic post pattern has been placed into the distal canal, which has a path of removal that does not conflict with the anticipated draw of a core. A nickel silver post, lightly lubricated with petroleum jelly on the coronal portion, is shown in the mesial canal. If any of these posts do not assume a stable position, they can be stabilized with pattern acrylic at the coronal end of the canal. This acrylic will become part of the core.

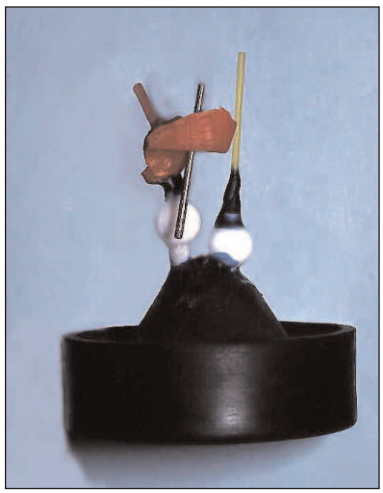

Figure 5. *Example of post and core assembly mounted on a sprue former prior to investing and casting.*

- Step 5: A core is in place after it was built up using a small brush to deliver increments of powdered acrylic pattern resin wetted with monomer.
- Step 6: The lubricated nickel silver post is withdrawn using a hemostat or similar instrument.
- Step 7: The nickel silver post is set aside in a cup for later use, and the core with attached post pattern is withdrawn as a single unit.
- Step 8: The core-post unit is re-seated and rough-prepared with a diamond rotary instrument.
- Step 9: The core-post unit is then withdrawn again, and the nickel silver post is inserted into the hole from which it was removed. Figure 5 shows an example of a core-post unit mounted on a sprue former. Additionally, a plastic post pattern, size matched to the nickel silver post, is sprued on the same sprue former.
- Step 10: Thereafter, the assembly is invested and cast in a precious gold alloy (Figure 6). The cast core-post unit is placed in warm, concentrated nitric acid to dissolve the nickel silver post; this leaves a hole in the core (Figure 7). Non-precious and silver platinum alloys should not be used; they will be dissolved by the nitric acid (Whaledent).
- Step 11: The separately cast post can be tried into the hole. If the post does not easily slide into the hole, then the hole is reamed with a size matched twist drill (Figure 8).
- Step 12: Visual inspection with magnification may reveal positive bubbles that should be removed with a small rotary bur or stone.

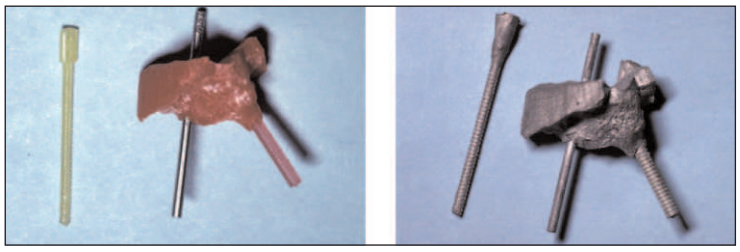

Figure 6. *Example of plastic patterns and their corresponding gold alloy castings.*

Figure 7. *Example of dissolving nickel silver posts with warm concentrated nitric acid.*

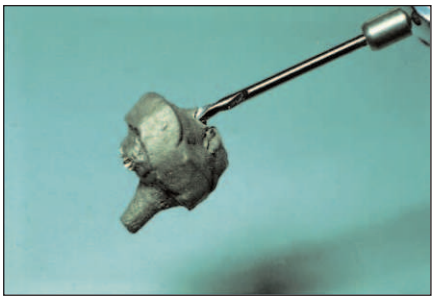

Figure 8. *Example of a size matched Parapost twist drill bit used to ream an accessory post hole.*

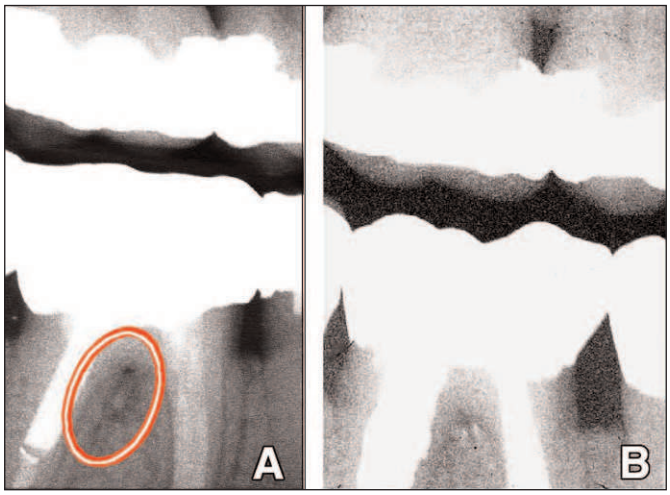

Figure 9 A and B. *Radiographs taken before (A) and after (B). Note that decrease in furcation radiolucency and excessive enlargement of distal canal in the second radiograph.*

