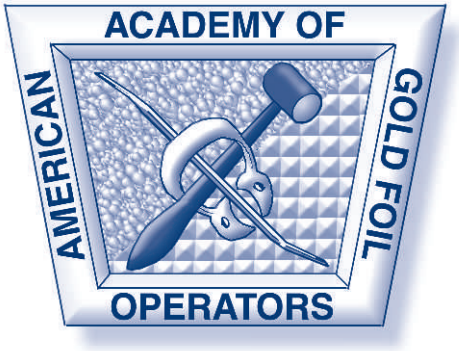
OPERATIVE DENTISTRY



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

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In Memoriam

Takao Fusayama

Dr Takao Fusayama, a scientist and teacher of true distinction, died on January 17, 2003 at age 86 of complications from pneumonia. During his illustrious career, he performed great work in the field of dentistry and provided several innovations to the profession. Dr Fusayama was dedicated to his calling and felt an extremely strong sense of responsibility and desire to develop science, education and health care promotion in dentistry.

Takao Fusayama was born in Gifu Prefecture on August 7, 1916. His father was a dentist, and Takao had no hesitation following in his father's profession, which was a very common Japanese tradition. He entered Tokyo Higher Dental School, founded in 1928 as the first governmental dental school in Japan and the forerunner of Tokyo Medical and Dental University (TMDU). In 1938, he graduated from the school, scoring the highest grades in the school's history. Immediately after graduation, Dr Fusayama joined the faculty of the school, but in the same year, he was called for army service, which was compulsory for all healthy Japanese youths. He entered the Imperial Guard Division, which was mobilized abroad after two years.

In 1941, while in French Indochina, the Pacific (Great East Asia) War broke out. He served as a radio platoon leader of the Divisional Signal Unit and Signal Company commander of the 4th Imperial Guard Infantry Regiment when Japan surrendered in 1945. After spending one year as a liaison officer managing Japan-Indonesian relations in Sumatra, he was repatriated to Japan. During these years, in his private time, he studied the folklore of the Batak race on the island and was helped by his outstanding ability in foreign languages—English and German, and some Chinese and Indonesian. In 1975, he published a book, *Mysterious Race, Batak*.

In 1946, as a means of supporting his family, Dr Fusayama worked for several months with his father in his hometown. However, he was soon invited to the Tokyo Dental School for Girls as a lecturer and was promoted to professor in 1947. In 1950, he was invited back to TMDU as a lecturer. Unfortunately, he spent the next two years and seven months fighting tuberculosis. A miraculous recovery ensued, and he returned to his position at the university.

Takao Fusayama
1916-2003

In 1955, Dr Fusayama received a PhD from Tokyo University for his thesis on the accuracy of the indirect dental stone model. Part of his thesis was published in the *Journal of the American Dental Association* (Vol 52). Dr Fusayama's was the first paper published in an international dental journal by a Japanese dentist after World War II. In 1956, Dr Fusayama received a Fulbright scholarship and studied with Professor Ralph W Phillips at the Indiana University School of Dentistry. He developed a lifelong friendship with Dr Phillips and worked to raise funds for an endowed faculty position at the Indiana University School of Dentistry (the Ralph W Phillips Scholar in Dental Materials), which honored the memory of Dr Philips after his death.

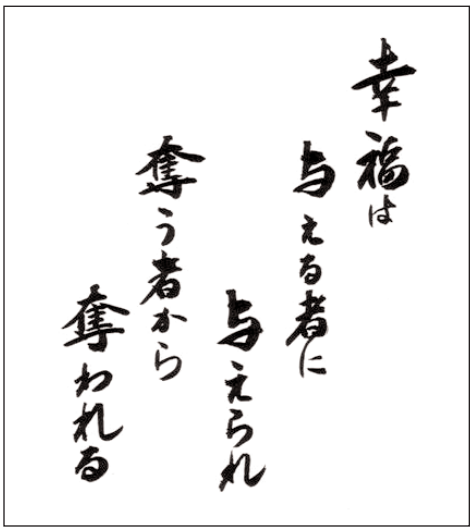
Dr Fusayama became professor and chairman of the Department of Operative Dentistry at TMDU in 1960 and served until his retirement in 1982. During the

1950s, '60s and '70s, the main research topic of the department was the improvement of cast and amalgam restorations. He developed a minimally invasive cavity design for amalgam and a new impression technique using agar and alginate materials. This technique is still widely used in Japan and some other countries.

In the 1970s, Dr Fusayama began studies on carious dentin and adhesive restorative materials. At that time, removal of caries was usually performed empirically, based on the perceived hardness of the remaining dentin. Dr Fusayama developed a practical classification of the dentinal carious lesion into two layers by histo-pathological and biochemical studies. The results were applied clinically through the development of a "caries detector" dye. At the same time, he began his research on resin adhesion, including the total etching technique that was developed in collaboration with the Kuraray Co. In 1978, the adhesive resin restoration was established as a conservative technique that minimized the removal of healthy tooth structure primarily based on Dr Fusayama's studies. This concept revolutionized dentistry and resulted in modern day minimally invasive caries treatment. Although Dr Fusayama's innovative concepts were not readily accepted, eventually his work prevailed and he was awarded the fifth American Dental Association Gold Medal for Excellence in Dental Research in 1997.

During his career, Dr Fusayama authored 25 textbooks, including two English-language books and more than 300 scientific articles. He received many honors in recognition for his groundbreaking research, including the Hollenback Award from the Academy of Operative Dentistry; the Wilmer Souder Award from the International Association for Dental Research; the Pierre Fauchard Academy Award; the List of Honor from the Federation Dentaire Internationale and the Shijuhosho Decoration by the Emperor of Japan. He was also awarded honorary memberships in the dental associations of USA, Italy and Korea and was presented with membership as the only dentist in the 135 year-old prestigious Japan Academy.

Dr Fusayama may have come across as a very disciplined personality when people first met him. However, everyone recognized his kind-heartedness and charity as they got to know him. Of course, he was very strict in what he expected of himself as a human being,



Written in Dr Fusayama's own handwriting, his motto translates into: "Happiness comes to one who gives to others, and leaves from one who deprives it."

teacher and professional. Many who knew him well must have learned from Dr Fusayama a philosophy similar to "BUSHI-DO" through watching his demeanor and lifestyle.

Dr Fusayama's cherished motto was "Happiness comes to one who deprives it." He gave happiness to many people in the world and it is believed that he led an extraordinarily happy and fruitful life. His spirit will live on in those who knew him and the colleagues whose lives his work has touched. His valuable accomplishments will be carved into the history of dentistry forever.

Dr Fusayama is survived by his wife, Setsuko, two sons, one daughter and many grandchildren. According to Dr Fusayama's will, his body was donated to Tokyo Medical and Dental University to enable medical and dental students to study human anatomy.

Junji Tagami
Professor
Cariology and Operative Dentistry
Tokyo Medical and Dental University

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Guest Editorial

How Do You Know What You Don't Know, If You Don't Know?

From a speech presented at the 2002 annual meeting of the Academy of RV Tucker Study Clubs.

This is a quote of an old friend, Dr Pat Fleege, who was a pediatric dentist. "How do you know what you don't know, if you don't know?" I apply this to the vast majority of dentists who have no concept of what is demonstrated regularly at operating study clubs throughout the world. Our profession has been very progressive in studying and teaching the new concepts that are changing almost day-to-day. However, our profession has not applied this same concern for learning and perfecting the clinical techniques that have been used very successfully for generations.

The fact is, there has been such a gap in the teaching of cast gold and gold foil procedures that much of what we did know has been lost. New techniques that have made cast gold a far more useful dental restoration have not been learned. Except for the relatively small group of dentists who are affiliated with the Academy of RV Tucker Study Clubs (approximately 600 dentists in the United States, Canada and Europe), this type of restoration is given almost no consideration in the treatment of patients or the education of students. I am aware that, even in prosthodontic graduate schools, our techniques are rarely taught, and we depend on these schools to provide our future teachers.

Although we know very well the advantages to the patients who have this type of treatment, we have seldom assumed an outreach attitude of concern that other dentists and patients become aware of what can be done with gold restorations. I assume some of the responsibility for this, in that I have long felt that if any dentist really wanted to learn, he or she would seek us out. This approach has not been entirely a failure. All of you, and the rest of the members of our Academy, have had to seek it out, because the 50 study clubs that have been operating for as long as 27 years are doing so only because individuals took the initiative to meet criteria and organize, sometimes under difficult circumstances. Some study clubs are located where there are very limited clinical facilities, so they must meet in private offices, and some must have a mentor come from half way around the world. So, my opinion has been borne out that if there is a "strong enough will, there is a way."

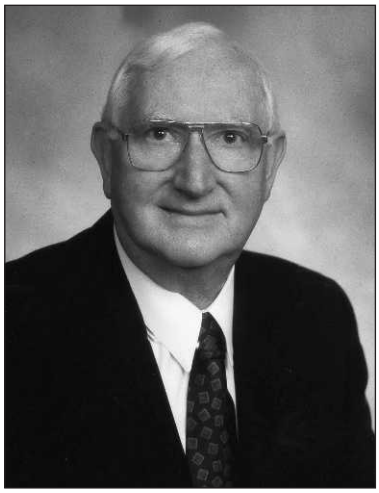
Although I have a special regard for those individuals who have sought this type of ongoing training and have made the effort and sacrifice to learn cast gold techniques as all of you have, I could be accused of having developed a rather "soft" approach to recruiting more colleagues into study club activity. I believe that we should demonstrate some concern for those who have never been exposed to what can be accomplished with fine gold restorations. "How do you know what you don't know, if you don't know?"

I am now asking the officers and directors of our Academy to take a new approach. As I observed the most remarkable operations at this annual meeting's clinical session, where the finest gold restorations of any in the world were accomplished, I could only wish that many more dentists could see and appreciate the excellence of this type of dentistry. This is, by far, the closest to lifetime dentistry that we could ever provide. Would it not be a good thing if others in our pro-

fession, who have never had the opportunity, could watch the operations being performed and the final results as we, perhaps, take them for granted?

Ours is a rather cloistered organization, but I would ask that we change policy to invite guests of the individual members, both to our annual meeting and to the individual study club sessions. I am very grateful to Dr Dennis Miya, Director of the Tucker Institute, for his excellent work in setting up the courses for dentists from around the world. Perhaps our leaders can recognize other means of exposing those outside our study clubs to this special kind of dentistry because "How do they know what they don't know, if they don't know?"

Richard V Tucker, DDS



Richard V Tucker

Commentary

I would like to thank Dr Tucker for allowing me to print his speech as a Guest Editorial in this journal. I was present at this meeting and, as always, was amazed at the quality of the work presented. Dr Tucker's presentation to the Academy, as well as my own experience with study club activities, has given me the idea for my previous editorial, "Where Have All the Mentors Gone?" (*Op Dent* **28-1**, 2003). I also felt that the quote, "How do you know what you don't know, if you don't know?" was applicable to so many things that I wanted to share it with all of you.

Michael A Cochran
Editor

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Non-Carious Cervical Lesions

INTRODUCTION

The Academy of Operative Dentistry has developed the following recommendations for diagnosis, treatment and prevention of non-carious cervical lesions. Patients should be informed of the possible etiologies, implications of the presence of these lesions, methods of minimizing their occurrence, treatment alternatives and expected outcomes. Failure to appropriately prevent and treat non-carious cervical lesions can result in continued loss of tooth structure, tooth sensitivity, weakening of the tooth, the need for endodontic therapy, or tooth loss and the occurrence of additional lesions in other teeth.

Clinical Assessment and Diagnosis

A. Etiology

The etiology of non-carious cervical lesions is not completely understood. Several different causes have been implicated, and there is evidence to support each. It can be said with a high degree of certainty, however, that the cause is multifactorial (Bader & others, 1996).

That acid causes erosion of tooth structure is well established. The term dental erosion describes the process and the results of loss of dental hard tissue, chemically etched away from the tooth surface by acid and/or chelation, without bacterial involvement (ten Cate & Imfeld, 1996). Many common foods and beverages (citrus fruits and juices, soft drinks and wines) have been shown to have an acidic pH in the range that will dissolve enamel (Lussi, Portmann & Burhop, 1997; Amaechi, Higham & Edgar, 1999; Larsen & Nyvad, 1999; Künzel, Cruz & Fischer, 2000; Lussi & others, 2000; Meurman & Vesterinen, 2000). A pH below 5.5 has been shown to dissolve enamel (Meurman & ten Cate, 1996). Acids also cause the softening and loss of dentin (Shellis, 1996). Acidic drinks supplemented with adequate calcium and phosphate do not erode enamel (Larsen & Nyvad, 1999; Lussi & others, 2000). In addition, fluoride in acidic liquids and fluoride pre-treatment have been shown to reduce acidic erosion of enamel and dentin (Sorvari, Pelttari & Meurman, 1996; Thiradilok, 1976).

Wear or abrasion of tooth structure is also implicated as a factor in the occurrence of non-carious cervical lesions. Wear of tooth structure results from the mechanical rubbing of teeth by some object or objects. In the cervical area, this may be due to the toothbrushing techniques, equipment or pastes, or it may be the result of habits the patient has. Frequency of toothbrushing has been implicated in the progression of wedge-shaped defects (Lussi & Schaffner, 2000). Enamel is very resistant to wear, so abrasion is slow. Once exposed, dentin wears much faster (Forss, Seppa & Lappalainen, 1991). Its wear rate is many times greater than that of enamel (Stookey & Schemehorn, 1979; Stookey & Muhler, 1968).

There is evidence that the loss of tooth structure from abrasion is accelerated when acid softening or dissolution of enamel and dentin occurs prior to or during mechanical abrasion of those tooth tissues (Attin & others, 2000; Ganss, Klimek & Friedrich, 1999; Attin & others, 1997; Davis & Winter, 1980). If the tooth surface is not exposed to mechanical abrasion during the softened state, remineralization reverses the softening after a period of exposure to saliva (Jaeggi & Lussi, 1999). Most Americans have been taught to brush after meals; this may not be the best guidance in relation to the development of non-carious cervical lesions. A delay of as little as one hour after an acid challenge, with teeth exposed to saliva, can increase their resistance to abrasion (Attin & others, 2000). The use of a fluoride-containing dentifrice several times a day has been shown to be beneficial because of the fluoride exposure (Larsen, 2001). That exposure could occur without abrasion through rinses instead of brushing.

Much attention has been directed to the role that tooth flexure from occlusion may play in the development of non-carious cervical lesions. A non-carious cervical lesion that is believed to be caused by

tooth flexure has been called "abfraction," meaning "to break away." So many articles have implied or stated that occlusal forces cause non-carious cervical lesions (Grippo, 1991; Grippo & Massi, 1991; Lee & Eakle, 1984) that this theory has become accepted as fact. There is ample evidence that teeth flex or bend (Powell, Nicholls & Shurtz, 1977; Powell, Nicholls & Molvar, 1980; Sakaguchi & others, 1991) and that a point of stress concentration is the cervical area (Palamara & others, 2000; Kuroe & others, 2000). While there is a limited, but growing, body of evidence that suggests that tooth flexure may be a factor in the initiation and advance of non-carious cervical lesions (Pintado & others, 2000; Whitehead, Wilson & Watts, 1999; Braem, Lambrechts & Vanherle, 1992), it has not been clearly substantiated as a cause. Additional evidence is needed to determine the role that so-called "abfraction" plays.

As stated previously, it is certain that the etiology of non-carious cervical lesions is not the result of one single factor. The etiology is multifactorial, with acid and other tooth-softening chemicals, abrasion, and very possibly tooth flexure each playing a role to a greater or lesser degree from patient to patient and tooth to tooth (Bader & others, 1996; Piotrowski, Gillette & Hancock, 2001).

B. Clinical Features and Methods of Assessment

Although the specific causes of a particular shape and depth of a lesion cannot yet be determined accurately, wedge-shaped lesions are believed to be more associated with abrasion than with erosion, whereas lesions that are wider occlusogingivally than they are deep are believed to be associated more with erosion than abrasion (Lussi & Schaffner, 2000; Bergström & Lavstedt, 1979).

Indications for Treatment

Unless the enlargement of a non-carious cervical lesion has been halted, some intervention is needed. If the lesion is relatively small and the tooth is not sensitive, treatment should usually consist of efforts to remove the causal factors. A determination of the primary cause(s) should be made and, if the patient will agree and comply, the cause should be reduced or eliminated.

For lesions influenced by acidic dissolution or softening of tooth structure, the most frequent acid challenge should be assessed. The patient should be counseled to avoid the acidic substance or, in some way, to bring about a buffering of the acid. If an acid challenge, such as from fruit juice or soft drink ingestion, is followed by brushing within a few hours, the time of brushing should be altered. Patients with conditions such as gastro-esophageal reflux disorder (GERD) should be counseled to seek medical attention for the problem.

THERAPEUTIC GOALS

The first treatment goal for patients with non-carious cervical lesions should be to remove the etiological factors. The lesion itself should then be evaluated to determine if the destruction appears to have halted, if gingival tissue repositioning or connective tissue grafting is needed to protect the tooth from future problems, or if a restoration should be placed.

Therapeutic Considerations

The first therapeutic measure to be considered in treatment of these lesion(s) is to help the patient eliminate or reduce/modify the most likely cause. If acid is believed to be part of the etiology, the source should be found and eliminated. Exposure to fluorides will reduce the softening effect of acids (Larsen, 2001). A combination of fluoride and Xyitol has been shown to have a greater positive effect on remineralization than either agent used alone, and there is an additive effect in the reduction of tooth erosion from acidic drinks (Amaechi, Higham & Edgar, 1999).

Sources of abrasion should be detected and modified to reduce any abrasive effect. Brushing methods may be modified and harder brushes and more abrasive toothpastes exchanged for soft brushes and less abrasive toothpastes.

Although loss of tooth structure due to tooth flexure (abfraction) is not yet well supported by research findings, occlusal stress may be a factor in producing at least some lesions. Therefore, occlusal stresses on the affected tooth/teeth should be evaluated and reduction of heavy lateral forces considered. Reduction of laterally-directed stresses on the tooth will reduce the concentration of force in cervical areas. This may help reduce the loss of cervical tooth structure (Kuroe & others, 2000).

Dentin is much more subject to loss due to acid attack and wear than enamel. When cervical dentin is exposed, either from loss of overlying enamel or from loss of gingiva covering the root, tooth structure loss may accelerate unless etiological factors are determined and eliminated. The determination and subsequent elimination of etiologic factors may not always be possible. In instances when esthetic considerations allow and loss of cervical tooth structure is minimal or lesions are shallow, exposed dentin may be covered with gingival tissue via a coronally positioned flap (Saletta & others, 2001); or a connective tissue grafting procedure (Cordioli & others, 2001; Novaes & others, 2001; Majzoub & others, 2001).

Exposed dentin in the cervical area, with or without the loss of tooth structure, may be sensitive. Dentin sensitivity may be caused by thermal changes from foods, beverages or air passing over the area, and/or mechanical stimulation (touch). If there is no loss or

minimal loss of tooth structure, placement of a restoration should be low on the list of treatments. Several non-restorative treatments of sensitive cervical dentin have had significant success.

One method of treating cervical sensitivity is to reposition the gingival tissue coronally. A semi-lunar flap is created and placed over, and coronal to, the exposed dentin. The stabilized tissue heals in place and protects the cervical area against further loss of tooth structure and against sensitivity. Another method of treating cervical sensitivity is the connective tissue graft as mentioned previously.

Chemical means of treating cervical sensitivity have also demonstrated a significant level of success. Application of fluoride solutions or other fluoride-containing compounds has been shown effective. In one study (Thrash, Jones & Dodds, 1992), a 0.717% fluoride solution containing 1.09% sodium fluoride, 0.4% stannous fluoride and 0.28% hydrofluoric acid was applied to sensitive dentin twice daily for two weeks. This regimen brought about a significant reduction in sensitivity. In addition, a 0.4% stannous fluoride gel was found to significantly reduce dentinal sensitivity (Thrash & others, 1992; Blong & others, 1985). When this fluoride treatment was followed by home use of a 0.4% stannous fluoride gel, the relief was sustained for a long period of time (Thrash, Dodds & Jones, 1994). Dentifrices containing potassium nitrate, potassium chloride or strontium chloride have also been shown to reduce dentinal sensitivity (Silverman & others, 1996; Silverman, Gingold & Curro, 1994; Salvato & others, 1992).

Several studies have shown that some resin bonding agents (Swift, May & Mitchell, 2001; Dondi Dall'Orologio & others, 1999; Ferrari & others, 1999), the primers of some bonding agents (Wantanabe & others, 1991) and resins designed specifically for the purpose can be effective in reducing or eliminating sensitivity.

Iontophoresis is a technique that uses low-amperage, direct electrical current to introduce ions or ionized drugs such as fluoride into the tooth. This technique has been used successfully to reduce or eliminate dentinal sensitivity (McBride, Gilpatrick & Fowler, 1991).

Some non-carious cervical lesions may require placement of a restoration of some sort. The decision to restore the area will be dependent upon several factors, as follows:

- Inability to eliminate or greatly reduce the rate of lesion progression through elimination of etiological factors.
- Lesion is esthetically unacceptable to the patient.
- Significant sensitivity of exposed dentin to cold liquids, foods and air.
- Depth of the lesion threatens the strength of the tooth and the integrity of the coronal-radicular unit.

Selection of Restorative Material

When a restoration is needed, there are several materials from which to choose, each with its own advantages and disadvantages. The restorative materials range from direct gold (foil), chosen for its durability, to resin composite, selected for its relative conservation of tooth structure, ease of placement and esthetic appearance. The following are possible choices of restorative materials and some considerations for or against selecting:

Direct gold (foil)—This material has proven its durability over the last century and has often been used in cervical areas. However, tooth preparation for its placement is not conservative, the procedure is exacting and requires excellent moisture control and the material is not esthetic. The cost of the restoration is also comparatively very high.

Amalgam—Amalgam has been used for restorations in cervical areas for many years. It has good durability and is relatively inexpensive. Its drawbacks include its unesthetic appearance and its need for a preparation that destroys sound tooth structure to assure adequate bulk of material and mechanical retention.

Resin composite—Bonded resin composite restorations, without cavity preparation, are proving to be excellent for restoring teeth that have non-carious cervical lesions (Duke, Robbins & Snyder, 1991; Leinfelder, 1994; McCoy & others, 1998). Resin composite is offered in a variety of shades and translucencies for matching tooth color, and it can provide an extremely natural, esthetic appearance. Bonding to the tooth, whether to enamel or dentin, has proven so successful that macro-mechanical features in the preparation are generally unnecessary (Duke & others, 1994; McCoy & others, 1998). The preparation does not need depth or specific form because bulk of material is not needed. Metallic and ceramic materials do require bulk for strength. Placement is relatively easy, and tooth preparation is more conserving of sound tooth structure than other restorative techniques. Resin composite restorations can exhibit marginal staining and/or dislodgement over time if not adequately bonded.

Glass-ionomer, resin-modified glass-ionomer and polyacrylate-modified resin composite (Compomer)—These materials are not used as frequently as resin composite because some tend to be more difficult to place and do not have the wear resistance or tensile and compressive strength of resin composite.

Although tooth colored, they generally do not offer the excellent esthetic qualities provided by resin composites. To offset these disadvantages, however, they provide chemical and mechanical bond to tooth structure, release fluoride and can be recharged with topical fluoride to provide long-term fluoride release (Wood, Maxymiw & McComb, 1993; Haveman, Burgess & Summitt, 1999). This fluoride release may be especially beneficial in mouths at high risk for caries. There is growing evidence of the benefits of fluoride-releasing materials in helping to diminish caries occurrence (Haveman & others, 1999; ten Cate, Buijs & Damen, 1995; Jacobson, Strang, & Stephen, 1991).

Indirect Restorations—Indirect, laboratory-fabricated restorations have also been used in the cervical area. These have been made from gold alloys, ceramics and resin composite materials. They have been successful in limited situations when carefully selected, but they require greater tooth reduction, specific preparation shape regardless of the lesion shape and more appointment time. They are more expensive. As a general rule, they are not indicated for restorations placed due to cervical lesions, either carious or non-carious.

OUTCOMES ASSESSMENT

A satisfactory result of non-restorative therapy is the absence of progression of any lesions present in the mouth, the absence of sensitivity associated with any lesion and the absence of occurrence of any new lesions. Satisfactory outcome for a restoration is the absence of loss of tooth structure adjacent to the restoration, the long-term retention of the restoration without loss of contour or esthetics and absence of sensitivity associated with the restored tooth.

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Clinical Research

Clinical Evaluation of In-Office and At-Home Bleaching Treatments

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Clinical Relevance

An ADA-accepted at-home bleaching treatment is more effective, more acceptable to patients and requires less chairtime compared to an ADA-accepted in-office bleaching treatment.

SUMMARY

This three-month, single-blind clinical study compared two whitening treatments, at-home with 10% carbamide peroxide and in-office with 35% hydrogen peroxide, for the degree of color change of teeth, color relapse and tooth and gum sensitivity. The degree of color change and color relapse was evaluated by using a colorimeter, shade guide and color slide photography. Teeth and gum sensitivity were self-evaluated by the subjects, who recorded daily the tooth and gum sensitivity they experienced during the two weeks of treatment and one week post-treatment.

A 14-day at-home treatment was compared with 60 minutes of in-office treatment (two appointments, each with three 10-minute applications). The at-home treatment produced significantly lighter teeth than the in-office treatment during all active-treatment periods and follow-up visits according to all three-color evaluation methods. Color relapse for both treatments stabilized by six weeks. At-home treatment resulted in statistically significant higher gum sensitivity than in-office treatment during the latter part of the first week. For tooth sensitivity there were no significant differences between the treatments. Eighty four percent of the subjects reported at-home treatment to be more effective and 16% found no difference between the treatments. There were no subjects who reported the in-office treatment to be superior in tooth whitening to the at-home treatment.

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INTRODUCTION

Cosmetic dentistry has become an important part of restorative dental practice in recent years. The appearance of teeth is very important to patients of all ages and is often associated with a perception of health and fitness. Cosmetic procedures have become more available as standards of living have improved. Dentistry has also succeeded in reducing the frequency and severity of caries and periodontal diseases, which has led to the preservation of natural teeth even in older patients. Since white teeth are believed to be associated with health and beauty, lighter-colored teeth have become desirable. It is up to our profession to offer the treatment to allow patients to achieve their goals safely. Vital tooth bleaching can be performed with a high rate of success as a more conservative measure than restorative treatment, such as porcelain veneers, crowns or composite bonding (Barghi, 1998).

In-office vital tooth bleaching has been used for many years in dentistry and is known to be a reliable technique for quickly lightening discolored teeth (Faunce, 1983; Jordan & Boksman 1984; Nathanson & Parra, 1987). Today, patients have the choice of having the procedure done in-office or at-home. At-home vital tooth bleaching also has been shown to produce a significant perceivable change in color, reducing chair time and, therefore, it has become very popular (Jones & others, 1999; Kihn, Barnes & Romberg, 2000; Swift, May & Wilder, 1999).

This study evaluated the degree of color change of teeth, color relapse and tooth and gum sensitivity associated with ADA-accepted in-office and at-home tooth whitening agents.

METHODS AND MATERIALS

At the screening visit, subjects were evaluated to determine if they met the inclusion (Table 1) and exclusion (Table 2) criteria. During the same appointment all subjects had two alginate impressions of their maxillary arch taken with Jeltrate Plus (Caulk Division, Dentsply International Inc, Milford, DE 19963, USA). Study models were made from Silky-Rock stone (Whip Mix Corp, Louisville, KY 40217, USA). One model was used to fabricate the night-time bleaching tray for at-home bleaching according to the manufacturer's recommendations.

The labial surfaces of anterior teeth of the study model were blocked out from approximately 1.0 mm incisal to the gingival margin to the incisal edge with LC Block-Out Resin (Ultradent Products, Inc, South Jordan, UT 84095, USA). This created reservoirs for the bleaching gel in the custom tray. The custom maxillary tray was fabricated from a 0.035-inch soft tray (Sof-Tray, Ultradent Products, Inc) by a vacuum form-

Table 1: *Inclusion Criteria*

1. Have all six maxillary anterior teeth darker than B-54 and lighter than B-85 on the Trubyte Bioform Color Ordered shade guide.
2. Have no maxillary anterior teeth with more than 1/6 of the labial surface of their natural tooth covered with a restoration.
3. Be at least 18 years of age.
4. Be willing to refrain from the use of tobacco products during the study period.

Table 2: *Exclusion Criteria*

1. History of any medical disease that may interfere with the study or require special consideration.
2. Use of tobacco products during past 30 days.
3. Past use of professionally-applied or prescribed in-office or at-home bleaching agents.
4. Gingival index score greater than 1.0.
5. Pregnant or lactating women.
6. Tetracycline stained teeth.

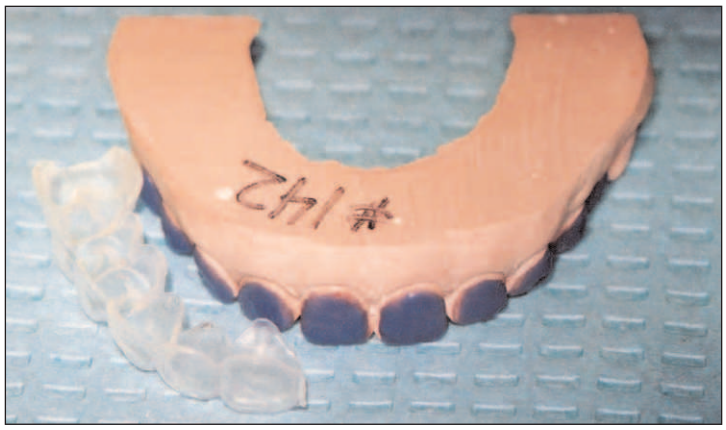

Figure 5. *A soft tray was custom-made and cut in half for each patient to use for half-mouth arch only.*

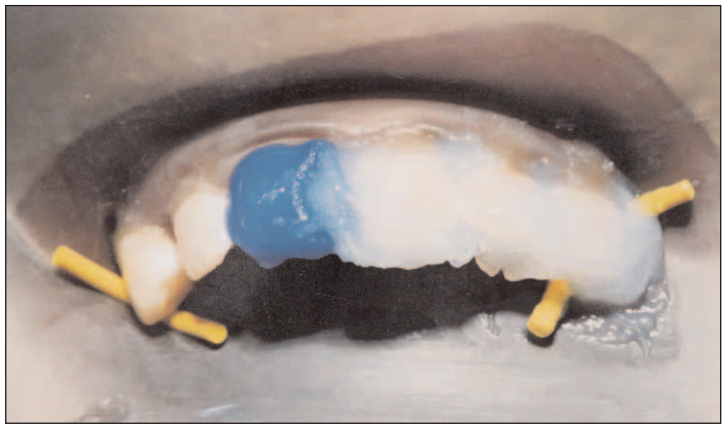

Figure 6. *In-office treatment was performed for half of the maxillary arch that was not treated by at-home bleaching.*

ing technique. The excess was trimmed on the labial and lingual surfaces just incisal to the free gingival margin. The trays were then cut in half between teeth #8 and #9 (Figure 5). Patients received only the side intended for at-home bleaching. The second study model was used to construct a positioning jig with full palatal coverage to ensure proper repositioning of the colorimeter. The Eichhold Coupling System (Mokhlis, 1999; Panich, 1999) with Pindex dual-pin precision attachments (Coltene/Whaledent Inc, Mahwah, NJ 07430, USA) was used in this study.

Twenty subjects qualified to participate in this study; 19 completed the study. One subject was excluded from the study because the facial anatomy did not allow for proper positioning of the colorimeter. Patients signed a consent form approved by the Institutional Review Board at Indiana University-Purdue University Indianapolis (IUPUI). All subjects received a prophylaxis by a licensed hygienist or dentist at least one week prior to beginning the study. Extrinsic dental stains were removed with a fluoride containing dental prophylaxis paste (Nupro Supreme, Dentsply Int, York, PA 17404, USA).

During the baseline appointment and at one, two, three, six and 12 weeks, color evaluation was performed using three methods: 1) subjective shade guide matching by an independent experienced evaluator using the Trubyte Bioform Color Ordered shade guide (Dentsply Int); 2) by comparing clinical photographs recorded on Elite Chrome 100 35 mm slide film (Kodak, Rochester, NY 14650, USA). The slide photographs were projected to an image of 3.0 X 4.5 feet in size and were compared for color changes by two independent evaluators. The evaluators categorized the left and right side of the maxillary arch into one of four gradients: 0–no difference, 1–slight difference, 2–moderate difference, 3–large difference. 3) Objective color measurements using a color measuring device (Chroma Meter CR 321, Minolta, Ramsey, NJ 07446, USA). The six anterior maxillary teeth were measured colorimetrically three different times at each appointment. The colorimeter measures the color of the teeth based on the CIE L*a*b* color space system. This system was defined by the International Commission on Illumination in 1967 and is referred to as CIELab (Commission Internationale de L'Eclairage, 1978). The L* represents the value (lightness or darkness), a* is the measurement along the red-green axis and b* is the measurement along the yellow-blue axis. Total color differences or distances between two colors (ΔE) was calculated using the formula: $\Delta E = [(\Delta L^*)2 + (\Delta a^*)2 + (\Delta b^*)2\ ]1/2$ (Commission Internationale de L'Eclairage, 1978).

All subjects were given a sensitivity sheet on which they indicated the level of tooth and gum sensitivity they experienced during the two weeks of treatment and for seven days after the treatment. Subjects recorded any tooth and/or gum sensitivity (indicating whether the left and/or right) in one of five categories: 1–none, 2–slight, 3–moderate, 4–considerable, 5–severe. Patients who experienced more than a moderate degree of sensitivity received potassium nitrate desensitizing gel (UltraEZ, Ultradent Products, Inc) and were instructed to place the gel on the sensitive side for 20 minutes three times a day.

Two commercially available bleaching agents, accepted by the ADA, were used in this study: Opalescence Tooth Whitening Gel with 10% carbamide peroxide (Ultradent Products, Inc) for at-home bleaching and StarBrite (Interdent, Inc, Los Angeles, CA 90232, USA) with 35% hydrogen peroxide for in-office bleaching. Manufacturers' instructions for handling and application were followed for all products used in this study. The bleaching treatments were randomly applied to the right or left maxillary anterior teeth. The side was determined by flipping a coin.

Custom-made at-home half-arch bleaching trays were tried in the mouth and adjustments were made so that they were retained snugly. Patients were instructed verbally and by hands-on practical demonstration regarding the use bleaching gels and custom-made bleaching trays according to manufacturers' instructions. Patients were asked to continue this procedure for 14 nights. All patients were asked to brush their teeth at least twice a day for oral hygiene standardization.

At the first week appointment a rubber dam was placed and the in-office procedure was accomplished on the side of the maxillary arch not treated by at-home bleaching (Figure 6). Both the subjects and all measurement personnel wore protective eyewear with side shields during the in-office tooth whitening procedure. The gel was mixed and applied to the teeth according to the manufacturer's recommendations. After five minutes the bleaching gel was stirred on both the lingual and facial tooth surfaces. The gel remained on the teeth for a total of 10 minutes. The teeth were then rinsed and dried. The entire procedure was repeated two more times. Total in-office bleaching procedure time was approximately 30 minutes. After completion of the bleaching procedure, the rubber dam was removed and the teeth were allowed to rehydrate for 15 minutes. A color evaluation was performed using all three methods to determine the color of the teeth. Patients used the at-home bleaching trays with the same instructions for another week and returned for the second in-office bleaching appointment.

During the week two appointment, the same protocol was followed as at the first week's appointment. At this appointment, subjects discontinued at-home bleach-

ing. Subjects returned all used and unused syringes with bleaching gels to ensure completion of the at-home bleaching. Subjects returned in three, six and 12 weeks from baseline evaluation for the same type-color evaluation that was conducted during the baseline evaluation.

Subjects completed a questionnaire at their last evaluation visit, recording their personal responses to the questions: 1) did they notice a difference in the color of upper teeth between the right and the left sides and 2) what was their overall impression of the effect of at-home bleaching at two weeks and 12 weeks.

STATISTICAL METHODS

The products were compared for differences in baseline mean L*, a*, b* and shade guide rank using repeated measures analysis of variance (ANOVA), with fixed effects for product, tooth type and the product-by-tooth type interaction. The products were compared for differences in mean ΔL*, Δa*, Δb*, ΔE and Δ shade guide rank using repeated measures ANOVA, with fixed effects for product, tooth type, time and all interactions between the three factors and baseline measurements as covariates. ANOVA, with fixed effects for product, day and the product-by-day interaction and random subject effects, were used to compare daily gum and tooth sensitivities. Wilcoxon Sign Rank tests were used to determine significance of differences in tooth color by slide assessment, tooth lightness and tooth and gum sensitivity in the subject survey.

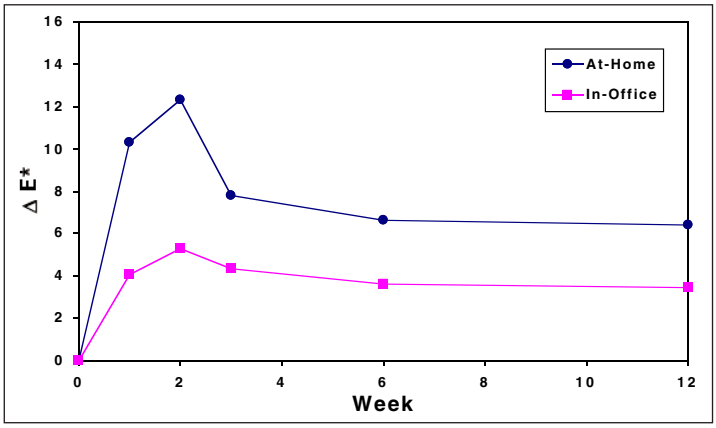

Figure 1. *ΔE for at-home and in-office treatments.*

RESULTS

Nineteen subjects completed the study. Ten were assigned the at-home (Opalescence 10% CP) bleaching treatment on the right side of the maxillary arch and nine used the at-home bleaching treatment on the left side. Opposite sides of the maxillary arches were treated with the in-office (StarBrite 35% HP) bleaching gel.

Chroma Meter Data

The products did not have significantly different baseline L* (p=0.56), a* (p=0.76) or b* (p=0.52). The at-home treatment had significantly more color change in ΔL*, Δa*, Δb* and ΔE overall (p=0.0001) and at each individual follow-up examination (p=0.0001) than the in-office treatment (Figure 1, Tables 3 and 4).

Shade Guide Data

The Δ shade guide rank order for teeth that received the at-home treatment was significantly different from the teeth that received the in-office treatment overall (p=0.0001) and at each follow-up examination (p=0.0001) (Figure 2, Table 4).

Clinical Slide Data

The assessment of clinical slides showed no significant differences between the right and left sides at baseline (p=1.00). At-home treatment was significantly lighter than in-office treatment at every follow-up examination (p=0.0001) (Table 6).

Sensitivity Data

At-home treatment had significantly higher gum sensitivity than in-office treatment for day four (p=0.0042), day five (p=0.0001), day six (p=0.0269) and day seven (p=0.0269); the overall test, combining all days, was also significant (p=0.0378) (Figure 3). The overall test, combining all days for tooth sensitivity, did not reach statistical significance (p=0.0631). None of the tooth sensitivity comparisons for the individual days were significant between treatments (p>0.15) (Figure 4).

Table 3: *Mean ΔL*, Δa* and Δb* with Standard Deviations (SD) for At-Home (At-H) and In-Office (In-O) Treatments*

Week	Mean ΔL*				Mean Δa*				Mean Δb*			
	At-H	SD	In-O	SD	At-H	SD	In-O	SD	At-H	SD	In-O	SD
1	8.57	2.92	3.43	1.72	-1.22	0.42	-0.19	0.26	-5.08	1.52	-1.49	1.05
2	10.16	3.56	4.45	1.86	-1.43	0.53	-0.54	0.28	-6.18	1.56	-2.49	1.03
3	5.90	2.85	3.10	1.69	-1.12	0.58	-0.60	0.45	-4.32	1.98	-1.71	2.36
6	4.92	2.81	2.75	1.18	-0.90	0.54	-0.53	0.34	-3.78	1.18	-1.57	1.02
12	4.91	2.61	2.55	1.43	-0.93	0.45	-0.51	0.27	-3.46	1.34	-1.57	1.04

Survey Data

According to the subjects survey data, at-home treatment produced significantly lighter teeth than in-office treatment (p=0.0001), 84% of subjects reported at-home treatment to be more efficient and 16% reported no difference between the treatments. No subjects found the in-office treatment to be superior to the at-home treatment. Sixteen percent of the subjects found the in-office treatment to cause more sensitivity than the at-home treatment, 74% found no difference between treatments and 11% found that at-home treatment caused more sensitivity than in-office treatment (Table 5).

Table 4: *Mean ΔE and Δ Shade Guide (SG) with Standard Deviations (SD) for At-Home (At-H) and In-Office (In-O) Treatments*

Week	Mean ΔE				Mean ΔSG			
	At-H	SD	In-O	SD	At-H	SD	In-O	SD
1	10.30	2.60	4.05	1.62	-12.98	4.19	-9.14	3.72
2	12.32	2.89	5.32	1.93	-15.98	2.50	-10.54	4.13
3	7.83	2.77	4.33	1.95	-14.75	3.53	-10.60	4.57
6	6.64	2.48	3.63	1.12	-13.75	3.35	-9.42	3.53
12	6.39	2.39	3.48	1.27	-13.95	3.94	-9.93	4.46

Table 5: *Subjects Survey for Lighter Side and Difference in Sensitivity*

In-Office Lighter	No Difference	At-Home Lighter
# of subjects (%)	# of subjects (%)	# of subjects (%)
0 (0%)	3 (16%)	16 (84%)
In-Office More Sensitivity	No Difference	At-Home More Sensitivity
# of subjects (%)	# of subjects (%)	# of subjects (%)
3 (16%)	14 (74%)	2 (11%)

Table 6: *Slide Assessment for At-Home and In-Office Treatments*

Week	In-Office Lighter	No Difference	At-Home Lighter
	# of subjects (%)	# of subjects (%)	# of subjects (%)
0	0 (0%)	19 (100%)	0 (0%)
1	0 (0%)	0 (0%)	18 (100%)
2	0 (0%)	0 (0%)	19 (100%)
3	0 (0%)	0 (0%)	19 (100%)
6	0 (0%)	0 (0%)	19 (100%)
12	0 (0%)	0 (0%)	19 (100%)

DISCUSSION

There is a general belief among the general population and anecdotal evidence among dentist practitioners that in-office bleaching is superior to at-home bleaching. Some manufacturers claim that high concentration hydrogen peroxide bleaching agents are superior and faster compared to the low concentration carbamide peroxide at-home products (Technical Specifications, 2001). However, no published studies that compare these two treatments are available in the scientific literature. Haywood and Berry (2001) have stated that the efficacy of in-office bleaching may not be as good as at-home bleaching treatment.

Objective Evaluation

At two weeks, average ΔE reached 12.32 for at-home treatment and 5.32 for in-office treatment. Clinically, this could mean that patients would need longer in-office treatment time to achieve the same results as with at-home treatment. The average ΔE for at-home treat-

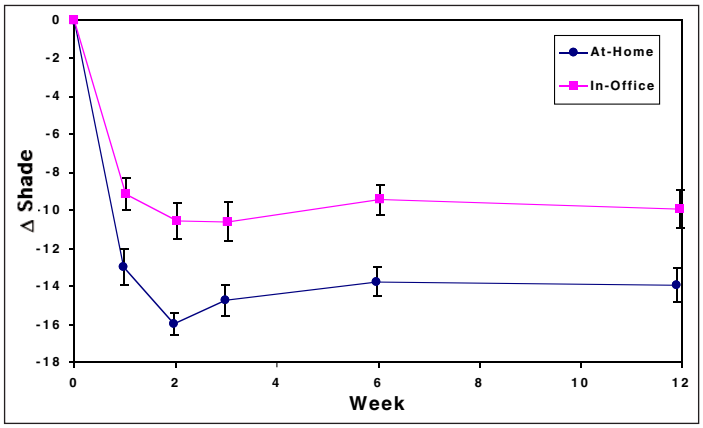

Figure 2. *Δ shade for at-home and in-office treatments.*

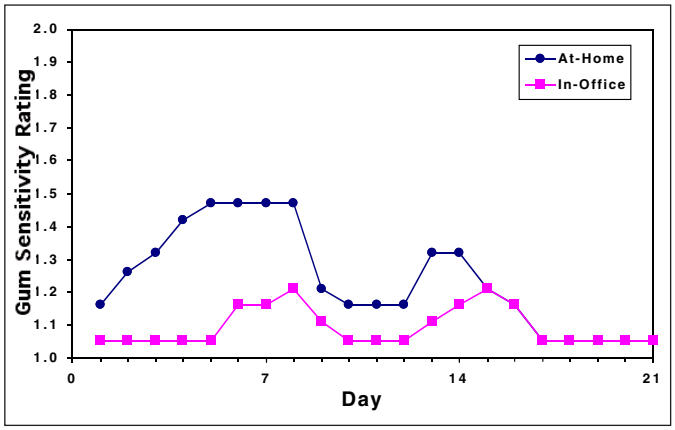

Figure 3. *Gum sensitivity for at-home and in-office treatments.*

ment obtained in this study agrees with the average ΔE obtained by Matis and others (1998), who reached an average 10.4 for average ΔE after two weeks of bleaching with 10% carbamide peroxide. Another study by Mousa (1998) investigated tooth color change with 10% and 15% carbamide peroxide. He found that after two weeks of bleaching with 10% carbamide peroxide, average ΔE reached 8.79.

