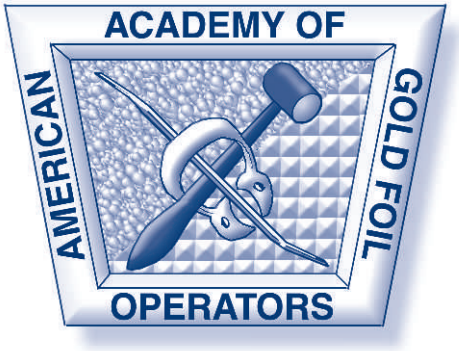
OPERATIVE DENTISTRY



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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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Guest Editorial

"I have all my teeth, my friends don't have theirs."

In 1992, Dr Miles Markley invited the late Wilmer Eames, John Smedley and the two of us to examine 20 of his patients. They were patients who had entered Dr Markley's practice in the years 1934 through 1945 and had remained in his practice until his retirement in 1973. These patients remained in the same practice after Dr Markley's retirement under the care of Dr David Hartman, who had previously been Dr Markley's associate and had the same philosophy of practice.

The four of us were given the charts of these elderly patients and, over a two-day period, we probed, picked, looked at, radiographed and photographed the patients' dentition. We were amazed to find that, during the years these 20 patients had been under the care of Dr Markley and his successor, only one tooth had been lost. Most of the restored teeth we examined had been restored only once, soon after the patient had entered Dr Markley's practice. The restorations had survived an average of approximately 50 years and were still serviceable. All the patients gave Dr Markley credit for their retaining functional dentitions for so long, and most told us stories of Dr Markley's relentless insistence on good home care and non-cariogenic diets. As we said goodbye and thanked these patients for coming in (during a significant snow storm), their universal reply was that it was their privilege, because they still had all their teeth and their friends did not.

In today's world, dentistry seems to be looking at short-term rather than the long-term results. The same criticism has been leveled at the business world and other areas, so our profession is not unique. Anterior resin-composite restorations are replaced frequently because of slight stain at a cavosurface margin, because "better materials" are now available. Amalgam restorations are replaced because of "detectable" margins, and gold crowns are replaced due to poor buccal contours even though the periodontium is stable.

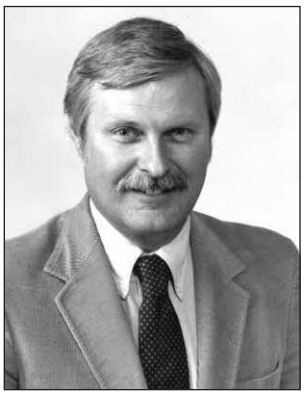
Much of this "replacement dentistry" is done because of the "philosophy" of the practitioner, which is often passed to the dentist during dental school. As Bader and Shugars (1995) have pointed out, dentists routinely place crowns on teeth, because they have old amalgam restorations with deteriorating margins, suspected recurrent caries and are thought to be at risk for fracture. The authors call this remarkable, because none of the assumptions are based on strong supporting evidence. Dentists display such a low agreement on diagnosis that a second dentist is not likely to agree with the first. Secondary caries is equivocal, and dentists are unaware of the rate of fracture of teeth that have these amalgam restorations or of the rate of periodontal or endodontic involvement of teeth that have been crowned.

Frankly, possibly due to the way third-party payers disperse payment for procedures, most dentists seem to be much more likely to replace restorations than to maintain or repair existing ones. For dental amalgam, long-term clinical studies have concluded that recurrent caries around amalgam in adults is infrequent; in one study, it was as low as 1% in 15 years (Akerboom & others, 1993). Yet, surveys show that 50% of amalgams are replaced by the 10-year point (Mjör, 1997). And this is worldwide (Mjör, Moorhead & Dahl, 2000; Mjör & Toffenetti, 1992). Bogacki and others (2002) report that amalgam restorations are replaced by a new dentist seven times more often than if the patient continues to be seen by the dentist who placed the original restorations. Does a new dentist have better vision and insight than the previous dentist? Or is this the result of poor diagnostic skills or a poor understanding of oral diseases and a lack of criteria to determine when to keep or replace?

We would do well to rethink our habit of routinely replacing restorations that are not significantly defective. When assessing an older restoration, why not ask: "Could this last another one to two years? Or could it be repaired?" The long-term problem we face is that a tooth can be restored only a finite number of times without its vitality, or the tooth itself, being lost. If a restoration has a life expectancy of seven years, then a restoration that is initially placed in the mouth of a 14

year-old would be replaced 10 times by age 85. But, it is unlikely that a tooth could withstand that amount of re-restoration. By the time the patient reaches the age of 35 to 40, a gold or porcelain crown is on the tooth and the timeline starts anew under less favorable conditions. The ideal solution is to prevent the need for an initial restoration. However, if the tooth has been restored via maintenance and repair, it could extend the life of the restoration to 15 or 20 years, then the tooth would only need re-restoration three or four times by age 65 to 70, at which time a crown could be placed, if necessary. And if we increase the longevity of the tooth to 30 to 50 years, well, you see the implications.

If Dr Markley could place restorations that last 50 years using the technology of the 1930s and 1940s, we should be able to do so today. And if we rethink the routine replacement of restorations that could be repaired or maintained, today's patients would be saying when they are in their 80s and 90s, "I have all my teeth."



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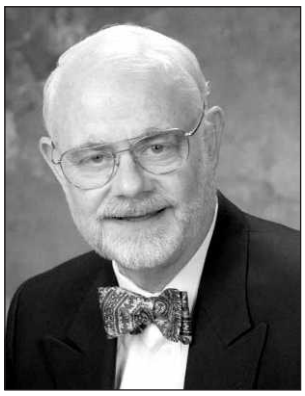

James Summitt, DDS, MSD

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Clinical Research

A Screening Test for Unstimulated Salivary Flow Measurement

M Fontana • S Zunt
GJ Eckert • D Zero

Clinical Relevance

Hyposalivation is the systemic condition that has one of the greatest influences on restorative treatment prognosis and outcomes. This article provides preliminary data to validate an easy method for assessment of hyposalivation in clinical practice.

SUMMARY

It is well established that saliva is an important factor for the health of both soft and hard tissues in the oral cavity. This study determined: 1) the correlation between unstimulated salivary flow assessed using the Modified Schirmer tear strip Test (MST), with gravimetric and volumetric measurements and 2) the MST value that would allow the most reliable identification of patients with severe (<0.1 ml/minute) and moderate (<0.2 ml/minute) hyposalivation. A retrospective clinical study was conducted using data from 90 patients seen at the Indiana University School of Dentistry. All patients had a sample of unstimulated whole saliva collected by drooling for five minutes for volumetric/gravimetric assessment, followed by placement of the Schirmer strip in the floor of the mouth for three minutes (MST). Results showed a non-linear association between the MST and volumetric/gravimetric methods, with moderate Spearman correlation coefficients (0.67-0.71). Analysis of ROC-curves suggests that a cutoff screening value of 25 mm/three minutes provides high sensitivity (77%) and positive predictive value (71%) without significantly affecting specificity (80%). In conclusion, this study supports use of the MST test as a screening tool for hyposalivation.

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INTRODUCTION

It is well established that saliva is an important factor for the health of both soft and hard tissues in the oral cavity. Oral complications as a result of salivary gland hypofunction include altered oral sensations, taste dysfunction, mucosal dryness and increased infections and abrasion, among other factors (Navazesh, 1994; Fox & others, 1985). Pain and diminished quality of life are also common complaints associated with salivary hypofunction. Additionally, chronically low salivary flow rate has been found to be one of the strongest salivary indicators for an increased risk of dental caries (Leone & Oppenheim, 2001).

Since edentulism has decreased significantly in the last three decades and the population of the US has been projected to increase from 250 million to 310 million people, while the elderly group will increase from 28 million to 64 million (Douglass, 1992), apparently the need for restorative dentistry for adults will increase in the future. Additionally, as the population ages, the regular use of analgesics, antiarrthythmics, antihypertensives and diuretics increases, while the use of antidepressants and antihistamines appears to remain constant (Billings, 1993). Many of these medications have the potential to affect salivary flow rate. It has also been reported that as the number of medications with potential antisalivatory effects increases, the salivary flow rate decreases (Billings, 1993; Thorselius, Emilson & Osterberg, 1988; Handelman & others, 1989). These facts support the importance of salivary flow assessment as one of the factors necessary in determining the caries risk status of patients and treatment plan choices and prognosis.

Many dentists tend to rely on the complaint of "dry mouth" to diagnose hyposalivation. Unfortunately, subjective complaint of dry mouth (xerostomia) often does not correlate with the objective findings of reduced salivary flow rate (Fox & others, 1985; Leone & Oppenheim, 2001). Therefore, the objective presence and extent of salivary gland hypofunction ideally should be assessed before an appropriate treatment plan (preventive and restorative) can be developed for a patient. However, dental practitioners rarely examine the salivary status of their patients, probably because of the cumbersome nature of the available sialometric methods and their lack of sensitivity (Lopez-Jornet & others, 1996; Fox, Busch & Baum, 1987; Davis & Marks, 1986). Bahleda and Fontana (2003) surveyed 250 randomly selected dentists in the Indianapolis area regarding their use and formal recording in dental charts of caries risk assessment and management strategies for their patients. Their results indicated that although the majority of the respondents stated they did some form of risk assessment (72%), only 27% documented the outcome in the chart. The most commonly reported assessed risk variable was caries activity, with 90% of the respondents assessing this, while the least commonly assessed variable was salivary flow (measured volumetrically or gravimetrically), with only 5% of the respondents assessing this.