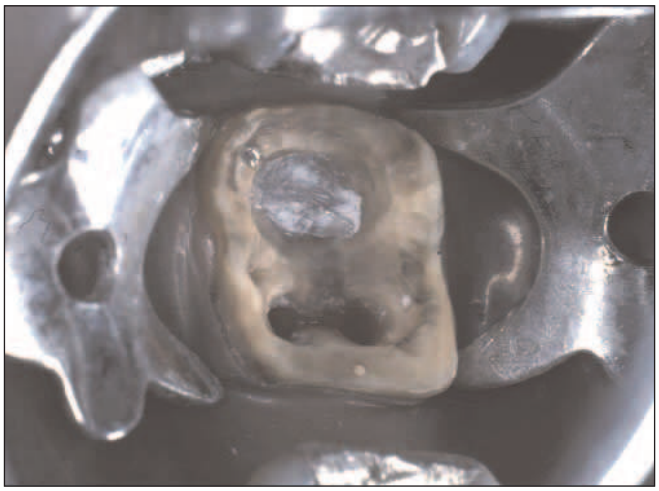

Figure 9C. *Remaining tooth structure.*

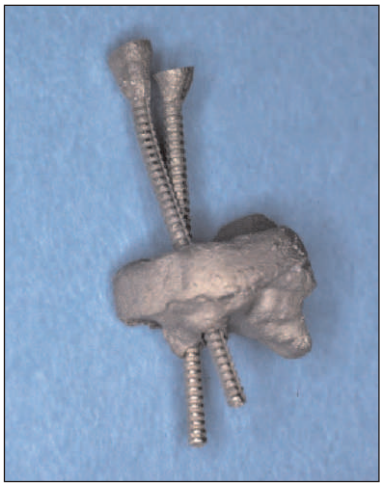

Figure 9D. *Cast posts and core.*

Figure 9E. *Cementation of divergent post & core assembly.*

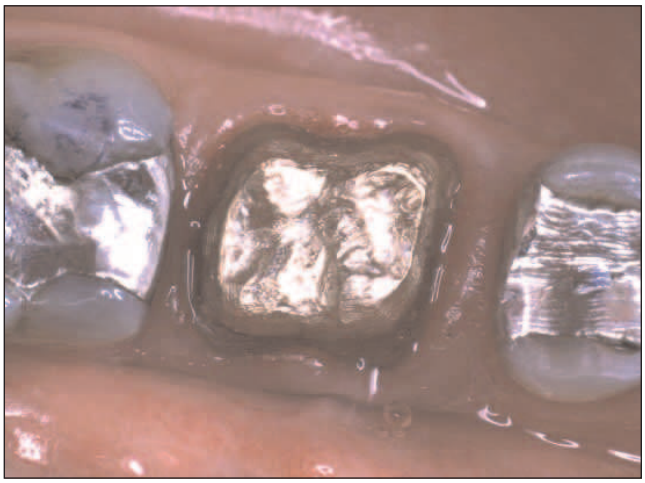

Figure 9F. *Occlusal view of completed crown preparation.*

• Step 13: The removable posts should not be shortened; they should remain at their full length at the try-in and during cementation. Note that the post lengths in Figure 3 were reduced in error, and they had to be recast prior to trial seating into the tooth. The length of these posts protruding from the occlusal surface should be noted at the try-in seating so that the dentist can determine that they are fully seated during cementation.

- Step 14: The cement chosen should allow for adequate working time for cementation of the parts. Zinc phosphate cement is easily controlled for this purpose. Nevertheless, it is necessary to work quickly. A lentulo spiral is used to introduce cement into the canals. After the cement has set, the protruding portions of the accessory posts at the occlusal surface are removed with a high-speed rotary instrument.

CLINICAL CASE EXAMPLE

See Figures 9A-G for illustrations. This patient reported with significant mobility on tooth #30. The tooth had an over-contoured crown, endodontic root fillings and a post in the distal canal. Sulcus depths were minimal. A bitewing radiograph showed a radiolucency in bone in the disto-occlusal portion of the bifurcation area.