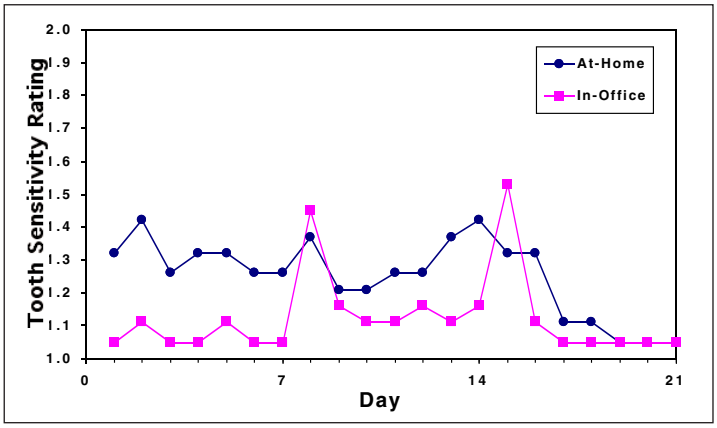

Figure 4. *Tooth sensitivity for at-home and in-office treatments.*

There is no available scientific literature to compare in-office (35% hydrogen peroxide) treatment results with results received for the same product with the chroma Meter CR-321 (Minolta) color-measuring device. In this study, color relapse began after bleaching treatments were finished and continued until the sixth week, after which there was no significant change in ΔL*, Δa*, Δb* and ΔE for either treatment, however, there were significant differences between the treatments.

The at-home treatment color relapse pattern agrees with a six-month *in vivo* study by Matis and others (1998), a six-week study by Mousa (1998) and a 12-week study by Mokhlis (1999). In-office treatment color change and color relapse was at a lower rate compared to the at-home treatment. Color stabilized by six weeks for both at-home and in-office treatments at a level significantly different from baseline. At six weeks ΔE

Figure 7 a-f: *Subject #14 was a 25 year-old female patient. The subject's right side was treated with at-home agent; the left side was treated with in-office agent.*

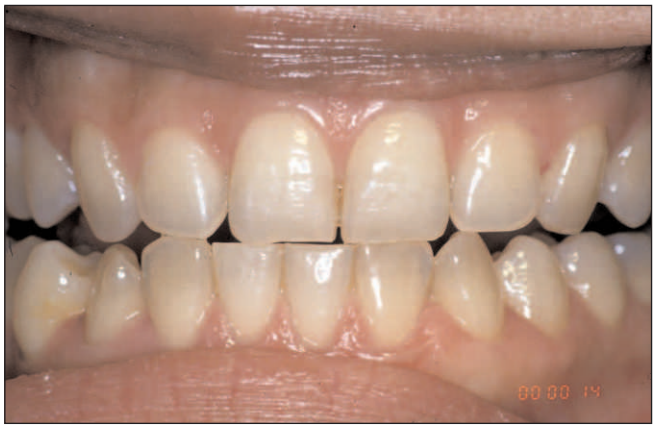

Figure 7a. *Baseline.*

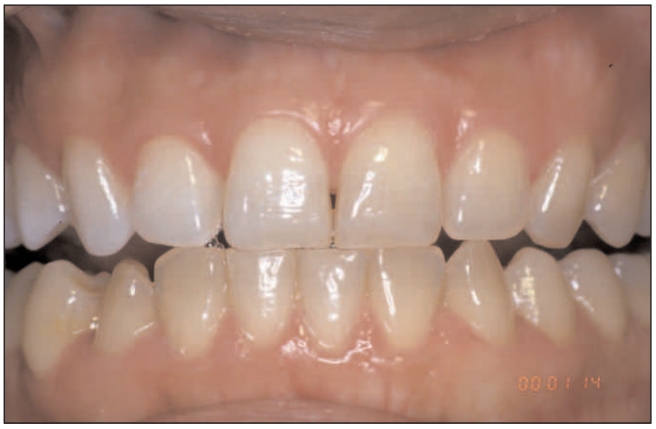

Figure 7b. *Week 1.*

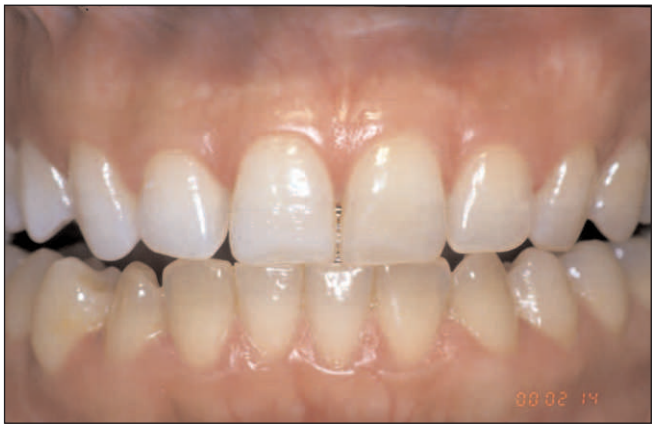

Figure 7c. *Week 2.*

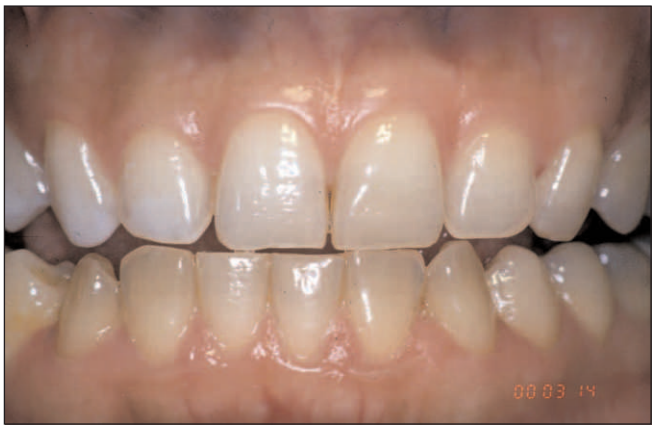

Figure 7d. *Week 3.*

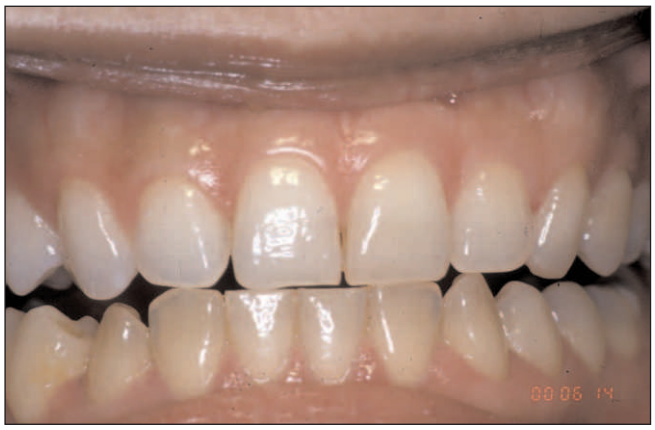

Figure 7e. *Week 6.*

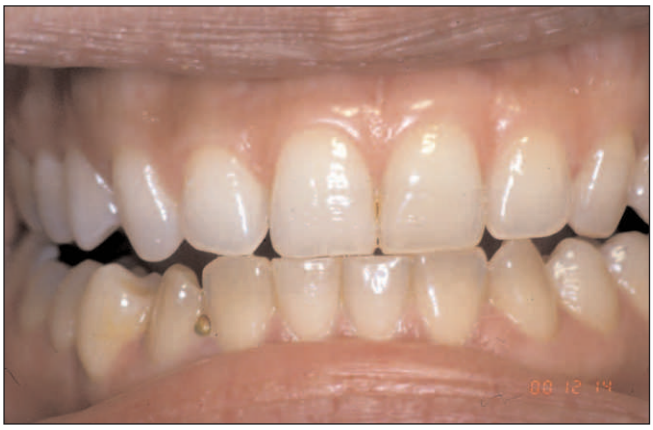

Figure 7f. *Week 12.*

decreased to 6.64 for at-home treatment and to 3.63 for in-office treatment. In the study by Matis and others (1998), the six-week ΔE value for 10% carbamide peroxide was 5.7. Mousa (1998) found that the six-week ΔE value was 5.13.

Subjective Evaluation

Subjective shade guide matching was performed using the Trubyte Bioform Color Order Shade Guide, which consists of 24 shade tabs. After two and six weeks of bleaching with at-home and in-office treatments, Δ shade guide rank reached the peak of -15.98 and -10.54, respectively. The Δ shade guide rank obtained in this study agrees with the study by Mousa (1998), where Δ shade guide rank reached the peak of -15.40 after two weeks of bleaching with 10% carbamide peroxide and with the study by Matis and others (1998), where peak Δ shade guide reached -17.5. In this study, during color relapse Δ shade guide rank decreased to -13.75 for at-home and to -9.42 for in-office treatment at six weeks. These results agree with studies by Mousa (1998) and Matis and others (1998), where sixth week Δ shade guide rank values decreased to -13.13 and -12.2, respectively. The subjective slide evaluation in this study showed a statistically significant difference between the treatments, both during the active treatment period and at each follow-up visit. The colorimeter and shade guide measurements agree and reinforce this finding (Figure 7 a-f).

Some subjects experienced mild gingival or tooth sensitivity associated with at-home bleaching, which lasted up to two weeks. One subject experienced moderate tooth and gum sensitivity during the first week of at-home treatment. The subject used desensitizing gel for 20 minutes before at-home bleaching treatment for a couple days, which reduced sensitivity.

CONCLUSIONS

A 14-day at-home treatment was compared with 60 minutes of in-office treatment (two appointments, each with three 10-minute applications). The at-home (10% carbamide peroxide) treatment sides were significantly different from the in-office (35% hydrogen peroxide) treatment sides during all active treatment periods and during follow-up visits according to all three color evaluation methods.

At-home treatment had significantly higher gum sensitivity than in-office treatment during the latter part of the first week of the study. For tooth sensitivity, there were no significant differences between treatments.

Eighty four percent of the subjects reported the at-home treatment to be more efficient and 16% reported no difference in lightness between the treatments. None of the subjects reported the in-office bleaching treatment to be superior to the at-home bleaching treatment.

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Laboratory Research

An *In Vitro* Comparison of Metal and Transparent Matrices Used for Bonded Class II Resin Composite Restorations

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WHM Raab • H Lang

Clinical Relevance

For bonded direct Class II restorations, the use of metal matrices should be favored because excess formation is significantly lower when compared to transparent matrices.

SUMMARY

This study compared excess formation of direct bonded Class II restorations using different matrix systems—metal or transparent. Sixty freshly extracted, non-carious, posterior human teeth were used. In all of the teeth, standardized MOD-cavities were prepared with the gingivo-proximal margins located 1.0-1.5 mm cervical to the cemento-enamel junction. The prepared teeth were randomly assigned to six groups. Half were restored using metal matrices and wooden wedges; the other half were restored using transparent matrices and reflective wedges. Three different material systems were used to fill the cavities: 1) a hybrid composite (Tetric) plus an adhesive bonding agent (Syntac Classic), 2) a flowable composite (Tetric Flow) plus Syntac Classic and 3) a compomer (Dyract AP) together with an adhesive bonding agent designed for compomers (Prime & Bond NT). After the specimens were preserved in saline solution, scanning electron microscopy (SEM) assessed the amount of overhang formation at the restoration margins. The data collected indicated the use of transparent matrices resulted in significantly higher amounts of excess material at the restoration margins compared with metal matrices. Moreover, there was no significant difference between the materials when the same matrix was used. All of the dental restorations examined displayed material overhang. Based on these findings, the authors concluded that the type of matrix exerts a major impact on overhang formation, with metal matrices resulting in significantly less excess material buildup.

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INTRODUCTION

Excess formation is a problem frequently encountered by dental practitioners who restore dental cavities with composite or compomer resin materials. The amount of excess material that builds-up at the restoration margins depends on the materials and the techniques used. Although fluid filling materials can penetrate small

gaps between the matrix and the tooth, increased pressure is required to apply highly viscous materials to the cavity walls if unfilled regions are to be avoided. Once excess material buildup has occurred, is extremely hard to detect and just as difficult to eliminate.

The problem of excess material—one with which every dental practitioner is familiar—has been implicated in a wide range of complications, including secondary caries and periodontal disease (Lang, Kiel & Anderhalden, 1983) and is of great clinical significance.

Transparent matrices and translucent wedges have been introduced to mitigate the consequences of polymerization shrinkage and, thus, the development of microleakage (Ciamponi, Del Portillo Lujan & Ferreira Santos, 1994; Lutz, Krejci & Barbakow, 1992; Lutz & others, 1986; Krejci, Sparr & Lutz, 1987; Koenigsberg, Fuks & Grajower, 1989; Kays, Sneed & Nuckles, 1991). Because such matrices and wedges are, by their nature, highly unstable, it is difficult to adjust them to the natural anatomic shape of teeth. As a result, gaps may occur between the matrix and the tooth, promoting the accumulation of excess material during filling procedures.

This study determined whether transparent or metal matrices result in the formation of greater overhangs when different restorative materials (composites, flowable composites and compomer resins) are used to restore Class II cavities.

METHODS AND MATERIALS

Sixty freshly extracted, non-carious, posterior human teeth (premolars and molars) were used in this study. The extracted teeth were cleaned thoroughly to remove both hard and soft deposits, then stored in saline solution for 3.5 months. Standardized Class II cavities were created in all specimens using a conventional handpiece (Intra Lux 3, KaVo Dental GmbH, Biberach, Germany) with a medium-grain diamond bur (Fig 837XL, ISO 806 314 112 524 014, Hager & Meisinger, Duesseldorf, Germany). All the Class II cavities were 3 mm wide; the gingival margins were 1.0-1.5 mm below the cemento-enamel junction. Each tooth was mounted between two artificial teeth in a stone cast to simulate the geometric configuration of the approximal site.

Table 1: *Experimental Design of the Study*

Group (n=10)	Filling Material	Type of Matrix	Type of Wedge
1	composite	metal	wooden
2	composite	transparent	reflective
3	flowable composite	metal	wooden
4	flowable composite	transparent	reflective
5	compomer	metal	wooden
6	compomer	transparent	reflective

The specimens were randomly assigned to six different groups (n=10) according to the type of restorative material and matrix used (Table 1). The material combinations included 1) a hybrid resin composite (Tetric) together with an adhesive bonding agent (Syntac Classic, Ivoclar Vivadent, Schaan, Lichtenstein), 2) a flowable resin composite (Tetric Flow) plus Syntac Classic and 3) a compomer resin (Dyract AP) together with a different bonding agent (Prime & Bond NT, Dentsply DeTrey, Konstanz, Germany). In half of the restorations, metal matrices (Hawe-Tofflemire, No 1002, Hawe/Neos Dental, Bioggio, Switzerland) were used in combination with wooden wedges (No 830, Hawe/Neos). In the other half, transparent matrices (Hawe-Lucifix Molarbands, No 778, Hawe/Neos) were used with translucent wedges (Hawe-Luciwedge medium, No 790 S, Hawe/Neos).

All cavity surfaces were etched with phosphoric acid gel (Conditioner 36, Dentsply, Konstanz, Germany). Prior to etching, all surfaces were dried with oil-free compressed air. All specimens were then etched with the 36wt% orthophosphoric acid gel for 30 seconds, rinsed with water for 30 seconds and dried with oil-free compressed air for five seconds. Syntac Primer was then applied to the cavity with a fine brush for 15 seconds. Gentle air drying was repeated. An adhesive bonding agent (Syntac, Vivadent, Lichtenstein) was applied to the surface for 10 seconds. Air drying was repeated again. Finally, a thin layer of bonding agent (Helio-Bond, Vivadent, Lichtenstein) was applied. The bond was polymerized for 10 seconds. All materials were used according to the manufacturer's instructions. All specimens were treated with a standard composite/compomer resin curing light (Spectrum TM 800, Model No 703, Dentsply DeTrey, Konstanz, Germany). Metal matrices with wooden wedges were applied to half of the teeth; the balance were restored using transparent matrices and translucent wedges. Matrix fit was checked with a magnifying glass (KF, Zeiss, Jena, Germany; magnification: 3.2). Afterwards, the resin composite (Tetric) was applied in horizontal increments with a thickness of about 1.0-1.5 mm each. When transparent matrices were used, the first layer was cured initially through the translucent wedge and later from occlusal (in each case for 20 seconds). When metal matrices were used, curing was carried out only from occlusal for 40 seconds. All subsequent layers were light cured for 20 seconds. The composite material was then light cured for 60 seconds.

In conformance with the study design, 20 Class II cavities were filled with Tetric Flow. Half of the teeth were restored using a metal

matrix and the other half were restored using a transparent matrix.

Similar bonding techniques were used for the compomer and resin composite specimens, respectively. The bonding agent (Prime&Bond NT, Dentsply DeTrey, Konstanz, Germany) was applied for 20 seconds, then air dried for five seconds. If the surface did not exhibit a uniform glossy appearance after applying the bonding agent, air drying was repeated. The adhesive bonding agent was then light cured for 10 seconds and the compomer material (Dyract AP) was then applied.

The teeth were examined by SEM with x200 magnification (DSM 950, Zeiss, Jena, Germany). The total length of the restoration margins [mm] and the length of the margins exhibiting excess material [mm] were measured. The authors determined the percentage of margin that exhibited excess material. Mean values and standard deviations were calculated; the intergroup differences were analyzed by the non-parametric Mann-Whitney U test. The authors presumed a statistically significant difference of $p \leq 0.05$. Statistical analysis was carried out with SPSS for Windows (V 8.0, SPSS Inc, Chicago, IL 60606, USA).

RESULTS

Figure 1 demonstrates the amount of overhang formation in each group. Among the dental restorations made with hybrid resin composites, a high-significant difference (p=0.001) was noted between the group with metal matrix (42.0 ± 13.2%) and transparent matrix (75.1 ± 17.4%).

Moreover, a greater degree of overhang formation (p=0.015) was noted at the margins of flowable composite restorations using transparent matrix (53.3 ± 18.7%) than at the margins of similar restorations using a metal matrix (31.9 ± 20.2%).

Using transparent matrices together with compomer resins (61.4 ± 15.5%) also resulted in a significantly higher (p=0.003) buildup of excess material than did the combination of metal matrices and compomers (36.4 ± 14.0%).

In addition, the effects of using both metal and transparent matrices were compared for the different restoration materials. There was no significant difference between hybrid composites, flowable composites and compomer resin materials. Nevertheless, overhang formation was detected at the margins of every restoration examined.

DISCUSSION

In recent years, transparent matrices have been introduced for restoring dental cavities. The advantages of this technique have been investigated in dental research (Rovatti, Cavalleri & Dallari, 1998). This study has shown that, in Class II cavities, resin composite materials can be cured from the proximal surface. This brings about the degree of polymerization

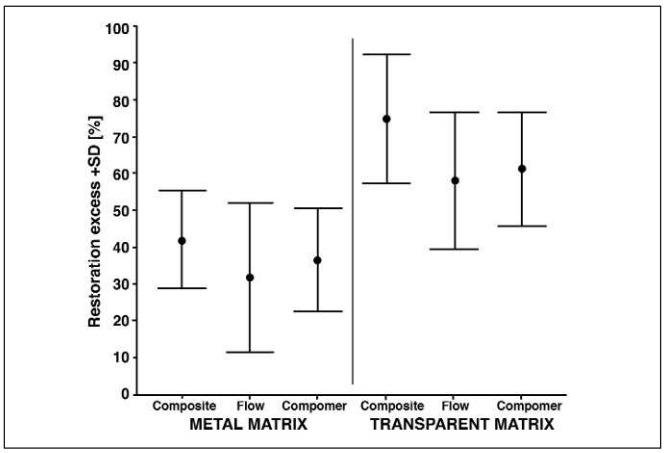

Figure 1. *Excess material buildup at the margins of dental restorations (Means ± SD for each group are shown).*

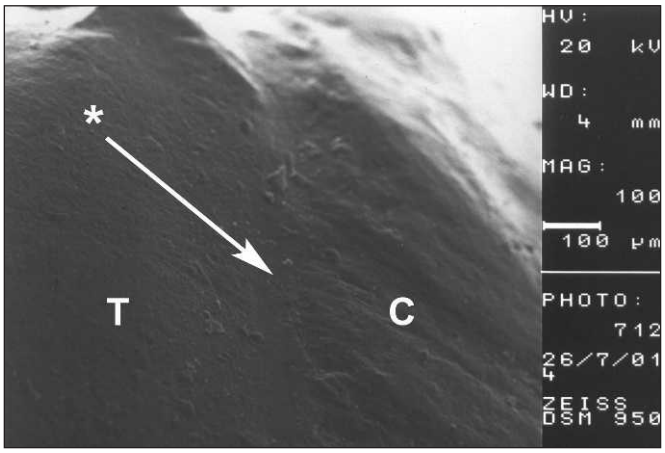

Figure 2. *Cavity margin without excess material (marked with *, T=Tooth, C=Composite resin).*

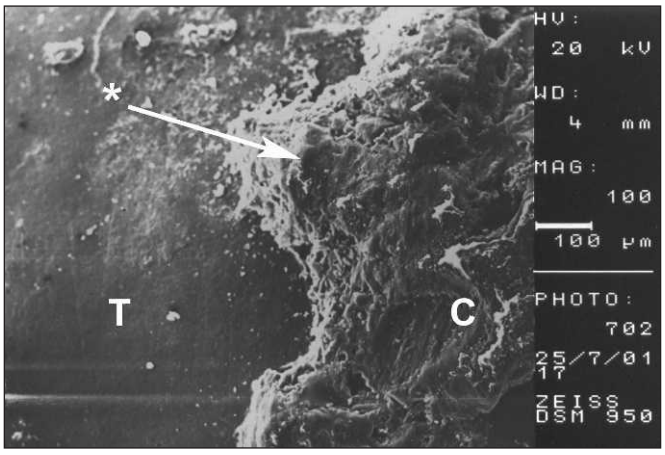

Figure 3. *Cavity margin with excess material (marked with *, T=Tooth, C=Composite resin).*

required for stable restorations. Kays and others (1991), Scherer and others (1989) and Dannheimer and others (1996) have concluded that using a clear plastic matrix with the light positioned at the gingival surface results in greater hardness in both the gingival and occlusal portions of the restoration. Asmussen and Peutzfeldt (1999) have found that the direction of shrinkage is directed towards the light source. When the filling is cured from occlusal, therefore, it shrinks away from the adhesive zone where the bonding of the restoration material occurs at the same time. It can be concluded that under these circumstances damage could occur to the adhesive bond. When the filling is cured from proximal, however, this danger is eliminated and microleakage is reduced (Ciamponi & others, 1994). Cvitko, Denehy and Boyer (1992) have observed that microleakage takes place as a consequence of polymerization shrinkage. Neiva and others (1998) have suggested that polymerization shrinkage may be one of the major factors directly responsible for marginal leakage at the tooth-restoration interface and may also influence the longevity of posterior resin restorations. Lutz and others (1986; 1992) have stated that the use of a transparent matrix and reflective wedges together with interproximal curing is indispensable for achieving sufficient polymerization and less shrinkage.

However, there are several disadvantages to using transparent matrices. With respect to adaptability, metal matrices are superior in that they can be better pre-contoured and firmly applied to the tooth surface. Neiva and others (1998) claim that this is one of the advantages of metal matrices. From an anatomical viewpoint, the metal matrix holds the proximal contour better than its transparent counterpart. Furthermore, transparent matrices are used with reflective wedges. These are very stiff and lack the ability of wooden wedges to adapt themselves to the natural anatomic tooth contour. As a result, reflective wedges can make contact to the matrix placed on the tooth at only one point. This permits the development of large gaps between the matrix and the tooth at the critical cervical cavity margin and generates substantial overhang formation during filling procedures. It is obvious that a buildup of tooth-colored restoration material at the approximal part will be difficult to detect. Even if correctly identified, this excess material is very troublesome to remove and, thus, poses a formidable clinical challenge.

This study determined whether transparent matrices, combined with reflective wedges, really generate significant overhang formation and whether there are differences between composite, flowable composite and compomer resins. In this study, the authors found a significantly higher buildup of excess material when transparent matrices were employed. This observation applies to the materials used. In addition, the authors compared the various materials with each other using the same matrix. These results revealed no significant difference.

Regarding these results, we need to reevaluate how overhang formation could be decreased. One possibility is to use condensable resin composites that do not readily penetrate gaps between the tooth and the matrix. Frankenberger and others (1999) have shown, however, that increased viscosity in resin composites can lead to a higher percentage of underfilled beveled margins. Furthermore, Peumans and others (2001) discovered that condensable resin composites do not achieve better proximal contact. Another possibility is to use magnifying glasses with a 2.5–3.2 magnification factor, as was done in this study. Using such magnifying glasses, Frankenberger and others (1999) reduced the percentage of excess material buildup by as much as 40%. Magnifying devices are even more effective when used for the entire filling procedure, that is, during application of the matrix, the actual restoration work and the removal of excess material. It is obvious that this requires greater handling efforts and a larger investment of time by the clinician.

CONCLUSIONS

Using metal matrices with wood wedges (instead of transparent matrices and reflective wedges) when restoring dental cavities with bonded Class II resin composite restorations may significantly reduce overhang formation. Dental practitioners should take this result into account when weighing the advantages and disadvantages of both types of matrices.

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Marginal Adaptation, Retention and Fracture Resistance of Adhesive Composite Restorations on Devital Teeth With and Without Posts

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Clinical Relevance

Using minimally invasive adhesive techniques to restore devital teeth is a promising alternative to conventional treatment modalities.

SUMMARY

This *in vitro* study generated data on the quality of marginal adaptation, fracture resistance and retention of several indirect adhesive composite configurations on root-treated premolars before and after a long-term fatigue test and compared these results to a control group of adhesive onlays on "vital" teeth.

Six root-treated extracted human premolars per group, with four different restorative configurations with and without adhesive fiber posts, were evaluated. Another group of six premolars, "revitalized" by using diluted horse serum to simulate pulpal fluid and restored with adhesive composite onlays, served as the control. Marginal adaptation before and after long-term occlusal loading (1,200,000 occlusal loading cycles at max 49 N) was assessed by using the replica technique and quantitative evaluation in SEM at 200x magnification. The number of lost restorations was recorded after loading. Fracture resistance and fracture patterns were evaluated by using a universal-testing machine on the fatigued samples.

No significant differences ($p>0.05$) between groups were detected before and after loading for the percentage of "continuous margin" at the total marginal length. Loading had a significant ($p<0.05$) effect on the percentage of "continuous margin" for the total marginal length of two groups only. No significant difference ($p>0.05$) for fracture resistance was detected and no lost restorations were observed.

The results suggest that for both the less decayed and the more significantly decayed devital teeth, the minimally invasive adhesive restorative approach is promising.

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INTRODUCTION

Restoring devital teeth represents a major challenge for the practitioner, because it requires profound knowledge in endodontics, periodontics and restorative therapy (Morgano & others, 1994). The situation is further complicated by the fact that clinical concepts regarding the restoration of devital teeth have often been based on empirical philosophies due to the lack of sound scientific data (Cariso & others, 1987; Morgano & others, 1994; Robbins, 2001). Among the three factors mentioned above, restorative therapy for endodontically treated teeth may be the most important. Nonrestored, endodontically treated teeth are prone to fracture and coronal leakage, leading to bacterial contamination (Torabinejad, Ung & Kettering, 1990; Ray & Trope, 1995; Ricucci, Grondahl & Bergenholtz, 2000; Tronstad & others, 2000). In restored, endodontically treated teeth, catastrophic failures are mainly induced by failed restorations—in most cases, crown fractures due to secondary caries—leading to extraction. Tooth loss due to periodontal reasons or failed endodontics are relatively scarce (Vire, 1991; Fuss, Lustig & Tamse, 1999). Poor endodontic therapy leads to failure irrespective of the quality of restorative treatment. However, success of a well done endodontic treatment is significantly increased by a good quality coronal restoration (Bishop & Briggs, 1995; Tronstad & others, 2000).

Traditional restorative therapy of devital teeth involves a combination of root canal filling, conventionally cemented prefabricated or custom made metallic post with a metallic core and coverage with a conventionally cemented full crown (Colman, 1979). Under the conditions of proper planning and meticulous clinical work, this restorative complex may serve for decades (Nanayakkara, McDonald & Setchell, 1999). However, the traditional method of restoring devital teeth comprises several drawbacks and risks that have given rise to serious criticism (Stockton, Lavelle & Suzuki, 1998). One drawback is the considerable treatment time spent on such complex restorations, making them extremely costly (Shugars & others, 1997). Another drawback is the considerable amount of sound tooth structure that often has to be sacrificed (Sornkul & Stannard, 1992). In particular, the risks are root perforations and root fractures due to placing radicular posts and the decementation of posts (Nanayakkara & others, 1999; Fuss & others, 2001; Gher & others, 1987; Alhadainy, 1994; Fuss & Trope 1996).

In the 1980s, unfilled resins were proposed as luting agents for metallic posts to increase retention (Goldman & others, 1984). However, the true breakthrough in the field of modern restoration of endodontically treated teeth was the introduction of the adhesive technique, especially propelled by the development of efficient dentinal adhesives (Van Meerbeek & others, 2001). Because the retention of adhesive restorations is mainly based on adhesion and does not require macroretentive elements (Tjan, Munoz-Viveros & Valencia-Rave, 1997), minimally invasive preparations with maximal conservation of dental tissues can be realized (Robbins, 1990; Tjan & others, 1997; Bindl & Mörmann, 1999). In addition, the insertion of radicular posts often becomes obsolete.

Although the trend towards minimally invasive restorations is overwhelming, even in modern adhesive concepts, full coverage crowns with post and cores are recommended for restoring seriously damaged teeth and cuspal coverages for minimally damaged posterior devital teeth are deemed necessary (Smith & Schuman, 1997). However, in view of the new possibilities given by adhesive techniques, the question arises whether these guidelines are still justified.

Therefore, this study generated data on the quality of marginal adaptation, fracture resistance and the retention of different types of indirect adhesive restorations on devital teeth before and after a long-term fatigue test. These results were to be compared to a control group of adhesive composite onlays on "vital teeth." The working hypothesis was that marginal adaptation before and after loading would not be significantly different between the control and experimental groups and there would be no significant difference in respect to fracture resistance among the different groups and no lost restorations.

METHODS AND MATERIALS

Specimen Preparation

Thirty caries-free extracted human premolars with completed apexification, stored in 0.5% thymol solution for at least three months at 4°C until initiation of the experiment, were used for this study. They were randomly divided into five equal groups.

The root length of each tooth was adjusted to fit into the chamber of the mechanical loading device (Department of Cariology, Endodontics & Pedodontics; Laboratory of Electronics of the Faculty of Medicine; University of Geneva) (Figure 1). After sealing the apex of each tooth with a filled light-curing dentinal adhesive (Optibond FL, Kerr Corp, Orange, CA 92867, USA), all the specimens were fixed with light-curing composite (Herculite XRV, Kerr) on custom-made metallic holders (Provac; FL-9496 Balzers, Liechtenstein) and their root bases were further stabilized with self-curing acrylic resin (Technovit 4071, Heraeus-Kulzer GmbH, D-61273 Wehrheim, Germany). In the reference group that simulated vital teeth, a metallic tube was inserted into the pulp chamber in the upper third of the root and sealed with a filled light curing dentinal adhesive

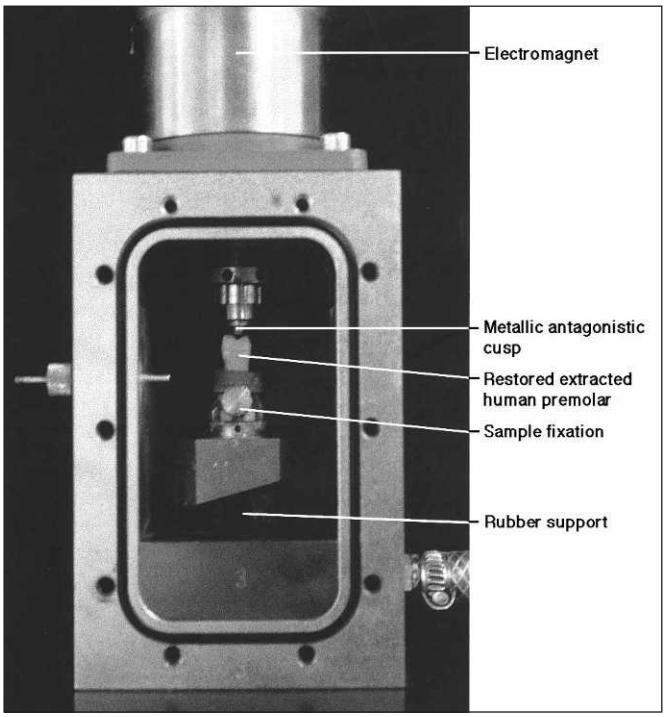

Figure 1. *One out of the six chambers of the custom made device was used for occlusal loading of the samples.*

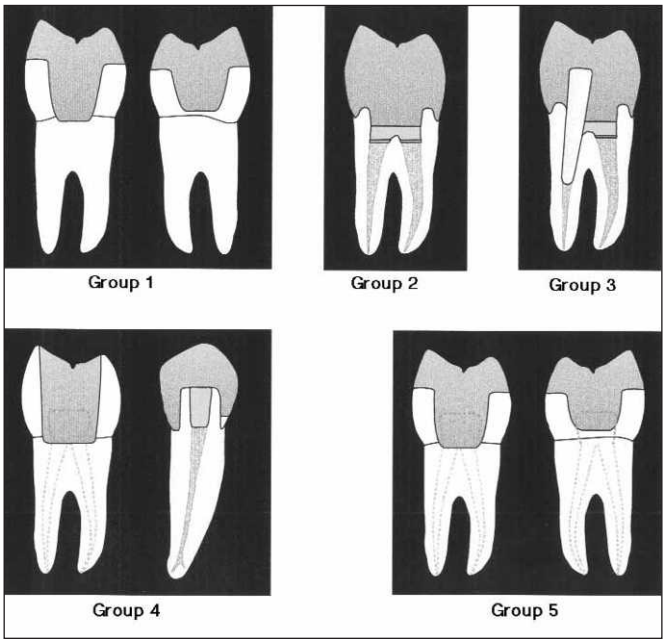

Figure 2. *Schematic representation of the experimental groups.*

(Optibond FL). This tube was connected by a flexible silicone hose to an infusion bottle placed 34 cm vertically above the test tooth. The infusion bottle was filled with horse serum diluted in a 1:3 ratio with 0.9% NaCl (Pashley & others, 1981) to simulate the dentinal fluid under normal hydrostatic pressure of about 25 mm Hg (Tao & Pashley, 1989). Twenty-four hours before starting the cavity preparation, using a three-way valve, the pulp chambers were evacuated with a vacuum pump and, subsequently, bubble-free filled with the diluted horse serum. At that moment, the intrapulpal pressure was maintained at 25 mm Hg throughout testing, that is, during cavity preparation, restoration placement, finishing and stressing.

Group 1 simulated the restoration of vital teeth. Class II cavities (MOD) were first prepared by using coarse diamond coated burs (Universal Prep Set, Intensiv SA, CH-6962 Viganello, Switzerland) in a high speed handpiece (Intramatic Lux 2 24LN, KaVo, D-88400 Biberach, Germany) under profuse water spray cooling with proximal margins located 1.0 mm below (mesial) and 1.0 mm above (distal) the cementum-enamel junction. The standardized dimensions of the tapered preparations were 4.0 mm in width and 1.5 mm in depth at the bottom of the proximal box, and 4.0 mm in width and 3.5 mm in depth for the occlusal isthmus, all walls having 10° of divergence against the occlusal plane. Subsequently, 2.0 mm of the lingual and the buccal cusp were reduced, thus, creating an onlay cavity. All internal cavity surfaces and cavity margins were finished under the stereo microscope (MZ6, Leica, D-6330 Wetzlar, Germany) at a 15x magnification.

In Group 2, endodontically treated non-vital teeth with completely destroyed clinical crown were simulated. The root canal preparations were performed using NiTi rotary instruments (Hero 642, MicroMega, F-25000 Besançon, France) in a low speed handpiece (Intramatic Lux 29LN, KaVo, D-88400 Biberach, Germany) under intermittent rinsing with 5% NaOCl. An epoxy sealer (AH Plus, Batch 0102000063, Dentsply Caulk, Milford, DE 19963, USA) and the vertical guttapercha condensation technique (Obtura II, Obtura Corp, Fenton, MO 63026, USA) were used as the canal obturation system. An 0.5 mm layer of glass-ionomer cement (KetacBond, 3M-ESPE, St Paul, MN 55144, USA) was applied on top of the filled root canal to facilitate future re-entry. The clinical crown was completely removed and the remaining tooth prepared as follows: A central inlay 2 mm in depth was cut into the pulp chamber and a chamfer 1.0 mm width and 1.5 mm in height was prepared around the entire tooth periphery, 1.0 mm below the cemento-enamel junction.

In Group 3, the same preparation was used as for Group 2. However, instead of using a glass-ionomer layer on top of the root filling, the root canal was prepared to fit an adhesive post to a length of 7.5 mm.

Group 4 represented devital teeth with inlay restorations. The dimensions of the preparations corresponded to those of Group 1 but without occlusal reduction of the cavity walls, and the root canal treatment was

conducted according to the procedure described in Group 2.

Group 5 corresponded to Group 4, but both cusps were reduced by 2.0 mm, creating a devital onlay situation (Figure 2).

Restorative Procedures

A composite (Targis, Batch No enamel 10563 and dentin 13330, Ivoclar-Vivadent AG, FL-9494 Schaan, Liechtenstein), a dual-cured luting composite (Variolink II, Batch No base 14589 and catalyst 15619, Ivoclar-Vivadent AG, FL-9494), an organic silane (Monobond S, Batch No 07717, Ivoclar-Vivadent AG, FL-9494) and a multi-functional adhesive (Syntac Classic, Batch No Primer 05853, Adhesive 05896, Helibond 05896, Ivoclar-Vivadent AG) were used for all groups.

Except for Group 3, the adhesive system was applied and light-cured (Optilux 501, Demetron/Kerr Corp, Danbury, CT 06810, USA), and relative intensity was measured with the Curing Radiometer Model 100 (Demetron/Kerr Corp) > 1000 mW/cm^2) for 60 seconds on the cavity surfaces according to the manufacturer's instructions in order to seal the cavity. Thereafter, cavity margins were finished with a fine diamond bur (Geneva Prep Set, Batch S9901, Intensiv SA, CH-6962 Viganello, Switzerland) according to the principles of the selective bonding technique (Krejci & Stavridakis, 2000), where complete adhesion is confined to the cavity margins only.

In Group 3, fiber-reinforced composite posts (Vectrispost, Size S, Batch No ZZ9265, Ivoclar-Vivadent AG, Schaan, FL-9494) were inserted into the root canals, and the preparations for all groups were then replicated by using a polyvinylsiloxane material (Aquasil light and heavy, Dentsply Caulk) in custom made trays. All impressions were poured with hard stone (Fujirock; Fuji, GC Europe NV, B-3000 Leuven, Belgium), resulting in individual stone dies. Two thin layers of a water soluble glycerine gel (Model separator, Batch No A12029, Ivoclar-Vivadent AG, FL-9494) were applied on each die as a separating medium, and indirect composite restorations were fabricated on these dies by using an incremental technique. Each increment was light cured for 20 seconds (Optilux 501). The finished workpieces were coated with glycerine and subjected to a light and heat post-curing process (Programm P1, Targis Power, Ivoclar-Vivadent AG, FL-9494). The bond between the composite workpieces and the posts in Group 3 was assured by sandblasting the surface of the post with 50 microns Al_2O_3 at 2 bar pressure, silanization (Monobond S, Batch No 07717, Ivoclar-Vivadent AG, FL-9494) and applying a bonding agent (Heliobond, Batch No 05896, Ivoclar-Vivadent AG).

The workpieces were adhesively luted. For this purpose, the enamel margins were acid etched for 30 seconds using 37% phosphoric acid (Total Etch, Batch No 30606, Ivoclar-Vivadent AG) (Munechika & others, 1984). After rinsing and drying, Primer, Adhesive and Heliobond were applied in a thin layer on enamel and dentin according to the manufacturer's instructions and Heliobond was pre-cured for 60 seconds (Optilux 501). The internal surfaces of the composite workpieces were sandblasted with Al_2O_3 at a pressure of 2 bar, silanized (Monobond S) and coated with bonding resin (Heliobond). Variolink II dual cured composite served as the luting agent that was light-cured (Optilux 501) from the oral, facial and occlusal direction, each mesially and distally, for 60 seconds.

Finishing and polishing was performed immediately after polymerization of the luting composite by using fine diamonds (Geneva Prep Set) and finishing discs with descending abrasives (Sof-Lex, 3M-ESPE).

Mechanical Loading

The stress test was initiated after seven days of storage in water at 37°C in the dark. All specimens were submitted to 1,200,000 cycles with maximum 49N loading force by using artificial cusps made of stainless steel with a hardness similar to natural enamel (Vicker's hardnesses: enamel = 320-325; steel = 315); the diameter of the cusps was 4 mm and they contacted the occlusal surface of the restoration about 1.5 mm out of the central fossa. The axial force was exerted at a frequency of 1.5 Hz, following a half-sinus curve. By having the specimen holders mounted on a rubber disc, a sliding movement of the tooth was produced between the first contact on an inclined plane and the central fossa (Krejci & others, 1990; Dietschi & others, 1995). These conditions are believed to simulate approximately five years of clinical service (Krejci & Lutz, 1990).

Marginal Adaptation

Before and after the stress test, gold sputtered (SCD 030, Provac, FL-9496 Balzers, Liechtenstein) epoxy resin replicas (Epofix, Stuers, D-2610 Rodovre, Denmark) of all samples were fabricated by using polyvinylsiloxane impressions (President light body, Coltène-Whaledent AG, CH-9450 Altstätten, Switzerland). They were subjected to the quantitative evaluation of marginal adaptation at a standard 200x magnification in a SEM (XL20, Philips, NL-5600 Eindhoven, Netherlands) by using a custom made module programmed within image processing software (Scion Image, Scion Corp, Frederik, MA 21703, USA). The following criteria were applied and reported as percentages relative to the entire marginal length: "Continuous margin," "marginal opening," "marginal tooth fracture," "marginal restoration fracture," "overhangs" and "underfilled margins." The data were sub-

mitted to parametric statistical analysis by using ANOVA and Sheffe's F test at a 95% level of significance.

Retention

Lost restorations were recorded after completing the load cycle and their number was reported per group.

Fracture Resistance

The fracture resistance test was performed using a universal testing machine (Instron Model 1114, Instron Corp, High Wycombe, HP 12 357, Great Britain) on the fatigued samples. After embedding the teeth up to 2 mm beneath the CEJ in cold curing resin (Epofix), a spherical headpiece 5.0 mm in diameter was used to apply axial compression force in the middle of the occlusal surface of the samples (Figure 3). The crosshead speed was 1.0 mm/minute and compression force was applied until the specimen fractured.

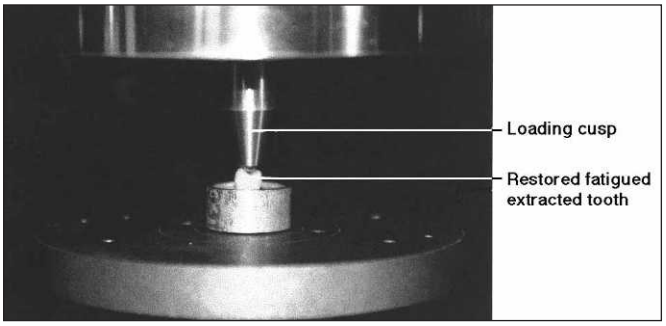

Figure 3. *This figure represents the fracture strength test design.*

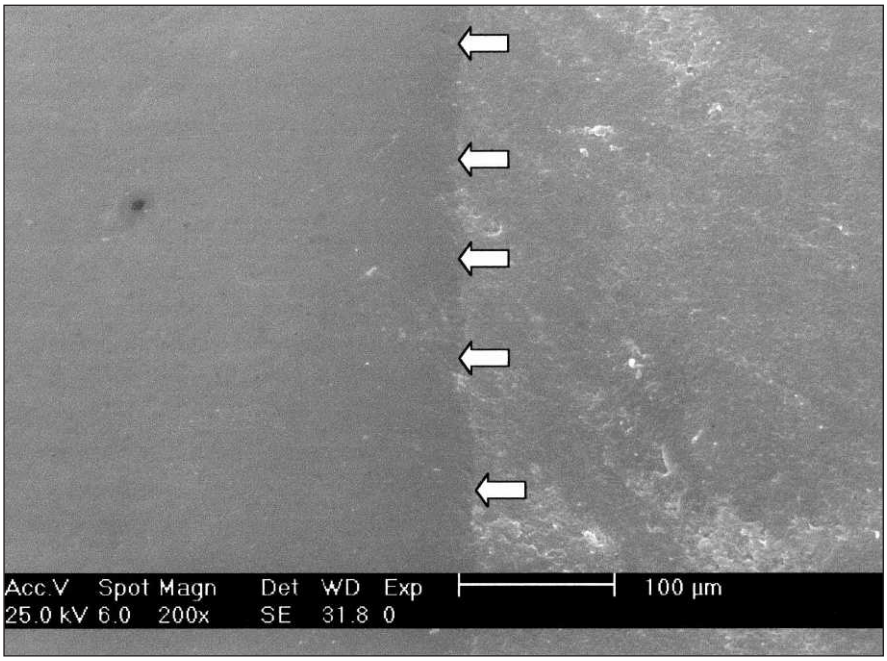

Figure 4. *The continuous margin (flashes) at the axial wall of an onlay of Group 4 after loading. Left: enamel; right: restoration (SEM, original magnification 200x).*

The data of the fracture resistance evaluation were submitted to ANOVA and Student-Newman-Keuls test.

RESULTS

All restorations were in place after completing the stress test, meaning that retention amounted to 100% for all groups.

The results of the marginal adaptation at the interface between the tooth and luting composite are represented in Tables 1 through 3. No significant differences ($p>0.05$) between groups were detected before and after loading for the percentage of "continuous margin" (Figure 4) at the total marginal length (Table 1). The same was true for dentinal margins prior to loading (Table 2). After loading, however, significant ($p<0.05$) differences were found between Groups 3 and 4 and Groups 3 and 5 at dentinal margins, with Groups 4 and 5 showing the best marginal adaptation.

Loading had a significant ($p<0.05$) effect on the percentage of "continuous margin" for the total marginal length of Groups 2 and 4. However, only Group 2 showed a significant difference due to loading at the dentinal margins.