Attempts are being made to look for more user friendly, patient acceptable techniques (Lopez-Jornet & others, 1996; Wolff, Herscovici & Rosenberg, 2002). One new alternative method is the use of Schirmer tear test strips. These are commercially available and routinely used by ophthalmologists to measurement tear gland function and have been reported to be easy to use in the oral cavity (Davis & Marks, 1986; Zunt, Lee & Woo, 2002). Zunt and others (2002) suggested that a value of 10 mm/three minutes or less using these strips might be able to identify patients with severe hyposalivation.

The objective of this retrospective clinical study was to provide preliminary data for the validation of a very simple, inexpensive method that relies on use of the Schirmer tear strips called the Modified Schirmer Test (MST) to measure whole, unstimulated salivary flow. The specific aims include: 1) determining the correlation between unstimulated salivary flow assessed using MST with gravimetric and volumetric measurements; and 2) determining the best cutoff point for MST that would produce high sensitivity and specificity for identifying patients with severe (<0.1 ml/minute) and moderate (<0.2 ml/minute) unstimulated hyposalivation.

METHODS AND MATERIALS

Study Design/Inclusion and Exclusion Criteria

A retrospective clinical study was performed at the Indiana University School of Dentistry (IUSD). Included were 90 consecutive records of patients referred to the IUSD Dental Faculty Private Practice for oral and maxillofacial pathology consultation, including examination for diagnosis and management of oral disease between August and December 2002. All patients had a sample of unstimulated whole saliva collected for five minutes as part of IUSD's caries risk assessment protocols. Immediately after the volumetric/gravimetric assessment, each patient had the unstimulated flow measured using the MST. This test is routinely used by one of the authors for diagnosis and management decisions regarding hyposalivation (Zunt & others, 2002). At the time of their appointment, all patients or their guardians had provided written permission to use their records for publication in scientific journals or for the advancement of dental teaching as part of IUSD's informed consent. Institutional Review Board approval was obtained to use these records to obtain the information reported in this manuscript. Patients excluded from the current investigation were those on sialogogues, such as pilocarpine and cevimeline.

Patients ranged in age from 9 to 90 years of age (mean age: 57.4). Seventy-four patients were female and 16 were male. All 90 patients had been examined for collection of the data by a single experienced clinician (SZ) who routinely teaches and calibrates students at IUSD on the use of these techniques.

Procedures

Salivary Collection

At the time the data was collected, all patients were given the routine instructions not to eat, drink, chew, smoke, brush or floss for two hours prior to their appointment. At the time of their visit each patient sat quietly for at least five minutes before collection, then

had a sample of whole saliva collected by drooling (draining method) for five minutes into a funnel and pre-weighed tube. The patient swallowed all the saliva in his or her mouth prior to the collection exercise. During saliva collection the patient was seated with his/her head tilted down and eyes open (Navazesh & Christensen, 1982). The tube was capped, the volume, excluding foam, recorded, then the tube was weighed on a Mettler analytical balance. The weight was expressed as g/ml (assumption: 1 ml of saliva = 1g). The specimen was discarded after weighing.

Modified Schirmer Test Procedure

Between 5 and 10 minutes after the volumetric/gravimetric assessment, after sitting quietly, each patient had the unstimulated flow measured using a commercially available calibrated paper test strip (ColorBar Schirmer tear test strip, Eagle Vision, TN, USA) with a dye-impregnated color bar that marks the fluid front. The strip had mm marks ranging from 2 mm to 35 mm. Patients were asked to swallow all the saliva in their mouths at the beginning of the test, then not to swallow their saliva during the test. The MST strip was held vertically in the anterior floor of the mouth for three minutes, making sure that only the rounded, dye-impregnated portion of the strip got wet. The patient's mouth remained opened during the procedure, with the tongue touching the roof of the mouth. The results (mm/three minutes) were recorded immediately when the strip was removed from the mouth and the strip was discarded.

Statistical Analysis

Spearman nonparametric correlation coefficients were computed between the MST, gravimetric and volumetric measurements to assess the linear relationships between the variables, along with plots to determine any non-linear relationships. The following definitions of ordered categories were used:

Gravimetric Categories: ≤ 0.1 g/minute; ≤ 0.2 g/minute; >0.2 g/minute

Volumetric Categories: ≤ 0.1 ml/minute; ≤ 0.2 ml/minute; >0.2 ml/minute

MST Categories: ≤ 1 mm/3 minute; ≤ 10 mm/3 minute; ≤ 25 mm/3 minute; ≤ 30 mm/3 minute; >30 mm/3 minute

Additionally, the authors used a Receiver Operating Characteristic (ROC)-curve approach to choose the appropriate MST cut-points to correspond to the gravimetric and volumetric hyposalivation cut-points (≤ 0.1 ml/minute and ≤ 0.2 ml/minute). The selected MST cut-points provided sensitivity and specificity estimates for diagnosing severe and moderate hyposalivation using MST, with gravimetric or volumetric measurements as gold standards.

RESULTS

The mean ± standard deviation values for all 90 patients were: for the MST= 19.8 ± 13.5 (at 3 minute), with a range from 0 to 31; for the gravimetric= 0.28 ± 0.33 (gr/minute), with a range from 0 to 2.02 and for the volumetric= 0.30 ± 0.30 (ml/minute), with a range from 0 to 1.90. Patient percentages included in each of the hyposalivation categories were as follows: gravimetric ≤ 0.1= 39%, gravimetric ≤ 0.2= 52%, volumetric ≤ 0.1=40%, volumetric ≤ 0.2=58%, MST ≤ 1= 21%, MST ≤ 10= 34%, MST ≤ 25= 42% and MST ≤ 30= 48%.

The relationship between the gravimetric and MST techniques was non-linear (Figure 1). The same trend was noted between the volumetric measurements and the MST values. The Spearman correlation coefficients fell in a moderate range (0.67 to 0.71), showing that MST values or categories are somewhat related to gravimetric and volumetric values or categories (Table 1). Gravimetric and volumetric values correlated well (0.93), as expected.

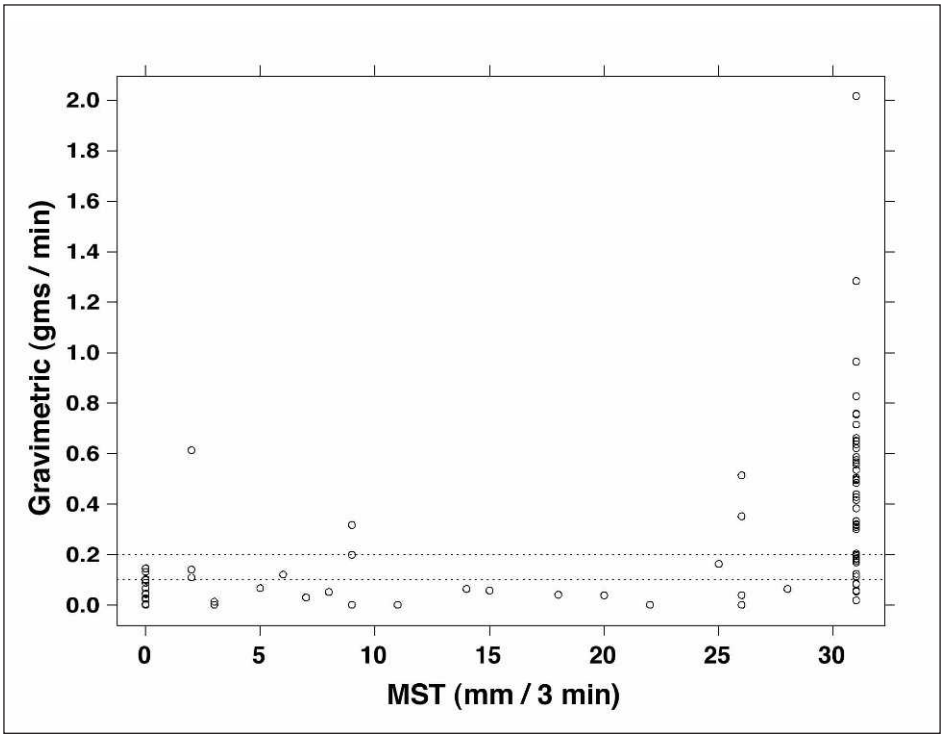

Figure 1. *The plot shows that the relationship between the gravimetric and MST values cannot be considered a linear relationship. The two horizontal dashed lines in the graph show the hyposalivation cutoff points under study (0.1 g/minute and 0.2 g/minute).*

ROC curves were plotted for the MST using the following parameters as gold standards for this test: gravimetric ≤ 0.1 (Figure 2), gravimetric ≤ 0.2, volumetric ≤ 0.1 and volumetric ≤ 0.2. The areas under the ROC curves in all cases ranged between 0.82 and 0.87, which were high enough to indicate that MST is worth further investigation as a test in place of the volumetric/gravimetric assessments.