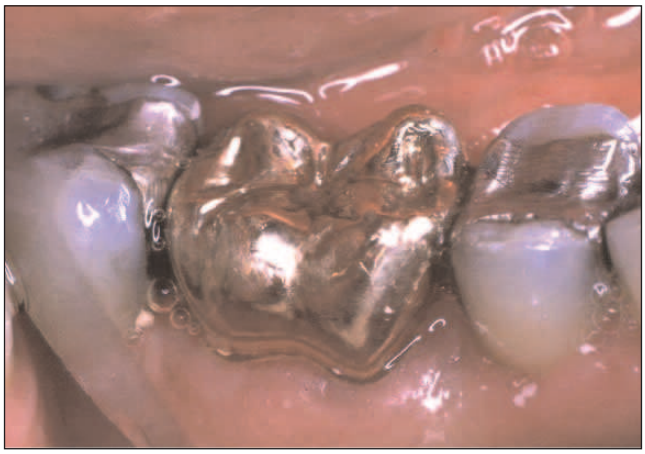

Figure 9G. *Full gold alloy crown restoration.*

Removal of the crown, core and post revealed an unsuspected perforation in the pulp chamber floor and extensive caries. Additionally, there was very little remaining clinical crown. Crown lengthening as a method for increasing crown resistance and retention would have exposed the furcation and, therefore, was not a preferred option. The perforation was addressed by overlaying it with dental amalgam. Access to the distal canal was limited, and the portion available was broad and of limited depth after post and caries removal. A custom acrylic post, attached to the core, was made for the distal canal. The two mesial canals were prepared to receive posts cast from Parapost plastic patterns. The posts, core and crown have been in place for over 15 years, functioning satisfactorily without sequelae. Following treatment, the mobility and furcation radiolucency were reduced.

SUMMARY OF DISADVANTAGES AND ADVANTAGES

Potential problems that the dentist may have with this method are as follows:

If a cement that sets too quickly is used, there may not be adequate time to seat or fully seat accessory posts. Once cemented, removal of the divergent post and core requires extensive grinding. Working with small pieces in the mouth presents a risk for aspiration or swallowing of these pieces; rubber dam is recommended to preclude these events, and careful cotton isolation is a less preferred option. Acrylic use in the mouth of a patient with acrylic allergy is not indicated. A lab procedure is required. The possibility of void or particle inclusions is a risk inherent in the casting method. Good ventilation and care is required for nitric acid use and storage.

Advantages to consider are the following: The lab procedure need not be, but can be done in dental offices with casting capability. A solid root-core union is established by this method; separation requires fracture. Restoration is facilitated for teeth that may otherwise be designated "non-restorable." Fabrication of all parts of the post and core assembly in the same casting mold may reduce the potential for corrosion potential and corrosion stress forces. The procedure predictably provides stability that lessens the amount of crown preparation needed to extend cervically onto tooth structure. This can reduce the need for, or extent of, crown lengthening with its accompanying encroachment into furcations. The technique is simple and practical.

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Case Report—Pre-Eruptive Intracoronal Resorption

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BS Wall • DL Leonard

INTRODUCTION

A pre-eruptive intracoronal resorption is a radiolucent lesion in the crown of an unerupted tooth. The reported incidence of pre-eruptive intracoronal resorption is 3% to 6% of the population and 0.5% to 2% of all teeth (Kupietzky, 1999; Seow, Wan, & Mc Allen, 1999b). Permanent molars have the highest incidence, followed by permanent premolars (Seow, 1998; Seow & Hackley, 1996; Seow & others, 1999b). While the cause of these defects is not firmly established, the current theory is dentin resorption. This theory is supported by histological studies that show osteoclasts, multinucleated giant cells and macrophages present in the lesions (Seow, 1998; Singer, Abbott & Booth, 1991). Resorptive cells may enter the developing tooth from the surrounding bone through breaks in the dental follicle or enamel epithelium and cause resorption until the tooth erupts (Johnson & Harkness, 1997; Kupietzky, 1999; Seow, 1998; Seow, Lu & Mc Allen, 1999a; Seow & others, 1999b). After eruption into the oral cavity, cariogenic bacteria may then enter the tooth through grooves in the occlusal surface and begin caries formation in the resorptive lesion (Seow, 1998; Johnson & Harkness, 1997). Early detection of the resorptive defect before eruption may prevent unnecessary loss of tooth structure. Many of these lesions go undetected until the permanent tooth has fully erupted. The purpose of this case report is to increase the dentist's awareness of this condition, recognize the radiographic and clinical findings of these lesions and suggest treatment strategies.

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CASE REPORT

A nine-year-old female patient reported for a routine examination in August 1998. Her medical history was non-contributory. She was in good oral health, with no history of caries or soft tissue defects at previous dental examinations. All of her primary teeth had exfoliated, and all permanent teeth had erupted except for second and third molars. Bitewing radiographs were taken at this appointment (Figure 1). A well-circumscribed radiolucent, occlusal lesion found at the dentino-enamel junction (DEJ) of the mandibular left second premolar