The predominant marginal defect in all groups was the pure marginal opening (Figure 5 and 6). However, several groups also showed some "marginal enamel fractures" (Figures 7 and 8), with a significant ($p>0.05$) increase after loading for Group 5 (Table 3).

No more than 3% of the "marginal restoration fractures," "overhangs" and "underfilled margins" were found before and after loading, with no significant differences among the groups.

The marginal adaptation at the interface between luting composite and workpiece was perfect in all groups, both before and after loading.

Very inhomogenous fracture patterns with no preferential fracture behavior were observed in all groups after the fracture resistance test. However, most fractures followed an almost axial direction through the restoration and radicular dentin. In addition, vast standard deviations were present in the quantitative measurements, so that despite rather large differences between the mean fracture force values, no statistical significance could be detected (Table 4).

DISCUSSION

The experimental groups in this study consisted of extracted human premolars because they represent a more severe situation than molars due to longer clin-

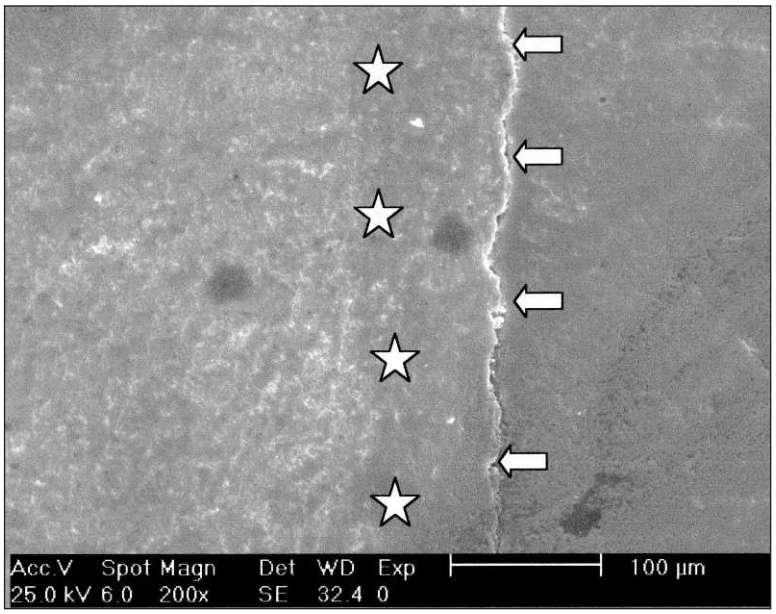

Figure 5. *Marginal gap (flashes) at the interface between enamel (right) and luting composite (middle) and the excellent adaptation (stars) between luting composite (middle) and composite restoration (left) at the axial wall of an onlay of Group 5 after loading (SEM, Original magnification 200x).*

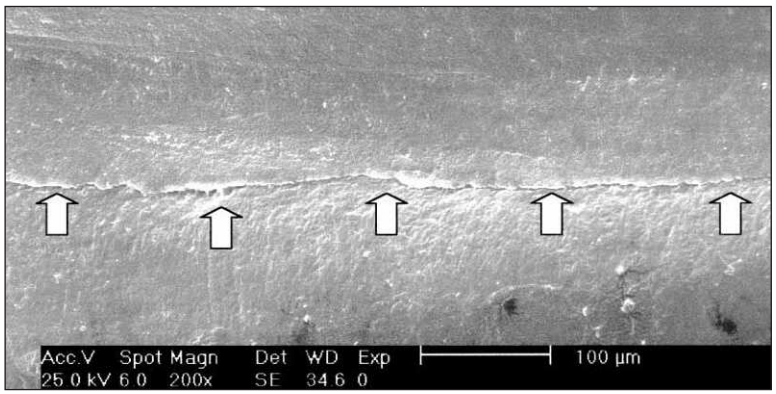

Figure 6. *Marginal gap (flashes) at the cervical margin between dentin and luting composite in Group 1 after loading (SEM, Original magnification 200x).*

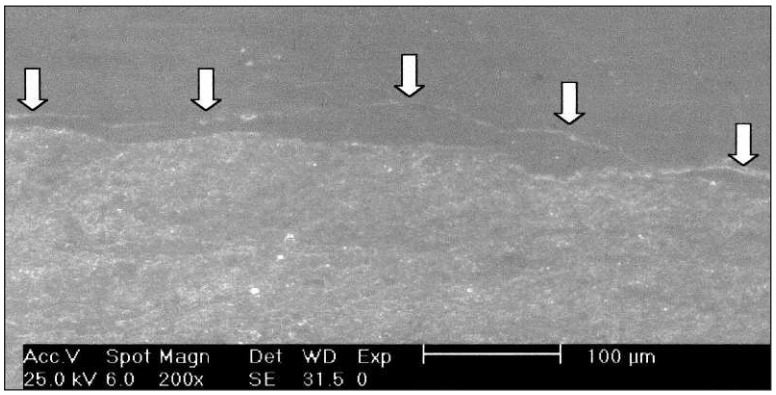

Figure 7. *Marginal enamel fracture (flashes) at the occlusal margin of Group 3 after loading. On the upper part of the photo: enamel; in the lower part: luting composite (SEM, Original magnification 200x).*

ical crowns and less dentinal surface for bonding (Robbins, 2001). In this way, an extreme clinical situation was simulated.

A consensus can be found in the newer literature that posts do not strengthen devital teeth (Assif & Gorfil, 1994; Stockton & others, 1998; Baratieri & others, 2000). However, they are still considered necessary for the retention of the restoration, especially in the case of severely damaged teeth (Zalkind & Hochman, 1998). Although this study might have some limitations in respect to its clinical relevance, the direct comparison between Groups 2 and 3 suggest that radicular posts have no relevant influence on retention if used in an adhesive restorative design. In these two groups with missing clinical crowns, the restorations remained in place with and without posts, and there was no significant difference between their marginal adaptation before and after loading. In addition, no significant differences in respect to fracture strength and fracture patterns were recorded. These results indicate that the posts might need re-evaluation. It is obvious that with conventional, non-adhesive restorations, such as amalgam or gold, posts increase retention in a relevant way (Standlee, Caputo & Hanson, 1978). However, this effect may become far less important where adhesive restorations are concerned. The crucial factor here may be the direction of the load. In this experiment, the axial forces were applied to the center of the crown, thus, simulating a normal occlusal situation on a premolar. Since it is well known that fracture resistance depends on the angle of applied load (Kern, von Fraunhofer & Mueninghoff, 1984; Christian & others, 1981; Plasmans & others, 1986) and axial forces are less detrimental than oblique forces (Loney, Moulding & Ritsco, 1995), future work needs to determine whether shear forces would change the outcome of the study. Another limitation of this study may be that occlusal loading was applied without simultaneous thermal cycling. It has been suggested that thermal cycling may further stress and weaken the adhesive bond, thus, decreasing fracture strength and increasing microleakage (Eakle, 1986).

Cuspal coverage is thought necessary for the conventional restoration of devital posterior teeth to avoid cusp fractures (Sorensen & Martinoff, 1984; Smith & Schuman, 1997). Comparing the results of Groups 4 and 5 suggest that this recommendation might be modified for adhesive restorations in the future. No cuspal fractures after the load test were seen in Group 4 with large inlay restorations on devital teeth, even though the buccal and lingual walls were very thin.

Marginal adaptation before and after loading and fracture strengths were not significantly different between Groups 4 and 5. In addition, the fracture patterns were never localized at the restoration/cusp interface and the amount of marginal enamel fractures was even lower in Group 4 than in Group 5. The adhesively restored teeth were apparently sufficiently strengthened to withstand the extensive occlusal loading applied during this experiment (Morin, Delong & Douglas, 1984). However, the same limitations for the interpretation of these results are true for Groups 2 and 3 because this study simulated axial loads only; the situation may be different with shear forces, especially if directly applied to the cusps (Uyehara, Davis & Overton, 1999) and, therefore, has to be evaluated in future experiments. In addition, the influence of thermal changes will have to be determined.

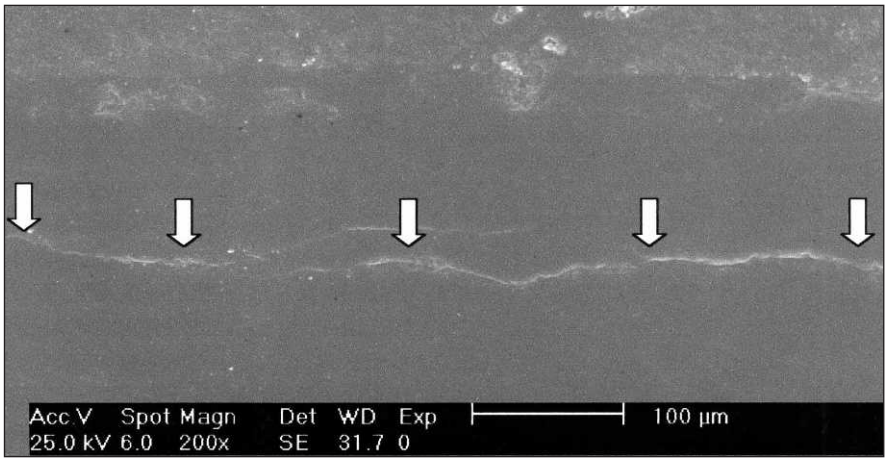

Figure 8. *Marginal enamel (flashes) at the cervical margin located in enamel of Group 5 after loading are featured. The upper part of the image is luting composite; the lower part is enamel.*

No significant difference in retention, marginal adaptation and fracture strength was seen between the "vital" (Group 1) and "devital" (Group 5) onlays. This agrees with a study where the presence of an endodontic access did not change the fracture strength of a tooth (Steele & Johnson, 1999). For the adhesive system used, it also agrees with another study where the simulation of dentinal fluid had no influence on the marginal adaptation of the adhesive system used in this study in enamel and dentin (Krejci, Kuster & Lutz, 1993). Dentinal adhesion was very successful in this study because the values of "continuous margin" in dentin in groups with dentin and enamel margin (Table 2) were similar to the values for the total marginal length (Table 1). In addition, the two groups with margins completely located in dentin (Groups 2 and 3) were not significantly different from the other groups.

Table 1: *Percentages of "Continuous Margin" for the Total Marginal Length Before and After Loading (Means ± SD)*

	Group 1	Group 2*	Group 3	Group 4*	Group 5
Before Loading	91.5 ± 2.8	94.9 ± 5.2	82.1 ± 11.2	91.3 ± 5.6	89.8 ± 8.0
After Loading	85.4 ± 11.9	82.5 ± 7.6	75.9 ± 17.9	72.6 ± 13.7	72.9 ± 6.3

p<0.05 before/after loading*

Table 2: *Percentages of "Continuous Margin" for the Dentinal Margins Only Before and After Loading (Means ± SD)*

	Group 1	Group 2*	Group 3	Group 4	Group 5
Before Loading	94.8 ± 8.8	94.9 ± 5.2	82.1 ± 11.2	100.0 ± 0.0	95.8 ± 5.2
After Loading	83.7 ± 22.7	82.4 ± 7.5	75.9 ± 17.9	88.2 ± 13.1	93.9 ± 6.5

p<0.05 before/after loading*

Table 3: *Percentages of "Marginal Enamel Fracture" for the Total Marginal Length Before and After Loading (Means ± SD)*

	Group 1	Group 2**	Group 3**	Group 4	Group 5*
Before Loading	4.0 ± 3.6	-	-	4.6 ± 4.1	3.7 ± 4.5
After Loading	5.6 ± 4.7	-	-	3.2 ± 3.1	12.3 ± 8.1

p<0.05 before/after loading*
***No enamel margins present*

Table 4: *Fracture Strength (Means ± SD)*

	Group 1	Group 2	Group 3	Group 4	Group 5
Load in kg	109.7 ± 32.5	88.5 ± 57.4	83.5 ± 20.3	82.3 ± 38.4	74.1 ± 30.7

The interface between composite and luting composite was excellent before and after loading. This confirms the results of an earlier study where sandblasting, silanization and applying a bonding agent resulted in a very good bond between a composite workpiece and a luting composite (Göhring, Peters & Lutz, 2001).

Substantial variations in fracture strength measurements make the interpretation of these results difficult. However, the inconsistent results agree with the literature and are probably due to natural variations in tooth morphology (Steele & Johnson, 1999). Though no

significant differences between the groups were detected, a certain trend was observed in the sense that the control, consisting of "vital" teeth (Group 1) was somewhat stronger than all the variations of the non-vital tooth restoration. It was also interesting to note that the fracture pattern of Group 3 did not differ from the fracture pattern of Group 2. This shows that the presence of a fiber-reinforced composite post had no relevant influence on the distribution of the axial forces.

CONCLUSIONS

Under the limitations of the experimental set-up, several conclusions may be drawn from this study: If normal occlusion is present, adhesive inlay restorations should be considered as the restorative treatment of choice for devital teeth without the need for posts and cuspal coverage. Posts may not be necessary for the restoration of largely destroyed teeth where the clinical crown is fully missing. The marginal adaptation in dentin, even after extensive occlusal loading, was similar to the marginal adaptation in enamel. This shows that dentinal adhesion may be as reliable as enamel adhesion, even under the influence of simulated dentinal pressure. No relevant difference was present between a "vital" and a "devital" restored tooth in respect to retention, marginal adaptation and fracture strength, showing that devital teeth may be treated in the same way as vital teeth. However, the conclusions drawn out of an *in vitro* study need to be backed up with controlled clinical trials before they can be used as recommendations for routine clinical work.

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Analysis of the Enamel/Adhesive Resin Interface with Laser Raman Microscopy

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Clinical Relevance

The depth and pattern of resin monomer penetration into etched enamel is different among the bonding systems employed.

SUMMARY

Adhesion of resin composites into enamel is currently believed to rely on infiltration of bonding resin into the porous zone, establishing micromechanical retention to etched enamel. This study investigated the change in chemical composition of the enamel/resin interface using a laser Raman microscopic system (System-2000, Renishaw). Two-step bonding systems, Mac Bond II (Tokuyama Corp), Clearfil Mega Bond and Single Bond (3M/ESPE) were employed. Resin composites were bonded to bovine enamel with bonding systems and sectioned through the bonded interface. The sectioned surfaces were then polished with diamond pastes down to 1.0 µm particle size. Raman spectra were successively recorded along a line perpendicular to the enamel/resin interface. The sample stage was moved in 0.2 µm increments on a computer-controlled X-Y precision table. Additional spectra from samples of enamel and cured bonding resins were recorded for reference. The relative amounts of the hydroxyapatite (960cm^{-1}, P-O), bonding agent (640cm^{-1}, aromatic ring) and alkyl group (1450cm^{-1}, C-H) in the enamel/resin bonding area were calculated. From Raman spectroscopy, a gradual decrease in hydroxyapatite was observed, and it was estimated to extend 2.2~2.6 µm for Mac Bond II, 1.2~1.6 µm for Clearfil Mega Bond and 5.2~5.6 µm for Single Bond. Furthermore, the enamel/resin interface represents a gradual transition of bonding agent from the resin to tooth side. Evidence of poor saturation of adhesive resin in etched enamel with Single Bond was detected. From the results of this study, non-uniform resin infiltration into etched enamel was detected and the degree of resin infiltration was found to be different among the bonding systems used.

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INTRODUCTION

Good adhesion of resin composite to tooth substrate has been sought for many years since reliable adhesion would produce less microleakage and a stable bond (Perdigão & Lopes, 1999). With the introduction of the acid-etch technique, micromechanical retention has

been achieved between the resin composite and etched enamel (Buonocore, 1955; Buonocore, Matsui & Gwinnett, 1968). In an effort to simplify bonding procedures, several new adhesive systems rely on the simultaneous etching of enamel and dentin with phosphoric acid or a self-etching primer (Van Meerbeek & others, 2001). The systems that use phosphoric acid with an adhesive are called one-bottle adhesive systems. They combine the functions of dentin primer and the bonding agent of three-step systems. On the other hand, self-etching primer systems combine the tooth surface etching and priming steps to simultaneously treat enamel and dentin followed by bonding agent application. A morphological study of the etched enamel surface demonstrated that applying the self-etching primer did not create a deep etching pattern, as did applying phosphoric acid (Perdigão & others, 1997). Laboratory data suggest either equal or reduced enamel bond strengths of self-etching primer systems compared to those of one-bottle adhesive systems (Miyazaki & others, 1998). Because of the differences in etching patterns of the enamel surface, further studies are required to investigate the penetration of resin monomers into the etched enamel surface of the one-bottle adhesive system and the self-etching primer system.

Morphological studies have been conducted to obtain a clearer picture of the enamel-resin interface (Ferrari & others, 1997; Gordan, Vargas & Denehy, 1998). Due to minimal thickness of the enamel-resin interface, methods to analyze this layer must have a very high resolution. Raman micro-spectroscopy is a useful analytical technique for studying the composition and structure of bonding of a sample (Tsuda & Arends, 1997). Since the acquired spectra are attributed to molecules rather than to single elements, and the laser beam can be focused to a very small spot size with the CCD camera, a high spatial resolution at the sample surface can be achieved (Miyazaki, Onose & Moore, 2002). Dehydration of the sample is not required and the measurements can be done under room conditions. Though penetration of resin monomers into decalcified dentin has been reported with the use of Raman microscopy (Suzuki, Kato & Wakumoto, 1991; Ozaki & others, 1992; Van Meerbeek & others, 1993; Wieliczka, Kruger & Spencer, 1997; Spencer & others, 2000), differences related to the extent of resin penetration into etched enamel have not been fully characterized.

This study analyzed the relationship between the penetration of adhesive resin and the degree of demineralization of bovine enamel using laser Raman micro-spectroscopy. The hypothesis tested was that penetration of adhesive resin into etched enamel corresponds to the depth of enamel demineralization.

METHODS AND MATERIALS

The bondng systems used in this study were two self-etching primer systems, Mac Bond II (Tokuyama Dental Corp, Tokyo 110-0016, Japan) and Clearfil Mega Bond (Kuraray Co, Ltd, Kita-ku, Osaka, 530-8611 Japan) and a one-bottle adhesive system Single Bond (3M/ESPE**,** St Paul, MN 55144, USA) as listed in Table 1. All adhesive systems were used according to the manufacturers' instructions. A curing unit (Optilux 500, Demetron/Kerr, Danbury, CT 06810, USA) was connected to a variable transformer in order to adjust the light intensity to 600 mW/cm^2 as measured with a dental radiometer (Model 100, Demetron/Kerr).

Mandibular bovine incisors were used as a substitute for human teeth. After removing the roots with a low-speed saw, the pulps were removed and final finish was achieved by grinding on wet 600-grit SiC paper and ultrasonic cleaning with distilled water.

For the self-etching primer systems, primer was applied on the enamel surfaces for 10 seconds and air dried followed by adhesive application and light irradiation for 10 seconds. For the one-bottle adhesive system, phosphoric acid was applied for 15 seconds, then rinsed off with distilled water. The bonding agent was applied on the blot-dried enamel surface followed by light irradiation for 10 seconds. The resin composites shown in Table 1 were placed into a Teflon mold (4 mm

Table 1: *Bonding Systems Used in This Study*

System	Conditioner (Main Components)	Lot #	Adhesive (Main Components)	Lot #	Resin	Lot #
Mac Bond II	Primer (MAC-10, HEMA, water, ethanol)	A: 021 B: 011	Bonding Agent (MAC-10, BIS-GMA, PI)	016	Palfique Estelite	J236
Clearfil Mega Bond	Primer (5-MNSA, HEMA, water, ethanol)	A: 038 B: 048	Bond (MDP, HEMA, BIS-GMA, filler, PI)	0056	Clearfil AP-X	00687A
Single Bond	Etchant (35% Phosphoric acid)	1UY	Single Bond (BIS-GMA, HEMA, polyalkenoic copolymer water, ethanol, PI)	9DE	Filtek Z250	0ABJ

MAC-10: 11-methacryloxy-11-undecarboxylic acid, HEMA: 2-hydroxyethyl methacrylate, BIS-GMA: bisphenol-glycidyl methacrylate, PI: photoinitiator, 5-MNSA: N-methacryloyl 5-aminosalicylic acid, MDP: 10-methacryloyloxydecyl dihydrogen phosphate

in diameter, 2 mm in high) on the treated enamel surface and light irradiated for 40 seconds. After storage in 37°C water for 24 hours, the specimens were embedded in self-curing epoxy resin (Epon 812, Nisshin EM, Tokyo, Japan) and stored at 37°C for 12 hours. The embedded specimens were then sectioned and the surfaces of the cut halves were polished with an Ecomet 4/Automet 2 (Buehler Ltd, Lake Bluff, IL 60044) using silicon carbide papers of 600, 1200 and 4000-grit size, successively. The surface was finally polished with a special soft cloth and diamond paste with a grit size of 1 µm (Buehler Ltd). At least three specimens from different bovine teeth were prepared for each adhesive system. Care was taken to avoid aggressive polishing which might change the specimen height, and no destructive effect was found around the enamel-resin interface where the Raman analysis was conducted upon inspection with an optical microscope.

Raman spectra were obtained with a computer-controlled laser Raman microscopy System-2000 (Renishaw, Gloucestershire, GL12 8JR, UK) equipped with a monochrometer and a back-thinned MPP-(multi-phase pinned) type CCD (charge coupled device) camera. Fluorescence resulting from electronic excitation of the organic components sometimes dominated the weaker Raman signal when a short wave length laser was used. To overcome the problem of this background noise, a He-Ne laser (GLG-5900, NEC Co, Tokyo, Japan) tuned to a wavelength of 632.8 nm with an output level of 75 mW was used as an excitation source. In addition, the light generated by a He-Ne laser did not contribute to initiating the polymerization reaction of visible-light cured resins that use campohroquinone as a photoinitiator (Taira & others, 1989). The focus of the laser beam in conjunction with the CCD (70% quantum yield) provided a spatial resolution of 0.6~0.8 µm. Instrument calibration was determined before data acquisition by comparison of spectra from pure Si (520 cm-1).

The sample was placed on a precision X-Y stage with a spatial resolution of 0.1 µm. An optical microscope (BH2-UMA, Olympus, Tokyo, Japan) allowed for visual identification of the position from which the signal was obtained. The laser beam was focused on the sample surface with a 100x microscope objective, taking care to avoid any defects that would disturb the measurement. The sample was moved perpendicular to the enamel-resin interface in steps of 0.2 µm, and the spectra were obtained at each position across the enamel-resin interface with an integration time of 120 seconds for each measurement. The number of measurement points across the enamel-resin interface were 50 (10 µm) for the self-etching primer systems and 100 (20 µm) for the one-bottle adhesive system. Raman spectra of unaltered enamel and cured bonding agents were also recorded as references for each obtained spectra. The acquired spectra in the region of interest were analyzed using a curve-fitting program with the Raman microscope software (Renishaw), and relative amounts of hydroxyapatite and bonding resins in the interface were calculated. The measurements were done three times with different teeth for each bonding system, and the reproducibility of this technique was confirmed.

RESULTS

The Raman spectrum obtained from bovine enamel in region of 500-1800 cm^{-1} is shown in Figure 1. The intense peak at 960 cm-1 is associated with the P-O stretching vibration in the mineral component, mainly hydroxyapatite (HAP), of enamel, and this peak was selected as identifying the enamel substrate (O'Shea, Bartlett & Young, 1974).

The Raman spectra of cured adhesive resins are shown in Figures 2A-2C. Several intense peaks were observed; from these, 640 cm^{-1} assigned to the aromatic ring group of BIS-GMA (Wieliczka & others, 1997), and 1450 cm^{-1} assigned to the CH alkyl group (Xu & others, 1997), were selected as measures of the bonding resin. Representative Raman spectra of cured bonding resin of Clearfil Mega Bond, Single Bond and Mac Bond II were obtained.

The intensities of the selected Raman bands scanned across the resin-enamel interface of Mac Bond II are shown in Figure 3A. A gradual decrease in intensity of HAP from the enamel to the resin side was observed. On the other hand, peaks in the BIS-GMA and alkyl group gradually increased across the enamel-resin interface. From these curves, HAP removal seemed to start at 2.4 µm and end at 4.8 µm, and the resin penetration corresponded to this same region. The width of demineralization was 2.2-2.6 µm based on the Raman spectroscopic observation.

The intensities of the selected Raman bands scanned across the resin-dentin interface of Clearfil Mega Bond

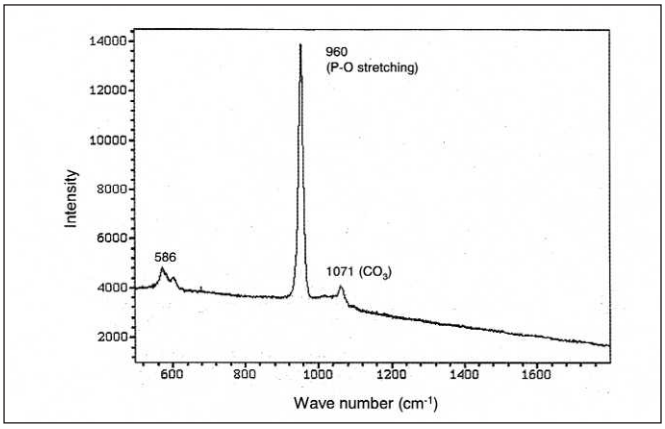

Figure 1. *Typical Raman spectrum obtained from bovine enamel in the region of 500~1800 cm^{-1}. The strong peak of P-O stretching vibration from hydroxyapatite was observed at 960 cm^{-1}.*

Figure 2: *The Raman spectrum of cured bonding agents in the region of 500~1800 cm^{-1}. Several intense peaks were observed in this area.*

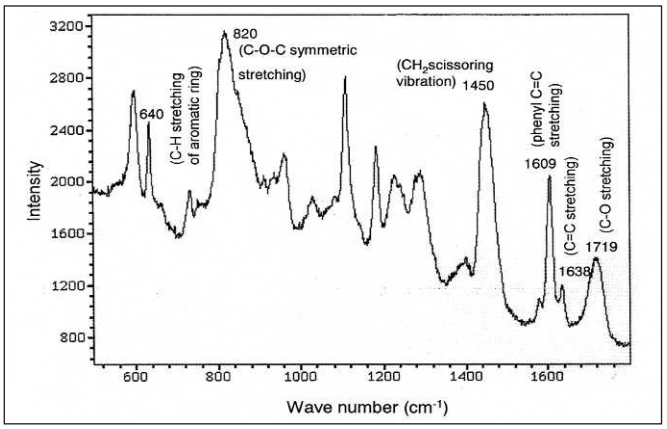

Figure 2A*: Mac Bond II.*

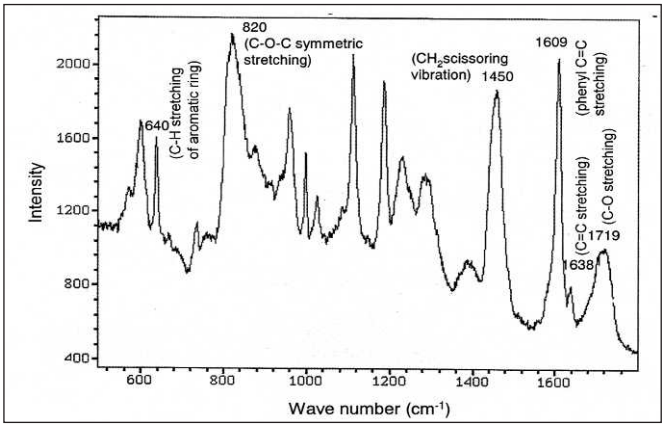

Figure 2B: *Clearfil Mega Bond.*

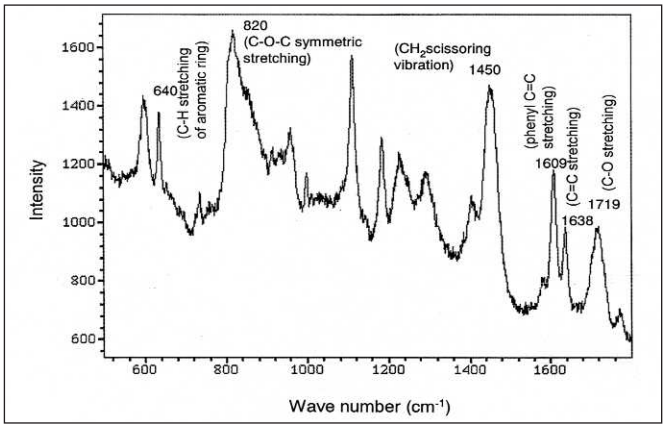

Figure 2C*: Single Bond.*

Figure 3: *Representative intensity curves of the selected Raman bands scanned across the resin-dentin interface.*

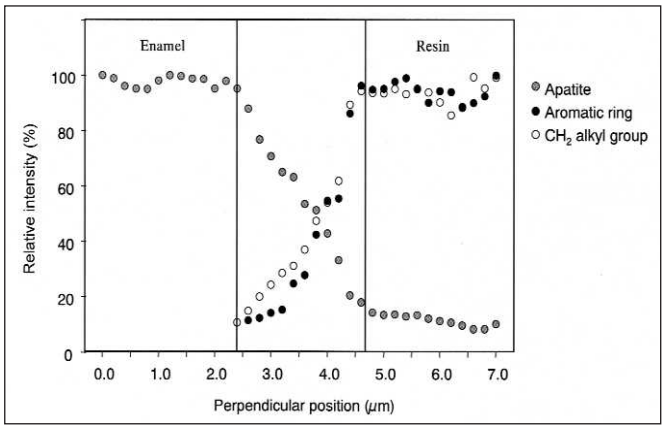

Figure 3A*: Mac Bond II.*

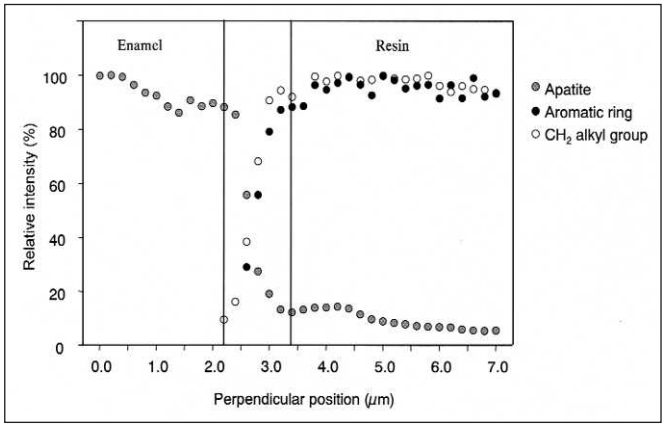

Figure 3B: *Clearfil Mega Bond.*

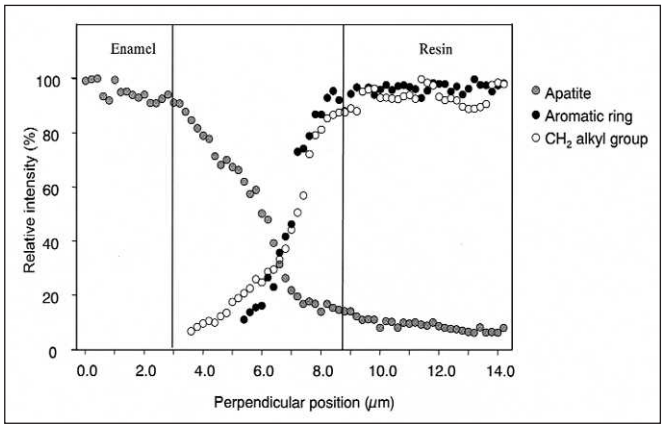

Figure 3C: *Single Bond.*

are shown in Figure 3B. The same tendencies of demineralization and resin penetration were observed as with Mac Bond II. From these curves, mineral removal seemed to start at the position of 2.2 μm and end at 3.2 μm, and the resin penetration corresponded to this same region. The width of demineralization was 1.2-1.6 μm, approximately half the width seen with Mac Bond II.

The intensities of the selected Raman bands scanned across the resin-enamel interface of Single Bond are shown in Figure 3C. A gradual change in Raman intensity of the HAP peak was observed. The mineral loss seemed to start at 2.8 μm and end at 8.4 μm. The width of demineralization was 5.2~5.6 μm. A gradual increase in the CH band was observed from 3.6 to 8.4 μm. The penetration of BIS-GMA began at 3.6 μm and increased to the adhesive resin surface. An area of demineralized enamel without CH_2 penetration was observed and the width of this zone was approximately 1.6 μm. The aromatic ring groups also failed to penetrate into the deeper portion of demineralized enamel. The depth of demineralization was deeper with the use of phosphoric acid etchant. However, resin failed to penetrate into the deeper demineralized enamel.

DISCUSSION

Although it is desirable to use human extracted teeth for bonding research (Nakamichi, Iwaku & Fusayama, 1983; Fowler & others, 1992), in Japan, it has become increasingly difficult to obtain intact extracted human teeth for laboratory studies. To compare the data found in the bovine enamel bond-strength test, bovine teeth were used as a substitute for human teeth in this study.

Laser Raman spectroscopy was used to analyze the bonding structure of samples and to determine their composition. Using this technique, the problems associated with morphological analysis of the dentin-tooth interface with infrared spectroscopy can be avoided (Tsuda & Arends, 1997). Raman spectra can be acquired from moist specimens under normal conditions without ultra-thin sectioning. One of the disadvantages of Raman microscopy is that the background from fluorescence dominates the weaker Raman signal. This problem of high background noise was avoided by using a He-Ne laser as an excitation source. In addition, the light generated from a He-Ne laser (632.8 nm) does not contribute to initiating the polymerization reaction of visible-light cured resins.

Self-etching primers were applied to tooth surfaces before applying bonding agent to improve wettability of the tooth surface and penetration of the bonding agent into etched enamel which creates resin tags (Barkmeier, Los & Triolo Jr, 1995; Ikemura, Kouro & Endo, 1996; Gordan & others, 1997). The depth of selective HAP removed during self-etching primer application may depend on the type and concentration of acidic monomers employed. Applying the self-etching primer only resulted in a shallow etching pattern and might be a result of limited penetration of the self-etching primer into the enamel microporosities or from precipitation of calcium on the enamel surface masking the etch pattern, then interfering with resin penetration (Perdigão & others, 1997). If the resin does not completely infiltrate the etched enamel, a region of weakened enamel prism structure may result.

The representative Raman bands scanned across the resin-enamel interface showed transitional changes in the hydroxyapatite and resin component, indicating that minerals removed by the self-etching primer were gradually replaced by resin. The line scans of the enamel-resin interface suggested that the depth of dentin demineralization was 2.2-2.6 μm for Mac Bond and 1.2-1.6 μm for Clearfil Mega Bond. Though the alkyl groups of resin monomers, including HEMA, penetrated the entire depth of the etched enamel, the BIS-GMA could not penetrate into the deepest area and the depth of this BIS-GMA unsaturated layer was estimated to be about 0.2 μm. For Single Bond, the depth of the BIS-GMA unsaturated layer was estimated to be 2.0-2.4 μm. Incomplete penetration of HEMA into the deepest portion of etched enamel was also detected. HEMA is included in most dentin adhesive systems to wet the dentin substrate and facilitate subsequent penetration of adhesive resins that can polymerize with other monomers in order to create a stable bond. Even with the relatively low molecular weight of HEMA, it seems impossible for it to diffuse into the entire depth of phosphoric acid etched enamel with Single Bond.

Harmony between the depth of demineralization and the extent of resin monomer penetration is the key to creating a high-quality bonding interface between resin and enamel. Poor infiltration of adhesive resin into the demineralized dentin leaves nano-spaces in the hybrid layer (Sano & others, 1994; Spencer & Swafford, 1999), and such a region may be susceptible to degradation from oral fluids (Sano & others, 1999). The same circumstances might occur in the resin-enamel interface. It is possible that a region of demineralized dentin that was not infiltrated by the resin monomers exists with self-etching primer systems. The self-etching primer is not rinsed off, in contrast to conventional phosphoric acid etchant. After applying the self-etching primer, the primed enamel surface is air dried because the primer contains solvents that adversely affect polymerization of the bonding agent (Miyazaki & others, 1999). After evaporation, functional monomers and HEMA remain on the etched surface, followed by curing of the bonding agent. The question then remains, how much resin monomer is required to infiltrate the etched enamel in order to prevent microleakage and reduce bond stability.

When the resin fails to completely infiltrate deeper portions of enamel prisms, the bond strength between the resin and enamel might be weakened after long-term service in the oral environment (Miyazaki & others, 1998). From the ultra-morphological examination of the enamel-resin interface, empty spaces in the hybridized area of enamel were found for an adhesive system (Perdigão & others, 1998). The deficient impregnation of

etched enamel with resin might allow water to leach into the bonding resin, resulting in resin swelling and plasticization (Söderholm, 1991). Water may accelerate hydrolysis of bonding agent and extract poorly polymerized resin oligomers (Bastioli, Romano & Migliaresi, 1990). The decrease in mechanical properties of resin may contribute to the decrease in enamel bond strengths. Though different types of etched enamel were found, no significant differences in the enamel bond strengths were found with varying durations of enamel etching (Uno & Finger, 1995). This phenomenon was explained by observing that the failure mode after the bond strength tests was cohesive failure in bonding resin close to the enamel interface. After long-term storage in water or thermal cycling, the enamel bond strength decreased and the failure pattern changed from cohesive to adhesive failure. The differences in penetration of bonding resin into etched enamel may affect long-term bonding durability.

SEM observation gives only morphological information about the resin-enamel interface; changes in chemical content with distance as revealed by Raman microscopy might help to better understand the mechanism of bonding to tooth substrate. Further research is needed to better define the nature of adhesion to the enamel surface with two-step bonding systems.

CONCLUSIONS

1. Laser Raman microscopy used in this study served as a useful tool to study the gradual changes in chemical character across the enamel-resin interface.
2. The resin-enamel interface represents a gradual decrease in the relative amount of adhesive from the resin side to enamel side.
3. In the deepest regions of demineralized areas, evidence of poor saturation with the adhesive resin was detected.

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Antimicrobial Properties of Self-Etching Primer-Bonding Systems

ZC Çehreli • AS Atac • B Sener

Clinical Relevance

Despite the variety of results obtained from this preliminary study, self-etching primers were shown to produce antibacterial effects against the tested bacterial strains *in vitro*.

SUMMARY

Self-etching primers are now considered the new generation of dentin bonding systems that modify and incorporate the bacteria-containing smear layer into their bonding mechanism. The antibacterial effects of the self-etching primers Clearfil SE Bond, Mac Bond, Imperva FL Bond, One-Up BondF and Prompt L-Pop were evaluated using the bacteria *Streptococcus mutans* ATCC25175, *Peptostreptococcus anaerobius*, *Peptostreptococcus prevotii*, *Peptostreptococcus asaccharolyticus*, *Lactobacillus acidophilus*, *Lactobacillus catenaforme*, *Lactobacillus jensenii*, *Actinomyces odontolyticus*, *Porphyromonas endodontalis*, *Clostridium ramosum*, *Prevotella oris*, *Prevotella denticola* and *Fusobacterium nucleatum*, with a disk diffusion method. A single-bottle total-etch dentin adhesive (Excite) was used for comparisons and chlorhexidine (0.2%) was used as the positive control. After incubation, zones of inhibited bacterial growth were observed. One-Up BondF, Prompt L-Pop and Excite showed growth inhibition for all bacterial strains. The bonding agents of Clearfil SE Bond, Mac Bond and Imperva FL Bond were unable to inhibit the growth of *Lactobacillus jensenii* and *Actinomyces odontolyticus*, while the primers of these systems produced inhibition halos to all tested microorganisms greater than that of chlorhexidine.

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INTRODUCTION

Bacterial microleakage has been claimed to be the main cause of pulpal inflammation, necrosis and the eventual need for endodontic therapy after placement of restorations (Brännström & Nyborg, 1971), and the biological sealing of prepared dentin is now considered critical for successful restoration. The advantages and disadvantages of the smear layer, which normally contains non-specific inorganic contaminants and microorganisms (Czonstkowsky, Wilson & Holstein, 1990), and whether it should be removed from the instrumented dentin surfaces, are still controversial (Gwinnett, 1984). Today, many dentin bonding systems require phosphoric acid etching to condition instrumented cavity surfaces by removing the smear layer as a standard procedure in adhesive dentistry. The infiltration of adhesive resins into such demineralized dentin results

in a resin-dentin impregnation zone that not only provides strong bonding but also hermetic seal (Swift, Perdigão & Heymann, 1995). In addition, recent studies have shown both dental phosphoric acids and total-etch dentin bonding systems to possess considerable short-term antimicrobial activity (Settembrini & others, 1997; Atac, Çehreli & Sener, 2001).

Formation of a hybrid layer is a reasonable method to produce a resistant structure against bacterial invasion. Unfortunately, it does not occur in every instance. After dentin etching with an acidic gel, the high permeability of dentin close to the pulp is markedly increased because of 1) the enlargement of dentinal tubules, 2) removal of smear layer and smear plugs and 3) the hypertonic property of acidic gel (Perdigão & Lopes, 1999). All three factors may interfere with an intradentin permeation of fluid bonding agent into the intertubular dentin. This seems to result in an unprotected collagen fibrous network below the hybrid layer (Van Meerbeek & others, 1993), which becomes vulnerable to hydrolysis (Sano & others, 1994). Once such microgaps are formed, bacteria and their toxic products readily invade. The bacteria proliferate beneath the bonding interface, permeate the unsealed dentinal tubules and may cause a pulpal response (Atac & others, 2001).

New adhesive systems, namely self-etching primers that combine dentin conditioning and bonding in a single step, have been introduced. In these adhesive systems, the acidic part of the primer dissolves the smear layer and incorporates it into the mixture as it demineralizes the dentin and encapsulates the collagen fibers and hydroxyapatite crystals. In this way, applying self-etching systems maintains the normal characteristics of dentinal tissue devoid of unprotected collagen with no increase in dentinal permeability (Watanabe, Nakabayashi & Pashley, 1994). However, incorporating the smear layer particles into the hybrid layer is one potential disadvantage of these systems from the viewpoint of the microbial content of the smear layer, which may further lead to pulpal infections. Therefore, the possibility of self-etching primers having antibacterial effects is attractive.

This study compared the antibacterial activity of five current self-etching primer systems against a single-bottle, total-etch bonding agent using a disk diffusion method.

METHODS AND MATERIALS

Table 1 shows the composition of the bonding agents used in this study. Antibacterial activity of each bonding system was evaluated against the following bacteria: *Streptococcus mutans* ATCC25175, *Peptostreptococcus anaerobius, Peptostreptococcus prevotii, Peptostreptococcus asaccharolyticus*, *Lactobacillus acidophilus, Lactobacillus catenaforme, Lactobacillus jensenii, Actinomyces odontolyticus, Porphyromonas endodontalis, Clostridium ramosum, Prevotella oris, Prevotella denticola* and *Fusobacterium nucleatum*.

Cultures were started from freeze-dried stocks into 10 ml of sterile brain-heart infusion broth (Difco Laboratories, Detroit, MI, USA) enriched with

Table 1: *Self-Etching Primer-Adhesive Systems Used in This Study*

Bonding Agent (Code)/Manufacturer	Class	Composition	
		Primer	Adhesive
Clearfil SE Bond (SE) Kuraray, Japan	SEP	*MDP, HEMA, Hydrophilic dimethacrylate, di-camphoroquinoneN, N-diethanol-p-toluidine, water*	*MDP, BIS-GMA, HEMA, silanated colloidal silica, hydrophilic dimethacrylate, di-camphoroquinoneN, N-diethanol-p-toluidine*
Mac Bond (MAC) Tokuyama, Japan	SEP	*MAC10, MDP, ethanol, water*	*MAC 10, HEMA, BIS-GMA, TEGDMA, photoinitiator*
FL Bond (FL) Shofu, Japan	SEP	*Water, photoinitiator 4-acryloxiethyltrimellitic acid (4-AET), HEMA*	*4-AET, HEMA, UDMA, TEGDMA, SiO_2 microfillers*
One-Up BondF (OB) Tokuyama, Japan	SEP	*Bonding Agent A* *MAC-10, photo-initiator, methacryloyloxyalkyl acid phosphate, BIS-GMA, TEGDMA* *Bonding Agent B* *MMA, HEMA, water, F-deliverable micro-filler, photo-initiator*	
Prompt-L-Pop (LP) 3M/ESPE, Germany	SEP	*Methacrylated phosphoric acid esters, camphoroquinone, Amino benzoate, Butyl hydroxy toluene, Water, Fluoride complex, Parabenes*	
Excite (EX) Vivadent, Liechtenstein	SB	*HEMA, dimethacrylates, phosphonic acid acrylate, silicone dioxide, stabilizers, alcohol*	

SEP=Self-etching primer/adhesive, SB=Single bottle (total-etch) adhesive
HEMA=hydroxyethylmethacrylate, BIS-GMA=bisphenyl glycidylmethacrylate, TEGDMA=Triethyleneglycol dimethacrylate, UDMA=urethane dimethacrylate MMA=methyl methacrylate
MDP=methacryloyloxydodecylpyridinium bromide

Vitamin K and hemin for the purpose of growing anaerobic bacteria. All isolates were grown under anaerobic conditions using an anaerobic chamber system for 48 hours before being used. The purity and viability of the bacteria were later confirmed by inoculating them into Schaedler agar and incubating them for 48-to-72 hours under anaerobic conditions. From these cultures, bacterial suspensions were prepared in sterile brain-heart infusion broth until a turbidity compatible with 0.5 MacFarland was obtained. This scale allowed the bacterial concentration of a suspension to be estimated by its turbidity; 0.5 corresponded to a concentration of $1.5x10^8$ (bacteria/ml) at an optic density of 550 nm.

Fifty microliters of each bonding agent was applied to sterilized paper disks (6 mm diameter, 1.5 mm thick), after which photopolymerization was performed using a light source (Translux EC, Kulzer, Germany) according to the manufacturers' instructions. For two component, self-etching adhesives (primer and bonding agent), each solution was tested separately (Table 1). Schaedler agar was used to determine the antibacterial activity of the test materials. For this purpose, 100 µl of each bacterial suspension was spread evenly in the agar plate using a sterile spreader. After the surface of the plates was dried, the specimen disks were placed on the plates using sterile tweezers. Following anaerobic incubation of the plates at 37°C for 48 hours, diffusion of the antibacterial components was determined using the inhibition zone produced around the disks. The diameter of the inhibition zone was measured (in millimeters) in three locations and the average calculated. The positive control used was a 0.2% aqueous solution of chlorhexidine that was added onto paper disks and tested as described for the bonding agents.