Given the results of the binary classifications, one way to possibly use the MST is as a screening tool (Table 2). MST ≤ 1 is a perfect or almost perfect (depending on the gold standard definition) indication of hyposalivation. MST between 1 and 25 is a good indication of hyposalivation. MST >30 is a good indication that there is no hyposalivation. In general, as the cutoff value considered was increased, the sensitivity of the test increased without a significant decrease in specificity.

DISCUSSION

The idea of using the Schirmer tear strips for measurement of salivary flow is not new. Davis and Marks (1986) used the strips placed near the parotid papilla for five minutes to measure salivary output of individuals in relation to age. They did not correlate these results with any other sialometric measure, but concluded that this was a quick, cost-effective system that could be used to measure flow rate. Since alterations in submandibular and sublingual function have the greatest impact on the sensation of dryness (Fox & others, 1987), and since these glands are also the main contributors (67%) to unstimulated whole saliva (Dawes, 1993) playing a key role in the health of oral soft and hard tissues, it was concluded that the ideal location for placing the Schirmer strips in the mouth to measure whole, unstimulated salivary flow rate was the floor of the mouth. Lopez-Jornet and others (1996) used the concept of these strips and devised a Whatman paper strip to study 159 healthy adult patients not suspected of having any salivatory problems. The strips were placed under the tongue, in contact with the mucosa, for five minutes. Although they found statistically significant correlations between the strips data (cm/minute) with unstimulated flow rate (ml/minute), the correlation coefficients were very weak (r<0.3). In a pilot study, Woo and Wai (1995) reported that from a pool of 40 patients, the Schirmer strips placed on the floor of the mouth for three minutes (which they called Modified Schirmer Test) could identify 100% of normal patients with readings between 15 and 35 mm in three minutes, while readings <15 mm would identify all head and neck radiation and graft-vs-host disease patients. No salivary flow data were reported in this abstract.

In this study, the authors tested the MST for measurement of unstimulated whole salivary flow rate using the salivary flow rate measured gravimetrically as the comparison gold standard. We decided to include volumetric data in the comparisons, since this data was obtained at IUSD using 15-ml tubes (labeled in 0.5-ml increments, starting at 1.5 ml), and many times estimations of volume have to be made by the clinicians. Our data show that the volumetric and gravimetric data correlated very well (0.93) regardless of possible inaccuracies in acquisition of the latter. The choice of

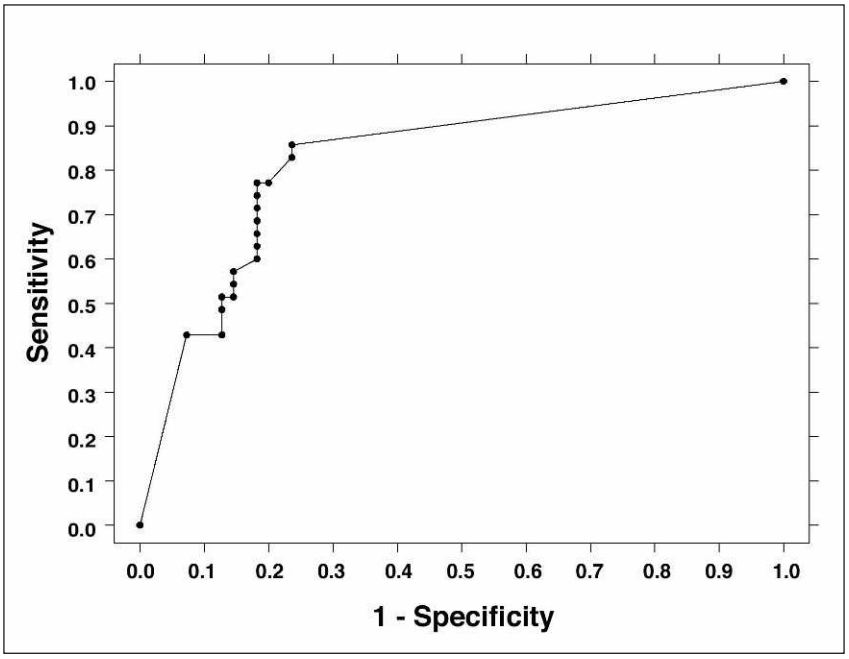

Figure 2. *The ROC curve allows for calculation of an area under the curve of 0.84 using the gravimetric measurement of 0.1 g/minute as the gold standard.*

Table 1: *Spearman Correlation Coefficients*

	Gravimetric	Gravimetric Categories	Volumetric	Volumetric Categories
Categories				
Volumetric	0.93	0.85		
Volumetric Categories	0.86	0.87		
MST	0.68	0.67	0.68	0.69
MST Categories	0.70	0.69	0.69	0.71

Spearman correlation coefficients are presented for the comparisons between the MST values and categories with the gold standards (gravimetric and volumetric values and categories).

Table 2: *Reliability of Different MST Screening Cutoff Values*

Gold Standard		MST≤1	MST≤10	MST≤ 25	MST≤ 30
Gravimetric 0.1 Cutoff	% Correct	73	73	79	80
	Specificity	93	82	80	76
	Sensitivity	43	60	77	86
	NPV	72	76	85	89
	PPV	79	68	71	70
Gravimetric 0.2 Cutoff	% Correct	63	72	80	81
	Specificity	100	81	95	89
	Sensitivity	37	58	69	75
	NPV	54	75	69	72
	PPV	100	68	95	91
Volumetric 0.1 Cutoff	% Correct	72	72	80	81
	Specificity	93	95	81	78
	Sensitivity	42	56	78	86
	NPV	70	61	85	89
	PPV	79	94	74	72
Volumetric 0.2 Cutoff	% Correct	69	76	83	84
	Specificity	100	93	93	88
	Sensitivity	40	60	74	81
	NPV	61	68	77	81
	PPV	100	90	92	88

Reliability data (% correct, sensitivity, specificity, NPV=Negative Predictive Value, PPV: Positive Predictive Value) is provided for different possible MST cut off point values, using different values (0.1 and 0.2) of hyposalivation measured either gravimetrically or volumetrically, as gold standards for comparison.

which cutoff value to use for our gold standard test as an indicator of hyposalivation has been the focus of research for many decades. In a review of the literature, Sreebny (2000) clearly reminded us that these numbers are not easy to define, since studies show that there is significant variation in the range of observed flow rates. The normal, unstimulated flow rate varies between 0.3 and 0.4 ml/minute (Sreebny, 1992, 2000), and although some variation can be found in the literature regarding this definition of "normal," most researchers and clinicians would agree that values ≤ 0.1 ml/minute should be considered abnormal (Navazesh, Christensen & Brightman, 1992; Erickson & Hardwick, 1978). Sreebny and Valdini (1988) reported that only 2% of subjects who complain of xerostomia had flow rates equal or below this number. Yeh, Johnson and Dodds (1998) studied more than 1,100 patients and concluded that the lower 10% of the population have flow rates that vary between 0.093 and 0.187 ml/minute, these values being lower for women (0.042 to 0.073 ml/minute) than for men. In the current study, the authors used both ≤ 0.1 gr/minute or ml/minute and ≤ 0.2 gr/minute or ml/minute as possible cutoff points of severe and moderate hyposalivation for comparison with the MST method.

Our data indicate that the relationship between the volumetric/gravimetric assessments and the MST method was non-linear, and the correlation coefficients fell in the moderate range (0.6-0.7) but were much higher than the values reported by Lopez-Jornet and others (1996). A possible explanation for the lack of linear relationship between the methods' data is that when there is a decreased flow rate, the amount of proteins in saliva increase (Lee & others, 2002) and there is a possible effect of viscosity, not only flow rate on the migration of the saliva in the paper strip, which needs to be further studied.

Ideally, a screening test would have both high sensitivity and specificity. The choice of cutoff value, among other factors, depends on the relative consequences of having too many false-positives or too many false-negatives. In the case of saliva, having high sensitivity (few false-negatives) is desirable, since failure to detect severe hyposalivation can have devastating consequences in the oral cavity. Additionally, although subjective assessments or complaining of dry mouth may not reflect actual salivary gland capabilities, there are some questions that can be asked to patients, and these questions have shown significant predictive value on salivary performance: "Does your mouth feel dry when eating a meal? Do you sip liquids to aid in swallowing dry foods? Do you have difficulty swallowing any foods?" Plus, a response of "too little" to the following question: "Does the amount of saliva in your mouth seem to be too little, too much or you do not notice it?" (Fox & others, 1987). This, in combination with a detailed medical history looking for medications or diseases that have an effect on hyposalivation, may aid clinicians in interpreting the results of this screening test and needs to be tested in the future. Based on current data, the authors suggest a cutoff point of 25 mm/3 minutes may be used to identify patients with moderate or severe hyposalivation. The increase from 10 mm, previously suggested by Zunt and others (2002), to 25 mm almost does not affect specificity (which is rather high), but it does significantly increase the sensitivity and positive predictive value of the test. The

MST was used by Zunt and others (2002) to identify salivary gland hypofunction in the management of oral mucosal diseases. The strips were placed on the floor of the mouth for three minutes to measure unstimulated flow rates, although the authors reported they have routinely used this method to also measure stimulated flow rates following either pilocarpine or cevimeline stimulation. Of the patients with clinical diagnoses of xerostomia or salivary gland hypofunction, 6% had an MST reading of >28 mm, 17% had MST readings between 24 and 27 mm, 26% had MST readings of 11 and 23 mm and 51% had MST readings of <10 mm. It would then seem that the choice of 25 mm would be able to identify more than 90% of all patients with these diagnoses.