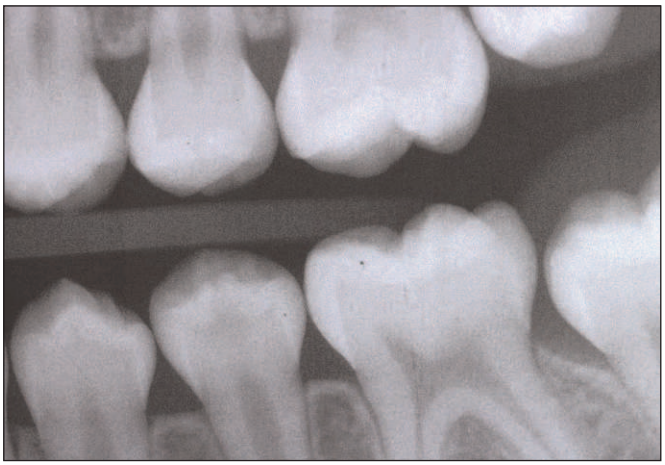

Figure 1. *Radiograph shows a well-circumscribed occlusal radiolucency of tooth #20.*

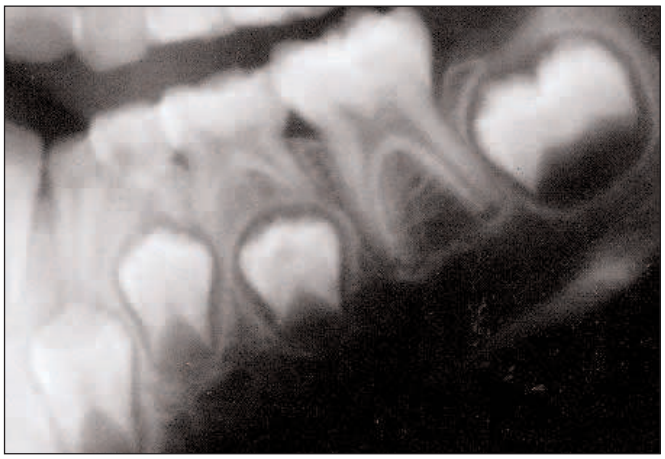

Figure 2. *Panoramic radiograph taken 31 months prior to Figure 1 shows pre-eruptive radiolucency on tooth #20.*

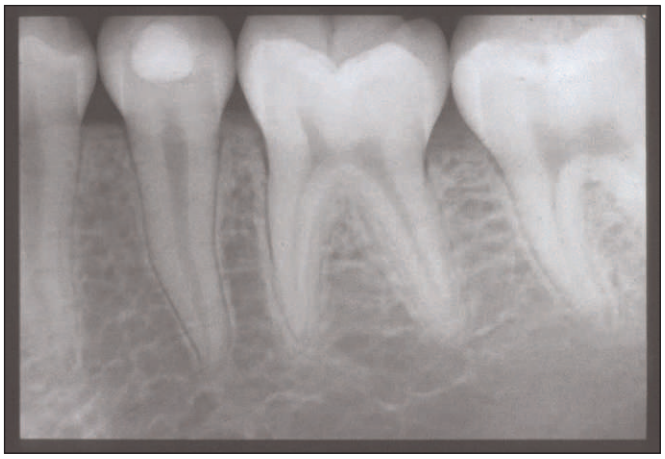

Figure 4. *Five-year follow-up.*

(tooth #20) extended approximately half-way through the dentin towards the pulp. The lesion was centered mesio-distally and spread along the DEJ. Clinical examination showed intact enamel over the defect, no clinical evidence of decay and no history of symptoms. A panoramic radiograph taken 31 months prior to this appointment showed a similar radiolucent lesion in tooth #20 prior to its eruption (Figure 2). The lesion appeared to have increased in size since that time.

The decision was made to restore tooth #20. Removal of the overlying enamel revealed a lesion in which the dentin had a chalky, grayish, dry and flaky consistency. This was removed with a slow speed round bur until hard dentin was reached. The lesion was restored with a bonded resin composite. Figure 3 shows the post-op radiograph. The tooth has been followed for five years and continues to test vital, remains asymptomatic and presents a stable radiographic appearance (Figure 4).