In each material group, the essays were conducted in nine samples per tested microorganism. The antibacterial activity of each test material was statistically analyzed using the Kruskal-Wallis test with a level of significance set at $p<0.01$. Intermaterial comparisons in conjunction with each bacterial species were statistically analyzed using the Mann-Whitney U test. Significance was determined at $p<0.001$.

RESULTS

Table 2 presents the zone of inhibition (in mm) produced by the test materials and the chlorhexidine control as means ±SD. For the two-component (primer and bonding agent) self-etching systems (SE, MAC and FL), the bonding agents showed remarkably lower

Table 2: *Diameter of Inhibiton Zones (in milimeters) Produced by Each Bonding Agent Against the Bacteria Tested (mean±SD)*

	Control (CH)	Excite	SE Primer	SE Bond	MAC Primer	MAC Bond	FL Primer	FL Bond	OneUp F	Prompt L-Pop
Streptococcus mutans	14.3±1.4	18.3±0.7	20.2±2.0	5.8±0.7	23.4±1.3	15.3±0.8	14.7±1.3	15.3±1.6	20.1±1.9	22.1±1.5
Lactobacillus mutans	11.6±1.0	14.8±1.3	22.0±1.6	12.3±1.5	21.8±1.5	12.4±1.1	11.7±1.2	12.4±0.7	12.4±0.8	20.7±1.3
Lactobacillus catenaforme	18.0±1.0	18.2±2.1	22.6±1.7	0.0±0.0	17.7±1.0	16.1±1.1	18.3±1.0	14.6±1.1	22.8±2.4	36.0±2.9
Lactobacillus jensenii	22.3±1.5	23.1±1.6	31.2±0.8	0.0±0.0	25.8±1.2	0.0±0.0	22.0±1.4	0.0±0.0	23.6±1.5	30.0±1.5
Clostridium jensenii	16.1±1.0	15.5±1.6	13.8±1.1	7.7±0.8	13.6±1.2	7.4±0.7	13.8±0.9	7.8±1.3	15.4±1.1	15.1±1.4
Porphyromonas jensenii	17.4±0.8	18.0±1.1	17.8±1.2	8.4±0.8	17.2±2.1	8.2±0.8	18.0±1.2	9.0±1.0	20.1±1.7	18.8±1.0
Actinomyces jensenii	17.3±1.1	18.1±2.0	14.5±1.3	0.0±0.0	15.1±1.2	0.0±0.0	15.0±1.3	0.0±0.0	14.1±1.2	14.6±1.3
Prevotella oris	18.5±1.8	19.8±1.2	23.1±1.4	15.2±1.2	19.4±1.9	15.0±1.4	18.7±2.1	19.4±3.2	22.3±1.8	21.8±1.6
Prevotella denticola	29.2±2.9	20.8±1.7	30.5±1.8	15.2±2.2	29.4±3.1	14.5±1.2	30.4±2.1	17.7±1.3	30.0±1.9	29.8±1.6
Peptostreptococcus asaccharolyticus	25.2±1.3	19.0±1.7	20.6±1.8	14.7±2.0	13.6±1.1	15.1±1.0	20.1±1.4	13.6±1.4	19.5±0.8	21.0±0.7
Peptostreptococcus anaerobius	17.1±1.5	16.5±2.3	15.8±2.2	13.5±2.7	15.5±0.7	11.7±2.1	23.6±1.2	14.6±1.4	18.1±1.1	16.4±1.3
Peptostreptococcus prevotii	23.2±0.8	19.4±1.3	17.1±1.2	13.6±1.4	15.8±1.2	11.7±1.3	15.0±2.2	14.7±2.9	19.0±1.6	18.1±2.1
Fusobacterium nucleatum	27.2±0.8	13.3±1.4	24.1±0.9	14.0±2.0	17.4±1.2	11.8±1.1	30.8±2.0	14.2±2.1	19.4±1.2	21.4±1.7

degrees of antibacterial action compared to their own primers (p=0.001) and with that of chlorhexidine (p=0.001). In addition, *Lactobacillus jensenii* and *Actinomyces odontolyticus* were markedly resistant to these three bonding agents (Table 2). SE bonding agent also failed to produce inhibition halos against *Lactobacillus catenaforme*.

However, as with the primers of two-component self-etching systems, the antibacterial effect against all tested microorganisms was achieved (Table 2). The intramaterial comparisons among these three systems showed FL primer to produce the greatest inhibition halo in conjunction with *Fusobacterium nucleatum* (p<0.001, Table 2). Similarly, SE primer was the most inhibitory agent against *Lactobacillus jensenii* (p<0.001). When compared with all materials, MAC primer produced the greatest inhibition halo against *Streptococcus mutans* (Table 2). However, the differences among MAC primer, SE primer and LP were not significant for this microorganism (p=0.001).

For the single-step self-etching systems, both OB and LP produced inhibition halos against all tested microorganisms (Table 2). LP was the most inhibitory bonding agent against *Lactobacillus catenaforme*. As for *Peptostreptococcus anaerobius, Peptostreptococcus prevotii, Peptostreptococcus asaccharolyticus*, *Actinomyces odontolyticus, Porphyromonas endodontalis, Clostridium ramosum, Prevotella oris, Prevotella denticola* and *Fusobacterium nucleatum*, the mean inhibition zones between OB and LP were not statistically significant. On the other hand, the antibacterial effects of both systems in conjunction with *Peptostreptococcus anaerobius, Peptostreptococcus prevotii, Peptostreptococcus asaccharolyticus* and *Fusobacterium nucleatum* were inferior to chlorhexidine (p<0.001).

EX was the only single bottle (total-etch) bonding agent tested in the study. It demonstrated antibacterial activity superior to chlorhexidine against *Streptococcus mutans* and *Lactobacillus acidophilus* (p<0.001), while differences between EX and chlorhexidine against *Peptostreptococcus anaerobius, Lactobacillus acidophilus, Lactobacillus catenaforme, Lactobacillus jensenii, Actinomyces odontolyticus, Porphyromonas endodontalis, Clostridium ramosum* and *Prevotella oris* were not significant (p>0.001), showing that the antibacterial effects of EX in conjunction with these species were similar to chlorhexidine. As for *Peptostreptococcus prevotii, Peptostreptococcus asaccharolyticus, Prevotella denticola* and *Fusobacterium nucleatum*, the mean inhibition zones obtained with chlorhexidine were greater than EX (p=0.0003, p=0.0003, p=0.0008 and p=0.0003, respectively). However, unlike the two component self-etching systems, this single bottle adhesive showed antibacterial effect against all tested microorganisms.

DISCUSSION

More than 500 bacterial groups are present in the oral cavity, but a relatively low number of species are related to caries or pulpal infections (Sundqvist, 1992). Using cultural techniques, a correlation has been suggested between the presence of specific anaeorbic oral bacteria (*Peptostreptococcus*, *Fusobacterium* and *Porphyromonas* species) and clinical symptoms in endodontically involved teeth (Gomes, Drucker & Lilley, 1994). In addition, Gram-negative bacteria (*Prevotella* and *Porphyromonas* species) have been detected in carious teeth when there are no signs of pulpal exposure, but when clinical signs of reversible pulpitis are present (Castillo & Liébana, 1995). Despite the evidence of *Streptococcus mutans* and lactobacilli are major human dental pathogens, several other species of bacteria have been associated with carious lesions. In this study, the reasoning behind including *Clostridium ramosum* was based on the concept that in caries development, acidogenic bacteria are substituted by proteolithic bacteria such as this microorganism (Atac & others, 2001).

Past concepts that pulp does not become infected until an actual carious exposure takes place have been challenged. According to many longitudinal investigations, a high number of primarily vital teeth remain which exhibit symptoms requiring endodontic treatment following restorative procedures (Zöllner & Gaengler, 2000). Often, pulp responses have been ascribed to the cytotoxic activity of particular restorative materials or subsequent microleakage, giving little regard to the possible effects of residual bacteria remaining in a prepared cavity that may lead to pulpal infections in the short-term. Although the smear layer in a prepared cavity is beneficial to serving as a cavity liner to reduce dentin permeability to the possible cytotoxicity of restorative materials (Gwinnett, 1984), the presence of the smear layer that contains entrapped bacteria (Brännström & Nyborg, 1971; Gwinnett, 1984) and denatured smear layer debris (Czonstkowsky & others, 1990; Settembrini & others, 1997) has been suggested as diffusing through the dentin floor of cavity preparations and increasing the level of pulp injury compared with total-etched dentin cavity restorations long before subsequent microleakage-oriented pulpal infection may occur (Çehreli & others, 2000a). Moreover, the adequate and homogenous removal of smear is difficult to achieve clinically, even with total-etch bonding systems (Çehreli & Altay, 2000b). This justified the authors' attempts to obtain preliminary data regarding the antibacterial effect of the new self-etching adhesives that incorporate bacteria-containing smear in their bonding mechanism (Watanabe & others, 1994).

The self-etching adhesives used in this study have demonstrated antibacterial action to varying degrees. Previous studies that focused on the antibacterial effect of commercially marketed adhesive systems have generally attributed this effect to their low pH environments (Meiers & Miller, 1996; Emilson & Bergenholtz, 1993). This fact was confirmed in this study by the acidic primers of the two-component self-etching systems (SE, MAC and FL) that showed antibacterial action against all microoorganisms tested (Table 2). However, these materials have also been known to fail to show an antibacterial effect against acid-tolerant species such as lactobacilli (Emilson & Bergenholtz, 1993). The bonding agents of SE, MAC and FL failed to produce inhibition halos against *Lactobacillus jensenii* in this study. In addition, the bonding agent of SE was unable to inhibit the growth of *Lactobacillus catenaforme*. As with the single-step self-etching adhesives, antibacterial activity were shown against all microorganisms. This result was not surprising, as these systems combine acidic primer and bonding agents in a single solution (Table 1). However, the antibacterial action achieved with OB and LP against *Peptostreptococcus anaerobius*, *Peptostreptococcus prevotii, Peptostreptococcus asaccharolyticus* and *Fusobacterium nucleatum* were inferior to chlorhexidine (Table 2).

Chlorhexidine is an antiseptic with a wide spectrum of action, and its use has been generalized over the past two decades for the chemical control of bacterial plaque and disinfection of therapeutic cavities. For this reason, it is adopted as a positive control for studies on bacterial growth or antibacterial activity (Settembrini & others, 1997). In this study, the antibacterial activity of chlorhexidine was greater than that of EX against *Peptostreptococcus prevotii*, *Peptostreptococcus asaccharolyticus, Prevotella denticola* and *Fusobacterium nucleatum*. Similar differences in favor of chlorhexidine were also found compared to the bonding agents of SE, MAC and FL. For the latter three systems, the results of this study may suggest that these inadequacies, compared to chlorhexidine, may be leveled or even surpassed readily by use of their acidic primers, which demonstrated better antibacterial activity than that of chlorhexidine (Table 2). As with EX, this difference could be compensated for by the antibacterial effect of its etchant (Email preparator, Vivadent, Schaan, Liechtenstein), which is applied on the cavity walls before applying the EX adhesive as part of the total etch protocol. In a recent study, 35% phosphoric acid gel demonstrated a wide range of antibacterial activity (Settembrini & others, 1997).

CONCLUSIONS

This study has shown that contemporary self-etching adhesive systems are capable of producing antibacterial effects to varying degrees *in vitro*, although any further interpretation of the results must be made with caution. Zones of bacterial inhibition in soft Schaedler agar do not necessarily relate to bacterial inhibition in hard, vital dentin. There might be some properties of dentin or the overlying restorative material that change the effectiveness of some of the materials tested. Thus, further studies are indicated to confirm these preliminary data, especially in conjunction with the involvement of smear-layer containing dentin, the amount or concentration of components released from such systems to inhibit bacteria, the duration of the effect and when it is applied *in vivo*.

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The Effect of Air Abrasion with Two New Bonding Agents on Composite Repair

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Clinical Relevance

Surface treatment with the air-abrasion method plus Optibond Solo application provides the highest shear bond strength in direct, visible, light-cured resin composite repair.

SUMMARY

This study evaluated the shear bond strength of repairs made with a composite (Herculite/Kerr) when two different surface treatment methods and two new generation bond materials were used. The bonding surfaces were prepared by sanding with 500-grit aluminum oxide sandpaper or air abrading with 25-micron aluminum oxide. Treated resin surfaces were acid etched, washed for one minute and air dried. Before adding the composite Herculite, either Optibond Solo (Kerr), Solobond M (Voco) bonding agent or no bonding agent (control group) was applied. The specimens were evaluated for shear bond strength after thermocycling. Fracture surfaces of some samples were also evaluated with SEM. Light microscope and SEM examination of fractured repair surfaces indicated mostly cohesive failure within the air-abraded group. The results showed that surface treatment with air abrasion plus Optibond Solo application had the highest shear bond strength.

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INTRODUCTION

The clinical performance of composite material has improved, with newer generation dentin bonding agents positively effecting success rates. On the other hand, repair of resin composite restoration due to fracture, abrasion, discoloration and color mismatch is naturally inevitable. In such conditions, repair is more practical than replacing the restoration because it reduces pulpal trauma and is cost effective.

Turner and Meiers (1993) stated that resin composite repairs occur regardless of the type of resin composite or technique used, whether macrofill, hybrid, microfill, chemical cure, light cure, direct or indirect. However, there is no consensus within dentistry regarding the best repair protocol to follow due to inconsistencies in materials and repair methods used in previous studies.

Although the importance of a good bond between the old and new resin material has been accepted, repair bond strengths have been variable and unpredictable, as stated in many studies (Boyer, Chan & Reinhardt, 1984; Pounder, Gregory & Powers, 1987; Puckett, Holder & O'Hara, 1991; Saunders, 1990; Swift, Le Valley & Boyer 1992; Swift, Cloe & Boyer, 1994).

Most studies have indicated that the surface roughness of composite has a greater influence on repair strength

than using a bonding agent (Kupiec & Barkmeier, 1996; Los & Barkmeier, 1994; Swift & others, 1992; Turner & Meiers, 1993).

Recent investigations have shown that air abrasion is considered a viable, safe alternative for preparation of tooth structure and removal of different restorations (Laurell & others, 1995; Rinaudo, Cochran & Moore, 1997).

This study determined the shear bond strength of repairs made with different surface treatment methods, where two new generation bonding materials were used. Fracture surfaces of some samples were also evaluated with SEM.

METHODS AND MATERIALS

Fifty-six Charisma hybrid resin composite (Heraeus Kulzer, South Bend, IN 46614, USA, shade B3) samples 6 mm in diameter and 5 mm in depth were prepared. Each stock sample was developed in a plastic canule using Teflon filling instruments. The composite was loaded into the canules with incremental technique and cured at each step for 20 seconds with Polofil Lux Halogen Light Unit (Voco-Cuxhaven, W Germany). These cylindrical samples were ejected from the canules and embedded in acrylic blocks, as a 2-mm part of the sample would be out. Sample test surfaces were sanded with 500-grit aluminum oxide sandpaper in a sanding jig to produce a flat surface perpendicular to the sample long axis. Samples were numbered and stored in saline at room temperature. They were aged by thermocycling for 300 cycles between 5°C and 55°C with a one-minute dwell time at each temperature.

Table 1: *Resin Materials and Test Groups*

Test Groups	Repair Sequence	Charisma Surface Preparation
Group 1	C/H	air abraded
Group 2	C/OS/H	air abraded
Group 3	C/SM/H	air abraded
Group 4	C/H	sanded
Group 5	C/OS/H	sanded
Group 6	C/SM/H	sanded
Control	C/C	none

Charisma (C) (Heraeus Kulzer, South Bend, IN 46614, USA)
XRV Herculite (H) (Sybron/Kerr, Romulus, MI 48174, USA)
Optibond Solo (OS) (Kerr Corporation, Orange, CA 92667, USA)
Solobond M (SM) (Voco, Cuxhaven, Germany)

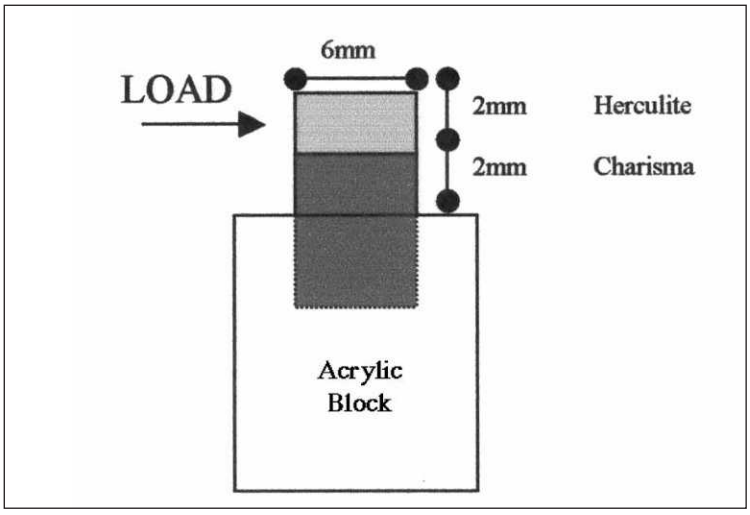

Figure 1. *Drawing to illustrate the shear bond strength test.*

Table 1 shows six different repair groups (n=8), each including composite samples that were investigated. Surface preparation methods, bonding agents and composites used in these groups are also seen in Table 1. In Groups 1, 2 and 3, air-abrasion unit (Micadent/Medidenta, Danville Engineering Inc, CA 94583, USA) was used with 25 micron aluminum oxide at 80 psi for three seconds using a continuous circular motion at 45° to the surface. Sample test surfaces in Groups 4, 5 and 6 were sanded by a 500-grit aluminum oxide sandpaper for six-inch strokes with a water-cooling abrasion machine (Minimet 1000 Grinder polisher, Buehler). A control test group (n=8) was separated to test the cohesive strength of the substrate resin.

The prepared surfaces were cleaned with 37% phosphoric acid for 10 seconds, rinsed for one minute with tap water, then dried with compressed air. In Groups 2, 3, 5 and 6, dentin bonding agents were applied according to manufacturers' instructions but not light cured prior to applying the repair composite. Plastic canules cut 2 mm in length with the same diameter (6 mm) were placed over the test surface to provide a consistent bonding area. Herculite resin composite (Kerr Corporation, Orange, CA 92667, USA, enamel shade A1) was applied as a repair material in 2 mm height with the help of an amalgam carrier, then light cured for one minute with a Polofil Lux Halogen Light Unit (Voco-Cuxhaven, W Germany) calibrated prior to use with Demetron Visible Light Analyzer (Demetron Research Corp, Danbury, CT, 06810, USA).

The repaired samples were thermocycled for 300 cycles between 5°C and 55°C with a dwell time of one minute at each temperature as before.

Samples were placed in a Hounsfield Tensometer (made in England, W 7584) with the brass washer parallel to and engaging the shearing pin (Figure 1). A crosshead speed of 2 mm/minute was used to fracture the repaired surface-interface. Shear bond strengths were calculated and recorded in megapascal units (MPa).

All the samples were investigated under light microscope x10 magnification to evaluate the fracture surface.

Table 2: *Bond Strength of Repairs by Bonding Agent/Methods*

	C/H	C/OS/H	C/SM/H	
	X ± SD	X ± SD	X ± SD	X ± SD
Air Abraded	18.2 ± 3.2	31.3 ± 8.3*	23.2 ± 5.2	24,23 ± 5,57**
Sanded	11.4 ± 3.2	13.5 ± 6.5	12.8 ± 5.4	12,57 ± 5,03

**means the statistical important group in all groups.*
***means the statistical important group when comparison of repair bond strength of air abraded and sanded groups.*

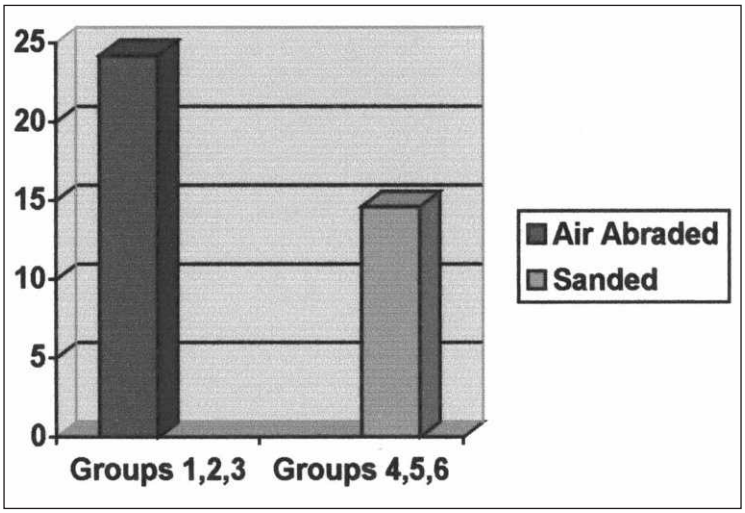

Figure 2. *Comparison of repair bond strengths of air abraded and sanded samples X±SD.*

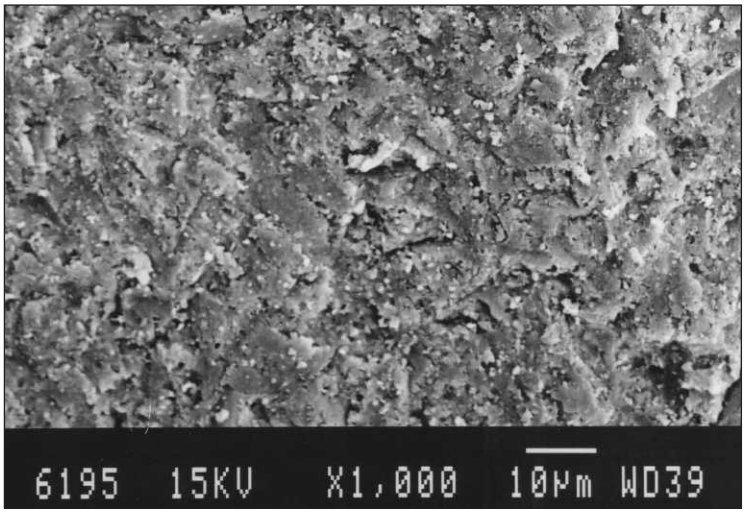

Figure 3. *Charisma surface air abraded with 25-micron aluminum oxide at 80 psi.*

The means and standard deviations for the samples were determined. The data were analyzed by analysis of variance using a factorial desings. Means were ranked by a Tukey interval calculated at the 95% level of confidence.

Selected samples from each test group were examined by scanning electron microscopy (SEM-JEOL JSM 5400) to evaluate the pre-bonding surfaces (n=4) and interfacial fractured surfaces.

RESULTS

Table 2 presents the bond values and the number of bonds that failed during thermocycling for all groups.

Charisma resin composite, which was air abraded, and Optibond Solo, which was applied (Group 2), had a significantly high mean shear bond strength value ($p \leq 0.05$).

Charisma resin composite, which was only sanded and repaired with Herculite resin composite without having a bonding agent (Group 4), or Optibond Solo, which was applied as the intermediary bonding resin (Group 5), had some failures during thermocycling as shown in Table 2.

Comparison of the air-abraded groups (1, 2 and 3) and the sanded groups (4, 5 and 6) showed that there were significant differences between the two surface preparation methods (Figure 2) ($p \leq 0.05$).

SEM Analysis

Table 3 shows the failure mode for specimens in all groups. The air-abrasion method with 25-micron aluminum oxide (Figure 3) created a homogenous but rougher surface than sanding with 500-grit sandpaper (Figure 4).

Figure 5 displays the cohesive failure where resin composite fracture occurred on repaired resin surface in the air-abraded group.

Figure 6 shows the adhesive fracture surface in sanded samples and the small islands of repair resin composite parts on it. In Figure 7, islands of the repaired composite on the fractured site and small voids on both composite surfaces were observed in the sanded group, indicating some cohesive failure again.

When these voids were examined with higher magnifications, microfractures were found in the interface surfaces (Figure 8).

DISCUSSION

The sanded Charisma surface appeared glossy smooth when using a light microscope and the SEM examination was intended to provide a bonding surface free of micromechanical retentive areas. However, air abrasion provides a rough, irregular surface with large microretentive areas and increases its wettability for adhesive system. Wetting of the surface to be repaired by a bonding agent to allow for optimal adaptation of the repair composite has been shown to be integral to developing a high bond strength (Causton, 1975; Boyer & others, 1984). Wetting is controlled by the surface-free energy differences between the substrate and bonding resin and the viscosity of the bonding resin.

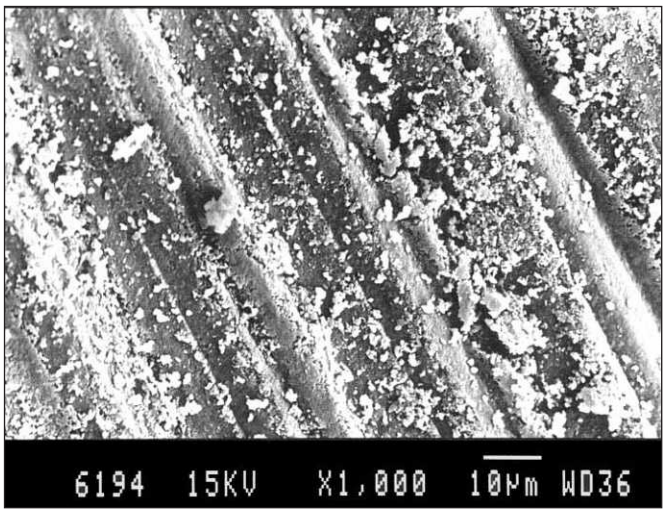

Figure 4. *Charisma surface sanded with 500-grit aluminum oxide sandpaper.*

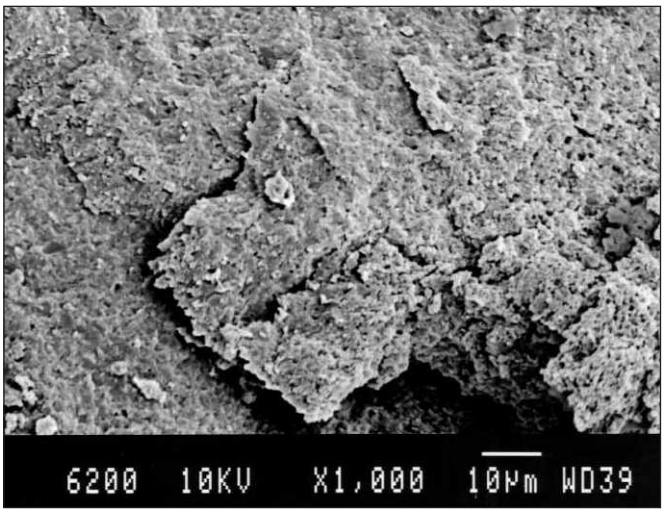

Figure 5. *The cohesive fracture in air abraded group.*

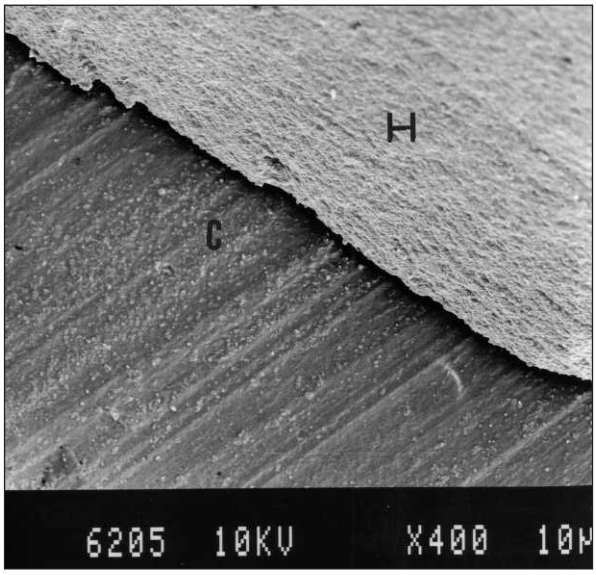

Figure 6. *The adhesive fracture in sanded group.*

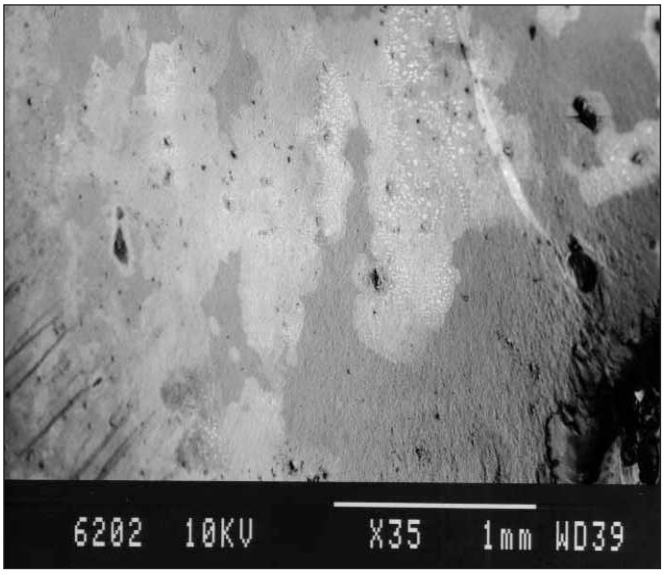

Figure 7. *Debonded surface of Charisma showing an island of repair composite.*

Table 3: *Mode of Bond Failure*

Group (n=8)	Adhesive	Cohesive	Mixed
1	1	6	1
2	-	6	2
3	1	5	2
4	5	-	1
5	5	-	2
6	6	1	1
7	-	8	-

This study indicated that surface roughness had more influence on the repair bond values than did the choice of a bonding agent.

Reports that showed roughening the surface of the composite with either a diamond instrument or aluminum oxide air abrasion had significantly ($p<0.005$) improved bond strength when compared to the 600-grit composite surface that supported the results of this study (Chalkley & Chan, 1986; Kupiec & Barkmeier, 1996; Turner & Meiers, 1993). Kupiec & Barkmeier (1996) concluded that using a bonding agent, alone, slightly increased the bond strength to these treated surfaces, confirming the importance of surface treatment techniques.

Dentin bonding agents are based on chloro-phosphate esters of the BIS-GMA with a surfactant and solvent added. The phosphate group may have the ability to hydrogen bond or condense with exposed silanol func-

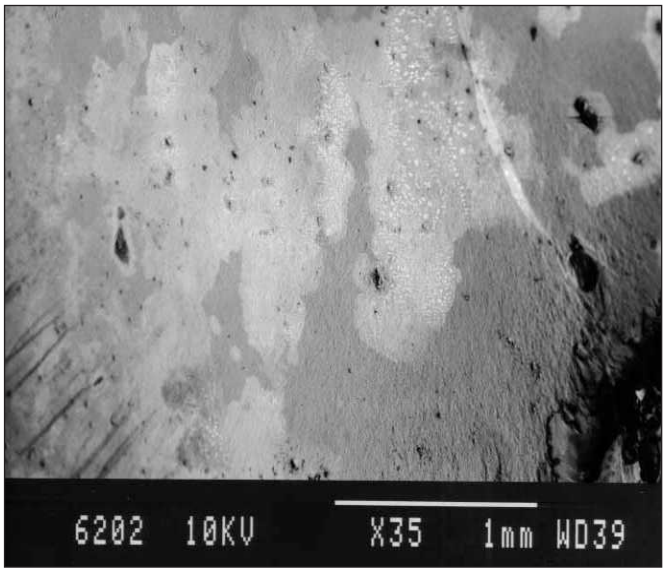

Figure 8. *Debonded repair composite surface, Herculite, showing a large void.*

tional groups in the inorganic phase. These possible reactions and the presence of surfactant and solvent to improve wetting and penetration would explain the higher repair bond strengths when dentin-bonding agents were used (Puckett & others, 1991).

Optibond Solo contains a resin with a filling of low elasticity that favors stronger, more long-lasting bonding by acting as a relatively flexible layer that absorbs stress due to contraction of the first layer of the composite. Another advantage of filled adhesives is that the film is thick enough to eliminate the problem of inhibition by oxygen (Perdigão, Swift & Lopes, 1999).

The bonding agent Optibond Solo (Group 2) provided significantly higher bond strength results (31.3 + 8.3 MPa) compared with Solobond (23.2 ± 5.2 MPa) in the air-abraded groups (Group 3) of this study. This difference may be due to the chemistry of the dentin-bonding agent.

The chemistry of composite substrate and repair material is another factor that effects bond strengths. Studies that show repairs involving combinations of urethane dimethacrylate-based composite and a BIS-GMA composite leaked to a greater extent than repairs involving similar materials, whereas, no effect was found on repairs when combinations of different BIS-GMA based composites were used (Eli & others, 1988; Ruyter & Svendsen, 1977).

Two different hybrid composites that contain a BIS-GMA matrix in order to limit the variations that effect the bond strengths of repairs were selected for this study.

Fractured surfaces examined with SEM showed mostly cohesive failure in the air-abraded group in this study. Kupiec and Barkmeier (1996) also indicated that failure types with and without bonding agent were 100% cohesive failures in the previously cured composite. The cohesive shear bond strength for the control group was considerably lower than for the manufactured information.

Voids and microfractures observed in the interface surface may result from polymerization stresses weakening the material. However, these voids and microfractures could not be differentiated as the cause, effect or being incidental to the cohesive failure from loading and fracture as stated by Turner and Meiers (1993).

CONCLUSIONS

1. The air-abrasion technique resulted in significantly higher bond strengths than sanding with 500-grit sandpaper.
2. Surface treatment with the air-abrasion method plus Optibond Solo application had the highest shear bond strength.

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Effect of Different Photoactivation Methods on the Polymerization Depth of a Light-Activated Composite

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S Consani • LC Sobrinho

Clinical Relevance

The use of distinct photoactivation methods promotes different patterns and depths of polymerization and, therefore, is considered an extremely important step during a restorative technique.

SUMMARY

This study verified the polymerization depth of the composite Z100 (3M Dental Products) by Knoop Hardness test using four photoactivation methods. Twenty cylindrical specimens (5 mm in diameter by 5 mm in height) were prepared in a metallic mold and photoactivated by one of the four methods: continuous light (520 mW/cm² for 40 seconds); stepped light (150 mW/cm² for 10 seconds followed by 520 mW/cm² for 30 seconds); intermittent light (cycle of 1 second, 1/2 second with the light on and 1/2 second with the light off for 60 seconds at 520 mW/cm²) and Xenon plasma arc (1370 mW/cm² for 3 seconds). Knoop hardness measurements were obtained on the surface, at the depths of 1.5 mm, 2.5 mm, 4.0 mm and at the bottom area by the HMV-2000 microhardness, with a load of 50 grams for 30 seconds. Twenty-five indentations were performed in each sample, and a total of five samples were prepared for each photoactivation method. The obtained values were submitted to ANOVA and Tukey's test at the 5% significance level. The results indicated that (1) for the continuous light method, the values of hardness were statistically superior in the surface area when compared to other areas. For the other methods, there were no statistical differences between the surface area and 1.5 mm; (2) the continuous and stepped light methods showed the highest mean Knoop Hardness Number in all areas; (3) the Xenon plasma arc method was not statistically different from continuous and stepped light ones on the surface at 1.5 mm and 2.5 mm of depth. However, a great decrease in hardness was observed in the deeper areas; (4) the intermittent light method showed intermediate results.

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INTRODUCTION

One of the inherent characteristics of resin composites that imposes restrictions to the restorative technique is polymerization shrinkage. The amount of shrinkage is

very high and creates stress at the tooth-restoration interface that may damage bonding and, therefore, enables the invasion of oral fluids and bacteria.

Several studies have been conducted in an attempt to reduce the rate of stress released by polymerization shrinkage through incorporating new techniques. Koran and Kürschner (1998) tried to minimize this stress by varying the photoactivation method. An initial polymerization in low light intensity, followed by exposure with higher light intensity, could minimize the observed shrinkage. This technique, according to the authors, would promote better marginal adaptation of the restoration due to rearrangement of the polymeric chains during the initial polymerization phase. However, a decreased light exhibition could diminish the composite's degree of cure and, thus, its hardness.

Therefore, aiming development of a new photoactivation technique, Stanford and others (1986) verified the influence of continuous-light versus stepped-light photoactivation concerning superficial and bottom hardness and the effect of photoactivation time. The results showed that the hardness obtained on the surface and at the bottom areas did not differ between the two methods. Similar results were shown by Uno and Asmussen (1991) and Feilzer and others (1995).

Recently, new photoactivation units were introduced in the market, offering reduction of the clinical time required for composite polymerization. This reduction in time is provided by the high light intensity of these units, which can reach around 1370 mW/cm^2 (Peutzfeldt, Sahafi & Asmussen, 2000). However, the decrease in time of composite photoactivation and the technique employed could lead to variations in the physical and mechanical properties of these materials.

Photoactivation achieves an adequate degree of cure in the entire extension of a polymerized composite increment. The Knoop hardness test has been used as an indirect method to verify the degree of cure and polymerization depth of a resin composite.

Therefore, this study determined the effect of four photo-activation methods on the polymerization depth of a restorative resin composite using the Knoop hardness test.

METHODS AND MATERIALS

Z100 restorative composite (3M Dental Products, St Paul, MN 55144, USA) was used to obtain samples submitted to different photoactivation methods. Five samples in a total of 20 samples were prepared for each photoactivation method.

The resin was placed in the cavity (5 mm diameter by 5 mm height) of a split copper mold and was covered with a mylar strip under a glass slab with a static load of 1 kg to remove the excessive material.

Following the glass slab removal, the samples were photoactivated according to one of four methods tested (Table 1): (1) continuous light (520 mW/cm^2 for 40 seconds); (2) stepped light (150 mW/cm^2 for 10 seconds followed by 520 mW/cm^2 during 30 seconds); (3) intermittent light (cycle of 1 second, 1/2 second with the light on and 1/2 second with the light off for 60 seconds at 520 mW/cm^2) and (4) Xenon plasma arc (1370 mW/cm^2 for 3 seconds.

After the polymerization procedure, the samples were removed from the copper mold and stored at 37°C and 95% ± 5% relative humidity for 24 hours. The representative samples of each group were then horizontally placed (lateral position) on a glass plate and polystyrene resin was poured on (Resapol T208, São Paulo, Brazil, 04735-000) to keep them fixed. Each group was flattened with carbide sandpaper of decreasing grit (100, 200, 320, 400, 600 and 1000) on an automated polisher APL-4 (Arotec Ind Com, São Paulo, Brazil, 06709-150) to obtain standardized surfaces. The cure resin was ground and polished until half the samples showed wear, exposing their whole extensions from the superficial tip to the bottom followed by polishing with diamond paste that contained 1 µm and 0.25 µm particles.

The areas that received direct incidence of light were marked so that the Knoop Hardness readings could be performed by an indenter (HMV-2000, Shimadzu, Japan) under a load of 50g for 30 seconds.

Five indentations were performed on five different regions: surface, at depths of 1.5 mm, 2.5 mm, 4.0 mm and at the bottom, in a total of 25 indentations for each sample. The values obtained in micrometers were con-

Table 1: *List of Investigated Photoactivation Methods with Their Outputs and Respective Manufacturers*

Photoactivation Method	Intensity of Light	Equipment	Manufacturer
Continuous Light	520 mW/cm^2 for 40 seconds	XL 3000	3M, St Paul, MN 55144, USA
Stepped Light	10 seconds – 150 mW/cm^2 30 seconds – 520 mW/cm^2	XL 3000	3M, St Paul, MN 55144, USA
Intermittent Light	520 mW/cm^2 in cycles of 1 second, 1/2 second with the light on and 1/2 second with the light off for 60 seconds	Optilux 150 (adapted)	Demetron Research Corp, Danbury, CN 06810, USA
Xenon Plasma Arc	1370 mW/cm^2 * for three seconds	Apollo 95E	DMD, Westlake Village, CA 91362, USA

**According to the manufacturer's information*

verted to Knoop Hardness Number (KHN) by indenter software.

The results of the Knoop hardness test were submitted to the ANOVA and Tukey's test at the 5% significance level.

RESULTS

Table 2 shows the average values for Knoop hardness in each depth for all methods of photoactivation analyzed. It was noted that, generally, there was a decrease in hardness, as depth increased. The continuous, stepped and intermittent light methods presented a more regular pattern of polymerization compared to the Xenon plasma arc method. The Xenon plasma arc method had a significant decrease in hardness values at deeper areas after a regular pattern of polymerization on the surface and at 1.5-mm depth, where no statically significant difference was verified ($p>0.05$).

Table 3 shows the average values for Knoop hardness of the photoactivation methods in each analyzed depth. It was verified that the values found for the continuous and stepped light photoactivation methods did not differ in depth ($p>0.05$). Photoactivation for intermittent light was not statistically different from the Xenon plasma arc except at the bottom areas; it presented statistically lower values compared to the continuous and stepped light methods ($p<0.05$). Photoactivation by Xenon plasma arc was not statistically different from the continuous and stepped light methods on the surface at depths of 1.5 mm and 2.5 mm ($p>0.05$). However, the results showed statistically lower values ($p<0.05$) in deeper areas when compared with the last two methods.

DISCUSSION

In this study, when comparing the Knoop hardness values on the surface at depths of 1.5 mm, 2.5 mm, 4.0 mm and at the bottom for each photoactivation method (Table 2), in general, a decrease in the values of hardness with an increase in depth was observed. The decrease in hardness values could be explained by the difficult light penetration into deeper areas, leading to a lack of polymerization. Therefore, a smaller degree of cure in this area would reduce hardness values. These results agree with De Lange, Bausch and Davidson (1980); Denyer and Shaw (1982); Onose and others (1985); Hansen and Asmussen (1993); Rueggeberg, Caughman and Curtis (1994); Sobrinho and others (2000).

Analyzing photoactivation by the Xenon plasma arc (Table 2) verified that the values obtained for this method on the surface are not statistically different from those at a depth of 1.5-mm ($p>0.05$). However, there was a significant decrease in values at deeper areas and an absence of polymerization at the bottom area.

On the other hand, analyzing the behavior of the other three photoactivation methods in each area (Table 2), it can be concluded that these methods present a more regular polymerization pattern, showing a gradual decrease in Knoop hardness values with an increase in depth.

Similarly, when comparing the methods in each analyzed area (Table 3), the fact that the photoactivation method can be verified by Xenon plasma arc does not present statistically significant differences ($p>0.05$) when compared to the methods of continuous and stepped light on the surface area and at depths of 1.5-mm and 2.5-mm. However, at the deeper areas, these values drop significantly and differ from the two previously mentioned methods. The significant decrease in hardness verified in deeper areas and the most irregular pattern of polymerization using the Xenon plasma arc method can be related to the time of light exposure. These results agree with other studies where the Xenon plasma arc method presented the lowest polymerization depth compared to the conventional method (Lim, Owens & Wells, 2001; Tonioli & others, 2001). Peutzfeldt and others (2000) concluded that the Xenon plasma arc photoactivation method showed similar or inferior polymerization depth compared to the conventional method.

This process can be explained through analysis of the total amount of energy released by each method during polymerization. Energy can be calculated as a product of the equipment's light intensity and the time of irradiation used. This calculation is similar to that used by Peutzfeldt and others (2000), where the unit was J/cm^2. As for the intermittent light method, determining that value is not possible due to the variation in light intensity during the polymerization cycle. However, when comparing the other three methods, the results for continuous light was a value of 20.8 J/cm^2, while for stepped light, it was 17.1 J/cm^2 of released energy. The total amount of released energy by the Xenon plasma arc method was relatively low (4.1 J/cm^2).

Halogen light equipment was used in the continuous and stepped light groups. This presents a broad spectrum of wavelengths. The filter in this equipment promotes partial selection of wavelengths among 400 to 500 nm. The high intensity equipment used was supplied by the manufacturer in two different tips. These tips allowed a specific passage of the wavelengths, 430 and 470 nm, depending on the current photoiniciator in the composition of the material. The composite used in the experiment presents camphorquinone as photoiniciator, activated in a wavelength peak of 468 nm.

Therefore, selective passage of the wavelengths and the high intensity of light of that equipment might have been responsible for the polymerization depth verified to a depth of 2.5 mm even for an exposure of only 3 seconds, when compared with the photoactivation methods by continuous and stepped light that have always presented the highest Knoop hardness values. However, in

depths of more than 2.5 mm, an accentuated decrease in Knoop hardness values can be observed for the Xenon plasma arc method. It can be hypothesized, therefore, that hardness not only depends on the amount of released energy, but also on the intensity and spectrum of emitted light by the photoactivation equipment. As shown in layers of thickness up to 2.5 mm, the Xenon plasma arc method did not differ from the other methods in this study despite their differences in released energy.

A comparative analysis between the results of the photoactivation methods by continuous and stepped light in each area (Table 3) demonstrated that these methods did not differ statistically in any depth ($p>0.05$). Therefore, the complementary exposure (30 seconds) under high light intensity (520 mW/cm^2) of the stepped light method compensates for the lower rate of initial cure obtained by its initial lower light intensity (150 mW/cm^2). Mehl and others (1995) verified the microhardness and marginal adaptation of composite restorations using the photoactivation method by stepped light. In this study, pre-polymerization was carried out on several levels of light intensity. It was noted that a 50% reduction in total intensity in pre-polymerization followed by a normal intensity polymerization for at least 20 seconds caused no significant differences in microhardness and promoted satisfactory marginal adaptation. Other studies have also verified that superficial hardness tends to remain constant when photoactivation methods by continuous and stepped light are compared (Burgess & others, 1999; Koran & Kürschner, 1998).