Considering this was a retrospective study, there are limitations that must be pointed out. The examiner was not calibrated other than by his or her own experience and personal training prior to collection of this data, and there is no reliability data regarding the accuracy of the examiner itself. Data from the different methods was always collected in the same manner, so a counterbalanced design was not tested. In addition, the sample size was not very large nor did it control for variables such as reasons for hyposalivation, among others that could have an effect on flow rate and viscosity of the saliva

CONCLUSIONS

Even when considering the limitations imposed on this study due to its design (retrospective study), the results provide preliminary data that validate use of the modified MST method for salivary assessment and support use of the MST method as a screening tool for hyposalivation in clinical practice.

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Randomized Clinical Comparison of Endodontically Treated Teeth Restored with Amalgam or with Fiber Posts and Resin Composite: Five-Year Results

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Clinical Relevance

Within the limits of this study, restorations with fiber posts and composite were found to be more effective than amalgam in preventing root fractures but less effective in preventing secondary caries; the overall failure rate was not significantly different for the two kinds of restorations.

SUMMARY

Prospective clinical studies comparing the results of different types of restorations of endodontically treated teeth are lacking. This study compared the clinical success rate of endodontically treated premolars restored with fiber posts and direct composite to the restorations of premolars using amalgam.

Premolars with Class II carious lesions were selected and randomly assigned to one of two experimental groups: (1) restoration with amalgam or (2) restoration with fiber posts and composite. One hundred and nine teeth were included in Group 1 and 110 in Group 2.

Patients were recalled after 1, 3 and 5 years.

No statistically significant difference was found between the proportion of failed teeth in the two experimental groups. Significant differences were observed between the proportion of root fractures (p=0.029) and caries (p=0.047), with more root fractures and less caries observed in the teeth restored with amalgam at the five-year recall. Within the limits of this study, it can be concluded that restorations with fiber posts and composite were found to be more effective than amalgam in preventing root fractures but less effective in preventing secondary caries.

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INTRODUCTION

Severely compromised root-filled teeth are often built up with a post and core before crown restoration. Sorensen and Martinoff (1984a,b) reviewed 1,273 endodontically treated teeth that had been restored from 1 to 25 years previously. Statistical analysis showed that coronal coverage did not significantly improve the rate of success for root-filled anterior teeth but did for premolars and molars.

In many cases where crowns are indicated, teeth are restored with metal-ceramic crowns. The preparation of a tooth for a metal-ceramic crown requires an interproximal and labial reduction of 1.5 mm (Sozio, 1977), an occlusal reduction of 2 mm (Preston, 1977) and a lingual reduction of 1.2 mm. The removal of tooth structure can be considerable, especially as many teeth have already sustained significant tooth structure loss due to caries, removal of old restorations and endodontic procedures.

Amalgam has traditionally been used for the restoration of endodontically treated posterior teeth and may be used either as the definitive restoration or as a core for a full coverage crown. The failure rate of endodontically treated and vital teeth was found to be of the same order when restored with extensive amalgam restorations without crown coverage in a 100-month prospective study (Plasmans, Creugers & Mulder, 1998). The only difference reported was that the failures observed in non-vital teeth restored with amalgam, metal dowels and retention in the pulp chamber more frequently led to tooth extractions compared to those of vital teeth; however, the difference between the fracture rate of vital and non-vital teeth was not compared.

Over the last four decades, the introduction of adhesive techniques has enabled the maximum amount of sound tooth structure to be preserved. Endodontically treated posterior teeth restored with amalgam (Hansen, Asmussen & Christiansen, 1990) or self-cured and light cured resin composite (Hansen & Asmussen, 1990) without crown coverage or post-placement have been studied retrospectively. No statistically significant difference was found between amalgam-restored MO/DO teeth and pooled MO/DO plus MOD resin restored teeth, whereas, teeth with MOD amalgam restorations had a higher failure rate than was found for resin-restored teeth. If a fracture occurred, the resin composite restored teeth failed less catastrophically and were more easily re-restored than teeth restored with amalgam. Endodontically treated teeth with complete loss of coronal tooth structure cannot be restored without the use of posts. The use of metal posts, which are much more rigid than the root, may result in an increase in the number of root fractures (Sorensen & Martinoff, 1984a,b). This led to a search for materials with an elastic modulus similar to that of dentin (Duret Reynaud & Duret, 1990a,b) and resulted in the development of fiber posts. A prospective study evaluated the success of 59 carbon-fiber post-composite core restorations covered with full ceramic crowns (Glazer, 2000); the average observation period was 28 months. There were no root fractures and the overall failure rate was 7.7%. Prospective studies comparing clinical results of different restorative techniques for endodontically treated teeth are lacking. The only prospective study on the restoration of endodontically treated premolars that is currently in the literature was conducted on fiber-post restored teeth (Mannocci & others, 2002). No similar study exists on the amalgam restorations of premolars. The only study providing information on premolars with similar loss of tooth structure restored with amalgam was a retrospective one (Hansen & others, 1990) where information on post reinforcement was not collected. Therefore, it was decided to compare these two types of restorations.

This study evaluated the clinical performance of endodontically treated teeth without crown coverage. Premolars restored with amalgam compacted into the root canals were compared with premolars restored with fiber posts and composite. The research hypothesis was that in endodontically treated teeth with limited loss of tooth structure, the placement of fiber posts using a resin composite luting agent would result in a lower failure rate and a different failure mode compared with teeth restored with amalgam.

METHODS AND MATERIALS

Written informed consent was obtained from each patient prior to participation in the study. The protocol of the study was approved by the appropriate institutional review board of the University of Siena. Patients were required to have one maxillary or mandibular premolar for which endodontic treatment was indicated. Patients had to be healthy and willing to return at regular intervals for evaluation. Only patients showing an orthodontic Class I occlusal scheme were included in the study. Only teeth without previous endodontic treatment presenting with a Class II carious lesion and intact cusp structure were included. The teeth were required to be in occlusal function following restoration and none were used as abutments for fixed or removable prostheses. Patients with shortened dental arches were excluded from the study. Patients wearing removable partial dentures were also excluded. Teeth were excluded if the periodontal attachment loss was greater than 40% of the root length. Patients were also excluded from the study if the Gingival Index score (Loe & Silness, 1963) was recorded as being greater than one. All patients received a course of oral hygiene instruction from a dental hygienist prior to commencement of the study.

A total of 219 patients referred to a private practice in Florence from January 1996 to August 1997 for the restoration of endodontically treated premolars was selected (116 women and 103 men). The age of the patients ranged from 32 to 63 years, with a mean age of 45. By tossing a coin, the selected patients were randomly assigned by an author different from the operator to one of the following two experimental groups. Teeth in Group 1 were endodontically treated and restored with amalgam. Teeth in Group 2 were endodontically treated and restored with fiber posts and composite; all participants assigned to either group received intended treatment: 109 teeth were included in Group 1 (26 first mandibular, 29 second mandibular, 28 first maxillary and 26 second maxillary premolars) and 110 teeth in Group 2 (28 first mandibular, 32 second mandibular, 24 first maxillary and 26 second maxillary premolars).

All mandibular premolars had one root canal and, of the maxillary premolars, 70 had one and 34 had two root canals.

All clinical procedures were carried out by the same operator; the teeth were isolated with a rubber dam both for root canal filling and restorative procedures.

Root Canal Treatment

Root canal treatment of teeth from both groups and the composite restoration of teeth from Group 2 were performed as described in a previous study (Mannocci & others, 2002). Root canal treatment was performed under local anesthesia with a chemo-mechanical technique. The root canal filling was performed with laterally condensed gutta-percha and endodontic sealer, (AH Plus, Dentsply De Trey, Konstanz, Germany).

All teeth were prepared and the roots filled at the same appointment. Teeth of both groups received a temporary filling with a zinc oxide, eugenol-free temporary filling composite material (Fermit, Ivoclar–Vivadent, Schaan, Liechtenstein).