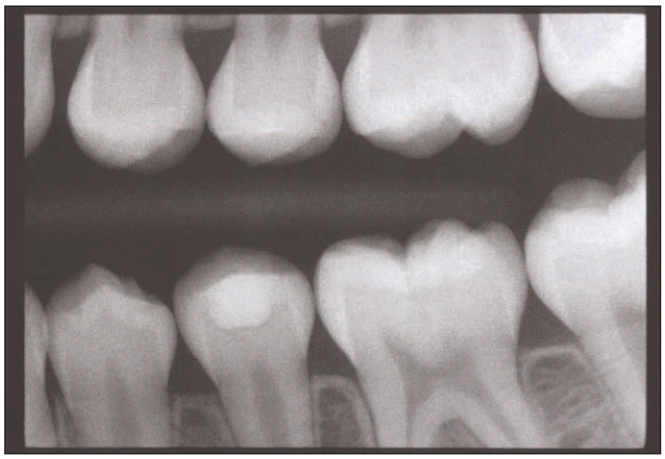

Figure 3. *Post-operative radiograph.*

DISCUSSION

Pre-eruptive intracoronal resorption is the preferred term to describe these lesions, because histological and clinical findings suggest that they are resorptive in nature (Seow, 2003). The term "pre-eruptive caries" was once used to describe coronal lesions in unerupted teeth (Seow & others, 1999a). This term reflects the fact that cariogenic bacteria had been cultured from pre-eruptive lesions after their eruption into the oral cavity. It was thought that caries in a primary tooth might infect the underlying permanent tooth. However, this could only occur if the overlying tooth had a periapical infection or if a fistula was present that allowed microbes access to the unerupted tooth (Johnson & Harkness, 1997; Singer & others, 1991). It is important to keep in mind that the tooth with the highest prevalence of these lesions is the permanent molar, which, of course, has no overlying primary tooth. Therefore, caries is not considered the primary cause of this lesion. However, caries may develop in these lesions after eruption.

A mechanism for resorption in these lesions may be ectopic positioning of the unerupted tooth or of an adjacent tooth. Seow and others (1999a) reported a 6% prevalence of pre-eruptive coronal lesions in ectopically positioned teeth. This was significantly higher than the 2% prevalence of teeth in the group that were not ectopically positioned. Seow and others (1999a) suggests that the ectopic positioning of the tooth may cause breaks in the dental follicle, allowing resorptive cells from the surrounding bone an opportunity to enter the developing tooth. Factors other than tooth positioning may also be involved, because the majority of ectopically positioned teeth show no defects. More research is needed to determine other etiologic factors.

Early diagnosis of pre-eruptive intracoronal resorption is elusive. Many of these defects are undiagnosed or have delayed diagnosis even though they can appear on the radiographs of unerupted teeth.

Radiographically, the pre-eruptive lesion appears as a well-circumscribed radiolucent lesion located at the DEJ (Seow, 1998; Seow & others, 1999a). The radiolucent area resembles caries, making diagnosis of these lesions difficult (Kunz, 1997; Seow, 1998; Seow & others, 1999a). Following eruption, caries may develop in the pre-eruptive lesion, obscuring the resorptive origin of the lesion. In this case, the overlying enamel appeared clinically intact and delayed the diagnosis. Also, in this case report and others, the defect was not diagnosed until the tooth had already erupted (Kunz, 1997; Kupietzky, 1999). Kunz (1997) reported that root canal therapy was necessary on a tooth that was later found to have a pre-eruptive intracoronal defect despite an intact enamel surface and an occlusal sealant. Seow and others (1999a) reported in their clinical observations that, out of 66 patients with clinically undetected occlusal caries ("occult caries"), 31 had pre-eruptive dentin radiolucencies. Seow (2000) hypothesizes an association between occult caries and pre-eruptive resorptive lesions. This possible association warrants further investigation.

Pre-eruptive intracoronal resorption lesions that do not become carious have a different clinical and histological appearance. Usually, the occlusal enamel surface is intact (Johnson & Harkness, 1997; Kupietzky, 1999; Seow, 1998; Singer & others, 1991; Wood & Crozier, 1985), with a chalky, grayish, dry, flaky dentin that has a different consistency from caries-induced demineralized dentin (Kunz, 1997; Seow, 1998). Histological examination has shown resorption (Grundy, Pyle & Adkins, 1984; Rankow, Croll & Miller, 1986) and scalloping of the enamel at the DEJ, suggesting osteoclastic activity (Seow, 1998; Singer & others, 1991). Johnson and Harkness (1997) performed a histological study of a pre-eruptive intracoronal resorption lesion found in a pre-molar extracted for orthodontic therapy. They noted that the margins of the dentin surrounding the debris-filled cavity had an irregular, "moth-eaten" appearance, which is suggestive of resorption. Upon histological examination of two separate lesions in teeth also extracted for orthodontic reasons, Singer and others (1991) found macrophages and giant cells lying in Howship's lacunae in the dentin. Histological studies have also reported intact dentin between pre-eruptive lesions and the pulp chamber, indicating that the resorptive cells are not related to pulpal tissue (Grundy & others, 1984; Rutar, 1997; Singer & others, 1991). Intact dentin has been observed between the base of the lesion and the pulp (Holan, Eidelman & Mass, 1994; Rankow & others, 1986; Wood & Crozier, 1985). In contrast, O'Neal, Gound and Cohen (1997) described a case in which the lesion communicated with a vital pulp.

Pre-eruptive intracoronal resorption has generally been noted to be progressive in nature, yet not aggressive (Johnson & Harkness, 1997). In the 31-month time period between radiographs, in this case, the lesion was found to increased in size, probably due to continued resorption prior to eruption. However, Kunz (1997) reported a case in which radiographs taken five months apart showed no progression of the radiolucent lesion prior to eruption.