In all analyzed areas, the values for the intermittent light photoactivation method were found to be statistically inferior to the continuous and stepped light methods. However, when compared to the Xenon plasma arc method, no statistical differences were found except for the bottom area. A possible explanation for the inferior results shown by the intermittent light method could be the methodology and equipment used. The equipment used in this study had the same light intensity for the continuous, stepped and intermittent light methods. Only the last method required modifications. When using intermittent light, the equipment accomplished a cycle of one second: 1/2 second with the light on and 1/2 second with the light off, as previously explained by Obici and others (2002). However, once the light was activated, it did not reach the equipment's maximum intensity immediately (520 mW/cm^2), thus, remaining in low intensity at first. The maximum intensity was briefly reached, though, for an insufficient period, since the light was on for just a 1/2 second according to its cycle. Therefore, the samples that were polymerized by the intermittent light method were subjected to a lesser amount of energy that could lead to a decrease in hardness, as previously verified.

From the results obtained in this study, one may conclude that the stepped light method can be considered an option for photoactivation as an attempt to minimize stress generated by composite polymerization shrinkage and avoid variations in its hardness in several depths. Also, it can be speculated that using a unit with high light intensity (greater than 520 mW/cm^2) for the intermittent light method or a greater exposure time for the light emitted by the Xenon plasma arc could cause the polymerization depth to equal the method used by continuous or stepped light.

CONCLUSIONS

Based on the results obtained, it can be concluded that:

1. For the continuous light method, the values of hardness were statistically superior in the surface area when compared to the other areas. For the other methods, there were no statistical differences between the surface area and 1.5 mm.

Table 2: *Mean Values of Knoop Hardness in Each Appraised Depth for All the Photoactivation Methods*

	Continuous	Stepped	Intermittent	Xenon
Surface	74.62 a (1.60)	72.16 a (2.76)	61.40 a (2.45)	69.90 a (1.98)
1.5 mm	64.02 b (2.97)	65.38 ab (2.72)	54.58 ab (3.85)	61.94 a (3.24)
2.5 mm	58.76 b (4.02)	60.08 b (4.06)	47.76 bc (5.02)	52.39 b (3.68)
4.0 mm	48.82 c (3.64)	53.60 b (4.19)	40.02 c (3.78)	35.58 c (2.30)
Bottom	38.94 c (3.53)	45.16 c (4.05)	25.00 d (1.70)	0.00 d (0.00)

Mean values followed by different letters in the column differ statistically among themselves for the Tukey Test at the level of 5%. ( )—Standard Deviation

Table 3: *Comparative Mean Values of Knoop Hardness for All the Photoactivation Methods in Each Appraised Depth*

	Surface	1.5 mm	2.5 mm	4.0 mm	Bottom
Continuous	74.62a (1.60)	64.02a (2.97)	58.76a (4.02)	48.82a (3.64)	38.94a (3.53)
Stepped	72.16a (2.76)	65.38a (2.72)	60.08a (4.06)	53.60a (4.19)	45.16a (4.05)
Intermittent	61.40 b (2.45)	54.58 b (3.85)	47.76 b (5.02)	40.02 bc (3.78)	25.00 b (1.70)
Xenon	69.90ab (1.98)	61.94ab (3.24)	52.39ab (3.68)	35.58 c (2.30)	0.00 c (0.00)

Mean values followed by different letters in the column differ statistically among themselves for the Tukey's Test at the level of 5%. ( )—Standard Deviation

2. The bottom area has always presented the smallest values of Knoop hardness in all methods, except for the continuous light method, which did not differ from the area of 4 mm.
3. The photoactivation methods of continuous and stepped light did not differ from each other in any analyzed area and presented higher values from those found for the intermittent light photoactivation method.
4. The Xenon plasma arc method did not statistically differ from continuous and stepped light on the surface and at depths of 1.5 mm and 2.5 mm. However, a significant decrease in hardness was observed in the deeper areas.
5. The intermittent light method showed intermediate results of mean hardness number compared to the other methods.

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Influence of Curing Methods and Materials on the Marginal Seal of Class V Composite Restorations *In Vitro*

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Clinical Relevance

Selection of a low shrinkage resin based composite appears to be more important for achieving a good marginal seal of Class V restorations than using specific light irradiation procedures.

SUMMARY

The study tests the hypothesis that soft-start irradiation improves, whereas, high intensity irradiation compromises the margin quality and marginal seal of Class V resin based composite [RBC] restorations. Box-shaped Class V cavities were prepared in extracted, human third molars with cervical margins located apical to the CEJ. Cavities were restored using a multi-step bonding agent (Optibond FL, Kerr), a thin layer of flowable resin composite and two increments of fine hybrid resin composite (Filtek Flow/Filtek Z250, 3M ESPE; Revolution f2/Herculite XRV, Kerr). Light irradiation was performed using either the standard (40 seconds) or the soft-start mode (40 seconds with exponential increase) of a quartz tungsten halogen or an LED curing light (Elipar Trilight, Elipar Freelight, 3M ESPE); for high intensity irradiation, a Plasma Arc curing unit was used with three irradiations of three seconds (Apollo 95E, DMDS). After 30 days of water storage and thermal cycling (n=2500, 5-55°C), margin quality was assessed in the SEM using the replica technique and marginal seal was evaluated using dye penetration ($AgNO_3$ 50%). Few differences were observed between the light curing protocols. However, less leakage was observed in the case of the lower shrinking RBC Filtek Z250.

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INTRODUCTION

Even today, with all the advances in dental materials, the major disadvantage of resin-based composites [RBC] is shrinkage during polymerization, which creates stress at restoration margins and, consequently, may result in margin fracture, marginal leakage and recurrent caries. In methacrylate-based systems, incorporating high volumetric filler loads (Yukitani & others, 1997) has reduced but not eliminated shrinkage. Alternative resin systems may allow the production of non-shrinking restorative materials in the future but are not yet commercially available. A variety of concepts have been recommended to reduce the side effects of polymerization contraction.

By using a variety of application techniques, the overall volumetric contraction can hardly be reduced because it correlates to the degree of cure (Rueggeberg & Tamareselvy, 1995; Silikas, Eliades & Watts, 2000). Under-irradiating photo-activated RBC may reduce their shrinkage, but at the same time, it will compromise their mechanical properties and biocompatibility (Rueggeberg & Craig, 1988; Pearson & Longman, 1989; Caughman & others, 1991; Ferracane, 1994), and, therefore, cannot be recommended.

Only prior to gel-formation, shrinkage will not create stress but rather may be compensated by flow within the material (Davidson & de Gee, 1984; Feilzer, de Gee & Davidson, 1990). Starting irradiation at reduced light intensity allows more time for flow to occur and was found to reduce contraction stress (Bouschlicher, Vargas & Boyer, 1997; Sakaguchi & Berge, 1998; Bouschlicher & Rueggeberg, 2000a; Bouschlicher, Rueggeberg & Boyer, 2000b) and improve the marginal seal of RBC restorations (Uno & Asmussen, 1991; Feilzer & others, 1995; Unterbrink & Muessner, 1995; Mehl, Hickel & Kunzelmann, 1997; Yoshikawa, Burrow & Tagami, 2001). As an alternative, curing speed may be reduced by modifying the photo-activating system (Venhoven, de Gee & Davidson, 1996; Watts & Al Hindi, 1999). On the other hand, not all investigations have confirmed the reduction of polymerization contraction (Sahafi, Peutzfeldt & Asmussen, 2001; Yap, Soh & Siow, 2002) and the improvement of marginal seal due to soft-start irradiation (Friedl & others, 2000; Hasegawa & others, 2001a).

According to Hook's law, contraction stress is not only influenced by shrinkage strain but it also depends on the modulus of the material. In accordance with their lower moduli, microfilled RBC were found to produce lower shrinkage stress (Bouschlicher & others, 1997) and a better margin quality (Kemp-Scholte & Davidson, 1990a) than hybrid RBC. The placement of low modulus intermediate layers between the dentin adhesive and the RBC turned out to be even more effective for improving the marginal seal of restorations (Kemp-Scholte & Davidson, 1990a; Kemp-Scholte & Davidson, 1990b). In recently published studies, this effect was verified only for particular brands (Ernst & others, 2002) or was not confirmed at all (Jain & Belcher, 2000; Loguercio & others, 2002).

Plasma arc curing units provide higher radiation intensities than quartz tungsten halogen lights and, therefore, are claimed to activate RBC in shorter periods of time, thus, saving clinicians' time. Due to the immediate start and fast progression of polymerization contraction, adverse effects on the margin quality of fast-cured restorations may be anticipated. However, this has not yet been confirmed experimentally (Stoll & others, 2000; Hasegawa & others, 2001b).

The latest generation of curing units use light emitting diodes (LEDs) as sources of light. LEDs feature a narrow spectral radiometric output and can be selected to optimally match the absorption characteristics of the most widely used photo-initiator, namely, camphoroquinone [CQ] (Mills, Jandt & Ashworth, 1999; Jandt & others, 2000; Knezevic & others, 2001; Kurachi & others, 2001). Due to the absence of infrared radiation, heating of the irradiated object is reduced to a minimum. The first commercially available LED curing units provide lower radiometric output levels compared to QTH or PAC lights and, therefore, are not recommended for reduced irradiation periods.

This study tests the hypothesis that soft-start irradiation improves, whereas, high intensity irradiation compromises the margin quality and marginal seal of Class V RBC restorations *in vitro*. In addition, the degree of cure produced by the different irradiation protocols is monitored. Standard and slow-start irradiation are performed using both a QTH and an LED unit, whereas, a PAC light is used for high intensity irradiation. Also, the study tests the hypothesis that restoration materials also influence the margin quality. Therefore, two brands of RBC were selected for the study so as to represent different amounts of polymerization shrinkage.

METHODS AND MATERIALS

Box-shaped Class V-cavities (5 x 3 x 2mm) were prepared on the buccal surfaces of extracted human third molars with cervical margins located slightly apical to the CEJ. At the coronal margin a bevel of 1 mm width was prepared. Enamel and dentin were etched using 35% H_3PO_4-gel (Ultra-Etch, Ultradent Products, South Jordan, UT 84095, USA) for 60 and 15 seconds, respectively. The cavities were carefully rinsed and excess

Table 1: *Resin Based Composites Used in the Study*

Name	Manufacturer	Type	Lot	Shade	Modulus[1]	Filler Load [by weight][1]	Filler Load [by volume]	Polymerization Shrinkage
Herculite XRV	Kerr	regular	006A30	A2 Dentin	11.0 GPa	87.1%	59%[1]	2.9%[3]
Revolution f2	Kerr	flowable	1-1086	A2	4.6 GPa	55.1%	41%[2]	3.9%[1]
Filtek Z250	3M ESPE	regular	1KF	A2	11.2 GPa	82%	60%[1]	2.4%[3]
Filtek Flow	3M ESPE	flowable	3700	A2	5.8 GPa	68%	47%[1]	4.1%[4]

[1] *Specification of the manufacturer*
[2] *St-Georges & others (2002)*
[3] *Determined using the deflecting disk technique 60 minutes after irradiation for 40 seconds at 800mW/cm² (Hofmann & others, 2002)*
[4] *Determined using the deflecting disk technique, specification of the manufacturer.*

water was removed using suction, leaving the dentin moist. A three-step dentin adhesive (Optibond FL, Kerr, Orange, CA 92867, USA) was applied according to the manufacturer's instructions and light-cured for 20 seconds using a QTH light at 800 mW/cm² (TriLight, 3M ESPE, 82229 Seefeld, Germany).

A small amount of flowable resin composite was applied using the steel syringe tips provided by the respective manufacturer and was spread over the dentin surfaces of the cavity using a dental explorer so as to achieve a thin, even layer. The bulk of the cavity was restored placing two increments of fine hybrid resin composite as shown in Figure 1. Two combinations of materials were used: Revolution f2/Herculite XRV (Kerr) and Filtek Flow/Filtek Z250 (3M ESPE), shade A2. Herculite features a higher contraction strain and a slightly lower modulus compared to Z250 (Table 1). Light irradiation was performed according to following protocols:

1. Halogen standard: 40 seconds @ 800mW/cm² using a QTH light (TriLight) [QTH Std].
2. Halogen ramp: gradual increase between 100 and 800 mW/cm² within 15 seconds, 25 seconds @ 800 mW/cm² (TriLight) [QTH Ramp].
3. LED standard: 40 seconds @ 350 mW/cm² using a LED curing unit (FreeLight, 3M ESPE) [LED Std].
4. LED ramp: gradual increase between 18 and 350 mW/cm² within 10 seconds, 30 seconds @ 350 mW/cm² (FreeLight) [LED Ramp].
5. High intensity irradiation: three irradiations of three seconds @ 1570 mW/cm² using a Plasma Arc curing unit (Apollo 95E, DMDS, 11560 Fleury d'Aude, France) [PAC].

Halogen standard irradiation was intended to serve as the control group for the study. The curing tip was always placed as close as possible to the cavities without touching the uncured materials. All restorations were placed by one operator following appropriate prestudy training. Restorative procedures were standardized as much as possible in a clinically related study. Despite standardization of the cavity dimensions, the variations typically found in extracted teeth did not allow for standardizing the exact volume of each increment.

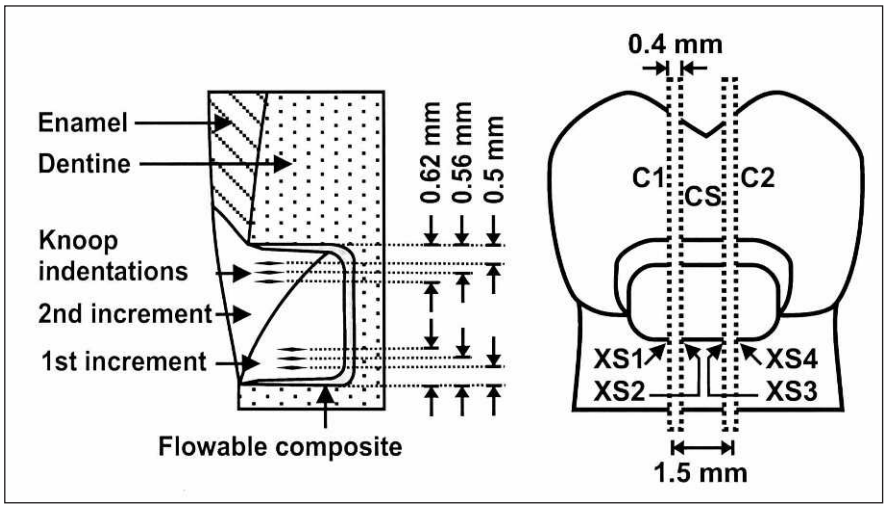

Figure 1. *Left: Cross section of the restorations; Right: Sectioning of the specimens for the evaluation of dye penetration (C1: first cut, C2: second cut, CS: central section, XS1/2/3/4: cross section 1/2/3/4).*

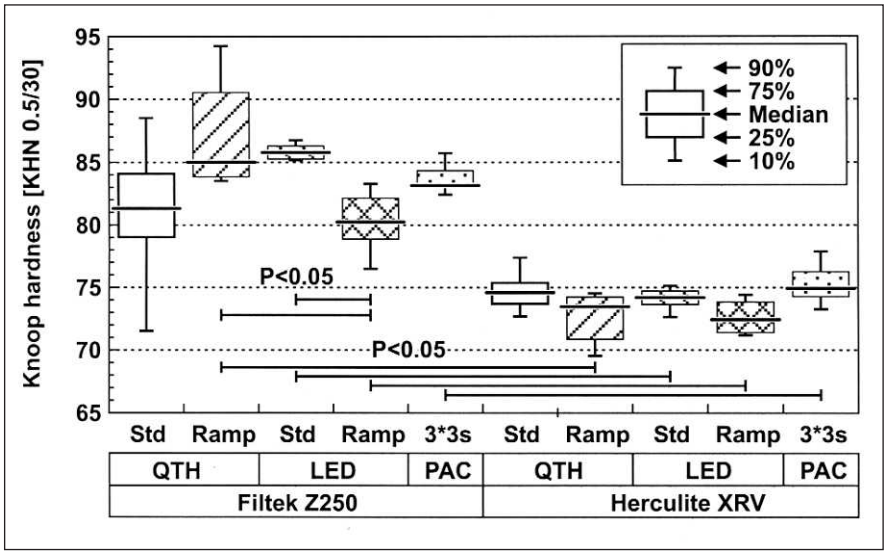

Figure 2. *Knoop hardness [KHN 0.5/30] in the different treatment groups. The horizontal lines with delimiters specify groups significantly different at a level of* p*<0.05 (results of multiple paired U-tests with Bonferroni-Holm adjustment).*

Specimens were stored in deionized water at 37°C for 30 days, and 2500 cycles between 5 and 55°C with a dwell time of 30 seconds at either temperature were carried out after day 15.

Margin quality was assessed in the SEM (DSM 940, Zeiss, 73447 Oberkochen, Germany) using the replica technique. The evaluation criteria were "Continuous margin," "Marginal opening" and "Swelling out." Areas showing underfilled margins or excess restorative material and areas not scorable due to limitations of the replica technique were excluded from evaluation. The length of the margin showing each of the different criteria was expressed as the percentage of the total cervical and coronal margin length.

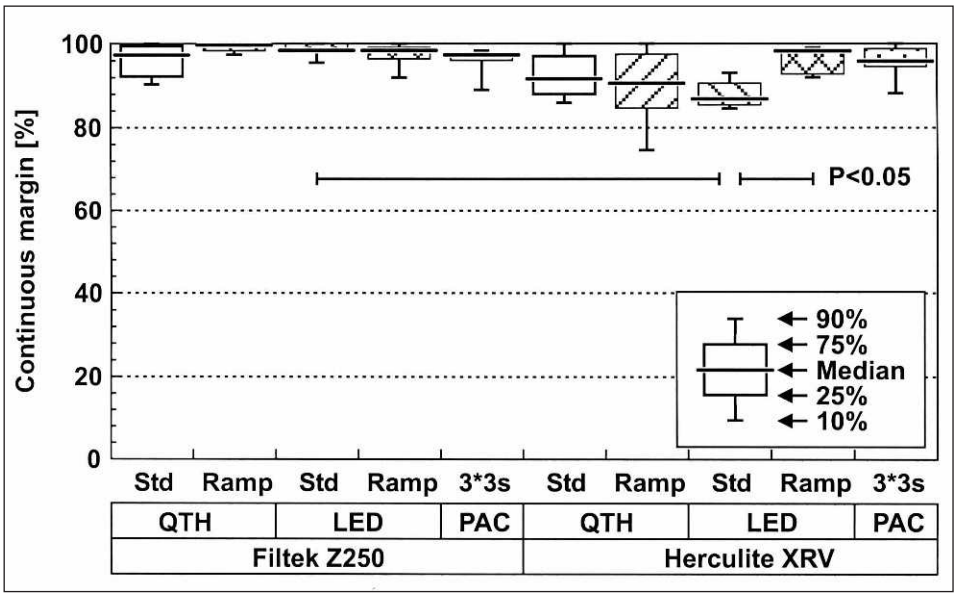

Figure 3. *SEM margin quality (percentage of "Continuous margin") at the coronal margins in the different treatment groups. Statistics as in Figure 2.*

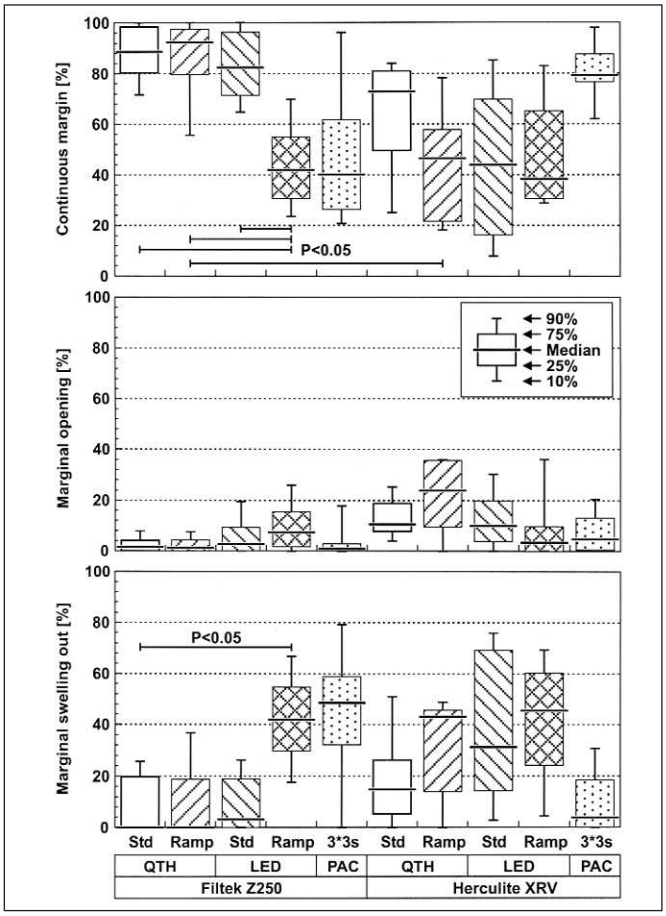

Figure 4. *SEM margin quality at the cervical margins in the different treatment groups. Top: percentage of "Continuous margin," middle: percentage of "Marginal opening," bottom: percentage of "Marginal swelling out." Statistics as in Figure 2.*

Specimens were coated using nail varnish, leaving the area 1 mm adjacent to the restoration and the restoration itself exposed. After immersion in 50% $AgNO_3$ solution for two hours, the specimens were cleaned using tap water and stored in developing solution for six hours using transparent vials placed on a slide sorter to provide simultaneous illumination. Two cuts 1.5 mm apart were made parallel to the long axis of the tooth and perpendicular to the restoration (Figure 1) using a water-cooled diamond saw (Woco 50 med, Conrad, 38678 Clausthal-Zellerfeld, Germany). Depth of dye penetration was measured on each of the four cross-sections using a travelling light microscope (Tessovar, Zeiss), and the maximum penetration value was recorded for the particular specimen.

On one surface of the central section, the surface hardness (Knoop) of the restoration was evaluated using a hardness tester (3212, Zwick, 98079 Ulm, Germany) applying a load of 5 N for 30 seconds. Three indentations were made 1 mm below the surface of the restoration at a distance of 0.5, 0.56 and 0.62 mm from both the cervical and the coronal cavity walls, with the results of the three cervical and coronal indentations being averaged separately.

For each combination of resin composite and curing mode, 10 specimens were prepared. Differences between the treatment groups were tested for statistical significance using multiple pairwise U-tests (Mann-Whitney) with Bonferroni-Holm adjustment at a $p<0.05$ level of significance. The following tests were selected *apriori*: comparison of any two curing modes for each of the resin composites, separately, and comparison of the two composites for each curing mode, separately. The differences between the hardness at the coronal and the cervical margins were tested for significance using the Wilcoxon test at a level of significance of $p<0.05$ for each treatment group, separately.

RESULTS

No significant differences were observed between the hardness at the coronal and the cervical margin (Wilcoxon: $p>0.05$). Consequently, the results were averaged. Figure 2 displays the hardness of the restorations for the different treatment groups. The data from this study, especially the results of the SEM evaluation, do not follow a normal distribution and are, therefore, presented as box plots. The horizontal lines represent

the median values, the boxes specify the inter-quartile range, whereas, the whiskers indicate the 10 and 90 percentiles, respectively. Z250 featured a higher hardness than Herculite (U-Test: $p<0.05$) for all curing modes except QTH Std. In the case of Z250, LED Ramp irradiation produced lower hardness values compared to QTH Ramp or LED Std irradiation. Regarding Herculite, no significant differences between curing modes were observed.

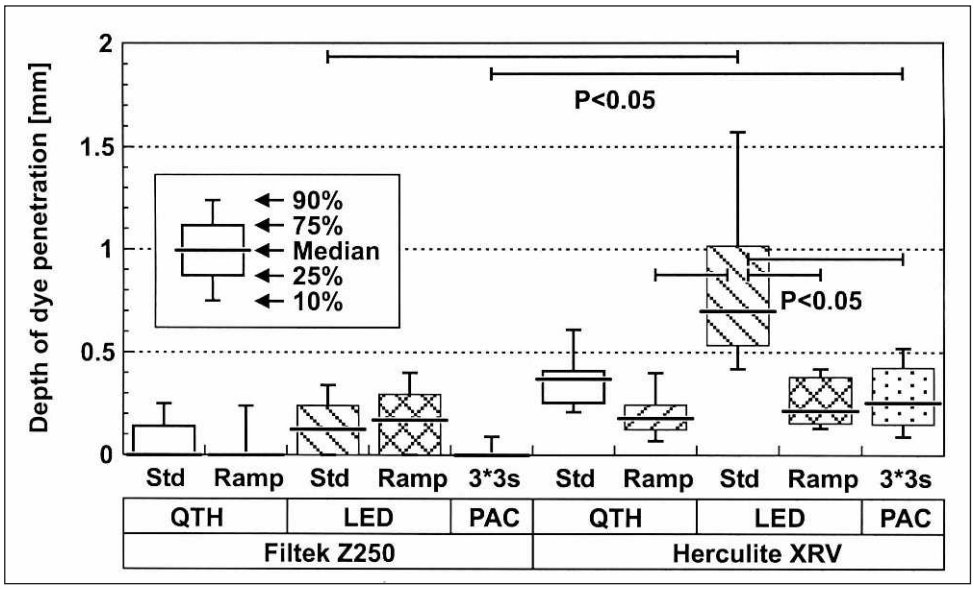

Figure 5. *Depth of dye penetration [mm] at the cervical margins in the different treatment groups. Statistics as in Figure 2.*

Margin quality at the coronal margin was very good, with median values of continuous margin ranging between 87% and 100% (Figure 3). Only LED standard irradiation of Herculite produced a lower percentage of continuous margin compared to LED Std-cured Z250 and to LED ramp-irradiated Herculite ($p<0.05$). The cervical margins featured lower percentages of continuous margin (Figure 4 top) compared to the coronal margins. In the case of Z250, LED ramp curing resulted in less continuous margin compared to LED standard and QTH standard and ramp irradiation. After QTH ramp irradiation, Z250 restorations featured more continuous margin than equally cured Herculite fillings. In the case of Herculite, the differences between the curing modes failed to reach the level of statistical significance.

The median values of marginal opening ranged between 1% and 7% in the case of Z250 and between 3% and 24% in the case of Herculite (Figure 4 middle); however, none of the differences was statistically significant. Swelling out at cervical margins reached median values of up to 48% (Figure 4 bottom), for Z250; it was significantly higher following LED ramp irradiation compared to QTH standard curing. No significant differences between curing modes were observed for Herculite.

Dye penetration values were higher for Herculite compared to Z250 (Figure 5); this difference was significant for the curing modes LED standard and high intensity irradiation (PAC). For Herculite, LED standard irradiation produced more leakage than any other curing mode except QTH standard curing. No significant differences were observed between curing modes in the case of Z250.

DISCUSSION

The data presented above failed to demonstrate that slow-start irradiation improves, whereas, high intensity irradiation compromises margin quality and marginal seal of RBC restorations. This contradicts reports by Uno and Asmussen (1991), Feilzer and others (1995), Unterbrink and Muessner (1995), Mehl and others (1997) and Yoshikawa and others (2001) and corresponds with those by Friedl and others (2000), Hasegawa and others (2001a) and Sahafi and others (2001).

These conflicting results may be explained by the differences between the irradiation protocols used, especially regarding the intensities used for the initial irradiation. Unterbrink and Muessner (1995) and Feilzer and others (1995) compared continuous low intensity irradiations of 250mW/cm^2 to higher intensity irradiations of 450mW/cm^2 or 650 mW/cm^2, that is, 56% or 38% of the maximum intensity, respectively. Uno and Asmussen (1991), Mehl and others (1997) and Yoshikawa and others (2001) report optimum margin qualities when initial irradiation is performed at 50%, 70% or 45% of the final intensity.

In contrast, the studies not confirming the advantages of soft-start irradiation used the pre-set step-curing protocol of a commercially available curing light (Elipar HiLight, 3M ESPE) with an initial irradiation of 10 seconds @150mW/cm^2 followed by 30 seconds @800mW/cm^2 (Friedl & others, 2000). Hasegawa, and others (2001a) specify intensities of 100 and 600mW/cm^2, Sahafi and others (2001) intensities of 100 and 750 mW/cm^2, respectively. In these studies, the initial irradiation features only 19%, 17% or 13% of the final intensity.

These considerations might elicit the hypothesis that the initial intensity should be about 50% of the final intensity rather than below 20% to produce a favorable

margin quality. On the other hand, with step-curing, contraction stress and contraction strain start later compared to full intensity (Bouschlicher & others, 2000b) or to full and half intensity irradiation (Hofmann & others, 2003). Therefore, more time is available for stress relaxation by flow and a favorable effect on margin quality could be expected.

The soft-start protocols of this study (ramp irradiation) started at 5% (LED) or 12.5% (QTH) of the final intensity, representing even lower initial intensities compared to step-curing as discussed above. With these protocols, contraction strain starts later and progresses more slowly compared to step-curing or standard irradiation at full or at half intensity (Hofmann & others, 2003; Hofmann, Hugo & Klaiber, 2002). These observations stimulated the hypothesis of this study which, however, was not supported. In addition, only minor differences between QTH and LED irradiation were observed.

In contrast, high intensity irradiation using PAC lights produces an immediate start and a fast progression of contraction strain (Hofmann & others, 2003). However, larger percentages of marginal opening or more dye penetration were not observed in this study, confirming previous reports regarding Class II and Class V restorations (Stoll & others, 2000; Hasegawa & others, 2001b). Evaluation of Knoop hardness confirmed that PAC irradiation provided an equivalent degree of cure compared to other curing protocols. Consequently, the margin quality of PAC irradiated restorations is not achieved at the expense of compromised mechanical properties and biocompatibility.

One study that compared different irradiation methods and different dentin adhesives found the latter to be more important for margin quality (Hasegawa & others, 2001a). The dentin adhesive selected for this study has been shown to provide excellent bond strength to dentin in many studies. In addition, applying a thin layer of flowable RBC may have contributed to perfectly polymerizing the dentin adhesive by replacing its oxygen inhibited layer without creating considerable contraction stress (Rueggeberg & Margeson, 1990; Unterbrink & Liebenberg, 1999). Moreover, the strain capacity of the flowable RBC may have helped to compensate for contraction stress during polymerization of the bulk of the restoration (Kemp-Scholte & Davidson, 1990b). It may be speculated that the combination of a highly efficient dentin adhesive with an intermediate layer of flowable RBC may have prevented the detection of irradiation effects on margin quality. On the other hand, several recently published studies did not observe better marginal seals of restorations compared to those without intermediate layers of flowable resin composites (Jain & Belcher, 2000; Loguercio & others, 2002), at least not for all combinations of materials (Ernst & others, 2002). However, applying flowable RBC was shown to improve internal adaptation and reduce the occurrence of interfacial voids (Frankenberger & others, 1999; Estafan, Estafan & Leinfelder, 2000; Chuang & others, 2001).

The mechanical properties of the two brands of RBC selected for this study suggest controversial influences on margin quality. The lower shrinkage strain of Filtek Z250 may be an advantage over Herculite (2.4% vs 2.9% one hour after QTH standard irradiation for 40 seconds @800mW/cm^2; Hofmann & others, 2002) and should favorably affect margin quality. On the other hand, the immediate start and faster progression of contraction strain observed for Z250 and its slightly higher modulus producing higher contraction stress might adversely affect restoration margins. In fact, Z250 restorations showed less dye penetration for all curing methods (statistically significant for LED Std and PAC) and higher percentages of continuous margin (except for PAC; statistically significant for QTH ramp). These results support the second hypothesis of this study and may indicate that a low shrinkage material may be more important to achieving a good marginal seal than its modulus, shrinkage strain kinetics or variations effected by light curing techniques.

CONCLUSIONS

Within the limits of this study, soft-start irradiation did not improve, whereas, high intensity irradiation did not compromise margin quality and marginal seal of Class V RBC restorations. Using low-shrinkage materials appears to be more important for a good marginal seal than variations created by different light curing techniques.

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An Assessment of Encapsulated Versus Hand-Mixed Glass Ionomer Restoratives

GJP Fleming • DM Zala

Clinical Relevance

When compared with a conventional mixer, Rotomix mixer significantly decreased the level of porosity of selected glass ionomer restoratives; however, the initial viscosity of the system determined the efficacy of the mixing process and the porosity levels achieved.

SUMMARY

Capsulation should enable uniform proportioning and mixing of dental restoratives so that functional properties of the cementitious mass will not be susceptible to clinically induced variability. Mechanical mixing induces a definite pore distribution determined by the viscosity of the system. This study evaluated the mixing process on the performance of a range of glass ionomer dental restoratives.

Mean compressive fracture strengths and standard deviations and the associated Weibull Moduli (m) were determined for six glass ionomer restoratives that were either encapsulated or mixed by hand. Working characteristics were assessed using an oscillating rheometer. Scanning electron microscopy and image analysis was used to investigate the influence of the mixing method on pore distribution.

The fracture strength data for some encapsulated restoratives resulted in significant differences compared with hand-mixing. Rotomix (compared with the Capmix mechanical agitator) resulted in increased Weibull moduli and 10% failure stress for the two restoratives that were investigated. Encapsulated restoratives that were prepared utilizing Rotomix or Capmix resulted in no significant differences for working characteristics; however, the setting time for the ChemFlex in Caps was extended compared with the hand-mixed ChemFlex.

Not all restoratives had reduced porosity and improved performance following mixing with a Rotomix. This suggested that optimization of the initial viscosity of the system by manipulating the individual proportions of the constituents may not have been appropriate for all the restoratives investigated. The increased viscosity for hand-mixed ChemFlex prepared to a consistency of 3.8 g/ml compared with encapsulated ChemFlex in Caps prepared to a consistency of 3.5 g/ml was responsible for the reduced setting time.

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INTRODUCTION

Dental restoratives are often placed in the posterior region of the mouth and have to withstand masticatory

forces and a wide range of thermal variations from ingested foods and drinks. It has been suggested that the optimum posterior glass ionomer restorative cement properties (consistency, solubility and disintegration, compressive strength, film thickness and setting characteristics) may be compromised by variations in temperature and relative humidity normally encountered in clinical practice when the materials are hand-mixed (Billington, Williams & Pearson, 1990). Capsulation enables uniform proportioning and mixing of the cement so that the properties of the restorative may be less susceptible to clinical variation. Encapsulated glass ionomer luting cements have been used since 1978 and enable the operator to provide cements with optimized properties under conditions normally encountered in clinical practice (Nomoto & McCabe, 2001; Mitchell & Douglas, 1997; Mount, 1994). However, a recent study that evaluated the effect of the mixing method on the compressive strength and porosity of dental glass ionomer cements (Nomoto & McCabe, 2001) suggested that although the method of mixing can markedly influence material properties, the relationship is not simple. Variations between materials were reported in the mixing method and properties, and the overall effect was dependent on the powder/liquid ratio, the initial viscosity of the cementitious mass and the mixing technique (Nomoto & McCabe, 2001).

Mechanical mixing of an encapsulated zinc phosphate luting cement resulted in air entrapment, with the level of viscosity determined by the mixing system (Fleming & others, 1999). Furthermore, the resultant pore distribution was compounded since the zinc phosphate cement-forming reaction was exothermic, as is the case for all acid-base dental cements. The main problem appears to relate to moderating the exothermic cement forming reaction and avoiding the formation of a porous friable weak cementitious mass indicative of vaporization porosity commonly seen with exothermic cements (Fleming, Landini & Marquis, 2002). These results highlight the necessity for further investigations to realize the properties of encapsulated cements produced by a process of mechanical mixing. While it is possible that the mechanical mixing process will induce a definite pore distribution related to the viscosity of the powder/liquid ratio, recently developed and marketed mechanical agitators claim reduced porosity in the cementitious mass. Conventional mechanical agitators such as Capmix (ESPE, Seefeld, Germany) manipulate the material mechanically in a vibratory mixer at between 4500 and 5000 cycles per minute (Bass & Wing, 1988). Rotomix (ESPE, Seefeld, Germany) is reported to employ a combination of rotational and centrifugal action to reduce porosity within the cement mass (Nomoto & McCabe, 2001). The manufacturers suggest that the capsule is maintained in a horizontal position during mixing, and the cementitious mass is manipulated in all directions so that, following rotational mixing, the mass is centrifuged for three seconds and porosity is reduced.

This study evaluated the process of mixing on the performance (in terms of compressive strength, porosity levels, cement morphology and working characteristics) of a range of glass-ionomer dental restoratives. A recently developed mixing machine (Rotomix, 3M ESPE, Seefeld, Germany) was compared with both its predecessor (Capmix, 3M ESPE) and the conventional hand-mixed counterparts, mixing the recommended proportions of powder/liquid.

METHODS AND MATERIALS

Table 1 lists the manufacturers' details and compositions of the powder and liquid constituents of the encapsulated and hand-mixed restoratives.

Cement Manipulation

The encapsulated and hand-mixed glass ionomer restoratives were manipulated according to manufacturers' instructions in a controlled environment (23 ± 1°C and 50 ± 5% relative humidity).

Encapsulated Restoratives

The capsules (KetacFil Plus Aplicap [3M ESPE], ChemFlex in Caps [Dentsply, DeTrey, Konstanz,

Table 1: *Manufacturers' Details on the Different Cement Types Under Investigation and the Recommended Ratios of Powder to Liquid*

Cement Type (Shade A3)	Manufacturers Details	Lot #	Mixing Ratio (g/ml)
KetacFil Plus Aplicap Encapsulated	3M ESPE, Seefeld, Germany	FW0063297	3.2/1.0
ChemFlex in Caps Encapsulated	Dentsply DeTrey, Konstanz, Germany	0103000197	3.5/1.0
Fuji IXGP FAST Encapsulated	GC Corporation, Tokyo, Japan	0106192	3.6/1.0
KetacFil Plus Hand Mixed	3M ESPE, Seefeld, Germany	101894	3.2/1.0
ChemFlex Hand Mixed	Dentsply DeTrey, Konstanz, Germany	0104000262	3.8/1.0
Fuji IXGP Hand Mixed	GC Corporation, Tokyo, Japan	0104031	3.6/1.0

Germany] and Fuji IXGP FAST [GC Corporation, Tokyo, Japan]) were activated for the appropriate time to rupture the membrane that separates the powder and liquid constituents that were placed in the appropriate mechanical agitator (Rotomix or Capmix) and vigorously mixed for an appropriate time (8 and 10 seconds, respectively). After mixing, the capsule was placed in an applicator and the resultant mass extruded by activating the lever.

Hand-Mixed Restoratives

Three glass ionomers (KetacFil Plus [3M ESPE], ChemFlex [Dentsply, DeTrey] and Fuji IXGP [GC Corporation]) were hand-mixed with the relative proportions of powder and liquid constituents determined from the manufacturers' data sheets. The relevant powder contents (for 0.25 ml of liquid) were weighed on a balance accurate to 100 µg. The appropriate volume of liquid was measured using a micropipette (Gilson, Villiers-le-Bel, France). The powder was separated into two halves, one-half was added to the liquid and mixed with a linear stirring motion within the first time increment before the remainder of the material was added, using a non-corrodible stainless steel mixing spatula. The total mixing time was within the manufacturers' instructions reported in the data sheets.

Compressive Strength

A single operator prepared the standard cylindrical encapsulated and hand-mixed glass ionomer specimens (height 6.0 ± 0.1 mm and diameter 4.0 ± 0.1 mm) for compressive testing using a Teflon split-mold assembly capable of holding up to five samples (Fleming & others, 2001, 1999). The mold was placed on the base of an aluminum jig that had been covered with an acetate strip to prevent the setting cement from adhering to the jig. The Teflon split-mold was coated prior to filling with a PTFE-dry film lubricant (RS Components, Northants, England) to facilitate removal of the hardened cement specimens. When preparing the glass ionomer restoratives, the powder was placed on one end of a cooled glass slab and manipulated with the appropriate volume of liquid according to the manufacturers' instructions. Within 60 seconds of mixing, the cement was transferred and packed to slight excess into the split-mold. The largest convenient portion of the hand-mixed glass-ionomer was conveyed to the mold and applied to one side with a spatula to consolidate the cement and avoid trapping air (Fleming & others, 2001, 1999). The encapsulated glass ionomer restoratives were applied to the mold directly from the syringe as recommended by the manufacturers'. The samples were covered with an acetate strip and isolated from the surrounding atmosphere with a glass slab and the apparatus was sealed with a G-clamp. Not more than two minutes after completing mixing, the entire assembly was transferred to a water bath maintained at 37 ± 1°C in an attempt to more closely simulate oral conditions. One hour after completing the mixing, the specimens were removed from the mold and the ends were ground on P800 carborundum (Silicon Carbide) abrasive paper (Struers, Glasgow, Scotland) using water as a lubricant to form a uniform contact between the specimens and test apparatus. The specimens were returned to the water bath and stored "wet" for an additional 23 hours before compressive loading 24 hours after completing the mixing. With the flat ends covered, the specimens were placed with a piece of wet filter paper to ensure that they were tested "wet." A compressive load was applied to the long axis of the specimen using "Instron Tensile Testing" apparatus (Instron Model 1185, High Wycombe, England) at a cross-head speed of 1 mm/minute. The maximum load to failure was recorded and the procedure repeated so that a minimum of 30 cylindrical specimens had been fractured for each sample group under investigation.

Working and Setting Times

The setting characteristics were determined using an oscillating rheometer (Bovis, Harrington & Wilson, 1971). Movement of the lower plate of the rheometer varied with the viscosity of the material and was measured by means of a transformer with an electronic oscillator and a built-in demodulator that provided DC output that was fed to a chart recorder. A zero time mark was made on the recording paper and the recorder started at the beginning of the mix. The specimens, mixed according to manufacturers' instructions within 20 seconds of completing the mixing, were transferred to the lower plate of the rheometer. The upper plate was lowered into position so that the thickness of the material between the plates was 1 mm. This produced a trace on the chart recorder that was wide when the material was fluid, with the width decreasing as the material set (Bovis, Harrington & Wilson, 1971). Wilson (1966) empirically assessed the working time as the time at room temperature (23 ± 1°C) when the width of the rheometer trace was approximately 95% of the initial width (Figure 1a). The setting time was when the mouth temperature rheometer trace (37 ± 1°C) became a straight line (Figure 1b). Three working and three setting time measurements were recorded for the encapsulated and hand-mixed restoratives.

Scanning Electron Microscopy (SEM)

Cylindrical specimens similar to those prepared for compressive testing were embedded transversally in a cold-cure resin prior to grinding and polishing. Grinding was achieved on successively finer grades of carborundum (Silicon Carbide) abrasive papers (P220, P500, P800 and P1200 for 25 seconds followed by P2400 and P4000 for 45 seconds) using an alcohol-based lubricant (DP-Blue, Struers, Glasgow, Scotland) on a Dap-7 Pedemin polishing machine (Struers,

Copenhagen, Denmark) at a force of 20 N. Polishing was completed using a DP Dac cloth with 6 µm diamond paste and lubricated with DP-Blue at a force of 25 N for 240 seconds (Fleming & others, 2001, 1999). The polished samples were mounted on aluminum stubs and gold-coated for 45 seconds using Desk II gold coating apparatus (Denton Vacuum, NJ 08057, USA). Three specimens were prepared for each material, and a strip of silver "dag" was painted from the sample down the mold to contact the stub. SEM examination was performed using a JEOL JSM 5300 LV (JEOL Ltd, Akishima Tokyo, Japan) scanning electron microscope in secondary electron mode to identify differences between morphology and microstructure of the restoratives.

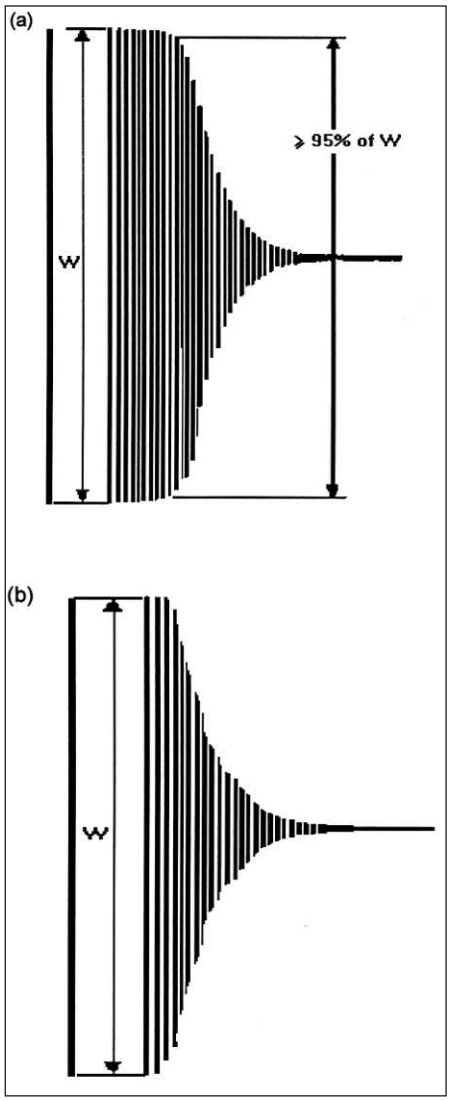

Figure 1. *Oscillating rheometer traces used to determine the setting characteristics of dental restoratives: (a) the working time was the time taken to reach approximately 95% of the initial trace width at 23 ± 1°C, and (b) the setting time was the time taken for the mouth temperature rheometer trace (37 ± 1°C) to deviate to the final straight portion.*

Image Analysis

For each material three cylinders were embedded in a cold-cure resin and ground with successively finer grades of carborundum abrasive papers prior to completing polishing according to the procedure outlined above. Cylinders were serially-sectioned transversally in order to maximize the number of fields in the analysis, and the resultant data was interpreted according to stereological methods (Weibel, 1979a,b) in accordance with the procedure described previously (Fleming & others, 2002; 1999). The procedure was applied in two random non-overlapping locations from the surface of each specimen and repeated approximately every 0.4 mm in depth. The spacing of 0.4 mm was utilized as this value was greater than the maximum pore diameter (determined empirically in a preliminary sample set) to prevent counting pores twice (Fleming & others, 2002; 1999). Given this spacing and accounting for the grinding and polishing of the top surface of the cylindrical specimens, the maximum number of sections was eight, so that a total of 16 fields per cylinder were analyzed. Optimas, version 4.0 (Media Cybernetics, Silver Spring, MD 20910, USA) automatically isolated the pores identified on a transmitted light microscope (Leitz, West Germany) fitted with a Cohu High Performance 4910 Series CCD Camera (San Diego, CA, USA) at 4x magnification. The values were based on the image intensity and returned the largest diameter in calibrated units. Frequency tables of the "largest diameters" were constructed to approximate the pore distribution.