Composite Restoration

One week after the root canal filling procedure, gutta-percha was removed to a depth of 7 mm or, whenever possible, to a depth equal to three quarters of the length of the root canal using Largo drills (Maillefer, Baillagues, Switzerland). The working length of the drills was controlled with silicone stops. The root canal walls were enlarged with low speed burs provided by the manufacturer for the preparation of a size 1 carbon fiber post (Composipost, RTD, St Egreve, France). Post diameter was 1.4 mm in the coronal part and 1.2 mm in the apical 2 mm. The depth of the post space preparation was the same as that of the gutta-percha removal. As a reference point, this depth was obtained by using a line painted on the shank of the burs at a distance 9 mm from the tip of the burs. The root canal

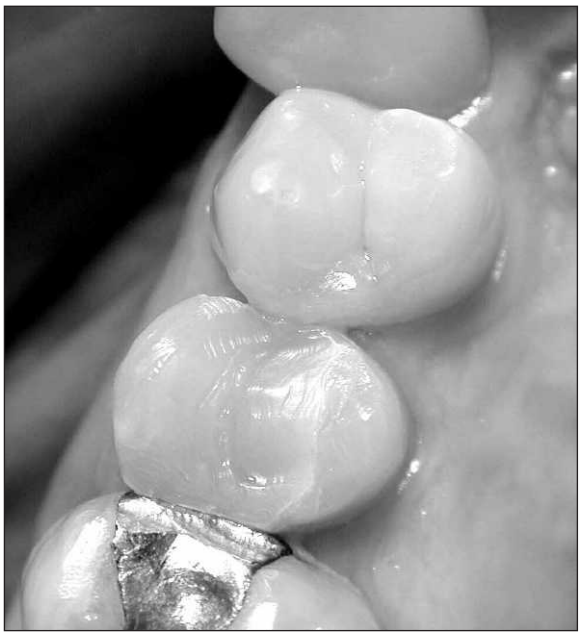

Figure 1. *Successful fiber-post + composite restoration of a maxillary second premolar at five-year recall. 1= fiber post + composite restoration.*

walls were etched with 32% phosphoric acid (All Etch, BISCO, Itasca, IL, USA) for 30 seconds, washed with water spray then gently air dried. Primer A and B (All Bond 2, BISCO) were mixed and applied in the canals. Dentin bonding material (All Bond 2 Pre-Bond Resin, BISCO) was applied in the canal. A layer of dentin bonding primer was applied on the carbon fiber posts, then equal volumes of base and catalyst of the luting composite (C&B, BISCO) were mixed for 10 seconds, according to the manufacturer's instructions. The cement was applied on the post surface, the post was inserted into the canal and the cement allowed to set for seven minutes. A number 1001 Tofflemire metal matrix band (Hawe Neos Dental, Bioggio, Switzerland) was positioned on the tooth; wooden wedges were used in order to improve the interproximal adaptation. Composite (Z100, 3M, St Paul, MN, USA) was placed incrementally in 2-mm layers. Each layer was exposed for 40 seconds with a visible light-polymerizing unit (Visilux 2, 3M) and no composite cusp coverage was performed (Figure 1).

Amalgam Restorations

One week after completion of the root canal filling procedure, the temporary restoration and gutta-percha were removed to a depth of 4 mm using Largo drills (Dentsply Maillefer, CH-1338 Ballaigues, Switzerland). A #1001 metal matrix band (Tofflemire) was placed around the tooth and wooden wedges were used to improve interproximal adaptation. The amalgam used was a palladium enriched, phase-dispersed amalgam alloy (Valiant PhD, Dentsply, Milford, DE, USA)

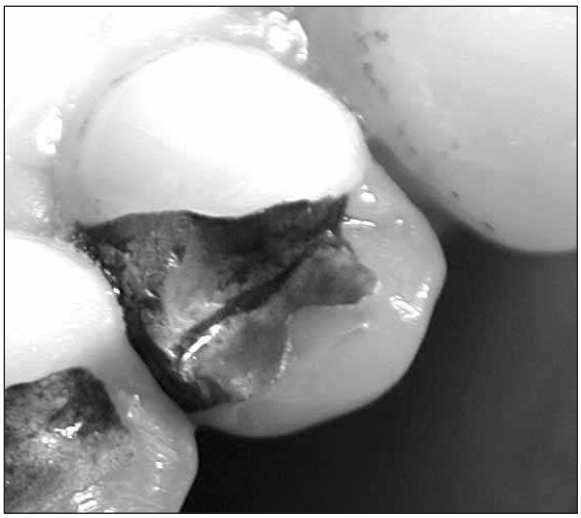

Figure 2. *Successful amalgam restoration of a maxillary first premolar at five-year recall.*

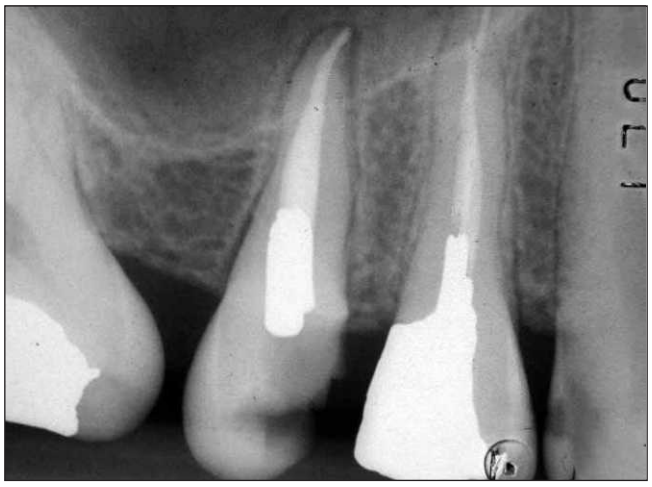

Figure 4. *Failed amalgam restoration of a second maxillary premolar at five-year recall. 3= Cervical fracture of the root. 4= Amalgam compacted into the root canal, this portion of amalgam remained into the root canal after tooth fracture.*

(Figure 2). No cavity liner was placed. In order to improve retention of the restoration, amalgam was compacted in the coronal third of the root canal from which the gutta-percha had been removed. A precise measurement of the diameter of the amalgam compacted into the root canal cannot be given, as the section of the coronal third of the root canals of mandibular and maxillary premolars is not circular but irregularly elliptical. In 70 teeth, the amount of dentin sustaining the buccal or lingual cusp was considered insufficient and, therefore, the cusp was covered with amalgam, the minimal cuspal reduction was about 3 mm.

Clinical Criteria for Success and Failure

The patients were recalled for examination after 1, 3 and 5 years. Causes of failure were categorized as root

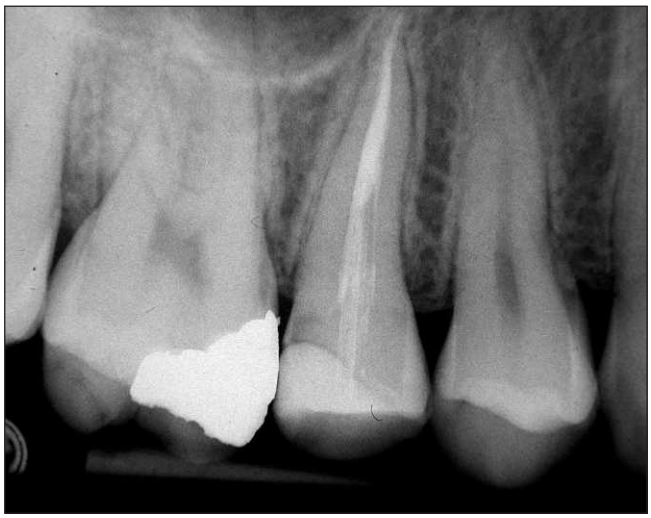

Figure 3. *Failed fiber post + composite restoration of a maxillary second premolar at five-year recall. 2= Caries at tooth-composite margin.*

fracture, post fracture, post decementation, clinical and/or radiographic evidence of a marginal gap between the tooth and restoration and clinical evidence of secondary caries contiguous with the margins of the restoration. Failure caused by root fracture was noted when, after extraction of a fractured tooth fragment, a fracture line involving the root was evident at inspection; the other failure modes were defined as described previously (Mannocci & others, 2002).

Clinical, radiographic and photographic assessments were performed as described previously by two calibrated examiners (Mannocci & others, 2002). Visual inspection was conducted using loops with fiber-optic coaxial illumination (Zeon Illuminator; Orascoptic Research, Madison, WI, USA) at 3x magnification, examination of the continuity of the margins of the restoration with the tooth structure was accomplished using an explorer (EXS6; Hu Friedy, Leiman, Germany) and periodontal probing was performed using a periodontal probe (Perio-Probe, ASA Dental 1-2, Lucca, Italy). Color slides (1:1 mirror shots) of the restorations were taken using standard film (Kodak EliteChrome 100, Eastman Kodak Company, Rochester, NY, USA). Periapical radiographic examination was performed using a paralleling technique at 65 kV and 8 mA. A radiographic extension cone (Orix AET, Ardet, Buccinasco, Italy) was used in combination with a paralleling device (Rinn XCP, Rinn Corp, Elgin, IL, USA). Ultra-Speed periapical 31 ¥ 41-mm dental films (DF-57, Kodak) were used. Radiographs were projected onto a 60 ¥ 90 cm screen. The clinical, radiographic and photographic examinations were performed immediately before restoration, immediately after restoration and at 1-, 2- and 5-year recall. The assessment took place immediately before reconstruction, immediately after and at 1 to 3 and 5-year recall.