There are several management strategies for pre-eruptive intracoronal resorption based on the size of the lesion. Small, pre-eruptive lesions can be radiographically monitored until the tooth erupts into the oral cavity and then restored with conservative operative procedures (Dowling, Fleming & Corcoran, 1999; Holan & others, 1994; Rutar, 1997; Seow & others, 1999b). For some small lesions, clinicians may choose to closely monitor the lesion and restore the tooth only if further progression is noted (Dowling & others, 1999). If patient compliance with monitoring is a concern, it may be best to restore immediately. When considering treatment, it is important to keep in mind that a tooth with pre-eruptive resorption is probably more vulnerable to decay and fracture due to the presence of undermined enamel. Large lesions approaching the pulp may be restored as soon as possible and might include an indirect pulp cap (Kupietzky, 1999). Some authors suggest that, for very large, progressive lesions, the tooth be restored in its unerupted state, removing an overlying primary tooth, if necessary (Dowling & others, 1999; Holan & others, 1994; Johnson & Harkness, 1997; Rutar, 1997; Seow & others, 1999a,b). Seow (1998) reported a case in which the lesion was so extensive that the coronal tooth structure fractured at the CEJ during orthodontic debracketing. Restoration of the tooth required endodontics, post and core and a crown. Similarly, Kunz (1997) reported carious activity extended into the pulp after the tooth erupted and required endodontic therapy. Currently, there are no reported cases of progression of pre-eruptive coronal radiolucent lesions after restoration (Seow, 2003).

CONCLUSIONS

In conclusion, dentists should examine radiographs for abnormalities in the number, size, shape and position of unerupted teeth as well as for intracoronal radiolucencies. With an accurate diagnosis of pre-eruptive intracoronal resorption, the lesion can be monitored and managed appropriately. Proper management will aid in preventing unnecessary loss of tooth structure.

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Departments

Faculty Positions

The University of North Carolina
Department of Operative Dentistry

The University of North Carolina seeks to fill a full-time, tenure-track or clinical-track position at the Assistant/Associate Professor level in the Department of Operative Dentistry. Responsibilities for this position will include pre-clinical and clinical teaching, service activities, intramural practice and research. Candidates with proven teaching abilities and advanced training or documented scholarly activity in operative dentistry or a related field are preferred. Salary will be determined commensurate with the applicant's qualifications and experience. Applications, including a curriculum vitae, and the names and addresses of four references, along with any nominations or inquiries, should be directed to Dr Harald O Heymann, Chair of Search Committee, Department of Operative Dentistry, School of Dentistry, CB #7450, University of North Carolina, Chapel Hill, NC 27599-7450 or http://www.dent.unc.edu/depts/academic/oper/

Application deadline with be open until the position is filled. As an Equal Opportunity/Affirmative Action Employer, the University is committed to equality of educational and employment opportunities and actively seeks to promote diversity through its recruitment endeavors.

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School of Dentistry

The School of Dentistry at the Oregon Health & Science University is seeking an energetic, motivated and qualified individual for a full-time position at the level of Assistant/Associate professor in the Department of Restorative Dentistry, Division of Operative Dentistry. Experience in teaching, research, service and patient care, as well as excellent interpersonal and communication skills are preferred. Implant experience, especially teaching at the pre-doctoral level, is considered an additional asset but not a requirement. Specific teaching responsibilities will include participation at the pre-doctoral level in both the clinical and pre-clinical curriculum, with the opportunity to serve as course director. Candidates should posses a DMD/DDS degree. One day per week (0.2 FTE) will be available for participation in the Faculty Dental Practice. Collaboration in research opportunities is available and encouraged. Salary will be determined by credentials and experience. OHSU is an Equal Employment Opportunity institution. Interested candidates should submit a letter, curriculum vitae and references to:

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Director, Division of Operative Dentistry
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The UCLA School of Dentistry
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University of California, Los Angeles, School of Dentistry, Division of Restorative Dentistry, invites applications for a full-time, tenure track position at the Assistant Professor level. Responsibilities will include conducting and mentoring research in the restorative dental sciences as well as clinical and/or preclinical teaching. Candidates must have a strong scientific background and preference will be given to applicants with an established record of laboratory or clinical research in areas directly relevant to restorative dentistry and the potential for continued generation of extramural research support. Teaching experience is required. Candidates must posses a DDS or DMD from an ADA-accredited dental school. A PhD degree is preferred, but not required.

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The University of Minnesota
School of Dentistry
Division of Operative Dentistry

The University of Minnesota, School of Dentistry, welcomes candidates for a full-time, tenured or tenure-track position in the Division of Operative Dentistry. Responsibilities include clinical, pre-clinical and didactic instruction in operative dentistry, provision and monitoring of patient care, and participation in an expanding

research program. The selected candidate will participate in the School of Dentistry meetings, committees, continuing education and outreach, along with other opportunities. Applicants must have a DDS degree or equivalent and must have or be eligible for licensure in Minnesota. An MS, PhD or equivalent degree is highly desirable. A minimum of two years full-time teaching and/or patient care experience desired. Private practice in the School of Dentistry's intramural practice is available one day per week. Salary and rank will be commensurate with experience and credentials. The search will remain open until the position is filled.