Statistical Analysis

Multiple comparisons of group means were made utilizing a one-way analysis of variance (ANOVA) and a Tukey's multiple range test was employed at a significance level of $p<0.05$. The compressive fracture strength data was ranked in ascending order and a Weibull analysis (Weibull, 1951) was performed on the resultant data. The basic form of the Weibull distribution is

$$P_f = 1 - \exp\left[-\left[V\left(\frac{\sigma - \sigma_u}{\sigma_o}\right)^m\right]\right]$$

Equation 1

where σ_u, σ_o and m are all constants. m is known as the Weibull modulus and was given physical meaning by Trustrum and Jayatilaka (1979) as characterizing the "brittleness" of a material, a higher value of m indicates a close grouping of the flexure stress data, while a lower value represents a larger scatter in the flexure stress data. σ_u is the stress at which the failure probability approaches zero and is known as the threshold stress (MPa), σ_o is normally referred to as the normalizing or scaling constant and V is the specimen volume. P_f is the probability of failure that varies from 0 to 1. The number of specimens determined the Weibull fatigue constants (m and σ_o) as predicted by Ritter, Bandyopadhyay and Jakus (1981). Confidence limits for the restorative groups were calculated and differ-

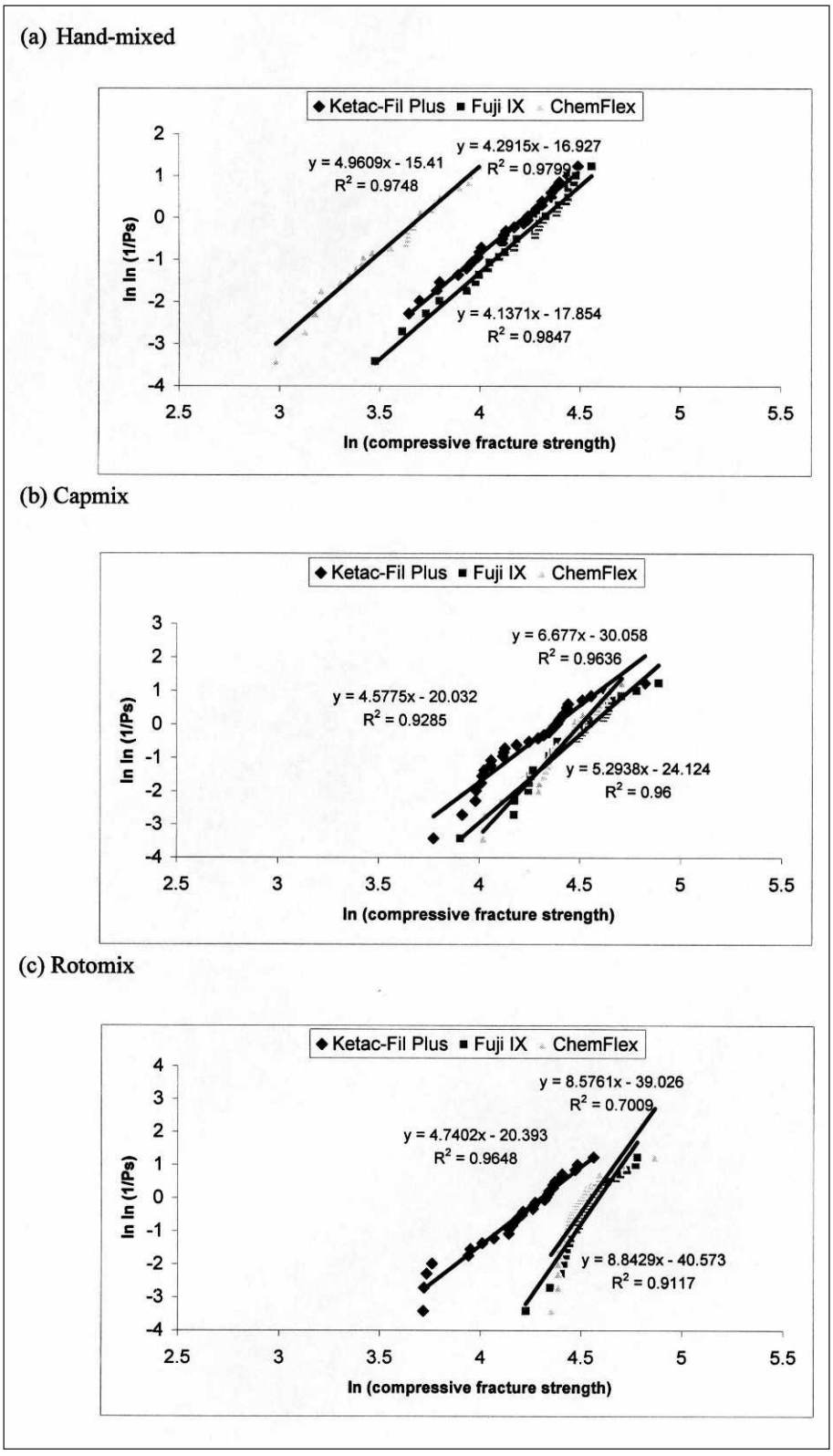

Figure 2. *Weibull plots illustrating the distribution of compressive strengths for (a) the hand-filled hand-mixed restoratives and the encapsulated restoratives manipulated with (b) Capmix and (c) Rotomix.*

ences considered as significant when the confidence intervals did not overlap. Stress at the 10% failure probability was calculated for each restorative and survival probability curves were examined to assess the distribution of fracture strengths.

RESULTS

Compressive Strength

The compressive fracture strength data for the encapsulated restoratives resulted in no significant differences between the means of groups prepared utilizing Rotomix or Capmix (Table 2) when the one-way ANOVA and Tukey test comparisons were analyzed ($p<0.05$). For two of the three restoratives (ChemFlex in Caps and Fuji IX GP FAST), using Rotomix compared with Capmix resulted in an increase in the Weibull modulus and the 10% failure stress (Table 2). However, mechanical mixing of KetacFil Plus Aplicap had no significant effect on the 10% failure stress or reliability of the fracture strength data (Table 2).

Analysis of the compressive fracture strength data resulted in a significant strength decrease for the hand-mixed ChemFlex and Fuji IX GP compared with their encapsulated counterparts (ChemFlex in Caps and Fuji IX GP FAST) at the 95% significance level. No significant strength decrease was identified for the hand-mixed KetacFil Plus compared with the encapsulated Ketac-Fil Plus Aplicap. The Weibull modulus decreased for ChemFlex and Fuji IX GP hand-mixed restoratives compared with the encapsulated ChemFlex in Caps and Fuji IX GP FAST specimens (Figure 2) and the 95% confidence interval of the Weibull moduli also failed to overlap. No significant differences were identified between the mean compressive fracture strength data and the reliability of the hand-mixed KetacFil Plus and encapsulated KetacFil Plus Aplicap.

Working Characteristics

In general, mechanical mixing reduced the working and setting times of encapsulated restoratives (Fuji IX GP FAST and KetacFil Plus Aplicap) com-

Table 2: *Physical Properties of Hand-Mixed and Encapsulated Glass Ionomer Restoratives*

Fuji IXGP			
Property	**Group**		
	Hand-mixed	Capmix	Rotomix
Compressive strength range (MPa)	32.4-95.4	49.7-133.09	68.4-119.2
Mean compressive strength (MPa)	67.9 (16.4)	87.9 (10.1)	93.2 (11.5)
Weibull modulus	4.1(0.7)	5.3 (1.0)	8.8 (1.6)
95% Confidence intervals	3.9-4.3	4.9 - 5.7	7.8-9.9
10% Failure stress (MPa)	41.8	65.2	81.5
R^2-value	0.985	0.960	0.912
KetacFil Plus			
Property	**Group**		
	Hand-mixed	Capmix	Rotomix
Compressive strength range (MPa)	35.2-89.1	43.5-124.2	41.2-95.9
Mean compressive strength (MPa)	62.0 (15.1)	72.7 (17.7)	67.61 (14.6)
Weibull modulus	4.3 (0.8)	4.6 (0.8)	4.7 (0.9)
95% Confidence intervals	4.1-4.6	4.1-5.1	4.4-5.1
10% Failure stress (MPa)	38.3	53.6	41.9
R^2-value	0.979	0.928	0.965
ChemFlex			
Property	**Group**		
	Hand-mixed	Capmix	Rotomix
Compressive strength range (MPa)	39.7-99.4	55.7-110.8	77.6-130.0
Mean compressive strength (MPa)	68.9 (17.2)	84.3 (13.3)	89.5 (10.9)
Weibull modulus	5.0 (0.9)	6.7 (1.2)	8.6 (1.6)
95% Confidence intervals	4.7-5.3	6.2-7.2	6.4-10.7
10% Failure stress (MPa)	43.6	60.9	80.4
R^2-value	0.975	0.964	0.701

pared with hand-mixing (Table 3). Encapsulated restoratives prepared utilizing Rotomix or Capmix highlighted no significant differences for working and setting times, although working times were lower utilizing Rotomix. The setting time for ChemFlex in Caps prepared by mechanical mixing was, however, extended compared with hand-mixed ChemFlex.

Scanning Electron Microscopy

SEM examination of the polished, encapsulated and hand-mixed restorative specimens at 750x to 2000x magnification revealed no discernible change in morphology of the restoratives. Examination by SEM provided an indication of the degree of porosity and filler particles in the hand-mixed and encapsulated restoratives. Figure 3, at 200x magnification, confirmed the porosity levels; however, a two-dimensional cross-section of a restorative cylinder does not provide any quantifiable information on the distribution of pores within the specimen and, as a result, image analysis studies were carried out.

Image Analysis

The image analysis system detected pores greater than 4 µm diameter and the "equivalent diameters" results (Table 4) provide an indication of pore distribution. It was not possible to compare porosity results across different restorative materials due to differences in initial viscosities of restoratives on mixing. However, Rotomix was more effective in removing porosity compared with Capmix for Fuji IX GP FAST and ChemFlex in Caps. Hand mixing also resulted in an increased number of larger pores (>26 µm) compared with mixing in a Rotomix for all restoratives.

DISCUSSION

Failure theories can be based on the surface or volume integral, where the surface integral accounts for surface imperfections due to porosity or cracks introduced during condensation and volume integral accounts for internal porosity and cracks (Fok & others, 2001). Measuring the tensile strength of brittle materials under uni-axial flexure conditions has serious disadvantages as stress in the loaded section is not uniform (Williams & Smith, 1971), varying from zero at the neutral layer to a maximum at the outer surfaces, which accentuates the effect of surface condition on measured strength (Rudnick, Hunter & Holden, 1963) and test results are in excess of the true tensile strength. Bi-axial flexural strength testing of posterior glass ionomers was also considered inappropriate as an alternative since testing induces tensile stress on the surface of specimens being tested, such that specimen failure is critically dependent on surface flaws. Compressive strength is important for dental cements when they are used for luting

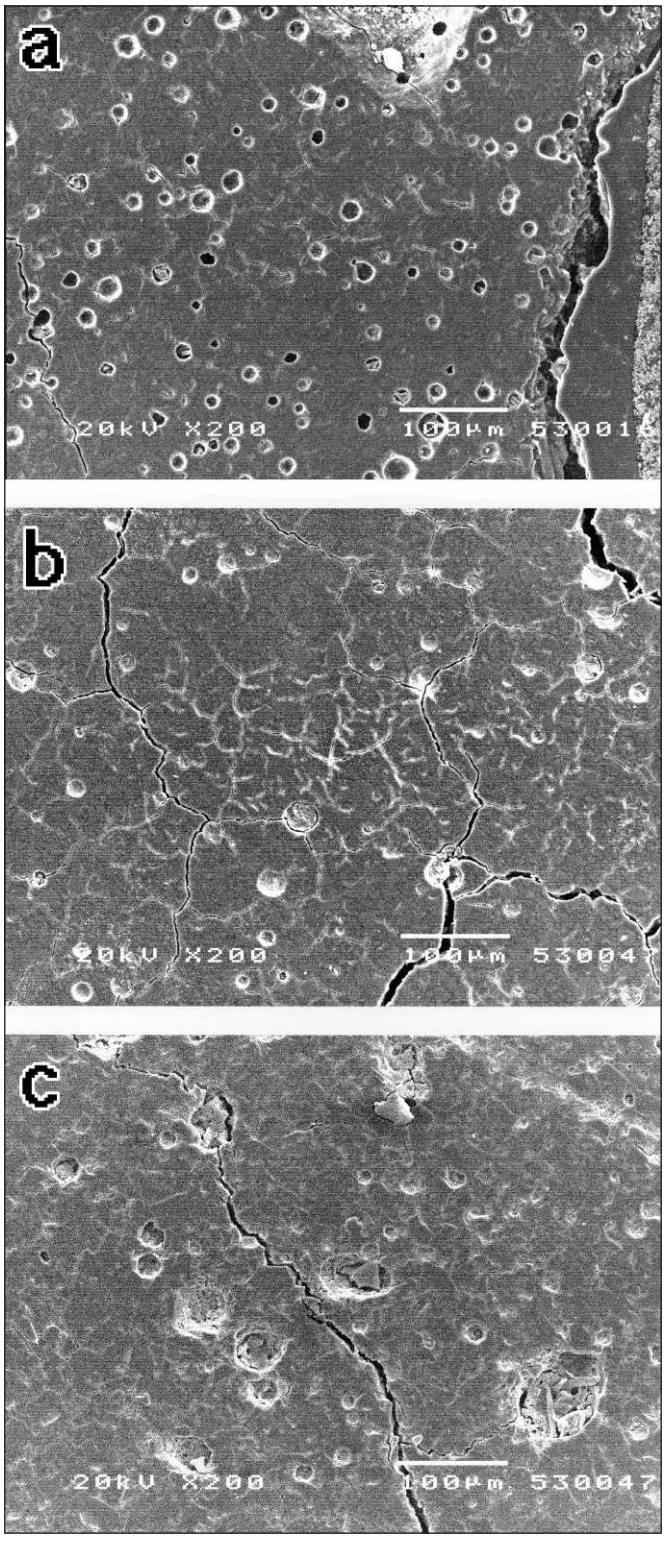

Figure 3. *Scanning electron micrograph of the polished surface of the encapsulated Fuji IXGP FAST restorative manipulated with (a) Capmix, (b) Rotomix and (c) hand-mixed Fuji IXGP restorative illustrating the porosity levels in the 2D cross-sections. Although some component of the surface cracking may have been caused by dehydration in the scanning electron microscope, it is noteworthy that no appreciable change in cement surface morphology occurred during examination under low-vacuum conditions.*

purposes or as cavity linings (Plant & Wilson, 1970). The compressive strength of posterior restorative cements becomes crucial due to masticatory forces encountered in service (White & Yu, 1993). Compressive testing has previously been shown to distinguish changes in bulk properties of brittle materials through changes in composition, levels of porosity and cement manipulation (Fleming & others, 2002; Nomoto & McCabe, 2001; Fleming & others, 1999).

A viscous mix is used for posterior restorative cements that contain a greater concentration of powder particles (compared with luting cements) since strength and lower solubility are important mechanical properties. Mount (1994) recommended capsulation as the ideal method for dispensing glass ionomer cements. Previous investigations on encapsulated zinc phosphate (Fleming & others, 1999) and glass ionomer luting cements (Nomoto & McCabe, 2001) have highlighted the fact that the cemetitious mass readily accepts air inclusions formed during the rapid mixing process compared with a slower hand-mixing regimen where the material is spatulated to avoid these inclusions.

The hand-mixing technique employed in manipulating the glass ionomer restorative cement should have resulted in an even distribution of unreacted glass filler particles in the plastic mass. However, if insufficient force was applied to the cement mass during spatulation, agglomerates form in the plastic cement mass rather than producing an even distribution of powder particles (Figure 3). These powder agglomerates have previously been shown (Fleming & others, 1999) to contain voids or pores that could manifest themselves as crack initiation sites when the material was stressed under load (Kelly, Campbell & Bowen, 1989; Kingery, Bowen & Uhlmann, 1976).

Voids could also be introduced following mixing, and it was proposed that when filling the mold with the hand-mixed restoratives (prepared to a posterior filling material consistency), it was possible that air bubbles may have become trapped, resulting in internal porosity. With viscous restorative consistencies, difficulties were previously experienced during filling of the split-mold. Therefore, hand-filled restoratives were more difficult to condense than encapsulated restoratives and they resulted in less consistent strength results, which were confirmed using the image analysis system where the total number of pores (>26 µm) was increased (Table 3).

The Weibull plots and survival probability distributions offer potential for identifying whether the failures at low stress levels are caused by the same defects as those that cause failure at high stress levels (Fleming & others, 1999). Weibull, by nature, facilitates the prediction of survival rates at different stress levels and, thus, intrinsically provides a basis for comparing the relative performance of different materials. During

Table 3: *The Setting Characteristics for the Glass Ionomer Restoratives*

	Hand-Mixed		Encapsulated (Capmix)		Encapsulated (Rotomix)	
	Working Time	Setting time	Working Time	Setting Time	Working Time	Setting Time
Ketac-Fil Plus	2.05 (0.08)	3.8 (0.1)	2.05 (0.05)	2.46 (0.07)	1.8 (0.1)	2.4 (0.09)
ChemFlex	2.5 (0.1)	3.1 (0.1)	1.9 (0.0)	3.4 (0.0)	1.63 (0.06)	3.58 (0.03)
Fuji IXGP	2.06 (0.06)	3.63 (0.06)	1.96 (0.15)	2.58 (0.03)	1.73 (0.06)	2.7 (0.0)

Table 4: *The Average Distribution of Pores Greater Than 4 mm Diameter (and standard deviations) Within Three Glass Ionomer Specimens Using the Serial Sectioning Technique*

Restorative	Fuji IXGP				KetacFil Plus			ChemFlex	
Pore Diameter (μm)	Hand-Mixed	Encapsulated	Encapsulated	Hand-Mixed	Encapsulated	Encapsulated	Hand-Mixed	Encapsulated	Encapsulated
	Hand-Filled	Capmix	Rotomix	Hand-Filled	Capmix	Rotomix	Hand-Filled	Capmix	Rotomix
4-6	17.3 (9.3)	32.7 (7.1)	33.7 (21.8)	39.3 (5.9)	56.3 (3.5)	40.7 (5.5)	26.7 (4.5)	47.3 (19.3)	26.7 (12.7)
6-10	16.3 (5.5)	31.3 (6.6)	23 (8.9)	33.6 (2.1)	33 (3.5)	16.3 (0.6)	24.7 (8.3)	54.6 (25.4)	28.3 (15.3)
10-14	5.3 (0.6)	13.3 (9.3)	4 (2)	10.3 (5.1)	12 (3.6)	8.3 (1.5)	11.3 (3.1)	21 (3.5)	12.7 (2.5)
14-18	3 (2.6)	3.3 (1.5)	1 (1)	5.3 (2.5)	7 (1)	5.3 (0.6)	4.3 (5.9)	14 (1.7)	6 (3.6)
18-22	1.7 (1.5)	6.7 (5.5)	1.7 (1.5)	2 (1.7)	9.3 (3.1)	2.7 (0.6)	3.6 (0.6)	5 (1)	5 (1)
22-26	2 (1.7)	2 (1.7)	0	2 (2)	5 (1.7)	1.3 (0.6)	3 (1)	3 (2.6)	2.7 (1.2)
26-30	0.7 (0.6)	2 (1)	0	2 (1.7)	1 (1)	1.3 (0.6)	2.3 (1.5)	2 (1)	3 (1.7)
30-34	0.7 (0.6)	1.3 (2.3)	0	0.6 (1.1)	1 (1.7)	1 (0)	0	2 (1)	1 (0)
34-38	0.7 (1.1)	0.7 (0.6)	0.3 (0.6)	0.3 (0.6)	0.3 (0.6)	0.3 (0.6)	0	1 (0)	0.7 (0.6)
38-42	0.3 (0.6)	0.7 (1.2)	0.7 (0.6)	0	1.3 (1.5)	1.7 (0.6)	1.3 (0.6)	1 (0)	0 (0)
>42	1.7 (0.6)	4.7 (2.1)	0.3 (0.6)	2 (1)	2.7 (3.1)	4 (2)	2.6 (1.5)	2 (0.7)	1 (1)
Maximum Pore	70.9 μm	140.2 μm	42.9 μm	118.9 μm	118.2 μm	121.9 μm	117.2 μm	48.7 μm	61.5 μm

compression testing, cracks propagate stably and twist out of their original orientation to propagate parallel to the compression axis so that failure occurs by the slow development of many cracks to form a crushed zone. Upon examining the Weibull plot for ChemFlex in Caps and Fuji IXGP FAST restoratives manipulated with Rotomix the R^2-value of the fracture, strength data was less than 0.95 such that the failure may have resulted from a modification of the defect mechanism on loading to fracture (Figure 2). The respective Weibull plots appeared to be modified at low stress levels indicative of where large pores introduced into the cement mass during the initial rotational action may have been broken down by the centrifugal action on mixing.

The combination of rotational and centrifugal action of the Rotomix mechanical agitator was, therefore, identified as beneficial to reducing porosity within some of the encapsulated restoratives that were investigated (Table 3). Therefore, low stress failures in Capmix could result from introducing larger pores compared with the Rotomix mechanical agitator for ChemFlex in Caps and Fuji IXGP FAST restoratives. Consequently, it is likely that centrifuging effects remove air voids to the surface of cement, and the effect on strength primarily depends on the ability of air to "escape" before mixing is finished. This centrifuging technique, after mixing, has been previously investigated for glass ionomer cements (Nomoto & McCabe, 2001), and the Rotomix system, combined with centrifuging, produced the greatest strength, although the effect of centrifuging was only statistically significant for one cement type. In this study, not all restoratives performed with increased reliability following a combination of rotational and centrifugal action on mixing, suggesting that initial viscosity of the cement mass determines performance.

The serial sectioning technique employed in this investigation offers the potential to identify the distribution of pores within restorative cement cylinders. An alternative technique used in the literature involved SEM images of the fractured specimen fragments (Nomoto & McCabe, 2001); however, the fracture plane is essentially a two-dimensional cross-section of the three-dimensional specimen. A single profile of the fracture plane of a cylindrical specimen is therefore not totally representative of the whole object from which it is derived (Weibel, 1979a,b). Alternative techniques used in the literature to assess the distribution of pores in a thin cement lute (of the order of 40 μm) are of little

use for restoratives that are routinely used in larger sections (Covey & Ewoldsen, 2001; Mitchell & Douglas, 1997). As a result, it is proposed that the porosity profile achieved utilizing the maximum number of fields in the analysis to interpret the resultant data according to the stereological method outlined previously (Weibel 1979a,b) provides a lower bond for the pore distributions within the specimens examined.

The oscillating rheometer (Bovis & others, 1971) was used to assess the impact of hand-mixing and mechanical agitation on the setting characteristics of glass ionomer restoratives. Increasing the powder content for a constant volume of liquid decreases the fluidity of the plastic cement mass because of the larger concentrations of powder particles and reduction in the amount of liquid available. In this study, the increased viscosity of hand-mixed ChemFlex cement (3.8 g/ml) compared with encapsulated ChemFlex in Caps (3.5 g/ml) limited movement of the lower plate of the oscillating rheometer.

The rheometer traces produced for setting times of ChemFlex converge to the final straight line quicker than the more fluid encapsulated material (ChemFlex in Caps) as shown in Table 3. Increasing the powder content of the cement would also be expected to decrease the working time for ChemFlex, however, this did not occur due to the increased energy of mechanical mixing compared with hand-mixing. This agrees with the literature that the speed of vibration of encapsulated glass ionomer cements is reported to be critical in relation to working time. By extending the mixing time and/or the speed of the amalgamator, the increased energy expended would be expected to increase the temperature of the plastic cement mass so that the working time of the cement may be reduced (Mount, 1994).

Alternatively, decreasing the mixing time and/or speed of the amalgamator may extend the working time, as all the liquid may not have been utilized, however, the physical properties would be decreased. Therefore, the decreased working and setting times for restorative materials prepared utilizing Rotomix, compared with Capmix, could be explained by the extended mixing regimen of rotational and centrifugal action utilized by Rotomix.

The oscillating rheometer had the advantage of being quick and simple to use, along with producing consistent test results. However, reliability of the test procedure is the subject of some controversy (Cook & Brockhurst, 1980; Vermilyea, Powers & Craig, 1977; Jacobsen, 1976; Jacobsen & Von Fraunhofer, 1974; Plant, Jones & Wilson, 1972; Bovis & others, 1971). The oscillating rheometer operated within a sensitivity range determined by the spring tension selected to restrict the motion of the lower oscillating plate (Jacobsen & Von Fraunhofer, 1974). If the chosen springs were weak, the initial changes during setting were emphasized; conversely, if the springs were too strong, the final stages of setting would be more discernible, so that a compromise between the two spring tensions was selected (Bovis & others, 1971).

Although the rheometer indicated resistance to shear forces within the setting material, small changes on setting may not be revealed until a certain viscosity (resistance to shearing force) was reached by the material under investigation (Cook & Brockhurst, 1980; Vermilyea & others, 1977; Jacobsen, 1976; Jacobsen & Von Fraunhofer, 1974). Vermilyea and others (1977) identified the viscosity of a zinc phosphate cement (the powder/ liquid mixing ratio was not supplied by the authors) decreasing by 11% when a rotating-t-bar spindle was used (whose continuous mixing motion stirred the cement) compared to a cylindrical bar (that did not interfere with the setting cement).

The authors suggested that the continued mixing motion of the rotating-t-bar spindle may have inhibited matrix formation and resulted in lower viscosities with prolonged working and setting times compared with those obtained by the cylindrical bar. Therefore, while the oscillating rheometer did not provide an exact measurement for the working and setting times of dental cements, it produced overestimated test results that provided an indication to the setting characteristics of the restoratives.

CONCLUSIONS

Mechanical mixing of dental restoratives resulted in air entrapment in the resultant mix that manifested as porosity within the restorative specimens on filling the sample molds. Utilizing a combination of rotational and centrifugal action in which the centrifugal action acts to remove porosity introduced during the initial rotational action has been identified as being beneficial to reducing porosity within some of the restoratives investigated. Not all restoratives had reduced porosity and, therefore, increased performance on mixing with a Rotomix mechanical agitator, suggesting that optimization of the initial viscosity of the system by manipulating the individual proportions of the powder and liquid constituents or by modifying the capsule design may not have been appropriate for some restoratives.

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Effects of Daily Fluoride Exposures on Fluoride Release by Glass Ionomer-Based Restoratives

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Clinical Relevance

Increasing daily fluoride exposure may enhance fluoride release from glass ionomer-based restorative materials.

SUMMARY

It is well documented that glass ionomer cements absorb and release fluoride following single fluoride exposures. This study examined fluoride release among three glass ionomer-based restorative materials following multiple daily exposures to three topical fluoride regimens. Using a Delrin mold, 32 cylindrical specimens, each of a glass ionomer (Ketac-Fil), resin-modified glass ionomer (Photac-Fil) and polyacid-modified resin (Dyract AP) were created. Each specimen was subjected to one of four daily treatments (n=8): (1) no fluoride treatment (control); (2) application of a fluoride dentifrice (1000 ppm) for one minute once daily; (3) application of the same dentifrice for one minute twice daily; (4) the same regimen as (3), plus immersion in a 0.05% sodium fluoride (NaF) mouth rinse (225 ppm) for one minute immediately following the second dentifrice application. Each specimen was suspended in a polyethylene test tube containing 1.0 ml demineralizing solution (pH 4.3) at 37°C for six hours, then transferred to a new test tube containing 1.0 ml remineralizing solution (pH 7.0) at 37°C for 18 hours. Fluoride treatments were completed at the time of transfer daily for seven days. Media solutions were buffered with equal volumes of TISAB II; fluoride levels were measured using a digital ion analyzer and fluoride electrode. Fluoride release decreased significantly from Day 1 to Day 3 for all materials regardless of fluoride treatment (Repeated Measures ANOVA, Tukey HSD, $p<0.05$). All specimens released significantly more fluoride in demineralizing solution than in remineralizing solution. For Days 2-7, Treatment 4 produced greater fluoride release than both the control and Treatment 2 for all three materials ($p<0.05$); For each material, the fluoride release produced by Treatments 3 and 4 was statistically similar on most days throughout the study. By Day 7, Photac-Fil demonstrated both the greatest total fluoride release and the greatest rechargability, followed by Ketac-Fil and Dyract AP. Although subsequent daily fluoride release never approached that of Day 1, increasing daily fluoride exposures enhanced fluoride release for all three restorative materials.

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INTRODUCTION

Fluoride has been well documented as a major contributing factor in the decline in the incidence and severity of dental caries (Haugejorden, Nord & Klock, 1997). Delivery of fluoride is accomplished by several means; most commonly, these include the fluoridation of public water supplies, the use of fluoridated dentifrices, mouth rinses, supplements and the professional application of topical fluoride agents.

Dental restorative materials facilitate the delivery of fluoride directly to susceptible tooth surfaces (Rawls, 1991; Donly & others, 1999). Glass ionomer cements contain fluoride that does not form a significant part of the polyacid salt matrix after cement maturation. This fluoride can then be released into the oral environment without a significant corresponding loss of structural integrity (Walls, 1986). Conventional glass ionomer cements consist of an aluminosilicate glass filler and a polyalkenoic acid, which set through a traditional acid-base reaction (Mount, 1999). Initially, the acid attacks the glass filler particles, and calcium and aluminum ions are released, forming a salt matrix from the polyacrylate chains within the cement (Naasan & Watson, 1998; Walls, 1986). Fluoride, which is incorporated into the glass filler during manufacturing, functions as an oxide scavenger and also acts to modify the fusion temperature of the glass melt (Wilson & McLean, 1989). Fluoride ions are released during the acid attack upon the glass filler and remain within the forming matrix even though they take no further part in the setting reaction. Due to the porous nature of the cement matrix, fluoride ions pass through without affecting its physical make-up (Walls, 1986).

Although fluoride release from glass ionomer restorations has been shown to occur for long periods (Forsten, 1990, 1995; DeSchepper & others, 1991; Hatibovic-Kofman & Koch, 1991; Forss, 1993; Perrin, Persin & Sarrazin, 1994), the greatest release takes place in the first 24-48 hours (El Mallakh & Sarkar, 1990; DeSchepper & others, 1991). Fortunately, glass ionomer cements have the ability to replenish fluoride through exposure to outside fluoride sources (Forsten, 1990; Takahashi, Emilson & Birkhead, 1993) and re-release it into the oral environment (Hatibovic-Kofman & Koch, 1991). It is through this release of fluoride that glass ionomer restorations may demonstrate an anticariogenic effect at the site of placement and throughout the entire mouth (Hicks, Flaitz & Silverstone, 1986).

Resin-modified glass ionomer cements were developed in response to clinical shortcomings of traditional glass ionomer cements, such as short working time, long setting times and less than optimal esthetic properties. Original glass ionomer cements were also very technique-sensitive and required special handling to avoid over-hydration and desiccation (Nicholson & Croll, 1997). Resin-modified glass ionomers differ from their traditional counterparts primarily by the addition of a chemically- or photo-initiated methacrylate resin (Fruits & others, 1996). The setting of these materials consists of two distinct reactions: an initial polymerization of the methacrylate groups, causing rapid development of a rigid matrix structure, and a secondary traditional acid-base setting reaction that begins during mixing and continues after the initial resin polymerization has been completed (Ruyter, 1992; Bourke, Walls & McCabe, 1992). Because of their rapid setting times, light-cured resin-modified glass ionomer cements are less sensitive to water dehydration and dissolution (Fruits & others, 1996).

Resin-modified glass ionomers demonstrate fluoride uptake and release patterns similar to those of conventional glass ionomers. Both conventional and resin-modified glass ionomers tend to release fluoride in an initial burst, with a rapid decline after 48 hours, then continue to release fluoride at significantly lower levels for several weeks or months (DeSchepper & others, 1991; de Araujo & others, 1996; Suljak & Hatibovic-Kofman, 1996). Forsten (1995) reported that resin-modified glass ionomer specimens, stored under running water for up to 11 months, had a fluoride uptake and release total that was equal to or higher than that of the traditional glass ionomer cements.

Polyacid-modified composite resins, or compomers, can be defined as resin-based materials that contain the essential components of traditional glass ionomer cements (acid-decomposable glass, polymeric acid) but at levels that may be insufficient to promote an acid-base reaction (McLean, Nicholson & Wilson, 1994). Because their composition is predominantly resin composite, these materials display physical, handling and esthetic properties that approach those of conventional resin composites (Christensen, 1997). Polyacid-modified resins are generally single-component materials dispensed from one compule or syringe that require no mixing. Studies suggest that these materials release fluoride, but at significantly lower levels than traditional glass ionomer cements (Forsten 1995; Bertacchini & others, 1999).

Many studies have examined the effect of fluoride exposure on glass ionomer-based restorative materials. Forsten (1991) studied the long-term fluoride release from four glass ionomer cements and one resin composite. For all materials, specimens that received a single fluoride exposure (immersion in 50 ppm fluoride for seven days) released more fluoride than non-fluoride-treated specimens at both seven and 15 months. Suljak and Hatibovic-Kofman (1996) studied fluoride release among three resin-modified glass ionomers and a polyacid-modified resin exposed to a 1000 ppm sodium fluoride solution. For all materials, fluoride release

increased significantly during the first 24-48 hours following fluoride exposure, then declined to a constant low level; subsequent fluoride exposures produced similar patterns of fluoride release. Takahashi and others (1993) examined *in vitro* fluoride release from five glass ionomer cements following single applications of sodium fluoride in varying concentrations. Over a 10-week period, all materials released significantly more fluoride following exposure to 2% sodium fluoride as compared to 0.2% and 0.02% sodium fluoride, suggesting that fluoride release from glass ionomers is dependent on the concentration of the original fluoride exposure. Similarly, other studies have reported the ability of glass ionomer cements to take up and subsequently release fluoride (Hatibovic-Kofman & Koch, 1991; Creanor & others, 1994; Forsten, 1995; Young & others, 1996; Vieira, de Souza & Modesto, 1999).

Historically, most *in vitro* fluoride release studies have been performed using a static immersion medium, most commonly distilled or deionized water or artificial saliva. Specimens are prepared and placed in test tubes or vials of the chosen solutions. Fluoride content of the storage medium is measured at baseline and again at predetermined intervals. The difference in fluoride levels is assumed to be fluoride release from the test specimen. However, *in vitro* fluoride uptake and release may be dependent on the storage medium in which specimens are suspended. El Mallakh and Sarkar (1990) compared the fluoride release of two conventional and two metal-reinforced glass ionomer cements in artificial saliva and deionized water. All materials released more fluoride in deionized water; however, the authors suggested that the levels of fluoride release observed in artificial saliva were probably more representative of the materials' actual clinical behavior, since this medium more closely reflects the chemical conditions of the oral environment. Forss (1993) studied fluoride release from one conventional, one metal-reinforced and five resin-modified glass ionomer cements stored in deionized water and lactic acid. All materials released significantly more fluoride when immersed in lactic acid. Carvalho and Cury (1999) compared the fluoride release of a conventional glass ionomer cement, a resin-modified glass ionomer cement and a polyacid-modified resin in three media: deionized water, artificial saliva and a two-part demineralizing/remineralizing solution. For all materials, daily fluoride release was greater in the demineralizing/remineralizing solution than in the other suspension media. The authors suggested that pH cycling offers a better representation of the dynamic process of caries formation and may be preferable to artificial saliva or deionized water for measuring the anticariogenic potential of dental materials.

Many studies have documented the long-term release of fluoride from glass ionomer cements (Forsten, 1977, 1990; Hatibovic-Kofman & Koch, 1991; Perrin & others 1994), as well as the effect of single fluoride applications on subsequent fluoride release (Forsten, 1991; Takahashi & others, 1993; Young & others, 1996). Less extensively studied, however, is the effect of repeated applications of different fluoride agents on fluoride uptake and release by glass ionomer, resin-modified glass ionomer and polyacid-modified composite resin restorative materials. It is not known whether daily tooth brushing with a fluoride dentifrice will optimally charge these restorations, or if the addition of supplementary fluoride sources will augment fluoride uptake and release. Therefore, this investigation sought to (1) compare the methods for recharging glass ionomer, resin-modified glass ionomer and polyacid-modified composite resin restorations, and (2) determine whether a saturation level exists wherein the exposure of the restoration to additional fluoride will no longer affect its fluoride uptake and release.

METHODS AND MATERIALS

One conventional glass ionomer restorative cement, one resin-modified glass ionomer and one polyacid-modified composite resin were chosen for this study (Table 1). Ninety-six specimens (32 of each material) were prepared by placing material into cylindrical Delrin molds (5 mm in diameter x 2 mm in height). The encapsulated materials (Ketac-Fil and Photac-Fil, 3M ESPE, St Paul, MN 55144, USA) were mixed according to the manufacturers' recommendations using a ProMix triturator (Caulk/Dentsply, Milford, DE 19963, USA), then injected into the test molds. Dyract AP (Caulk/Dentsply), which requires no mixing, was injected into the test molds directly from the unit dose compules as supplied by the manufacturer. All specimens were pressed between two glass slabs and cured as follows: Ketac-Fil specimens were allowed to harden for 15 minutes; Photac-Fil and Dyract AP specimens were polymerized for 60 seconds with an Optilux/Demetron model VCL 401 curing light (Demetron/Kerr, Romulus, MI 48174, USA) at an intensity of 400 mw/cm^2. Dental floss was incorporated into each specimen during fabrication to allow for suspension in the test medium. All specimens were stored in 37°C deionized water for 72 hours to permit more complete setting prior to beginning the experimental phase of the study.

Table 1: *Restorative Materials Used*

Material	Shade	Type	Manufacturer
Ketac-Fil	A-2	Glass ionomer	3M ESPE
Photac-Fil	A-2	Resin-modified glass ionomer	3M ESPE
Dyract AP	C-2	Polyacid-modified resin	Caulk/Dentsply

A pH cycling system (Carvalho & Cury, 1999) consisting of a demineralizing solution (Ca 2.0 mM, PO_4 2.0 mM and acetate buffer 75 mM, containing sodium azide, 0.02%; pH 4.3) and a remineralizing solution (artificial saliva; pH 7.0) was used as the suspension medium for storing the individual specimens. According to Featherstone and others (1986), this exposure mimics an *in vivo* high caries challenge environment. Each specimen was suspended in a polyethylene test tube containing 1.0 ml demineralizing solution at 37°C for six hours, then transferred to a new test tube containing 1.0 ml remineralizing solution at 37°C for 18 hours.

Test specimens were subjected to the daily fluoride exposure protocols listed in Table 2. The 32 specimens of each material were separated into four treatment groups (n=8) as follows: (1) no fluoride treatment (control); (2) application of a 0.243% sodium fluoride dentifrice (1000 ppm F) for one minute once daily; (3) application of the same dentifrice for one minute twice daily; (4) same regimen as Group 3 plus immersion in a 0.05% sodium fluoride mouth rinse (225 ppm F) for one minute immediately following the first dentifrice application. Fluoride treatments were completed at the time of transfer daily for seven days.

Media solutions were buffered with equal volumes of Total Ionic Strength Adjustment Buffer (TISAB II, Orion Research Inc, Beverly, MA 01905, USA). This reagent matches the ionic background of standards to sample by adding excess salt to both. By matching the conductivity of both solutions, offsets in readings and measurement errors are eliminated. Fluoride content was measured using an expandable ion analyzer (EA 94, Orion Research Inc) with a combination fluoride electrode (#94-09) and a reference electrode (#900100).

For each restorative material/fluoride treatment combination, mean (± SD) daily fluoride release in both remineralization and demineralization solutions was calculated. Total daily fluoride release for each specimen was calculated by adding the amount released in the demineralizing solution to that released in the remineralizing solution. For each material, the ability to "recharge" was defined as the difference in fluoride release between Group 4 (tooth brushing 2x/day and fluoride rinse) and Group 1 (control). Data were analyzed by repeated measures ANOVA and Tukey HSD post hoc tests α=0.05) to determine differences (1) within each treatment group over time and (2) among the four treatment groups during each testing period.

RESULTS

Mean (±SD) daily fluoride release data for each material/fluoride treatment combination are displayed in Table 3. This information is presented graphically in Figures 1-3 for Ketac-Fil, Photac Fil and Dyract AP, respectively. Figure 4 shows the daily fluoride release in demineralizing and remineralizing solutions for each material tested, and Figure 5 displays the daily fluoride rechargability of each material over the course of the study.

Repeated measures ANOVA revealed statistically significant dif-

Table 2: *Daily Fluoride Exposure Protocol*

1st Treatment			2nd Treatment
Group	**Tooth Brushing**	**NaF Rinse**	**Tooth Brushing**
1	Control – No Treatment		
2	✔		
3	✔		✔
4	✔	✔	✔

Table 3: *Mean (± SD) Daily Fluoride Release (ppm) (n=8)**

Material	Tx Group	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
Ketac-Fil	1	7.13 (1.11)	4.75 (0.35)	2.49 (0.10)	2.72 (0.24)	2.06 (0.11)	1.77 (0.34)	1.65 (0.25)
	2	7.87 (0.97)	5.96 (0.76)	3.31 (0.34)	3.92 (0.34)	2.31 (0.56)	2.42 (0.31)	3.16 (0.22)
	3	8.21 (1.54)	7.84 (0.10)	4.58 (0.51)	4.44 (0.44)	3.27 (0.30)	3.17 (0.29)	4.22 (0.41)
	4	7.57 (1.37)	7.72 (0.98)	4.01 (0.57)	4.60 (0.49)	3.32 (0.34)	3.16 (0.54)	4.04 (0.45)
Photac-Fil	1	9.02 (0.53)	7.34 (0.36)	3.20 (0.35)	3.41 (0.29)	2.03 (0.15)	1.88 (0.21)	1.70 (0.13)
	2	9.11 (0.56)	7.50 (0.35)	3.59 (0.33)	3.47 (0.63)	3.54 (0.34)	2.89 (0.11)	2.81 (0.14)
	3	10.39 (0.25)	9.12 (0.20)	4.33 (0.42)	3.99 (0.49)	3.41 (0.38)	3.47 (0.25)	4.12 (0.42)
	4	10.30 (0.50)	9.12 (0.42)	4.40 (0.31)	4.09 (0.23)	4.01 (0.20)	4.12 (0.27)	5.04 (0.32)
Dyract AP	1	7.18 (1.94)	5.80 (0.27)	2.87 (0.16)	3.70 (1.04)	2.63 (0.14)	2.58 (0.24)	2.73 (0.16)
	2	7.86 (0.18)	6.04 (0.20)	3.12 (0.20)	3.04 (0.26)	3.12 (0.14)	2.65 (0.16)	2.94 (0.20)
	3	7.84 (0.21)	6.76 (0.50)	3.46 (0.10)	3.34 (0.21)	2.70 (0.17)	2.81 (0.13)	3.02 (0.15)
	4	8.02 (0.50)	6.82 (0.35)	3.67 (0.20)	4.15 (0.27)	3.39 (0.09)	2.97 (0.13)	3.36 (0.16)

**Repeated Measures ANOVA and Tukey's HSD. Vertical lines connect values that are not significantly different (α= 0.05).*

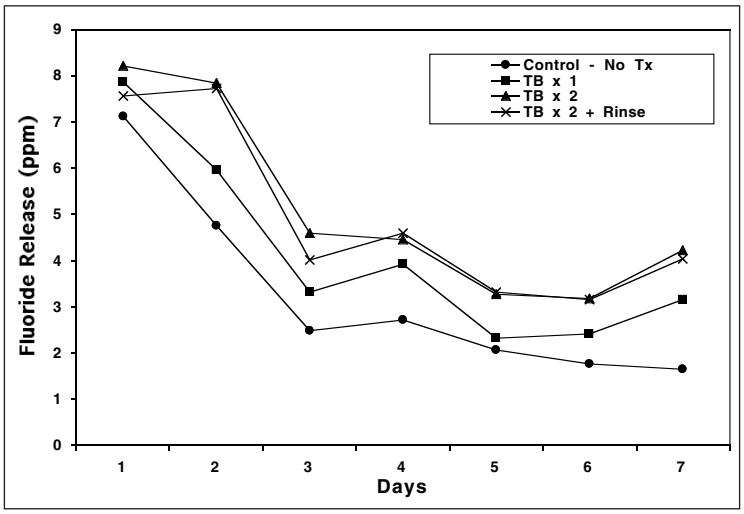

Figure 1. *Ketac-Fil – Fluoride release over seven days.*

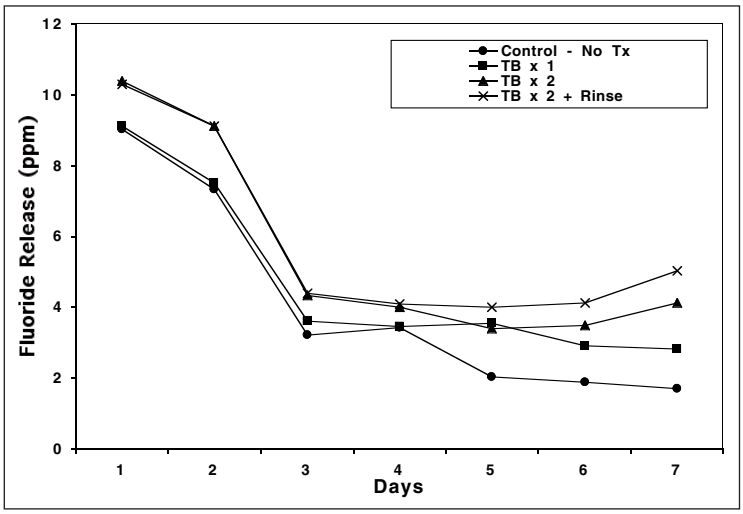

Figure 2. *Photac-Fil – Fluoride release over seven days.*

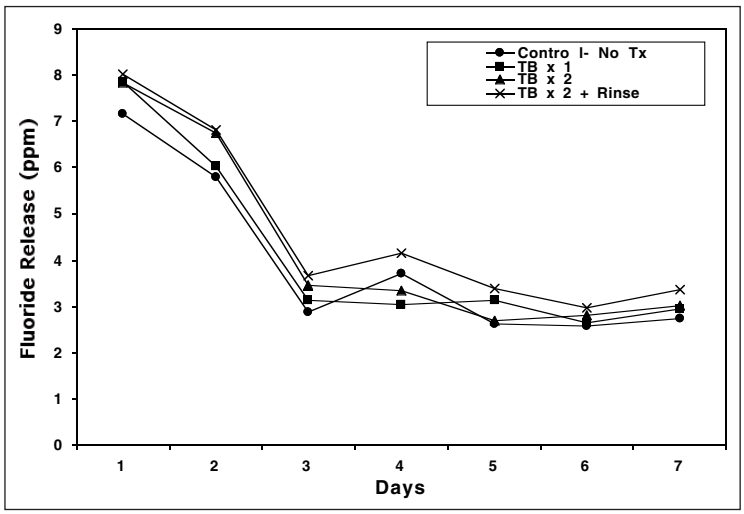

Figure 3. *Dyract AP – Fluoride release over seven days.*

ferences in fluoride treatment effect ($p<0.0001$), immersion media effect ($p<0.001$) and rechargability ($p<0.001$) for all materials.