Table 1: *Results at One Year Showing the Differences Between the Groups in the Proportions of Failed Teeth with 95% Confidence Intervals*

	Amalgam (Group 1)	Composite + fiber post Group 2	Δ	95% CI		p
Recall	107	109				
Failure	1	2	-0.009	from-0.056	to 0.035	1.00
Fracture	1	0	0.009	from-0.026	to 0.051	0.50
Caries	0	2	-0.018	from-0.064	to 0.019	0.50

Table 2: *Results at Three Years Showing the Differences Between the Groups in the Proportions of Failed Teeth with 95% Confidence Intervals*

	Amalgam (Group 1)	Composite + fiber post Group 2	Δ	95% CI		p
Recall	105	105				
Failure	3	3	0.000	from-0.055	to 0.55	1.00
Fracture	2	0	0.019	from-0.019	to 0.067	0.50
Caries	1	3	-0.019	from-0.072	to 0.027	0.62

Table 3: *Results at Five Years Showing the Differences Between the Groups in the Proportions of Failed Teeth with 95% Confidence Interval*

	Amalgam (Group 1)	Composite + fiber post Group 2	Δ	95% CI		p
Recall	100	97				
Failure	9	10	-0.013	from-0.100	to 0.073	0.81
Fracture	6	0	0.060	from 0.010	to 0.125	0.029
Caries	3	10	-0.073	from-0.152	to –0.002	0.047

Teeth lost due to trauma, endodontic or periodontal problems were considered as missing data. Patients who did not respond to one or more of the three recalls were excluded from the study.

Data Analysis

The proportion of fractures and failures between the two groups was compared using Fisher's exact test. The 95% confidence interval for the difference between the proportions was calculated using Wilson's method (Altman & others, 2000). The level of significance of 0.05 was used throughout the test.

RESULTS

Two patients from Group 1 and one patient from Group 2 failed to return at the one-year recall, two additional patients from Group 1 and four patients from Group 2 missed the three-year recall, five additional patients from Group 1 and seven patients from Group 2 failed to return at the five-year recall. These patients were excluded from the study.

No teeth were lost due to trauma, endodontic or periodontal problems. The only failure modes observed were due to caries (Figure 3) and root fractures (Figure 4). There were three failures (two from caries and one fracture) at one year, six failures (four caries and two fractures) at three years and 19 failures (13 caries and six fractures) at five years (Tables 1, 2 and 3). In the amalgam group, five of the six fractures involved cusps covered with amalgam. In three cases, the amalgam restorations remained intact and the cusp fractured; in the other three cases, the amalgam restorations also failed. In these cases, the amalgam compacted into the root canal and remained *in situ*, whereas, the remaining part of the restoration was involved in the fracture. No significant differences were found between amalgams and restorations with fiber posts and composite at the one- and three-year recall examinations (all $p>0.50$). At five years there were significant differences between the number of failures due to fractures ($p=0.029$) and the number of failures due to caries ($p=0.047$), with more root fractures in the teeth restored with amalgam and more caries in teeth restored with fiber posts and composite. No statistically significant differences were found for failure, fractures and caries when the analysis was conducted for maxillary and mandibular teeth separately (Table 4).

DISCUSSION

In this study, the recall rate after five years was 91.7% for Group 1 and 88.2% for Group 2. Similar recall rates have been reported in other long-term recall studies (Van Dijken, Olofsson & Holm, 1999). The 100-month survival rate of extensive amalgam restorations for both vital and non-vital teeth was found to be 88±2% in a study on extensive amalgam restorations (Plasmans

Table 4: *Results at Five Years Showing the Differences Between the Groups in the Proportions of Failed Teeth with 95% Confidence Intervals for Both Maxillary and Mandibular Teeth*

	Amalgam (Group 1)	Composite + fiber post Group 2	Δ	95% CI		p
Maxillary teeth						
Recall	47	52				
Failure	5	6	-0.009	from-0.138 0.125	to	0.88
Fracture	3	0	0.043	from -0.033 0.142	to	0.14
Caries	2	6	-0.052	from-0.173 0.073	to	0.36
Mandibular teeth						
Recall	53	45				
Failure	4	4	-0.13	from-0.140 0.103	to	0.82
Fracture	3	0	0.057	from 0.030 0.154	to	0.08
Caries	1	4	-0.070	from-0.189 0.027	to	0.12

& others, 1998). The survival rate of the amalgam group in this study was 91.3%; this higher survival rate may have been attributed to the selection of teeth with limited loss of tooth structure and to the shorter recall term. The survival rate of teeth restored with fiber posts and composite was 90%, this being somewhat less than that found in a previous two-year recall study (Glazer, 2000) on fiber post crown restorations (92.3%). This small difference might be explained by the longer follow-up time and supports the proposal that sound tooth structure should be preserved, if possible, and crown coverage avoided for premolars for the first five years after endodontic treatment. Post-core decementations may occur in crown-covered teeth restored with fiber posts and composite (Glazer, 2000; Mannocci & others, 2002). The absence of decementations in this study is another factor that favors the avoidance of coverage of composite cores with crowns, if possible. It has been demonstrated that fiber posts become more flexible after water immersion (Torbjoner & others, 1996; Mannocci & others, 2001a); contact of fiber posts with water may occur if there is leakage at the interface between the composite and the dentin (Mannocci & others, 2001b). In this case, it might be speculated that, for crown-covered teeth, rigid metal ceramic crowns transfer stress to fiber posts that have become more flexible. The flexion of fiber posts results in debonding of the adhesive from the tooth structure and post-core decementation. In teeth are left without crown-coverage, the less rigid composite restoration will probably wear more than the crowns and amalgam and, therefore, might transfer less stress to the fiber posts and the remaining tooth structure. This could account for the absence of post-core decementations and the absence of root fractures in fiber-post-restored teeth left uncovered. On the other hand, the wear rate was not included among the failure modes in this study, which might affect the long-term clinical performance of composite restorations without crown coverage.

The absence of root fractures in teeth restored with fiber posts and composite concurs with previous retrospective (Ferrari & others, 2000) and prospective (Glazer, 2000; Mannocci & others, 2002) studies on fiber post restorations. All failed teeth in this group were re-restored with adhesive techniques and maintained in clinical service, whereas, three teeth from the amalgam group had to be extracted due to root fractures. In accordance with Hansen and Asmussen (1990), it can be concluded that the failure mode of adhesively restored teeth was more favorable.

Cusp coverage of amalgam-restored teeth has been suggested in order to prevent root fracture (Robbins, 1990), but, to date, no controlled prospective study has demonstrated the effectiveness of cusp coverage for the prevention of root fractures. For this reason, in this study, only cusps with an amount of dentin considered to be insufficient were covered.

Procedures for finishing contact points and interproximal spaces were found to be less effective for composite restorations than for amalgam since they are performed on a less plastic material. This may lead to the formation of gaps that are difficult to detect and may result in plaque retention, hence, the higher incidence of secondary caries observed in the composite filling group.

Statistical analysis of the results did not support the first part of the research hypothesis—that in teeth with limited loss of tooth structure, endodontic treatment and build-up using fiber posts and composite results in a lower failure rate compared with endodontically treated teeth restored with amalgam. The second part of the research hypothesis—that the failure modes of the two groups were different—was upheld, as the number of root fractures was found to be significantly higher in teeth restored using amalgam and the number of teeth with caries was higher in teeth restored with fiber posts and composite.

CONCLUSIONS

It can be concluded that within the limits of this study conducted on endodontically treated premolars with limited loss of tooth structure, adhesive restorations were found to be more effective than amalgam in preventing root fractures but less effective in preventing secondary caries.

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The Performance of Air-turbine Handpieces in General Dental Practice

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Clinical Relevance

The long-term monitoring of air-turbine handpieces in service can reveal progressive changes in behavior which may be used as measures of deterioration. It is also demonstrated that adherence to a proper lubrication protocol can lead to bearing longevity several times that commonly experienced. Such investigations will permit clinicians to make informed decisions about the acquisition and maintenance of an essential item of equipment for clinical dentistry.

SUMMARY

Objective: To investigate variation in performance measures of fibre-optic, high-speed air-turbine handpieces during the course of daily use in general dental practice.

Materials and Methods: Four groups of five new high-speed fibre-optic handpieces were used in the routine treatment of patients over a period of 30 months by four general dental practitioners in two dental practices: Groups A, B: Super-Torque Lux 3 650B (KaVo, Biberach, Germany); Group C: BORA 898LE (BienAir SA, Bienne, Switzerland) and Group D: Toplight (W&H Dentalwerk, Burmoos, Austria). The dental practice teams had been rehearsed in the procedures to be followed before starting the study. Each dentist used the handpieces in strict rotation, while the groups were rotated monthly between practitioners. Four performance characteristics were measured before use, then at regular intervals: free-running speed (Hz) and bearing resistance (μNm) were measured using a purpose-built testing machine (Darvell-Dyson); illuminance (lux) and sound pressure level (dB(A)) were also measured. Handpieces were cleaned and lubricated in accordance with manufacturers' directions; all were autoclaved wet at 134°C for three minutes.

Results: Free-running speed showed an initial increase after use for Groups A, B and C, which may be associated with a decrease in bearing resistance. All handpieces in Group C suffered bearing failure between months 21 and 23, preceded by a substantial increase in noise, while those in Group D suffered failure of the fibre-optic system between months 18 and 24. Other deterioration due to use was identified but Groups A, B and D were still in use at month 30.

Conclusions: Variation in free-running speed, bearing resistance, illuminance and sound pressure level can be used effectively to monitor changes in air-turbine handpieces due to normal

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use. Although an increase in bearing resistance is associated with decreasing free-running speed, noise appears to be a useful indicator of imminent bearing failure. Assiduous adherence to manufacturers' directions for cleaning and lubrication may have contributed to increased bearing life.