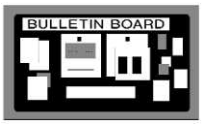
A Curriculum Vitae, letter of interest and three professional references should be sent to: Dr Gary Hildebrandt, Chair of the Search Committee, 8-450 Moos Tower, 515 Delaware St SE, Minneapolis, MN 55455. Applications may be faxed to (612) 625-7440 or e-mailed to: hilde014@umn.edu.

The University of Minnesota, School of Dentistry, is one of seven schools in our Academic Health Center, one of the most comprehensive academic health centers in the United States. The School of Dentistry consistently attracts high quality faculty and students and has achieved an international reputation for research, education and service. The University of Minnesota is an equal opportunity educator and employer.

The University of Texas Health Science Center at Houston Dental Branch

The University of Texas Health Science Center at Houston Dental Branch seeks applicants for a full-time tenure or clinical educator track position in the Department of Restorative Dentistry and Biomaterials at the Assistant or Associate Professor level. Applicants must have a DDS/DMD degree with prior teaching and/or private practice experience. Responsibilities include preclinical and clinical teaching to undergraduate dental students, research and service. The position is available August 1, 2005. Academic rank and salary are commensurate with qualifications and experience. The Dental Branch is located in the heart of the Texas Medical Center, which offers significant resources and important opportunities for collaboration and advanced training. The Dental Branch is one of six schools in the most comprehensive health science center in Texas. The University of Texas Health Science Center at Houston is an equal opportunity employer and a non-smoking environment. Women and minorities are encouraged to apply. Send a letter of application, a curriculum vitae and a list of three references to: Dr Arthur Jeske, The University of Texas Dental Branch at Houston, Department of Restorative Dentistry and Biomaterials, 6516 M D Anderson Blvd, Suite 493, Houston, Texas 77030.

Announcements



Ralph Phillips Student Research Award 2006

Applications are invited for the 2006 Ralph Phillips Student Research Award, sponsored by the Founders Fund of the Academy of Operative Dentistry. The Award is open to students at all levels wishing to undertake novel research relevant to contemporary operative dentistry.

The application should take the form of a protocol outlining the background, aims and hypothesis of the proposed research, the methodology to be employed, the anticipated work schedule and the expected impact of the work on the clinical practice of operative dentistry. The protocol should not exceed three double-spaced, typewritten pages and a budget sheet, including details for payment of the award. The budget for the proposed research may not exceed $6,000.

The recipient of the award will be required to present a table clinic reporting the findings of the research supported by the Award at the 2007 annual meeting of the Academy of Operative Dentistry to be held in Chicago, 22-23 February, 2007. Additional funds not to exceed $1,000 will be made available to offset the cost of attending the meeting.

A research mentor, who is a member of the Academy of Operative Dentistry, should be named and be a co-signatory to the application.

If the research supported by the Award leads to a research report intended for publication, it is expected that the report be submitted in the first instance to *Operative Dentistry*.

Applications for the award must be submitted electronically to Dr Nairn Wilson (nairn.wilson@kcl.ac.uk), Chairman of the Research Committee of the Academy of Operative Dentistry no later than 31st December 2005.

The award recipient will be announced during the 2006 Annual Meeting of the Academy of Operative Dentistry, which will be held in Chicago, 23-24 February, 2006.

Academy of Operative Dentistry European Section Conference:

New Horizons in Operative Dentistry
Friday 18th November 2005

Guy's Tower, GKT Dental Institute,
King's College London

The Academy of Operative Dentistry, European Section, is delighted to announce its 2005 Conference.

The Conference will be chaired by Nairn Wilson and Stephen Dunne, who will introduce speakers of international reputation, including Ivo Krejci, University of Geneva; Paul Lambrects, Catholic University of Leuven; George Vougiouklakis, University of Athens and Alfons Plasschaert, University of Nijmegen, who will be fully supported by presentations from the GKT Dental Institute representatives, including Paul Sharpe, Tim Watson, Avijit Banerjee and Christos Louca.

The highly topical subjects covered include air abrasion, lasers, photoactivated disinfection, dental caries including operative and non-operative management, the ADEE perspective on the teaching of conservative dentistry and stem-cell based tissue engineering of teeth. IADR-style posters will be displayed during lunch.

The Conference Dinner will be held on board the historic WWII Battle Cruiser HMS Belfast, which will bring the meeting to a memorable conclusion. In addition to enjoying a five-course meal and fine wines, delegates will have, by special arrangement, the opportunity to view the Cruiser's Operating Theatre, Sick Bay and Dental Surgery.