The three restorative materials demonstrated similar patterns of fluoride release over the course of the study (Figures 1-3). Fluoride release tended to be related to the dose of daily supplemental fluoride applied. For all materials, the greatest decrease in fluoride release occurred from Day 1 to Day 3. By Day 2, fluoride release was significantly greater in Groups 3 (TB x 2) and 4 (TB x 2 + rinse) than in Group 1 (control); the only exceptions occurred with Photac-Fil on Day 4 and Dyract AP on Days 4 and 6, when Groups 1 and 3 were similar. For each material, fluoride release in Groups 3 and 4 was statistically similar throughout the study, with the exception of Dyract AP on Day 5 and Photac-Fil on Days 5 and 7; for all materials, beginning at Day 2, fluoride release in Group 4 was significantly greater than in Group 1. Fluoride release in Group 2 tended to be slightly higher than Group 1 but lower than Groups 3 and 4 for all materials throughout the study (Table 3). Although increasing fluoride exposure resulted, in general, in increased fluoride release for each restorative material, by Day 3, total fluoride release was significantly greater for Ketac–Fil and Photac-Fil than for Dyract AP.

Immersion media played an important role in fluoride release for all materials. Regardless of fluoride treatment, mean fluoride release for all materials was significantly greater when immersed in demineralizing solution than in remineralizing solution (Figure 4). This was true even though the specimens were suspended in the remineralizing solution for three times longer than in the demineralizing solution.

For each material, the ability to "recharge" was defined as the difference in fluoride release between Group 4 (TB x 2 + rinse) and Group 1 (control). At Day 7 Photac-Fil showed the greatest recharge capability, Ketac-Fil showed the next highest and Dyract AP showed the least change in total mean fluoride release (Figure 5).

DISCUSSION

This study's results suggest that increasing fluoride exposure increases fluoride uptake and release by glass ionomer-based restorative materials. This confirms previous observations that glass ionomer cements can recharge after exposure to fluoride (Forsten, 1991, 1995; Takahashi, & others, 1993; Young & others, 1996). Vieira and others (1999) evaluated the effect of fluoride on glass ionomers and resin composite in a simulated high caries environment. They submitted specimens of traditional glass

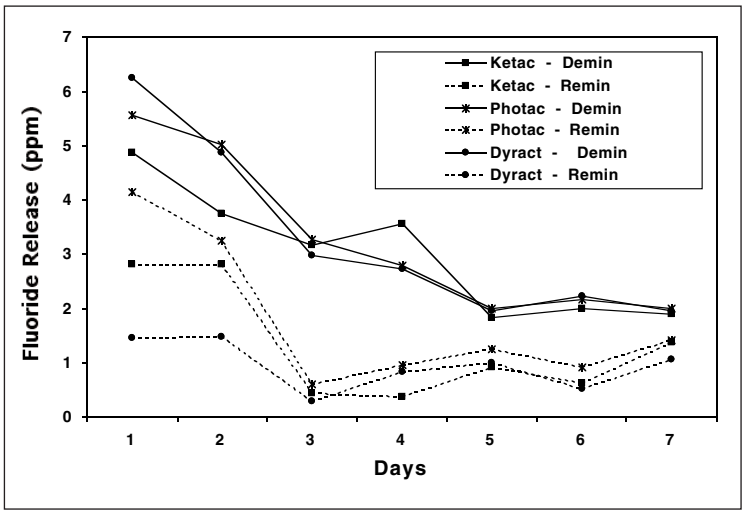

Figure 4. *Daily fluoride release (ppm) in remineralizing and demineralizing solutions.*

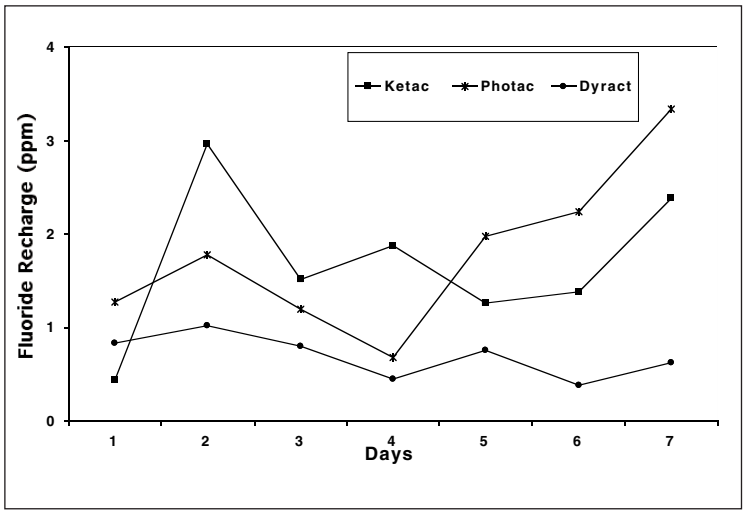

Figure 5. *Recharge of glass ionomer-based restorative materials following daily fluoride exposure.*

ionomer, resin-modified glass ionomer, polyacid-modified resin and resin composite to a 14-day demineralization/remineralization regimen, and on Days 8-14, applied a fluoridated dentifrice (1,100 ppm) to the specimens twice daily. Their results suggested that all materials were capable of absorbing fluoride from the dentifrice solution and later releasing it to the solution. This concurs with the results of the current study. In addition, the present study confirms the findings of others (Hatibovic-Kofman & Koch, 1991; Young & others, 1996), who concluded that the levels of fluoride necessary to allow uptake and its subsequent release into the environment could be found in a typical dentifrice.

Similarly, this study confirms the findings of Takahashi and others (1993), who demonstrated that fluoride release by glass ionomer materials increases with exposure to increased fluoride concentrations. In the current study, fluoride release was significantly greater in Group 4, which received the greatest daily fluoride exposure, than in Group 1, which received no additional fluoride, for each material on each day. Group 2, which received only one fluoride exposure per day, exhibited lower levels of fluoride release than the other treatment groups. In contrast, fluoride release in Group 3, which received two fluoride exposures per day, was generally very similar to that of Group 4, suggesting a possible saturation effect. The effects of higher concentration (for example, 5000 ppm) topical fluorides, and increased numbers of daily exposures, may provide additional insight into this phenomenon.

Compared to the remineralizing solution, the demineralizing solution appears to have eluted significantly more fluoride from all specimens. This result occurred even though the specimens were immersed in the demineralizing solution for only one-third as long as in the remineralizing solution. Other studies have demonstrated similar fluoride release patterns (Forss, 1993; Carvalho & Cury, 1999; Vieira & others, 1999). In traditional glass ionomer cements, fluoride is eluted from the free fluoride available in the matrix (Crisp, Lewis & Wilson, 1976). When exposed to an acidic challenge, both traditional and resin-modified glass ionomer cements may release additional fluoride. This may result from the dissolution of matrix-forming elements within the restorative material (Forss, 1993).

An interesting and unique finding of this study was the measure of the difference in fluoride release between Group 1 (control), which showed the lowest fluoride release, and Group 4 (TB x 2 + rinse), which showed the greatest fluoride release. This difference can be considered "recharging" (Suljak & Hatibovic-Kofman, 1996; Vieira & others, 1999) and was significantly different for each of the materials tested. It is worth noting that the resin-modified glass ionomer (Photac-Fil) showed the greatest rechargability, followed in rank order by the traditional glass ionomer (Ketac-Fil) and the polyacid-modified resin (Dyract AP).

It is important to note that although this study was designed to mimic daily occurrences of acid challenges and fluoride exposures seen with typical home care regimens, *in vitro* results may not be directly representative of *in vivo* results. Fluoride release measured from specimens immersed in a static medium may not take into account the dynamic nature of conditions found in the oral cavity. Although the majority of fluoride release studies are designed in this manner, there are some that attempt to more closely simulate intraoral conditions. A recent study by Carey and others (2000) measured fluoride release over time in a dynamic environment. Results indicated a fluoride release pattern similar to those reported here.

Finally, from Days 6 to 7 of this study, it appeared that, for all materials and treatment groups, the total amount of fluoride released was increasing. This may suggest that daily fluoride exposure exerts a cumulative or additive effect on subsequent fluoride release by glass ionomer-based restorative materials. Longer-term studies are needed to evaluate this trend and determine whether this increased fluoride release reaches a maximum. Moreover, the effects of additional increases in the number and/or concentrations of daily fluoride exposures should be evaluated.

CONCLUSIONS

Under the conditions of this study:

1. Typical home care fluoride regimens provided adequate fluoride exposure to produce measurable fluoride uptake and subsequent release in three glass ionomer-based restorative materials. Increasing fluoride exposure significantly increased fluoride release in all three materials.
2. Fluoride released from all materials was significantly greater in the acidic demineralizing solution than in the neutral remineralizing solution.
3. A resin-modified glass ionomer (Photac-Fil) showed the greatest rechargability, followed by a glass ionomer restorative (Ketac-Fil) and a polyacid-modified resin (Dyract AP).

Disclaimer

The opinions expressed in this article are the private views of the authors and should not be construed as reflecting official policies of the US Navy, Department of Defense or US Government.

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The Durability of a Fluoride-Releasing Resin Adhesive System to Dentin

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PNR Pereira • J Tagami • DH Pashley

Clinical Relevance

Fluoride released from adhesive resin could minimize the decrease in bond strength to dentin after months of storage in water.

SUMMARY

The durability of a fluoride-free (SE Bond) and a fluoride-containing (KBF) self-etching primer/adhesive system were compared by measuring the microtensile bond strengths (μTBS) of these adhesive systems to human dentin *in vitro*. After bonding, the restored teeth were serially sectioned into multiple slabs that were trimmed to a 1 mm² cross-sectional area at the bonded interface. For the three and six month specimens, half were fully covered with nail varnish (SE+, KBF+), while the other half were incubated at 37°C in water without any protective varnish (SE-, KBF-). The μTBS of the one-day specimens were 44.6 ± 11.2 MPa for SE Bond and 39.8 ± 8.0 MPa for KBF ($p>0.05$). When unprotected specimens were incubated in water for three and six months, the μTBS fell to 26.3 ± 8.8 MPa and 23.6 ± 10.7 MPa for SE-, respectively, but did not change in the specimens protected with nail varnish (SE+, 41.9 ± 12.8 MPa and 41.8 ± 9.8 MPa, respectively). In contrast, in specimens bonded with a fluoride-containing resin, KBF, the bond strengths of the unprotected specimens did not change over three and six months KBF-. Values were 32.4 ± 6.1 MPa and 36.8 ± 2.3 MPa, respectively. Similarly, varnish-protected KBF+ specimens did not change over three and six months (39.3 ± 13.6 MPa and 40.9 ± 14.7 MPa, respectively). The results indicate that decreases in bond strength over six months' storage are water-dependent but can be prevented by using fluoride-containing resins.

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INTRODUCTION

The quality and durability of bonds between adhesive resins and dentin are major considerations in the longevity of bonded restoration. Several reports of the

durability of dentin bonds *in vitro* have indicated that resin-dentin interfaces have degraded as a result of long-term water storage. This degradation of dentin bonds may occur due to the plasticizing effects of water on collagen (Maciel & others, 1996), unfilled resin (Calais & Söderholm, 1988), water sorption and/or hydrolysis of adhesive resin (Gwinnett & Yu, 1995; Burrow, Satoh & Tagami, 1996) or hydrolysis of collagen fibrils at the base of the hybrid layer (Kiyomura, 1987; Burrow & others, 1996; Hashimoto & others, 2000).

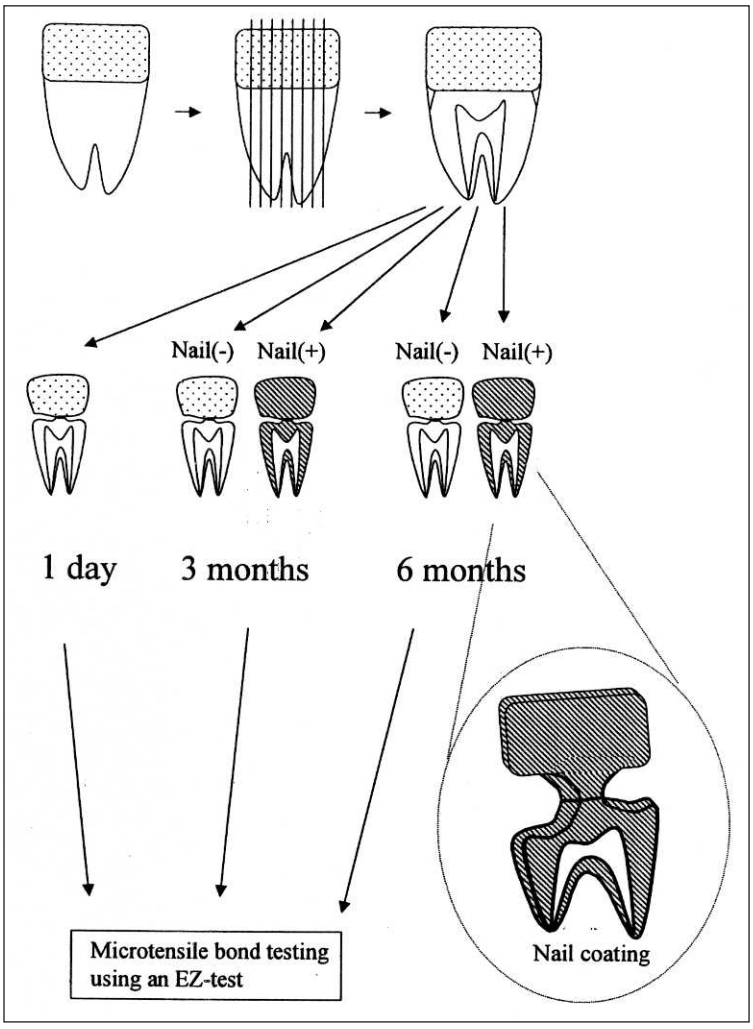

Figure 1. *Schematic showing how bonded composite buildups were vertically serially-sectioned into 0.7 mm thick slabs that were then trimmed to hour-glass shapes giving a 1.0 mm² bonded cross-sectional area. These were incubated in 37°C water for one day, three or six months prior to the μTBS test.*

Sano and others (1995) revealed a leakage pathway through nanometer-sized channels within the hybrid layer, which they termed "nanoleakage." They speculated that the nanolekage pathway might permit fluid penetration within the hybrid layer (Sano & others, 1999). Recently, Okuda and others (2002) investigated the relationship between nanoleakage and long-term durability of dentin bonds using self-etching primer adhesive systems. They concluded that nanoleakage gradually increased at the resin-dentin interface, so that the bond strength decreased over time.

Recently, manufacturers have developed various fluoride-releasing resin adhesives because fluoride is thought to exert anticariogenic activity by increasing enamel and dentin resistance to subsequent acid attack (Corpron & others, 1986; Dionysopoulos, Kotsanos & Papadogiannis, 1990) and by inhibiting carbohydrate metabolism in dental plaque (Norman & others, 1972). Moreover, (Saito, 1996) demonstrated that fluoride incorporated into MMA-TBB resin increased the durability of dentin bond. It had been demonstrated that the tensile bond strengths of MMA-TBB resin containing fluoride did not decrease after long-term (18 months) water immersion, while the tensile bond strength of the same resin system without fluoride decreased during the same immersion time. However, there are few reports of the durability of fluoride releasing adhesive materials to dentin, and the effect of fluoride on the long-term degradation of resin-dentin interface is unclear.

This study determined whether a fluoride-releasing adhesive could improve the durability of tensile bond strength to dentin following six months of water storage.

METHODS AND MATERIALS

Twenty extracted human third molars stored frozen were used in this study. The teeth were

Table 1: *Composition of Self-Etching Primer/Adhesive System*

ABF System		
Experimental anti-bacterial, F-releasing adhesive system		
	Anti-bacterial Primer (ABP);	MDP, HEMA, MDPB, dimethacrylates, photoinitiator, water
	Fluoride-releasing bonding agent (KBF);	MDP, HEMA, dimethacrylates, photoinitiator, Surface treated NaF, microfiller
Clearifil SE Bond		
	Bonding agent;	MDP, HEMA, dimethacrylates, photoinitiator, microfiller

Abbreviations: MDP = 10-methacryloyloxydecyl dihydrogen phosphate; MDPB = 12-methacryloylicydodecylpyridinum bromide HEMA = 2-hydroxyethyl methacrylate; NaF = sodium fluoride

thawed, cleaned of debris and the occlusal surface was ground flat to remove the enamel and expose middle dentin. The dentin surfaces were inspected to ensure that there were no remnants of enamel, then polished with 600-grit silicon carbide paper under running water.

All dentin surfaces were treated with a self-etching primer (ABP: experimental; Kuraray Medical Inc, Tokyo, Japan) for 20 seconds, followed by gentle air drying. The specimens were then randomly divided into two groups. One group was treated with a fluoride-releasing adhesive (KBF: experimental, Kuraray Medical Inc, Tokyo, Japan). The other group was treated with Clearfil SE Bond adhesive (Kuraray Medical Inc, Table 1). After light curing the adhesive for 10 seconds, the crown was built up incrementally with Clearfil AP-X (Kuraray Medical Inc) resin composite. The bonded assemblies were stored in tap water at 37°C for one day, then sectioned perpendicular to the bonded interface into approximately 0.7 mm-thick slabs with a diamond saw (Figure 1). Eight or nine slabs were cut perpendicular to the bonded interface from each tooth. The adhesive-dentin interface was trimmed to an approximate width of 1.4 mm using a fine diamond bur (c16ff, GC Ltd, Tokyo Japan) mounted with a handheld high-speed handpiece under copious air-water spray to produce a cross-sectional surface area of approximately 1 mm^2 for micro-tensile bond testing. After pooling all the slabs from each bonding system, they were randomly assigned to three time period groups (one day and three and six months). In the three- and six-month groups, the specimens were further randomly divided to two subgroups: in one subgroup, all the exposed surfaces of specimens were coated with fingernail varnish (Figure 1). The specimens in the other subgroup remained uncoated. The specimens of the three- and six-months groups were immersed in individual bottles containing water at 37°C. The water was changed daily until testing.

At specified time periods all specimens were subjected to microtensile bond test using a table-top material tester (EZ-test, Shimadzu Co, Kyoto, Japan) at a crosshead speed of 1 mm/min (Figure 1) using a viscous cyanoacrylate cement (Zapit, Dental Ventures of American, Corona, CA 91720, USA) to cement the mineralized endo of the specimens to the testing device. The specimens that were coated with nail varnish had the varnish removed prior to testing.

Bond strength data were subjected to three-way ANOVA (the three factors being fluoride, time and varnish) seeking statistically significant differences among the groups. When significant differences were found among the groups, they were identified and compared using Scheffe's test at the 95% level of confidence. Statistical analysis of the failure modes was performed using the Kruskal-Wallis rank test. Each failure mode was given score from 1 to 4 prior statistical analysis. A score of 1 was given for 90-100% failure in the bonded interface, a score of 2 for mixed failure of the bonded interface and the adhesive resin, a score of 3 for cohesive failure within the adhesive resin and a score of 4 for cohesive failure within the dentin.

All debonded specimens were fixed in 10% neutral formalin for at least eight hours prior to SEM examination. All fractured specimens were placed on SEM stubs and allowed to air dry. The debonded specimens were gold sputter-

Table 2: *Microtensile Bond Strengths Over Time*

Periods		One Day	Three Months	Six Months
Adhesive System	Nail-Coating			
ABP + KBF	(-)	39.8 ± 8.0(17)[a]	32.4 ± 6.1(14)[a]	36.8 ± 12.3(12)[a]
	(+)		39.3 ± 13.6(10)[a]	40.9 ± 14.7(14)[a]
ABP + SE	(-)	44.6 ± 11.2(17)[a]	26.3 ± 8.8(12)[b]	23.6 ± 10.7(12)[b]
	(+)		41.9 ± 12.8(10)[a]	41.8 ± 9.8(13)[a]

Values are mean ± SD(N). Groups identified by different superscript letters are significantly different (p<0.05). + designates specimens coated with nail varnish—designates uncoated specimens.
Abbreviations: ABP= Anti-bacterial primer; KBF= Fluoride-releasing adhesive; SE= Clearfil SE Bond adhesive that is fluoride-free.

Table 3: *Failure Modes After MicrotensileTest and Kruskal-Wallis Mean Rank*

	Interfacial	Mixed	Cohesive in Bonding Resin	Cohesive in Dentin	Kruskal-Wallis Mean Rank
ABP + KBF					
One day	0/17	7/17	10/17	0/17	72.0
Three months (-)	0/14	6/14	8/14	0/14	71.1
Three months (+)	2/10	3/10	4/10	1/10	64.6
Six months (-)	4/12	2/12	6/12	0/12	57.0
Six months (+)	2/14	6/14	6/14	0/14	58.6
ABP + SE					
One day	0/17	8/17	9/17	0/17	68.7
Three months (-)	2/12	3/12	7/12	0/12	66.8
Three months (+)	1/10	3/10	6/10	0/10	69.7
Six months (-)	2/12	3/12	7/12	0/12	66.8
Six months (+)	3/13	3/13	7/13	0/13	62.3

Interfacial = 90-100% of the failure occurred in the bonded interface; Mixed = Mixed failures in which a major fraction (>20%) of the fracture occurring at the bonded interface with the remainder of the failure occurring cohesively with in the bonding resin; Cohesive in bonding resin = 90-100% of the failure occurred in the bonding resin; Cohesive in dentin = 90-100% of the failure occurred in the underlying mineralized dentin. (+) designates specimens that were coated with nail varnish. (-) designates specimens that were not covered by nail varnish.
Abbreviations: ABP= Anti-bacterial primer; KBF= Fluoride-releasing adhesive; SE= Clearfil SE Bond adhesive that is fluoride-free.

Figure 2

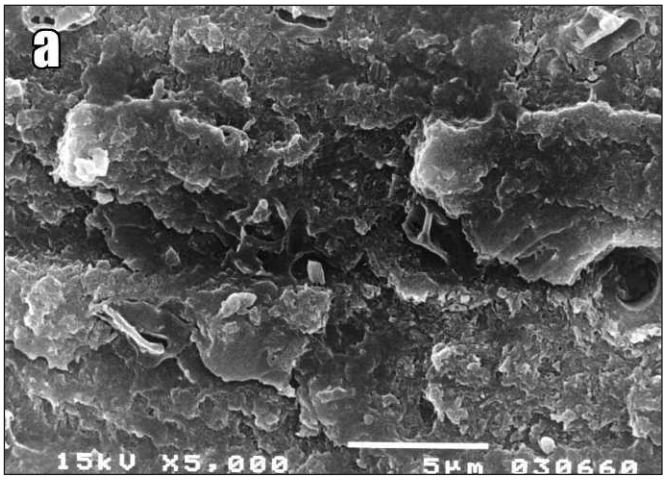

Figure 2a. *SEM micrograph of the dentin side of failed SE resin-dentin bond of one-day control group. Fracture occurred at the top of the adhesive-dentin interface. The resin and dentinal tubule are seen.*

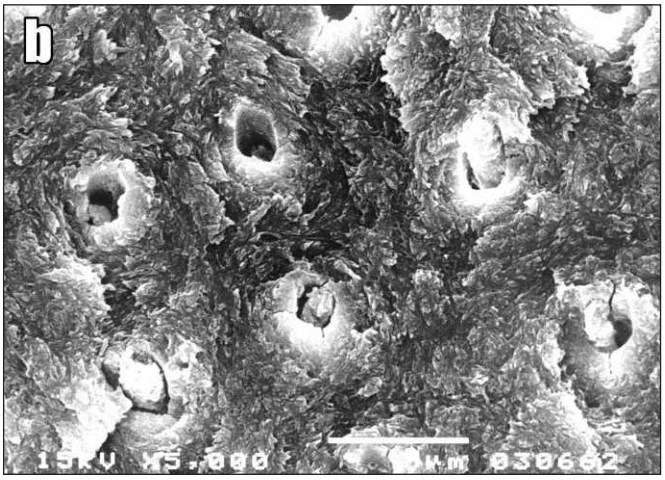

Figure 2b. *SEM micrograph of the dentin side of failed SE resin-dentin bond without nail-coating after six months. Fracture occurred within the hybrid layer. The resin tags in tubules are observed and few collagen fibrils are exposed.*

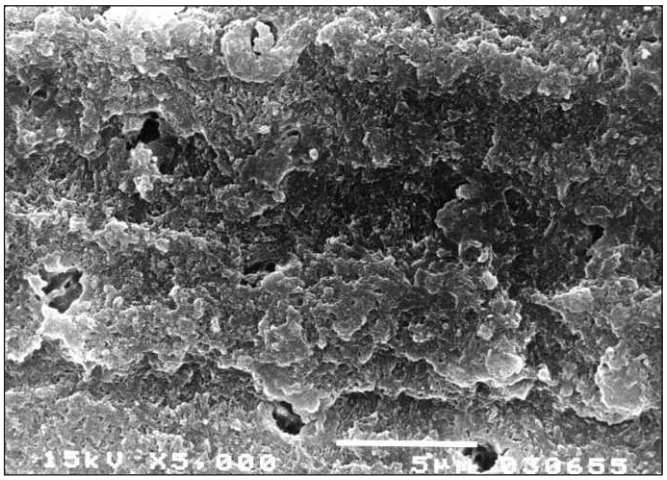

Figure 3. *SEM micrograph of the dentin side of failed KBF resin-dentin bond without nail-coating after six months. Fracture occurred at the top of the adhesive-dentin interface. The resin and dentinal tubules are seen.*

coated and observed with scanning electron microscopy (JSM-5310, JEOL, Tokyo, Japan) so that microscopic fracture patterns and the morphology of the debonded interfaces could be assessed. The failure modes were designated as interfacial if 90-100% of the failure occurred in the bonded interface, cohesive in adhesive resin if 90-100% of the failure occurred in the adhesive resin, cohesive in dentin if 90-100% of the failure occurred in the mineralized dentin and mixed failure when <20% of the fracture occurred adhesively between the bottom of the adhesive layer and the top of the hybrid layer, with the remainder of the fracture occurring cohesively within the adhesive resin.

RESULTS

Means and standard deviations of the microtensile bond strengths (µTBS) for the KBF and SE groups are summarized in the Table 2. The three-way ANOVA indicated that there were statistically significant differences for time (p=0.0004) and varnish (p<0.001) but not for materials (p=0.5369), and there were significant interactions between materials and time (p=0.0209), materials and varnish (p=0.0265) and time and varnish (p=0.005). There was no significant difference between the µTBS of the KBF and SE groups in the one day control groups (39.8 ± 8.0 vs 44.6 ± 11.2 MPa, respectively) (p>0.05). For the uncoated SE group, the µTBS decreased significantly after three and six months compared with the one-day control group (p<0.05), but there were no significant differences among the three time periods in the uncoated KBF group (p>0.05). In the groups covered with nail varnish, both adhesive materials gave similar µTBS after three and six months (Table 2) that were not significantly different from the one-day controls (p>0.05).

The modes of failure were not statistically different among all groups (p=0.9877) that were mainly classified as mixed failure of the bonded interface and the bonding resin or cohesive failure in bonding resin (Table 3). However, SEM observations of interfacial fractured sites of specimens bonded with SE without varnish protection showed morphological changes over time. In the SE- group, the interfacial failures were identified by the presence of residual adhesive resin on top of the hybrid layers in the one-day control (Figure 2a). In the three and six month SE- groups, there was a failure within the hybrid layer since collagen fibrils could be seen (Figure 2b). SEM observation of the inter-

facial failure sites of specimens bonded with KBF- without varnish protection showed that fractures occurred at the top of the adhesive-dentin interface because of the presence of residual resin on the hybrid layer during the test periods (Figure 3). For both adhesive materials that received varnish protection, the interfacial failure occurred at the adhesive-dentin interface after three and six months. Only one cohesive failure completely in dentin was observed in any of the groups (Table 3).

DISCUSSION

Previous *in vitro* studies indicated that resin-dentin interfaces degraded as a result of long-term water storage (Kiyomura 1987; Burrow & others, 1996; Shono & others, 1999; Okuda & others, 2001). The decrease in bond strength is thought to be due to the effect of hydrolysis at the interface of the bonding resin and the hybrid layer (Burrow & others, 1996; Armstrong & others, 2000). Sano and others (1999) reported similar results in their *in vivo* study. They demonstrated that the fractured interfaces of resin-dentin bonds obtained by using Clearfil LinerBond II after one year of function exhibited an increased porosity over time, whereas, bond strength remained unaltered. They speculated that the increased porosity at the bonded interface over time might have occurred via nanoleakage pathways that developed within the hybrid layer. Hashimoto and others (2000) investigated *in vivo* degradation of resin-dentin bonds in humans over one-to-three years using Scotchbond Multi-Purpose on cavities etched with 10% maleic acid. They reported that the bond strengths decreased significantly over time and the debonded surface showed complete loss of resinous materials between the collagen fibrils and/or depletion of collagen fibrils within the degraded hybridized dentin. Although some studies have used thermal and mechanical stressing of resin-bonded dentin, such experiments are usually conducted on intact teeth. In this study, the restored teeth were divided into many 0.7 mm-thick slabs to decrease water diffusion distances and accelerate the effects of aging. It would be technically difficult to mechanically stress these slabs. Future studies employing thermal and mechanical stressing should be conducted to determine whether they further accelerate time-dependent reductions in resin-dentin bond strength. Mechanical stressing may increase the uptake of water into the bonded interface.

In this study, the bond strengths of the uncoated SE Bond group decreased significantly ($p<0.05$) after three and six months of storage in water. The mode of failure on the dentin side of the adhesive bond was at the top of the hybrid layer in the one-day group, while failures in the three- and six-month groups were within the hybrid layer. The μTBS results agreed with Okuda and others (2002), who reported that the bond strengths of Clearfil LinerBond 2V gradually decreased and the fractured pattern changed over time. They demonstrated a highly significant inverse relationship between long-term bond strengths and nanoleakage. They reported that there was a high correlation (r=-0.92) between microtensile bond strength and silver penetration as an index of nanoleakage after nine months of water storage.

The most important pathway for nanoleakage in conventional total-etch adhesive systems is thought to be within a hybrid layer that has not been fully penetrated by resin, leaving spaces for fluid penetration (Sano & others, 1995). It was reported that nanoleakage in self-etching primer systems gradually increased at the resin-dentin interface following long-term water storage, presumably due to hydrolytic degradation at the interface as progressively more water permeates through nanoleakage space (Okuda & others, 2002). In this study, both varnish-coated groups exhibited stable bond strengths and unaltered fracture patterns and morphology over time. Apparently, the protective nail varnish prevented water penetration into any nanoleakage sites, protecting the resin-dentin interface from hydrolytic degradation. However, these results offer no specific insight into the cause of the reduced bond strengths in the SE- group specimens. The authors speculate that the reductions in bond strength in the SE- group might be due to slow demineralization of mineralized dentin just beneath the hybrid layer during prolonged water incubation. Water may not have had the same access to the mineralized dentin in the varnish-coated group (SE+). The slow release of fluoride in the KBF- group may have increased the local concentration of fluoroapatite in the mineralized dentin just beneath the hybrid layer. This less soluble mineral would prevent the release of hydrolytic enzymes from the mineralized matrix. It is even possible that fluoride uptake into apatite would prolong the fluoride-effect since fluoroapatite is far less soluble than NaF. Presumably, NaF-release from the KBF resin would be time dependent. No long-term (9-to-12 month) studies have been conducted to determine how long NaF release is sustained and how long any fluoroapatite that is formed remains after years of service. It is possible that the presence of fluoride in the KBF adhesive might have prevented loss of residual mineral from the smear layer that was incorporated into the hybrid layers produced by these self-etching primers systems (Tay & others, 2000a,b). Such studies require the high resolution that is available using transmission electron microscopy (TEM). No such TEM studies have yet been conducted in long-term experiments on the durability of resin-dentin bonds, although they are planned in the future. Although water sorption can plasticize resin, thereby, altering stress-concentrations during bond testing, the chemical composition of the two resins is similar, suggesting that the water sorption in both groups should have been similar. Thus, differences in

water sorption are probably not responsible for reductions in bond strength in the SE- group over time. Such water sorption studies should be conducted in the future to rule out this variable.

On the other hand, the uncoated KBF group also exhibited a stable bond strength and unaltered fracture pattern and morphology over time. KBF adhesive contains surface treated NaF crystals that have a prolonged fluoride-releasing ability (manufacturer's data). Some reports demonstrated that fluoride could be detected in the hybrid layer and the underlying mineralized dentin bonded with fluoride releasing resin materials after water immersion for several weeks (Ferracane, Mitchem & Adey, 1998; Han & others, 2002). Silanized NaF incorporated into BIS-GMA/TEGMA resins has been shown to be released from the polymerized resin for more than 20 weeks (Nakabo & others, 2002). Saito (1996) demonstrated that the tensile bond strengths of MMA-TBB resin containing fluoride did not decrease after long-term (18 months) water immersion, while the tensile bond strengths of the same resin system without fluoride decreased during the same immersion time. It was speculated that the fluoride somehow prevented the degradation of dentin, resulting in the improvement of long-term stability at the dentin interface. The hybrid layer created by the self-etching primer/adhesive system, Clearfil Liner Bond 2V, was only partially demineralized and contained apatite crystals scattered within the hybrid layer (Inoue & others, 1999). Fluoride released from the KBF adhesive might reduce the solubility of intrinsic calcium phosphates within the hybrid layer that would tend to solubilize in water over time, resulting in stable bond strength to dentin over time. Burrow, Inokoshi and Tagami (1999) reported that bonding resin absorbs a significant amount of water that may adversely affect the longevity of resin restorations. The susceptibility of the resin to hydrolysis is probably due to a low degree of conversion and cross-linking during polymerization (Shono & others, 1999). According to these previous studies, the weakest portion of the hybrid layer that causes the decrease in longevity seems to be the resin within the inter-fibrillar space. However, Vargas and others (2002) reported that the collagen fibrils of hybrid layers failed to take up heavy metal stains after four years of storage in water, suggesting that the collagen became denatured over time. Okuda & others (2002) proposed that the fall in resin-dentin bond strength was due to hydrolysis of ester bonds of the polymerized resin within the hybrid layer that gradually increased as water diffused through nanoleakage channels that became larger over time. The methacrylates used in dental resins were reported to be susceptible to enzyme-catalyzed hydrolysis by physiologically important esterases (Yourtee & others, 2001). The mineralized dentin matrix contains many enzymes trapped with the matrix (for example, alkaline phosphatase and metalloproteases, including collagenase) that may be released and activated during self-etching or, more slowly, during water incubation. These enzymes could attack the ester bonds of resins in a manner similar to cholesterol esterase (Santerre, Shajii & Leung, 2001) or they could attack collagen (Martin De-Las Heras, Valenzuela & Overall, 2000), or both. On the other hand, it has been reported that fluoride inhibits a number of enzymes including esterases, such as cholinesterase and liver esterase (Wiseman, 1970; Marcos & Townshend, 1995). Fluoride released from fluoride-containing adhesive within the hybrid layer might inhibit these enzymes from attacking the components of the hybrid layer, or the fluoride could prevent the enzymes from being released from the mineralized matrix due to its remineralization action.

Many researchers have demonstrated that the fluoride-releasing ability of glass ionomer cement offers resistance to secondary caries formation around restorations (Geiger & Weiner, 1993; Tam, Chan & Yim, 1997) due to the penetration of fluoride into mineralized dentin. It has usually been assumed that fluoride facilitated remineralization or prevented demineralization (ten Cate, 2000). Glass-ionomer matrices are formed by the reaction of polycarboxylic acids with the metallic salts in reactive glass-fillers and with mineralized hard tissues. These matrices do not normally infiltrate into the dentin matrix (Tay & others, 2001). Their retention and bond strength do not rely on hybrid layer formation. It is likely that such materials are less susceptible to the action of esterases or collagenases. It remains to be determined as to whether fluoride released by glass-ionomer cements could protect overlying composites from salivary esterases (Santerre & others, 2001). Further research is needed to clarify the effect of fluoride-releasing materials on the longevity of adhesive resin restorations in clinical practice.

CONCLUSIONS

Within the limitation of this *in vitro* study, the durability of dentin bonds created by an experimental fluoride-releasing adhesive was improved for the six-month study period compared with a control fluoride-free adhesive. When resin-dentin slabs bonded with the fluoride-containing and fluoride-free adhesive were completely covered with nail varnish, the μTBS of both systems to dentin showed little change over six months of water immersion, indicating that water penetration is a critical component.

Addendum

Since submission of this paper, the manufacturer has obtained all the necessary authorizations to market this product in the US. It will be called Clearfil SE Bond Plus and will be sold as a product separate from Clearfil SE Bond.

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Microwave Drying of High Strength Dental Stone: Effects on Dimensional Accuracy

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Clinical Relevance

Microwave radiation at 490W for 60 seconds to accelerate the drying of gypsum casts produced working dies as accurate as dies from air drying only. Thus, this procedure may be used when accelerated drying of casts is desired.

SUMMARY

High-strength dental stone is widely used to produce dies for the fabrication of restorations with the lost-wax technique. It is normal to wait at least 24 hours for casts to dry and gain sufficient strength prior to initiating laboratory procedures. This waiting time may be greatly reduced by using microwave drying. This study determined the optimum microwave energy density for preserving working die accuracy of a Type IV high-strength dental stone (Silky Rock; Whipmix). Cylindrical die specimens were fabricated according to manufacturer's instructions and allowed to set for one hour. The specimens were subsequently treated as follows: Group I (Control group)—air dried; Group II—microwaved at 700W for 40 seconds; Group III—microwaved at 490W for 60 seconds. The percentage weight loss of cylindrical specimens (n=6) and the percentage dimensional change (n=7) of die specimens in three axes (x, y and z) were determined at 30 minutes, 1 hour and 24 hours after air drying/microwaving. Weight loss was measured using an electronic digital balance, while dimensional changes were assessed using image analysis software. Data was subject to ANOVA/Scheffe's tests at significance level 0.05. No significant difference in percentage weight loss was observed between air drying for 24 hours and microwaved specimens at all time intervals. Although no significant difference in percentage dimensional changes was observed between specimens microwaved at 490W for 60 seconds and specimens air dried for 24 hours, significant changes in x, y and z dimensions were observed after microwaving at 700W for 40 seconds at various time intervals. Microwave radiation at 490W for 60 seconds is recommended for drying Type IV high-strength dental stone.

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Further investigations are required to determine changes in physical properties associated with the aforementioned microwave power density.

INTRODUCTION

High-strength dental stone (ADA Type IV stone) has been the most widely used material in dentistry to produce casts and dies used in the lost-wax technique for fabrication of crowns, bridges and implant-supported prostheses. Its popularity may be attributed to its low cost, ease of use and good dimensional accuracy. There are, however, several disadvantages to its use in the fabrication of dies. These include low abrasion resistance, inadequate tensile strength and the need for a waiting period prior to initiation of laboratory procedures (Rosenstiel, Land & Fujimoto, 1994). New or water-soaked casts need 24-to-48 hours to lose excess water and gain enough hardness and strength to be manipulated without damage (Luebke & Chan, 1985). Although water loss from 8-to-24 hours is only 0.6%, the strength is doubled (Anusavice, 1996). In addition, eliminating excess water prior to manipulation is necessary, as wet stone tends to clog up and reduce the cutting efficiency of instruments.

In dentistry, microwave radiation has been used for sterilization (Hume & Makinson, 1986), polymerization of acrylic resins (De Clerck, 1987; Reitz, Sanders & Levn, 1985), removal of wax from molds (Nomoto, Watari & Komamura, 1985) and shortening the dough stage of denture base acrylic resins (Kimura & others, 1983). Microwaves are short, high frequency radio waves that lie between very high frequency (infrared) waves and conventional radio waves. They are generated in special electron tubes (klystron or magnetron) with built-in resonators or solid-state devices that control frequency. Within an object, the microwaves orientate molecules, particularly water molecules, in a specific direction. The oscillating effect produced by the electron tube changes the orientation of microwaves millions of times per second. The water molecules begin to vibrate as they undergo equally rapid changes in direction. This vibration produces heat that can be used for purposes mentioned earlier. Microwave radiation has also been used for drying dental stone and other gypsum products (Luebke & Chan, 1985; Luebke & Schneider, 1985; Tuncer, Tufekçioglu & Çalikkocaoglu, 1993; Canay & others, 1999). Although the effects of microwave drying on hardness (Luebke & Chan, 1985) and strength (Luebke & Schneider, 1985; Tuncer & others, 1993; Canay & others, 1999) of different gypsum products have been reported, its effects on dimensional accuracy have not been investigated. This study determined the energy density (power ∗ time) for microwave drying of high-strength dental done and investigated the dimensional changes associated with microwave radiation.

METHODS AND MATERIALS

Pilot Study

A pilot study was conducted to determine the microwave energy density (power ∗ time) that produces a similar percentage water loss compared to air drying for 24 hours. Room temperature vulcanizing silicone (Protesil, Autrenal Medizintechnik, Koln, Germany) was used to fabricate molds of a stainless steel cylindrical (30 mm diameter, 10 mm height) master die. Six cylindrical Type IV high-strength dental stone (Silky Rock, Whip Mix Corp, Louisville, KY 40217, USA) specimens were fabricated according to the manufacturer's instructions and recommended powder:liquid ratio (100g powder:23 ml water) using the silicone molds. The stone was mixed at 360 rpm under a vacuum of 710 mm Hg for 30 seconds with a vacuum mixer (Multivac, Degussa, Dusseldorf, West Germany). After one hour, the stone specimens were removed from their molds and weighed with an electronic balance (EW-300A; AND, Tokyo, Japan). One specimen was left to dry for 24 hours under ambient room conditions (temperature 22.1 ± 1.2°C and relative humidity 60 ± 10%). A sling psychrometer (Sunbeam, Boca Raton, FL 32608, USA) was used to test the relative humidity. The remaining five cylindrical specimens were subjected to microwave drying. The microwave oven (R-3S56, Sharp, Tokyo, Japan) used had a maximum power output of 700W and a frequency of 2450 MHz. The five specimens were radiated at 700 W for 30, 40, 50, 60 and 70 seconds, respectively, and weight measurements were taken 30 minutes, 1 hour and 24 hours after microwave radiation. A container with 400 ml of water was placed in the microwave as a heat sink to protect the electron tube of the microwave if all the water was removed from the stone specimen. Only one specimen at a time was microwaved and the stone specimens

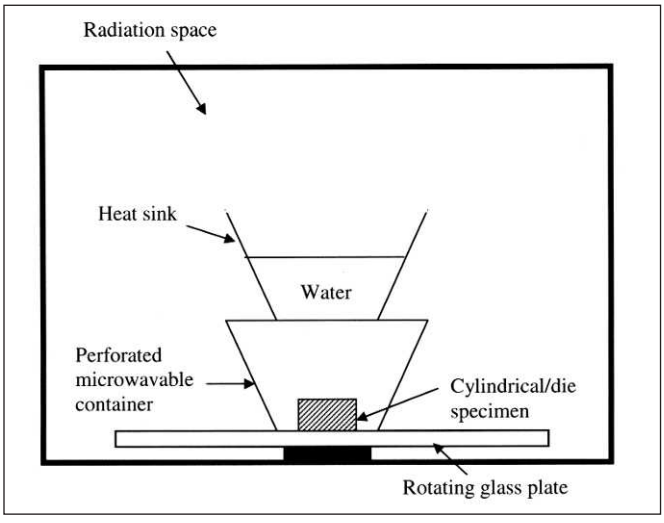

Figure 1. *Diagrammatic representation of the heat sink and specimen container in the microwave oven.*

were placed in the center of a rotating plate underneath the heat sink by using a customized microwavable container (Figure 1). The heat sink was changed for each new specimen. The test was repeated with another six specimens but at 70% of the maximum radiation output (490 W). Specimens radiated at 700 W for 40 seconds and 490 W for 60 seconds had comparable weight changes compared to the specimens that were air dried for 24 hours. These microwave power densities were thus selected for the experiment proper.

Determination of Weight Changes

Eighteen cylindrical (30 mm diameter, 10 mm height) Silky Rock specimens were made. They were allowed to set for one hour, removed from their molds and weighed (W_0) using the electronic balance. The specimens were then randomly divided into three groups and treated as follows: Group I (control)—air-dried under ambient room temperature and humidity (temperature 22.1 ± 1.2°C and relative humidity 60 ± 10%); Group II—microwaved at 700 W for 40 seconds and Group III—microwaved at 490 W for 60 seconds. The specimens were re-weighed 30 minutes, 1 hour and 24 hours after air-drying/microwave radiation. The percentage weight change at the various time intervals, which indicates the loss of excess water, was determined using the following formula:

$$\text{Percentage Weight Change} = \frac{W_x - W_o}{W_o} \times 100\%$$

W_x is the weight at the various time intervals and W_o is the weight immediately after removal from the mold. The mean percentage weight loss for the different treatments at the various time intervals was tabulated. Negative values for percentage weight change indicate the loss of excess water from the stone specimens.