INTRODUCTION

Since their introduction in 1957 (Dyson & Darvell, 1993c), high-speed turbine handpieces have been used extensively in dentistry for the preparation of teeth, including the removal of failed restorations. The original Borden handpiece underwent a number of modifications, but all relied on Borden's principle of a rotor carried by ball bearings (Dyson & Darvell, 1993c). Subsequent development of the air-bearing handpiece ostensibly avoided the inherent limitations of ball bearings but, while they are capable of reaching appreciably higher speeds, they have a number of disadvantages, including lower torque, higher air supply pressure and susceptibility to bearing instability.

The Super-Torque handpiece (KaVo, Biberach, Germany), introduced in the 1970s, had an enlarged turbine and thus increased torque, as well as rubber-mounted bearing races that may have helped to reduce vibration and therefore bearing wear. This development was, in turn, followed by the introduction of dynamically-balanced rotors which would be expected to reduce vibration and, thus, noise and so improve bearing longevity; this does not appear to have been documented.

In recent years, eliminating the need to use tools to insert and remove burs and introducing multiconnectors, fibre-optic illumination and multiple coolant nozzles as design refinements have increased ease of use. Walker and Morrant (1975) reported that there were no generally recognized methods of testing turbine handpiece performance, which remains the case, and they were among the first to measure and compare the main physical performance characteristics relevant to clinical use, namely torque and power.

Since the adoption of routine sterilization of handpieces in the 1980s, anecdotal evidence suggests that dentists believe that steam sterilization has a cumulatively damaging effect on handpiece bearings. As with performance characteristics, there are no recognized methods of testing the deterioration of bearings, fibre-optic illumination systems or other parts of the handpiece as a result of repeated autoclaving.

Eames and others (1979) attempted to establish a series of simple reproducible tests to evaluate handpieces in order to enable dentists to make informed purchasing decisions, and they were the first to study the effect of repeated autoclaving on performance. Angelini (1992) investigated the effects of a variety of sterilization and disinfection treatments, finding that some of the damage suffered by bearings at high temperature could be reduced and delayed by correct lubrication. However, the damage done to phenolic resin bearing retainers ("cages") was purely temperature-dependent, lubrication having no detectable effect. It was concluded that the decrease in handpiece performance related mainly to corrosion damage of the bearing itself and the heat-induced embrittlement of the bearing cage. It was acknowledged, however, that the study was under static conditions that did not reproduce usage in practice.

Worthington and Martin (1998) investigated the effect of repeated autoclaving on handpieces used in general practice, the criterion being "until a significant fall in performance was noted by each clinician." There was, however, no description of the performance measures used, and the assessments appear to have been entirely subjective. Wirthlin and others (1998) concluded that high-speed handpieces subjected to repeated autoclaving fail more rapidly than those that are not. Leonard and Charlton (1999) found that the rate of deterioration was often very rapid after 120 to 150 autoclave cycles.

Dyson and Darvell (1993a,b,c; 1995a,b; 1997; 1999) have reported on several aspects of the development, design and performance characteristics of high speed turbine handpieces and have categorized the cutting operations that are common in clinical practice. With regard to the test methods employed, they identified a number of shortcomings in previously published studies; for example, suggesting that tooth preparation involves one or more of the following types of cutting action: plunge cutting—as in endodontic access cavity preparation; slot milling—to section an old restoration and surface reduction—such as shaping a tooth surface in crown preparation (Dyson & Darvell, 1993c). In addition, they enumerated the key design and performance characteristics of these machines and suggested means by which the performance variables might be reliably measured.

Tooth cutting is rarely a continuous process, and there are large variations in the load applied to the tooth according to the tissue being cut and stage of the preparation. This results in considerable and continual variation in instrument speed and power usage. In contrast, a number of previous studies have involved either static testing (Eames & others, 1979), the use of non-dental tissue as the cutting substrate (Walker & Morrant, 1975) and non-standard burs or continuous running with no variation in the load applied to the instrument. Dyson and Darvell (1993c) concluded "for as long as there is a lack of data on duty cycles employed in actual practice, the clinical relevance of laboratory cutting studies will continue to be open to question."

Free-running speed has been measured using a number of techniques, including mechanical and magnetic tachometers (Lammie, 1951; Wirthlin & others, 1998; Leonard & Charlton, 1999). However, these methods load the system, extracting some energy from it and reducing the speed, while acoustic systems (Lammie, 1951; Morrant, 1962) suffer from considerable difficulties (Dyson & Darvell, 1995b, 1997, 1999). Stroboscopic methods have been used (Lammie, 1951; Angelini, 1992; Brockhurst & Shams, 1994; Wirthlin & others, 1998; Worthington & Martin, 1998) but their accuracy is limited when speed is not precisely constant and cannot be used to track changes continuously. A non-contact, opto-electronic technique is the approach of choice as non-loading, accurate and capable of recording continuously (Darvell & Dyson, 2005).

Previous studies in this field have therefore suffered from a number of flaws or deficiencies in terms of both the test methods and equipment. In laboratory studies, the limitations have been a result of the failure to simulate clinical usage, while in practice-based studies, failure criteria have been ill-defined. This study addressed a number of these failings by applying the approaches developed by Dyson and Darvell (2005) to monitor the behavior of handpieces in clinical use and by including two additional factors: noise and light output.

METHODS AND MATERIALS

Four groups of five new high-speed fibre-optic handpieces were used at the manufacturers' recommended working pressures. Groups A, B: Super-Torque Lux 3 Turbines 650B (KaVo, Biberach, Germany) ("KaVo"), 2.8 bar; Group C: BORA 898LE (BienAir SA, Bienne, Switzerland) ("BienAir"), 2.6 bar; Group D: Toplight (W&H Dentalwerk, Burmoos, Austria) ("W&H"), 2.2 bar. Each handpiece was laser-engraved with a group code and individual identification number. In turn, each group was allocated to one of four general dental practitioners (GDPs) in two practices, each GDP also being supplied with an autoclave (Sterimaster, KaVo) that was used only for autoclaving the unwrapped-handpieces in steam at 134°C for three minutes.

Within a group, the handpieces were used in strict rotation in the provision of everyday dental treatment (restorative, fixed and removable prosthodontics, endodontics) and once a month the groups were rotated among the four GDPs. At each change, supply air pressure was re-adjusted using one pressure gauge as reference. Before being put into service and after each use, the handpieces were cleaned and lubricated. With no bur in place, the exterior was cleaned with a soft nylon bristle brush and water. The manufacturer's instructions were followed using the appropriate proprietary internal spray lubricating-cleanser for one second, keeping the can upright after shaking (KaVospray, Part Number 411 9630, KaVo; Spraynet, Part Number 930-01-24, BienAir; Service Oil 900, Part Number 02690000, W&H). During this process, a clean tissue was held around the head to absorb excess spray and collect debris. If the tissue appeared to be soiled (discolored oil), the spray was repeated until there was no discoloration evident. Medical grease lubricant was applied before and after autoclaving for Group C. Product-specific details follow.

Groups A, B: KaVo

Excess oil was wiped off and the handpiece stood head-up in an instrument stand for five minutes to allow excess oil to drain prior to autoclaving.

Group C: BienAir

After cleaning, the turbine head was greased using the proprietary device (Lubrimed, Part Number 107.37.01, BienAir). The handpiece and greaser were then autoclaved together, after which, when cool, the handpiece was lubricated again with the sterile grease. The handpieces were stored head-up in an instrument stand. Prior to use, the turbine was run slowly up to speed to distribute the grease.

Group D: W&H

The handpieces were allowed to stand for five minutes vertically, head down, in an instrument stand (Part Number 01376600, W&H) prior to autoclaving.

All routines were established during a pilot run using other handpieces in the same practices to ensure that the teams were familiar with the protocols and procedures.

Free-running speed, bearing resistance and stall torque were measured using a purpose-made combined instrument (Darvell-Dyson Handpiece Tester, Dental Materials Science, The University of Hong Kong) (Darvell & Dyson, 2005) intended to allow reliable, routine monitoring of these performance characteristics.

Sound was monitored in broadband real-time sound level measurement mode (Model 573-C2 Sound Level Analyser, CEL Instruments, Hitchin, Hertfordshire, UK; 5 ~ 140 dB, 10Hz ~ 30 kHz; +0.5, -3.0 dB) in free-field conditions at 45 cm from the center of the turbine using a jig to position the handpiece reproducibly. For ease of pressure adjustment to the manufacturer's recommended value, the compressed air was supplied to the handpiece via the Darvell-Dyson test rig. The turbine was run up to full speed (free running) and the sound level monitored over five seconds; the peak value in dB(A) was noted.

The light output of the fibre-optic was measured using a light sensor (model 180-7133, RS Components, Corby, Northants, UK) in a purpose-built enclosure that accommodated the head of the handpiece, including the exit windows of the fibre optic system and

excluding ambient light. The handpiece was run up to speed normally so as to switch the light on and the average illuminance (a lightmeter function) over the next five seconds was recorded.

Except for stall torque, each variable was measured on each handpiece prior to putting it into service, then for Groups B-D at 1, 11 and 15 months, and thereafter at three-month intervals to 30 months. Group A handpieces were brought into service at month 15 and monitored monthly. Stall torque was measured before all handpieces were put into service (this character depends on handpiece and turbine design and is not subject to change).