For further information and booking information, please contact: Cynthia Casimir, Primary Dental Care Secretary, GKT Dental Institute, Denmark Hill Campus, Caldecot Road, London SE5 9RW

Tel: 00 44 (0)207 346 3585 Fax: 00 44 (0)207 346 3826

Email: cynthia.casimir@kcl.ac.uk

Instructions to Contributors

The newly revised and expanded Instructions to Contributors is posted on the Operative Dentistry Home Page: **http://www.jopdent.org/**

Click on Contributors for the most up-to-date way to submit your research papers electronically.

NOTE: As of May 1, 2005, all papers must be submited electronically.

Operative Dentistry Home Page

We hope all our readers will take advantage of the information available by accessing our Internet home page. Our address is: **http://www.jopdent.org/**

The home page contains a search engine and buttons that, hopefully, will lead you to answers to any questions you may have related to ***Operative Dentistry*.** These are:

Journal: leads to information on the Editorial Staff and Editorial Board; a complete index of journal volumes; a compilation of direct gold references; highlights of the current issue and a more detailed look at published Editorials and Clinical Pearls.

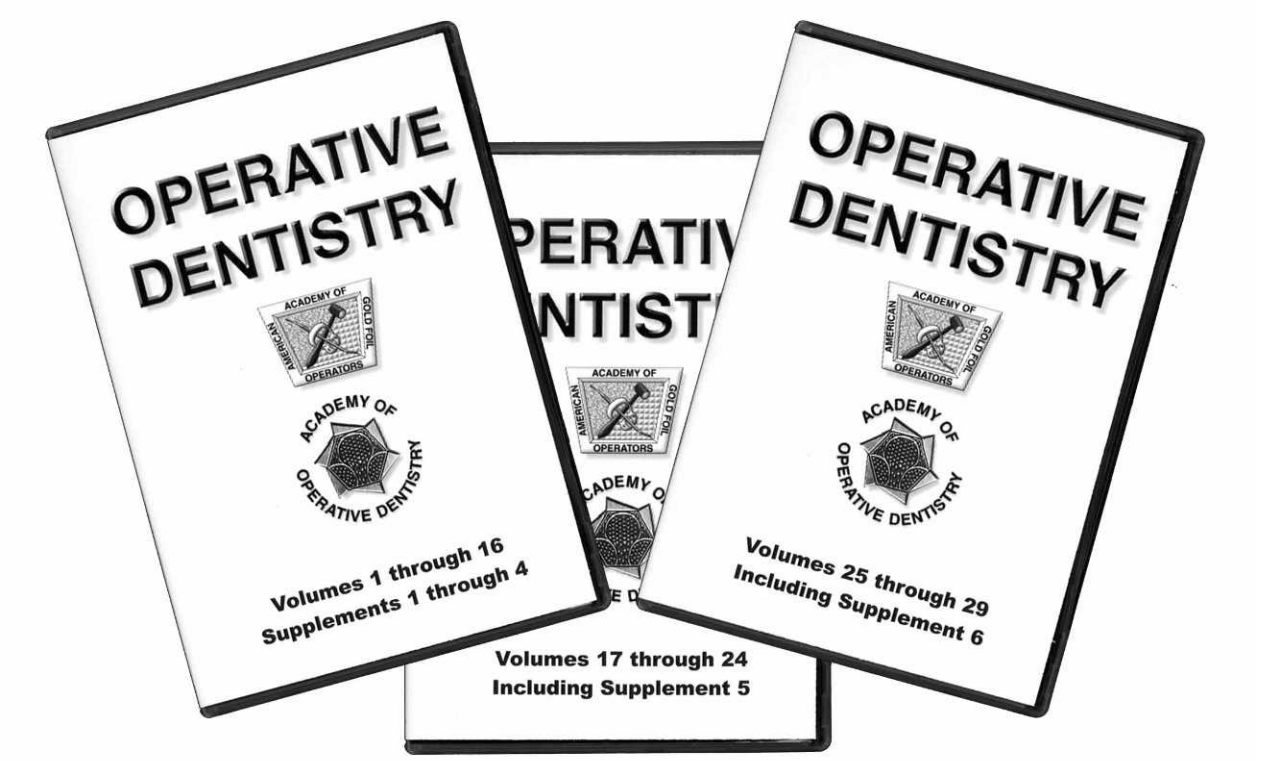
Subscribe: leads to complete information on subscription rates; purchasing back issues, reprints, and bound volumes; and subscription and change of address forms.

Affiliates: provides links to the American Academy of Gold Foil Operators, the Academy of Operative Dentistry, the AADS-Operative Section, and our Corporate Sponsors. In addition, membership applications for the journal's parent academies are available for downloading.

News: announcements of interest to our readers, including meeting information, advertised faculty positions, and upcoming CE courses.

Authors: complete instructions for contributors to the journal.

Reviewers: Link for our Editorial Board to submit manuscript reviews electronically.



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CLINICAL RESEARCH

LABORATORY RESEARCH

CLINICAL TECHNIQUE/CASE REPORT

DEPARTMENTS

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Periodicals