Determination of Dimensional Changes

To determine the effects of microwave drying on the dimensional accuracy of stone dies, a stainless steel master die was fabricated as described by Bailey, Donovan and Preston (1988). The frustum-shaped master die was machined from a 12.7 mm diameter stainless steel rod to a vertical dimension of 12 mm from the base of the master die to the occlusoaxial line. A 13 mm diameter base with a 1.5 mm shoulder was fabricated and resulted in a 10 mm diameter die at the axiogingival line angle. A 4.77-degrees convergence angle produced a 9 mm occlusal end diameter. Two perpendicular lines intersecting at the center of the occlusal surface and terminating at their intersections with the occlusoaxial line angle were scribed in the occlusal surface. A circle (7 mm diameter) was scribed in the occlusal surface by using the intersection of the perpendicular lines as its center. The four points at which the circle intersected the perpendicular scribed lines were used to record the dimensions in the occlusal plane. Two lines, each perpendicular to the long axis of the die, were scribed circumferentially in the surface of the axial wall of the die. One line was located 1 mm gingival from the occlusoaxial line angle and the other 1 mm occlusal from the axiogingival line angle. Another line was scribed parallel to the vertical axis of the die. This line intersected one of the perpendicular lines crossing the occlusal surface and the two circumferential lines previously scribed in the axial wall. The measurement between the circumferential lines along the vertical axis line is referred to as dimension z. The occlusal line intersecting the vertical axis line is referred to as dimension x, and the occlusal line perpendicular to dimension x is referred to as dimension y (Figure 2). To minimize optical errors when reading the dimension z, an alignment platform that compensated for the taper of the frustum was fabricated to hold the

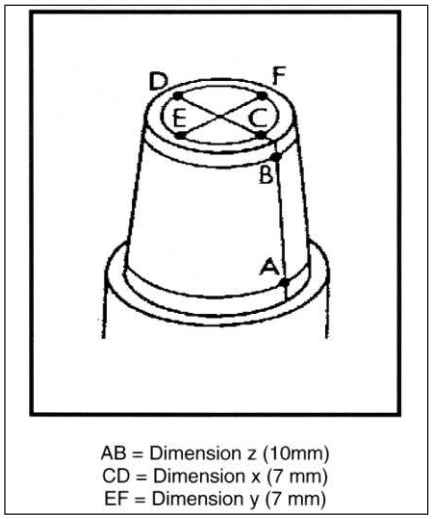

Figure 2. *Diagrammatic representation of the master die.*

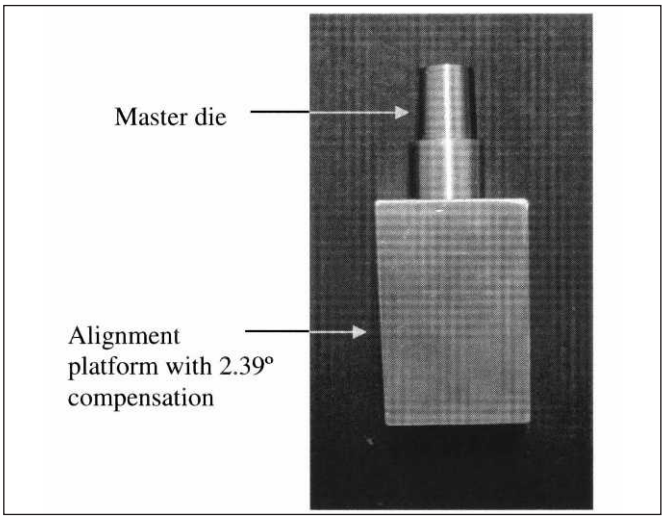

Figure 3. *Side view of the alignment platform with the master die in place.*

specimens during measurements (Figure 3). The alignment platform also had four localization markers machined into it to allow for repeatable positioning of the custom tray during impression taking.

Stone die specimens were individually fabricated at ambient room temperature (22.1 ± 1.2°C) from impressions of the master die. Custom acrylic trays (Tray Resin II; Shofu Inc, Kyoto, Japan) were fabricated using a reusable metal spacer/relief of 2 mm. The custom trays were aligned to the master die by means of the four localization markers on the alignment platform and were fabricated five days prior to impression making. Polyvinylsilox-ane impressions (Aquasil; Denstply Caulk, Milford, DE 19963, USA) of the master die were made using a one-stage, double-mix technique (high and medium viscosities). Adhesion of the impression material to the tray was achieved using tray adhesive and perforations. The impressions were allowed to set and left for one hour prior to fabrication of the stone dies.

Table 1: *Mean Percentage Weight Change Associated with the Various Treatments*

	Time		
Group	**30 minutes**	**1 hour**	**24 hours**
Group I	-0.57 (0.14)	-1.14 (0.36)	-3.77 (0.06)
Group II	-3.14 (0.47)	-3.39 (0.48)	-4.83 (0.52)
Group III	-3.73 (0.67)	-3.99 (0.691)	-4.61 (0.16)

Group I (control) = air dried at ambient room conditions; Group II = microwaved at 700 W for 40 seconds; Group III = microwaved at 490 W for 60 seconds.

Table 2: *Mean Percentage Dimensional Change Associated with the Various Treatment Groups*

Group		**Time**		
		30 minutes	**1 hour**	**24 hours**
Group I	**Dimension x**	0.19 (0.02)	0.39 (0.06)	-0.66 (0.42)
	Dimension y	0.72 (0.07)	1.19 (0.17)	-0.79 (0.35)
	Dimension z	-0.15 (0.11)	1.40 (0.19)	-0.40 (0.17)
Group II	**Dimension x**	-1.02 (0.40)	-1.80 (0.36)	-1.48 (0.42)
	Dimension y	-0.79 (0.19)	-1.44 (0.30)	-1.16 (0.27)
	Dimension z	-0.79 (0.18)	-0.94 (0.15)	-1.10 (0.25
Group III	**Dimension x**	-0.62 (0.08)	-0.63 (0.21)	-1.22 (0.25)
	Dimension y	-0.59 (0.15)	-0.94 (0.14)	-0.68 (0.12)
	Dimension z	-0.61 (0.17)	-0.51 (0.16)	-0.59 (0.17)

Group I (control) = air-dried at ambient room conditions; Group II = microwaved at 700 W for 40 seconds; Group III = microwaved at 490 W for 60 seconds.

Table 3: *Results of Statistical Analysis*

Variables	**Differences**
Mean Percentage Weight Change	No Significant Difference
Mean Percentage Dimensional Change in Dimension x	Group II at 1 hour & 24 hours < Group I at 24 hours
Mean Percentage Dimensional Change in Dimension y	Group II at 1 hour < Group I at 24 hours
Mean Percentage Dimensional Change in Dimension z	Group II at all time intervals < Group I at 24 hours

<denotes statistically significant differences (Results of one-way ANOVA/Scheffe's post-hoc test at p<0.05).

Twenty-one Silky Rock die specimens were fabricated. The die specimens were allowed to set for one hour, removed from their molds and measured in the x, y and z dimensions (M_o). Measurements were made at x 0.8 magnification using a computerized digital image analysis system. This consisted of a stereo microscope (SZ40, Olympus, Tokyo, Japan), which was attached to a CCD camera (SSCDC58AO, Sony Corp, Tokyo, Japan) and computer software (Microimage Version 4.0, Olympus Optical Co, Hamburg, Germany). The system had a measuring accuracy of 0.001 mm. For each specimen, the microscope image was digitized, captured, saved and measured in the following sequence: dimension z, dimension x and dimension y. The alignment platform ensured that dimension z was parallel to the horizontal axis when measurements were taken. The die specimens were subsequently divided into three groups and treated the same as the first part of this study. Dimensions x, y and z were re-measured 30 minutes, 1 hour and 24 hours after air drying and microwave radiation at 700 W for 40 seconds and 490 W for 60 seconds. All computerized optical measurements were conducted by one operator. The percentage dimensional change was determined using the following formula:

$$\text{Percentage Dimensional Change} = \frac{M_x - M_x}{M_o} \times 100\%$$

M_x represents measurements of dimensions x, y and z of individual stone dies at the various time intervals and M_0 represents measurements of dimensions x, y and z of the dies immediately after removal from the molds. The mean percentage dimensional change in dimensions x, y and z for the different treatments at the various time intervals was tabulated. Positive values indicate an increase, while negative values indicate a decrease in dimensions. Mean percentage weight and dimensional change at the various time intervals after microwave radiation was compared to percentage weight and dimensional change after 24 hours of drying

in air using one-way ANOVA and Scheffe's post-hoc test at significance level 0.05.

RESULTS

The mean percentage weight loss and dimensional changes associated with the various treatments are shown in Table 1 and 2. Table 3 shows the results of statistical analysis. At all time intervals no significant difference in weight changes was observed between microwaved specimens and specimens that were air dried for 24 hours. Although no significant difference in percentage dimensional change was observed for Group III, significant differences were observed for Group II when compared to the control group (Group I) at 24 hours. For dimension x, significant differences were observed at one and 24 hours. For dimension y, significant difference in percentage dimensional change was observed at one hour. For dimension z, significance differences were observed at all time intervals.

DISCUSSION

This study was undertaken to determine a suitable microwave energy density for the drying of Type IV high-strength dental stone in a microwave oven and to evaluate the dimensional accuracy of stone dies associated with microwave drying. A one-hour waiting time prior to microwave radiation was adopted to allow for setting of the stone and loss of excess water. Microwave ovens cannot be used to dry extremely wet or water-soaked cast or dies, as the boiling of free water may crack the casts or dies (Luebke & Chan, 1985). Mahler (1951) stated 7% excess water remained in air-dried gypsum materials one hour after mixing. This amount of excess water appears acceptable for microwave drying provided specimens are located under the heat sink as described in this paper. In the authors' earlier pilot studies, placing the heat sink in the center of the rotating glass plate and specimens at the periphery resulted in crack formation of the latter. Denser high-strength stone has been shown to be more susceptible to crack formation than more porous gypsum materials such as plaster and Type III stone (Luebke & Chan, 1985). The greater porosity of plaster and Type III stone allows the escape of steam formed during the microwave drying process. Luebke and Chan (1985) suggested that fewer cracks and holes in gypsum products might be obtained by microwave drying at two hours after mixing because less excess water would remain in the materials. Investigations have, however, not been performed to determine the ideal time after the mixing of dental stone before microwave drying.

The microwave energy densities used in this experiment (700 W for 40 seconds and 490 W for 60 seconds) differed from those used in previous studies. The energy densities that were employed ranged from 1450 W for five minutes to 55 W for 15 minutes (Luebke & Chan, 1985; Luebke & Schneider, 1985; Tuncer & others, 1993; Canay & others, 1999). Frequency of the microwave ovens was the same (2450 MHz) despite the large variations in power and time. Although the microwave oven used in this study allowed for low power radiation (50% of maximum power), this power level was not selected, as elimination of excess water was inadequate even after 10 minutes of radiation when earlier pilot studies were conducted. This finding corroborated that of Tuncer and others (1993), who found that increasing exposure time from five to 15 minutes is not effective for drying gypsum products when low power level is used. Several other observations were also made from earlier pilot studies. It was found that the further specimens were placed from the center of the rotating plate, the longer it takes to remove excess water in the stone. Cracks formed from placing specimens at the periphery of the rotating plate tended to radiate towards the center of the microwave oven. The placement of less than 400 ml of water resulted in the stone specimens cracking. The volume of water required for use as a heat sink may vary depending on the volume of stone being microwaved.

The lack of statistical significance in weight changes between microwaved specimens and those that were air dried for 24 hours does not imply that microwaved specimens are dimensionally stable or accurate. In addition, microwave radiation could also compromise the physical properties of high-strength stone. While one study reported no difference in strength between microwaved and air dried specimens at 24 hours (Luebke & Schnieder, 1985), others found that microwave radiation decreased strength and hardness of gypsum materials (Luebke & Chan, 1985; Tuncer & others, 1993). The apparent discrepancy in results can be attributed to the different microwave energy densities used. As temperatures approach 45° to 90°C, the calcium sulphate dehydrate formed during the setting reaction could revert to the hemihydrate as follows: $2CaSO_4$, $2H_2O \leftrightarrow (CaSO_4)_2$, $H_20 + 3H_2O$ (O'Brien, 1997a). This reaction is, fortunately, slow even at 90°C, and complete conversion takes about 12 hours (Khalil, Hussein & Gad, 1971). Nonetheless, the aforementioned could result in changes in physical properties and dimensions especially when high power radiation is used for long exposure periods.

The setting reaction of gypsum causes a decrease in the true volume of the reactants early in the setting process when the mix is fluid. However, once the mix becomes rigid (marked by the loss of surface gloss), an expansion is observed that results from growth pressure of the gypsum crystals that form (O'Brien, 1997a). The latter accounts for the positive values for percentage dimensional change generally observed at 30 minutes and one hour for air-dried specimens. After 24 hours, shrinkage was observed with the air-dried spec-

imens. Despite this relative shrinkage, the linear setting expansion of high-strength dental stone is approximately 0.10% (O'Brien, 1997a). Although the dimensional changes of dental stone are assumed to be isotropic, this assumption is not always justified. If restraint is imposed in some directions but not others, dimensional changes can be far from isotropic (Teraoka & Takahashi, 2000). Results of this study suggest that dimensional changes of high-strength stone may not be isotropic. This agreed with the findings of Chaffee, Bailey and Sherrard (1997), who assessed the dimensional accuracy of high-strength stone when used for producing single dies.

Although significant differences in dimensions x, y and z were observed between specimens that were microwaved at 700 W (high power) for 40 seconds and specimens that were air dried for 24 hours, no significant difference was observed when the specimens were microwaved at 490 W (medium power) for 60 seconds. High power radiation could result in greater vibration of the water molecules, leading to the generation of more heat. In addition to possible changes in physical properties, the latter could result in more water loss and shrinkage as was observed in this study. The clinical fit of crowns fabricated from dies that are dried by high power microwave radiation could be compromised as they may be smaller than the tooth preparations. When high power radiation was used, significant differences in dimension z were observed as early as 30 minutes after microwave drying. The 30-minute waiting period prior to the first measurement was necessary to allow for cooling of specimens. This avoids measurement errors due to thermal expansion of the stone and facilitates handling of the specimens. Significant differences in dimensions x and y were observed only after one and/or 24 hours. This observation may be partly accounted for by the low thermal conductivity of gypsum (O'Brien, 1997b). Microwave drying of high-strength dental stone should, therefore, be done at medium power (around 490 W). Radiation times may need to be varied depending on the volume of stone to be dried. The latter, together with the effects of microwave drying on abrasion resistance and other physical properties, warrants further investigations. The effect of microwave drying on the dimensional accuracy of full-arch casts also needs to be studied. Medium power microwave drying can save considerable time in the production of dies for the fabrication of restorations with the lost-wax technique.

CONCLUSIONS

Within the limits of this study, the following conclusions can be drawn:

1. Microwave drying of high-strength dental stone at 700 W for 40 seconds and 490 W for 60 seconds resulted in similar excess water loss as air drying for 24 hours.
2. Differences in dimensional changes for specimens microwaved at 700 W for 40 seconds and specimens that were air dried for 24 hours were statistically significant.
3. Differences in dimensional changes for specimens microwaved at 490 W for 60 seconds and specimens that were air dried for 24 hours were not statistically significant.
4. Microwave radiation at 490 W for 60 seconds is acceptable for drying Type IV high-strength dental stone.

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Influence of Polymerization Technique on Microleakage and Microhardness of Resin Composite Restorations

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GMB Ambrosano • LAF Pimenta

Clinical Relevance

The conventional technique for polymerization, used in association with a "packable" resin composite, provides similar resin-tooth interfacial seal to Soft-Start and better seal when compared to PAC; however, for a microhybrid resin composite, all techniques for polymerization present the same result.

SUMMARY

This study evaluated the influence of three polymerization techniques on microleakage and microhardness of Class II restorations using a microhybrid (Filtek Z250) and a "packable" resin composite (SureFil). The techniques, their respective light intensities and time used in relation to the resin composites, are: Conventional (C)—800mW/cm² for 40 seconds; Soft-Start (SS1)—75mW/cm² for 10 seconds plus 518mW/cm² for 30 seconds; Soft-Start (SS2)—170mW/cm² for 10 seconds plus 518mW/cm² for 30 seconds and Plasma Arc Curing (PAC)—1,468mW/cm² for three or six seconds. One hundred and fifty-two "Vertical Slot type Class II cavities" at the mesial and distal surfaces were prepared and divided into eight groups (n=19). After the restorative procedures, the samples were thermocycled (1,000 cycles at 5°C and 55°C), then immersed in 2% methylene blue dye solution for four hours. The microleakage was evaluated and the results analyzed by the Kruskal–Wallis and Multiple Comparisons tests. Ten samples from each group were randomly selected, embedded in polyester resin, polished and submitted to the Knoop microhardness test. ANOVA (split-plot) and Tukey's test ($p<0.01$) revealed significant differences among depths: the hardness at the top surface was significantly higher followed by the middle and bottom surfaces. There was no significant difference in microleakage among the techniques when microhybrid resin composite was employed. However, when using a "packable" resin composite, the conventional technique for polymerization was comparable to Soft-Start and better than PAC.

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INTRODUCTION

Since their introduction to the market in the 1970s, light curing resin composites have been used for restorations, making dental procedures more conservative and able to serve esthetic demand. However, some material shortcomings, such as reduced wear resistance, marginal staining and excessive polymerization shrinkage and the sensitivity of the technique, have not been eliminated despite extensive research (Leinfelder, 1995). The success of the clinical performance of light curing resin composites is directly related to adequate polymerization and light intensity, which are crucial factors in obtaining optimal physical properties (Bayne, Heymann & Swift, 1994).

During the setting process, polymerization shrinkage of a resin composite can create forces that may disrupt the bond to cavity walls (Davidson, de Gee & Feilzer, 1984; Donly & others, 1987; Carvalho & others, 1996). This competition between contracting forces built up in the polymerizing resin and the bonds of adhesive resins to the wall of the restoration is one of the main causes of marginal failure and subsequent microleakage (Davidson & others, 1984; Mandras, Retief & Russel, 1991). Bond strength must be greater than contraction stress in order to obtain stable marginal adaptation. Microleakage permits the passage of bacteria, fluids, molecules and toxins and could encourage dentinal hypersensitivity, pulp inflammation, secondary caries and pulp necrosis (Kidd, 1976; Opdam & others, 1998).

Some studies have shown a relation between polymerization shrinkage and light intensity (Feilzer & others, 1995; Silikas, Eliades & Watts, 2000). As a result, different light units have been introduced into the market to minimize or control the polymerization shrinkage of composites.

Conventional lamps instantly provide maximal light intensity, which causes the resin composites to harden and produce a considerable increase in viscosity of the material (Goracci, Mori & Casa de'Martinis, 1996). Composites cured at low light intensity have been shown to have a better marginal adaptation (Mandras & others, 1991; Uno & Asmussen, 1991). The theory is that a slower rate of conversion maintains a longer pre-gel phase, allowing for a better flow of the material, which decreases contraction stress in the filling material. However, this low intensity may affect the surface hardness and may be insufficient for ensuring mechanical stability (Unterbrink & Muessner, 1995; Pimenta, 1999).

Pre-polymerization at low intensity, followed by the final cure at high intensity, can allow for the flow of resin composite during setting. This method can reduce the width and length of marginal gaps without interfering with the physical properties of the restorations (Uno & Asmussen, 1991; Mehl, Hickel & Kunzelmann, 1997).

Now available, high-intensity light units based on a plasma system can reduce the long cure time and provide optimal properties in resin composite in a few seconds (Peutzfeldt, Sahafi & Asmussen, 2000; Park, Krejci & Lutz, 2002). However, the use of units with such high intensities could create more contraction forces and, consequently, marginal failure (Brackett, Haisch & Covey, 2000).

New methods of polymerization with varying intensities and curing times are on the market; therefore, it is necessary to analyze the effectiveness in the control of marginal adaptation and the quality of polymerization. This study evaluated the microleakage and microhardness of Class II resin composites using three available polymerization techniques—Conventional (Optilux501, Demetron/Kerr, Danbury, CT 06810, USA), Plasma Arc Curing (PAC, APOLLO 95E Elite, DMD Corp, Westlake Village, CA 91362, USA) and Soft-Start (Variable Intensity Polymerization, BISCO Inc, Schaumburg, IL 60193, USA) and two different resin composites—a microhybid (Filtek Z250, 3M Dental Products, St Paul, MN 55144, USA) and a "packable" (SureFil, Dentsply/Caulk, Milford, DE 19963, USA).

METHODS AND MATERIALS

Microleakage Test

Seventy-six extracted bovine incisors were initially stored in a 2% formaldehyde buffered solution (Eick & Welch, 1986; de Castro, Hara & Pimenta, 2000; Gallo & others, 2001), after which debris was removed from the teeth. The crowns of the bovine teeth were cut off 5 mm above the cement-enamel junction (CEJ) with a double-faced diamond disk (KG Sorensen Ind Com Ltda, Barueri, SP 06442-110, Brazil).

"Vertical Slot type Class II cavities" at the mesial and distal surfaces were prepared with #245 carbide burs (KG Sorensen Ind Com Ltda) with a high-speed water-cooled handpiece (Kavo do Brasil AS, Joinville, SC 89221-040, Brazil). The burs were replaced after every 10 preparations to maintain uniformity. Butt-joint cavities had the following dimensions: 1.5 mm axial deep by 3 mm bucco-lingual wide and the gingival margin was located 1 mm apical to the CEJ.

In all groups, enamel and dentin etching with 35% phosphoric acid was performed for 15 seconds. Single Bond (3M Dental Products) adhesive system was applied following manufacturer's instructions. The resin composites SureFil (Dentsply/Caulk) and Filtek Z250 (3M Dental Products) were inserted in three horizontal increments and each increment was polymerized on the occlusal surface according to the following groups (n=19):

GROUP 1: SureFil (Dentsply/Caulk) resin composite and Conventional (C) polymerization technique for 40 seconds, each increment, showing an average intensity of 800 mW/cm^2;

GROUP 2: SureFil (Dentsply/Caulk) resin composite using Soft-Start (SS1) polymerization technique (Variable Intensity Polymerizer, BISCO, Inc) showing an average initial intensity of 75 mW/cm^2 for 10 seconds and 518 mW/cm^2 for the subsequent 30 seconds;

GROUP 3: SureFil (Dentsply/Caulk) resin composite using Soft-Start (SS2) polymerization technique (Variable Intensity Polymerizer, BISCO, Inc) showing an average initial intensity of 170 mW/cm^2 for 10 seconds and 518 mW/cm^2 for the subsequent 30 seconds;

GROUP 4: SureFil (Dentsply/Caulk) resin composite using Plasma Arc Curing (PAC, APOLLO 95E Elite, DMD Corp) polymerization technique (APOLLO 95E Elite, DMD Corp) showing an average intensity of 1,468 mW/cm^2 for six seconds each increment, following manufacturer's instructions for this resin composite;

GROUP 5: Filtek Z-250 (3M Dental Products) resin composite and Conventional (C) polymerization (Optilux501, Demetron/ Kerr) for 40 seconds each increment, showing an average intensity of 800 mW/cm^2;

GROUP 6: Filtek Z250 (3M Dental Products) resin composite using Soft-Start (SS1) polymerization technique (Variable Intensity Polymerizer, BISCO, Inc) showing an average initial intensity of 75mW/cm^2 for 10 seconds and 518mW/cm^2 for the subsequent 30 seconds;

GROUP 7: Filtek Z250 (3M Dental Products) resin composite using Soft-Start (SS2) polymerization technique (Variable Intensity Polymerizer, BISCO, Inc) showing an average initial intensity of 170 mW/cm^2 for 10 seconds and 518 mW/cm^2 for the subsequent 30 seconds;

GROUP 8: Filtek Z250 (3M Dental Products) resin composite using the Plasma Arc Curing (PAC, APOLLO 95E Elite, DMD Corp) polymerization technique showing an average intensity of 1,468 mW/cm^2 for three seconds for each increment, following the manufacturer's instructions for this resin composite.

Following the restorative procedure, the teeth were stored in water at 37°C for 48 hours. All restorations were then finished with Sof-Lex (3M Dental Products) fine and ultra fine finishing disks and all specimens were thermocycled in a thermal cycling machine (MCT2-AMM instrumental, CA 94928, USA) for 1000 cycles at 5 ± 2°C and 55 ± 2°C with a dwell time of 60 seconds in distilled water and a five-second transfer time. Next, the apices and coronal surfaces were sealed with epoxy resin (Araldite, Brascola Ltda, São Bernardo do Campo, SP 09771-190, Brazil) and the teeth were coated with two applications of fingernail polish up to 1 mm from the gingival margins. All teeth were immersed in a freshly prepared aqueous 2% methylene blue solution (pH 7.0) for four hours at 37°C, then washed in water. Finally, each tooth was sectioned vertically through the center of the restoration with a diamond disk (KG Sorensen Ind Com Ltda) at low speed.

Microleakage at the gingival margin was evaluated by two observers with an optical stereomicroscope (Meiji Techno Co, LTD, Iruma-gun Saitana 356, Japan) at 70x magnification and scored using the following criteria (Figure 1):

0 - No dye penetration.

1 - Dye penetration that extended up to 1/3 of preparation depth.

2 - Dye penetration greater than 1/3, up to 2/3 of preparation depth.

3 - Dye penetration extending to the axial wall.

4 - Dye penetration past the axial wall.

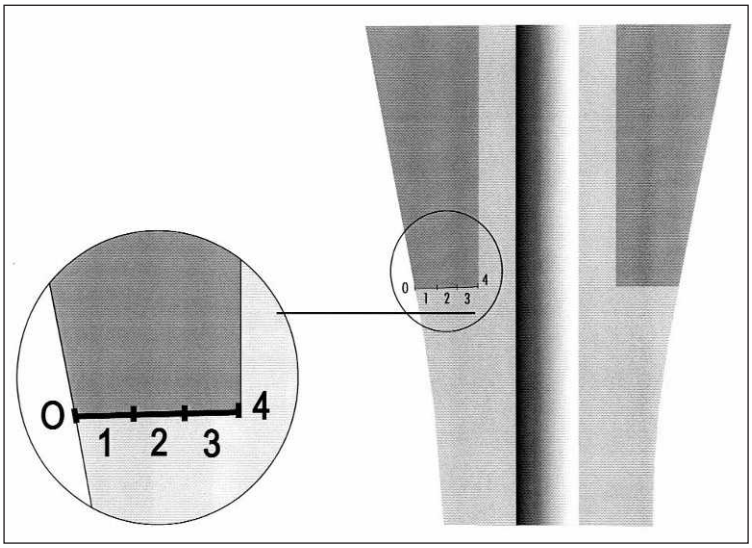

Figure 1. *Diagram of microleakage evaluation criteria.*

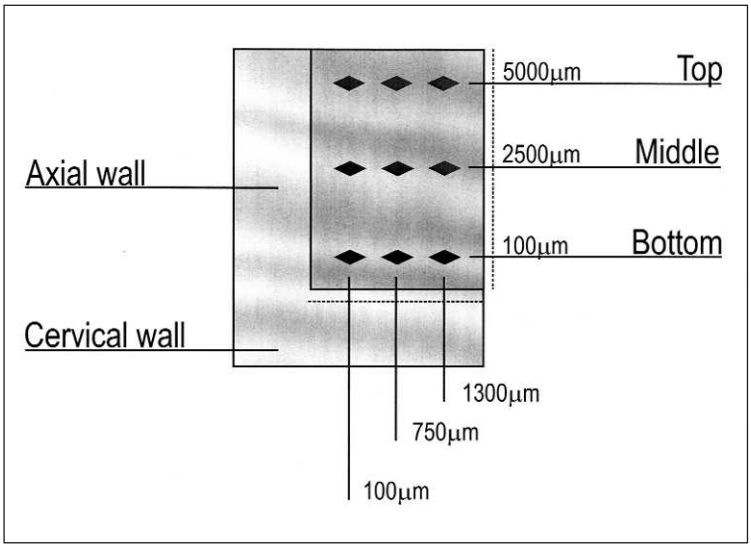

Figure 2. *Diagram of Knoop indentation locations.*

The results were analyzed by the Kruskal–Wallis and Multiple Comparisons tests.

Knoop Microhardness Test

After the microleakage evaluation, 10 sectioned restorations of each group were randomly selected and cut off with a double-faced diamond disk (KG Sorensen Ind Com Ltda). Twenty-six groups of three and one group of two restorations were placed in a 3/4 inch diameter PVC ring filled with self-curing polystyrene resin (Piraglass, Piracicaba, SP 13424-550, Brazil). The embedded restorations were ground on a water-cooled mechanical grinder (Maxigrind, Solotest, São Paulo, SP 01328, Brazil) using 400, 600 and 1000-grit Al_2O_3 abrasive paper (Saint-Gobain Abrasivos Ltda, Guarulhos, SP 07111150, Brazil). The restorations were polished on a mineral oil-cooled grinder using felts with diamond pastes of 3 µm and 1 µm (Equilam, Diadema, SP 09960-500, Brazil).

The Knoop microhardness test (Microhardness Tester, Future Tech FM-1E, Future Tech Corp, Tokyo 140, Japan) was performed using a 25g load for 20 seconds. The indentations were placed at 100, 2,500 and 5,000 µm from the gingival margin, and at 100, 750 and 1,300 µm from the axial wall (Figure 2). The larger diagonal length of indentation was measured with a monitor (9M 100A Teli, Tokyo 140, Japan) and the values transformed to Knoop Hardness Numbers (KHN).

The microhardness means for each depth and experimental group was calculated and submitted to the ANOVA split-plot and Tukey's test that was used to compare Knoop microhardness among groups, depths and resin composites.

RESULTS

Microleakage Test

None of the groups showed complete prevention of dye penetration. The results of the statistical analysis are summarized in Table 1.

Analyzing the data, the SureFil (Dentsply/Caulk) "packable" resin composite showed better results when using the Conventional technique. The SS1 and SS2 techniques presented intermediate results, although they showed no statistical differences from PAC, which demonstrated the worst scores. The Conventional technique for polymerization provided a similar resin-tooth interfacial seal to that of Soft-Start (Variable Intensity Polymerization, BISCO Inc) and a better seal when compared to Plasma Arc Curing (PAC, APOLLO 95E Elite, DMD Corp).

For Filtek Z250 (3M Dental Products) resin composite, there was no significant difference in leakage among the different methods of polymerization.

Knoop Microhardness Analysis

No significant differences in microhardness were observed between the resin composites (p=0.1701) and the C, SS1, SS2 and PAC unit polymerization techniques (p=0.7103).

The results showed no significant interaction among the resin composites vs light units (p=0.9111), resin composites vs depth (p=0.3511), light units vs depth (p=0,2646) and light units vs resin composite vs depth (p=0.4173) in microhardness values.

The Tukey's test (p<0.01) revealed significant differences in microhardness in relation to depth/thickness of resin. Hardness at the top surface (5,000 µm) was significantly higher, followed by the middle (2,500 µm) and bottom (100 µm) surface, which showed lower KHN means (Table 2). These findings were similar for both resins and curing techniques.

Table 1: *Results of Microleakage Evaluation*

Groups	Medium Ranks	
G5. Z250/Conventional	55.4737	**a**
G6. Z250/SS1	55.4737	**a**
G1. SureFil/Conventional	63.1316	**ab**
G7. Z250/SS2	70.0263	**abc**
G8. Z250/PAC	81.6579	**abcd**
G2. SureFil/SS1	87.6316	**bcd**
G3. SureFil/SS2	96.8947	**cd**
G4. SureFil/PAC	101.7105	**d**

Kruskal-Wallis test: Significant difference (p<0.05)
Same letters were not statistically different

Table 2: *Means and Standard Deviations Knoop Hardness Number (KHN) for the Different Cure Modes, Resin Composite and Depth*

Resin Composite	Cure Mode	Depth					
		Bottom (100 µm)		Medium (2,500 µm)		Top (5,000 µm)	
		Mean	SD	mean	SD	mean	SD
SureFil	C	100.06	25.44	107.45	13.59	112.82	11.36
SureFil	SS1	103.69	13.46	112.30	8.66	109.04	11.12
SureFil	SS2	95.94	16.12	100.64	20.73	109.13	11.76
SureFil	PAC	95.20	20.76	100.43	21.0	120.20	10.33
Z250	C	99.15	15.08	100.73	16.21	100.67	13.06
Z250	SS1	94.23	22.42	108.75	26.46	109.20	18.85
Z250	SS2	96.44	15.03	104.04	7.89	105.97	12.11
Z250	PAC	97.65	16.46	99.80	19.25	105.80	13.82
Mean		97.80 C		104.27 B		109.1A	

Tukey´s test (p<0,05) indicates statistical difference for means followed by distinct letters

DISCUSSION

Some techniques for reducing shrinkage stress and, consequently, marginal leakage have been suggested (Kays, Sneed & Nuckles, 1991). These include using reflective wedges (Lutz, Krejci & Barbakow, 1992), incremental restorative techniques (Tjan, Bergh & Lidner, 1992; Applequist & Meiers, 1996) and variations in light intensity (Uno & Asmussen, 1991; Feilzer & others, 1995; Unterbrink & Muessner, 1995). A lining material with a low-modulus of elasticity, such as a glass ionomer (Aboushala, Kugel & Hurley, 1996), a new generation of dentin bonding (Goracci, Mori & Bazzucchi, 1995; Nakabayashi & Saimi, 1996) or a flowable composite lining has also been proposed by some authors, mainly in association with the "packable" resin composite (Ferdianakis, 1998; Chuang, Liu & Jin, 2001).

The influence of using different kinds of light units with varying intensities during polymerization to reduce microleakage was evaluated in this study using a "packable" and a microhybid resin composite.

None of the methods or restorative materials eliminate microleakage in the face of thermal changes and differences in the coefficient of thermal expansion between dental tissues and the restorative material. These results were also observed in other studies (Liberman, Gorfil & Ben-Amar, 1996; Pimenta, 1999).

Both resins behaved differently when subjected to the same polymerization technique. While the microhybrid presented statistically similar results for all methods, the "packable" did not. In association with PAC units (G4) and SS2 (G3), the "packable" was statistically different in relation to C (G1) and SS1 (G2). The "packable" presented a high elasticity modulus that can cause more strain in the interface during polymerization (Davidson & others, 1984). Another reason may be that the "packable" composite may not adapt well to the dentin bonding agent and cavity preparation walls (Meiers, Kazemi & Meier, 2001).

The high microleakage scores that were found when the "packable" was compared to the microhybrid might indicate that the filler particle technology of the "packable" composite could translate into increased post-gel linear shrinkage stress directed at the margins (Meiers & others, 2001). Stress arising from post-gel polymerization shrinkage may produce defects in the composite-tooth bond, leading to bond failure and, consequently, post-operative sensitivity, microleakage and recurrent caries (Yap, Soh & Siow, 2002; Meiers & others, 2001). The more satisfactory results found for the microhybrid resin when compared with the "packable" in this study could be explained by the lower post-gel shrinkage as revealed by the manufacturers.

Different studies have indicated that Soft-Start (Variable Intensity Polymerization, BISCO Inc) light curing units can be used to improve marginal integrity and decrease marginal gap (Uno & Asmussen, 1991; Goracci & others, 1996). However, according to the results of this study, less leakage was not observed when the Soft-Start technique (Variable Intensity Polymerization, BISCO Inc) was used compared to Conventional and Plasma Arc (PAC, APOLLO 95E Elite, DMD Corp). Other studies also reported these results (Sahafi, Peutzfeldt & Asmussen, 2001; Yap & others, 2002; Yap, Ng & Siow, 2001). For both pre-polymerizations, starting with 75 mW/cm^2 (G2 e G6) or 170 mW/cm^2 (G3 e G7), the groups presented no statistical differences between the resins. However, the association of the "packable" with SS2 (G3) was not similar to SS1 with the microhybrid resin (G6).

The "packable" resin composite cured with Plasma Arc (PAC, APOLLO 95E Elite, DMD Corp) curing showed the highest leakage scores. However, it was not statistically different from Plasma Arc (PAC, APOLLO 95E Elite, DMD Corp) with the microhybrid (G8), which behaved similarly with all techniques. Several studies have shown that high and fast curing rates tend to produce excessive polymerization stresses on adhesive bonds, resulting in poor marginal adaptation along gingival or dentinal margins (Brackett & others, 2000; Uno & Asmussen, 1991; Mehl & others 1997). This study's results seem to show that the low flow capacity of "packable" resin composite might be responsible for these values.

In this study, the microhardness of resin composites was measured in different depths as an indirect method for evaluating the relative degree of conversion (Mehl & others, 1997). The effective cure of resin composite is vital, not only to ensure optimum physical-mechanical properties (Asmussen, 1982), but also to ensure that clinical problems do not arise due to cytotoxicity of inadequately polymerized material (Caughman & others, 1991). In general, higher hardness values are an indication of more extensive polymerization (Helvatjoglou-Antoniadi & others, 1991).

According to the results, the resin composites SureFil (Dentsply/Caulk) and Filtek Z250 (3M Dental Products) presented similarly when the C, SS1, SS2 and PAC unit polymerization techniques were used.

There was a significant difference in depth among the bottom (100 µm), middle (2,500 µm) and top (5,000 µm) surfaces. For all techniques, microhardness was higher at the top surface. This can probably be explained as a result of the relationship between irradiation distance and effectiveness of polymerization (Pires & others, 1993). The depth of cure was reduced by increasing the distance between the light tip and composite surface (Hansen & Asmussen, 1997). The degree to which light activated composite polymerizes is proportional to the amount of light to which the material is exposed (Rueggeberg, Caughman & Curtis, 1994). The top surface of the

material was nearer to the light force than the subsequent resin composite layers; in this way, light transmission did not suffer any interference and the intensity was not reduced. However, at the middle and bottom surfaces the light intensity was greatly reduced due to light scattering, thus, decreasing the effectiveness of polymerization (Ruyter & Oysaed, 1982). One way to compensate for this is to increase the light exposure time, which can provide better hardness results (Ota & others, 1985; Yap & others, 2001).

Although some studies demonstrated that three seconds of curing time was insufficient for optimal curing of composites when the Plasma Arc (PAC, APOLLO 95E Elite, DMD Corp) technique was used (Park & others, 2002), the results found in this study showed similarities among C, SS1 and SS2 for the microhybrid resin composite.

Despite the great advances in light units that present new polymerization techniques, the conventional method is still preferred. Providing adequate polymerization and satisfactory infiltration scores, the Conventional method may be similar to Soft-Start (Variable Intensity Polymerization, BISCO Inc) and better than PAC, although each material had different characteristics.

CONCLUSIONS

The results of this study allow the authors to conclude:

1. None of the techniques could eliminate microleakage;
2. For Filtek Z250 (3M Dental Products) microhybrid resin composite, all the polymerization techniques showed similar leakage results;
3. For SureFil (Dentsply/Caulk) "packable" resin composite, only the Soft-Start polymerization technique (SS1) (Variable Intensity Polymerization, BISCO Inc) with a 10-second initial intensity of 75mWcm^2, followed by 30 seconds at 518mW/cm^2, decreased microleakage to levels similar to the Conventional technique;
4. All polymerization techniques presented similar results in microhardness values, but the top surface always presented high values followed by the middle and bottom surfaces.

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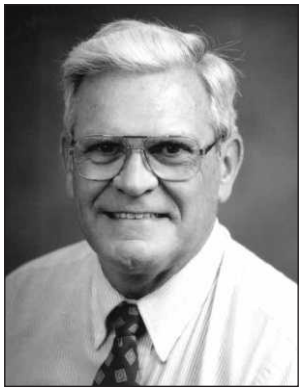
Academy of Operative Dentistry
Award of Excellence
Dr Lawrence L Clark

Dr Clark received his DDS from the University of Maryland School of Dentistry in 1961 and his MS in Operative Dentistry from the University of Texas Health Science Center in Houston in 1969. During his distinguished career in the United States Air Force, Dr Clark directed the Air Force General Practice Residency Program, served as consultant to the Surgeon General and retired in 1980 as Colonel. He then embarked upon a second and equally distinguished career in dental academics, beginning at the University of Mississippi School of Dentistry, serving as Professor and Director of the General Practice Residency Program. He was also Coordinator of Mississippi's elective course in Direct Gold, another indicator of his commitment to this admirable discipline.

In 1985, Dr Clark joined the faculty at the University of Florida College of Dentistry, directing their Advanced Education in General Dentistry Program. Soon he was teaching Operative Dentistry throughout the College curriculum. It was through this effort that he won, again, the admiration and love of his students. Upon his retirement from Florida, Larry was selected by the graduating class for very special honors indeed, a testament to his expertise, exceptional ability as a teacher and his uncompromising standards of ethical behavior.



Lawrence L Clark

Equally important to Larry's background of many presentations and publications is his history with the Academy. He served tirelessly for many years as Editor of *The Condenser*, and also served admirably as our President in 1985. Dr Clark's vocation is DENTISTRY! His calling is MENTORING! Throughout his military and academic careers he has counseled, encouraged and taught. He has always been the ideal teacher. He repeatedly received "Teacher of the Year" awards at both Mississippi and Florida. It is significant that so many of his former students still keep in touch and continue to seek his advice both in clinical care and in the ethical treatment of patients.

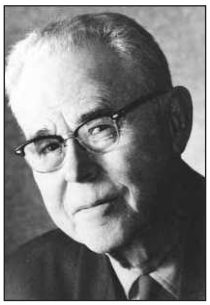
It is an honor for the Academy to present the 2003 Award of Excellence to this optimum dentist, esteemed professional and incomparable teacher…Dr Lawrence L Clark.

Richard Wilson

Academy of Operative Dentistry

Hollenback Memorial Prize

Dr John W Osborne



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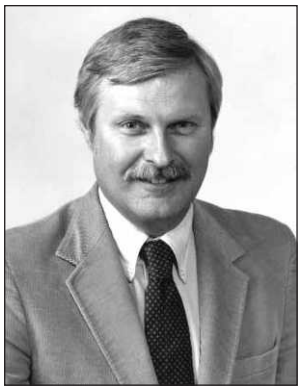


John W Osborne

Dr John Osborne is a pioneer in clinical research of dental materials. He received his DDS in 1963 and MSD in 1968, both degrees from Indiana University. His academic career started as an Instructor in the Department of Operative Dentistry and Dental Materials at State University of New York at Buffalo where he advanced to Associate Professor in 1971. In 1973 he became Associate Professor of Dental Materials at Indiana University. He moved on in 1978 to become Professor in the Department of Restorative Dentistry at State University of New York at Stony Brook. Since 1987 he has been Professor and Director of Clinical Research, Department of Restorative Dentistry, University of Colorado Health Science Center. He has also been part-time in private practice.

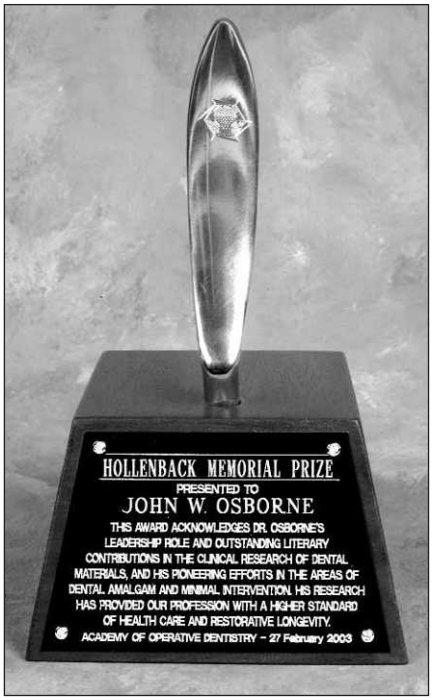
John has received many awards, including teaching awards. He has lectured extensively and presented numerous research reports. As a gifted and enthusiastic lecturer he has been invited to lecture in many parts of the world. He was cited by our own journal, *Operative Dentistry* in 1990, as one of 25 authors in this century for "outstanding literary contributions" whose "whole body of literature is a classic in Operative Dentistry." He has also received the Floyd Payton Award for Excellence in Clinical Research from the Dental Materials Group of the American Association for Dental Research.

While Dr Osborne is best known for his enduring clinical trials on dental amalgam, his clinical studies extend into the broad scope of materials available to our profession. The range is from luting agents, to composite resins, to glass ionomers and encompasses the spectrum of materials in between these classes. His efforts in clinical and laboratory investigations also include materials which, due in part to his findings, never found a way to the market place, as they did not meet the gold standard for our profession.

Dr Osborne's research has led to broader resources for general practitioners and an evolution in the fundamental processes for development of better dental restorations. The scale to assess ditched margins of amalgam restorations based on John's illustrations was instrumental in the development of non-gamma-two amalgams and has been used worldwide in clinical research. In addition, Dr Osborne's innovative ideas were the inspiration for publications in the areas of minimal intervention restorations; a revision of extension for prevention, and Operative Dentistry education. The fruits of his labor have served to upgrade the dental profession and have given us the ability to offer our patients a continually improved standard of dental care.

It is with great pride that the Academy of Operative Dentistry has selected Dr John Osborne as the recipient of the 2003 Hollenback Memorial Prize. It is an honor for me to present this award in recognition of his determined and passionate spirit in service to both the profession of dentistry and to the Academy.

Ivar Mjör



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All material submitted for publication must be submitted exclusively to *Operative Dentistry*. Manuscripts not following the form outlined below may be returned for correction and resubmission.

Manuscripts

❒ Submit an original typed manuscript and three copies. The manuscript should include a short title for running headlines. Any identifying information (author's names, etc) should be on a separate page and not a part of the manuscript. Authors with English as a second language should consider having their manuscript reviewed for grammar, syntax and punctuation prior to submission.

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❒ Identify the corresponding author and provide a complete address, fax number and e-mail address.

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❒ Proprietary names of equipment, instruments and materials should be followed in parenthesis by the name, company, city and state or country of the source or manufacturer.

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❒ The editor reserves the right to make literary corrections.

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References

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