Graphical analysis and regressions were done in software (SigmaPlot v9, SPSS, Chicago IL, USA), as were statistical calculations (SPSS v 8; SigmaStat v 2.03, both SPSS). The critical value for statistical significance was set at p=0.05; no corrections for multiple tests were applied.

RESULTS

The results of the trials are shown in Figures 1 through 4. All handpieces in Groups A, B and D remained fully mechanically-usable at the end of the study, although the fibre-optic system of all of Group D had failed by month 25; each handpiece in these groups had been through 820 autoclave cycles by month 30. The handpieces in Group C all suffered bearing failure (seizure) between 21 and 23 months and were therefore unusable beyond that point; they had been through about 560 autoclave cycles each.

Baseline values (Table 1) for most characters differed significantly between Groups (1-way Analysis of Variance): free-running speed (p=0.0018), stall torque (p=1.3 x (10^{-5}), illuminance (p=2.2 x (10^{-10})) and sound pressure level (p=1.7 x (10^{-4})), while bearing resistance did not (Kruskal-Wallis on ranks) (p=0.184).

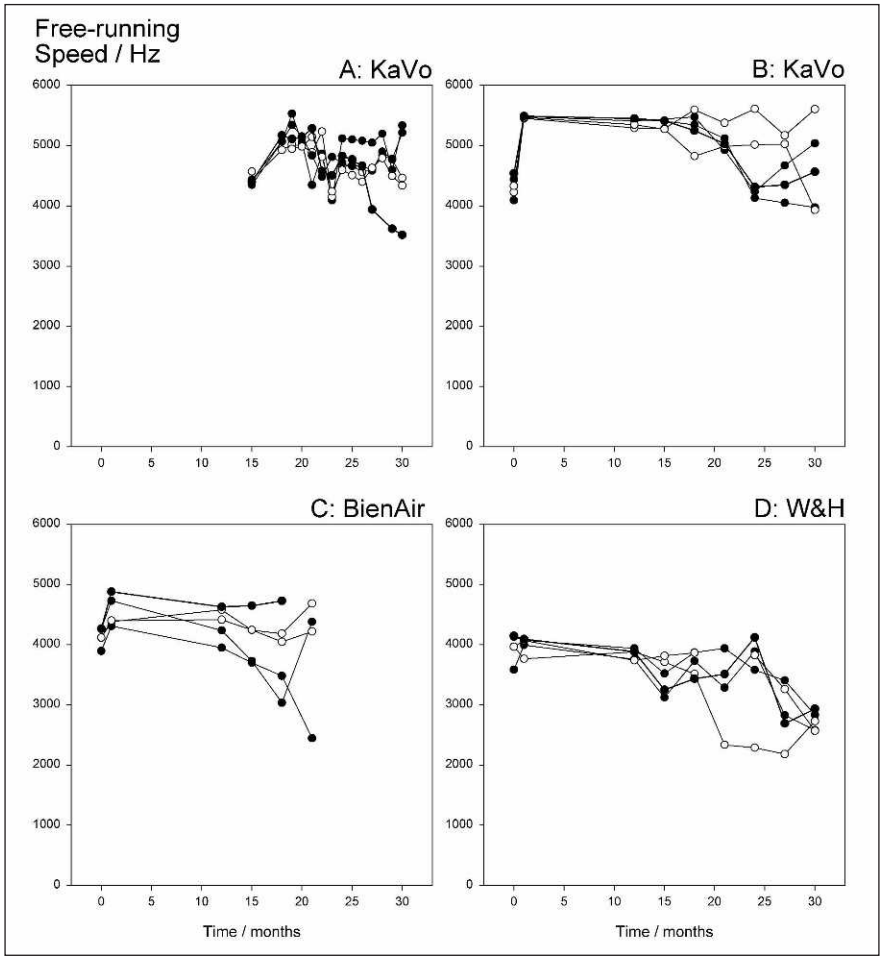

Figure 1. *Variation of free-running speed with time of use.*

Table 1: *Baseline Values for the Measured Performance Characteristics: Median (range) Within Groups*

Group (air supply pressure/ bar)	Free-running Speed/Hz	Stall Torque/ mNm	Bearing Resistance/ µNm	Illuminance/ Lux	Sound Pressure Level/dB(A)
A (2.8)	4442 (4357–4573)	1.30 (1.23–1.34)	16 (7–27)	4340 (4160–4560)	73.5 (69.5–80.6)
B (2.8)	4327 (4095–4541)	1.30 (1.27–1.34)	17 (16–18)	3896 (3650–4080)	68.2 (65.3–72.5)
C (2.6)	4131 (3895–4265)	1.13 (0.96–1.24)	19 (4–70)	2110 (2040–2170)	80.4 (77.5–83.5)
D (2.2)	3958 (3965–4146)	0.97 (0.92–1.03)	21 (1–89)	3476 (2890–3880)	76.6 (73.1–81.7)

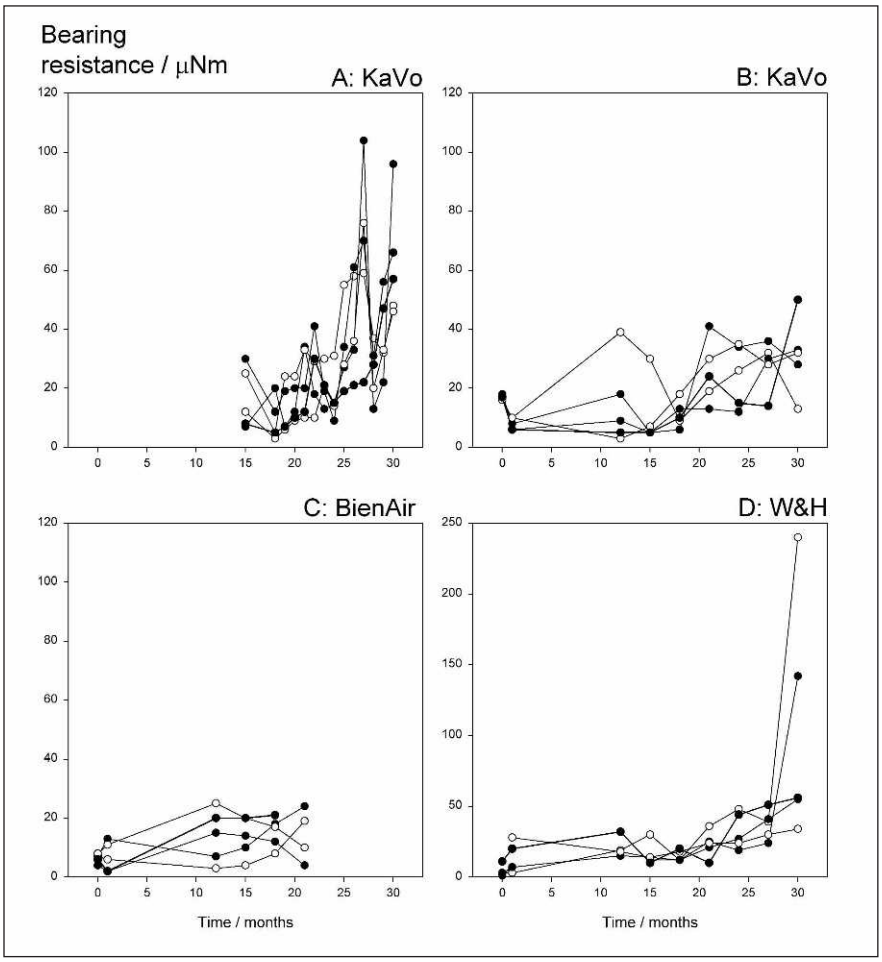

Figure 2. *Variation of bearing resistance with time of use.*

Free-running speed showed a general increase in value at the first reading after baseline for Groups A, B and C, although only that for Group B attained statistical significance (paired *t*-test: p=5.0 x (10^{-4})). Overall, there was clearly increasing scatter in values with time, and within each group, a small but statistically-significant decline on average (Table 2), although individual handpieces varied considerably.

Bearing resistance also showed increased scatter with time and a significant trend for Groups A, B and C (Table 3), although the slopes for Groups A and B differed significantly (*t*-test, p=1.2 (10^{-4})). Substantial jumps in value occurred for Group D in two cases at the last monitoring point (30 months); these were excluded from the calculation. There was no corresponding change in the free-running speed for these two handpieces. In Group D, there was a moderately strong (r^2=0.403) but highly-significant (p=1.1 (10^{-5})) correlation between bearing resistance and free-running speed. The others were non-significant (A: r^2=0.015, p=0.298; C: r^2=0.055, p=0.222) or significant but weak (B: r^2=0.166, p=6.6 (10^{-3})).

Sound pressure level showed no trend for Groups A and D, a very slight but significant overall increase for Group B, but a very large and highly significant increase from about month 15 for Group C (Table 4).

Illuminance showed a very marked and highly significant drop at the first monitoring point after baseline in each case (paired *t*-test, p<0.004). The trend after that was for a highly significant decline in each group overall except Group A (Table 5), for which group the rise from the minimum at month 20 was followed by a plateau.

DISCUSSION

The organization of a long-term trial of this type, across practices and involving several changes of personnel, when close monitoring of adherence to the protocol is precluded, presents its own challenges for subsequent data analysis. Indeed, there are a number of features of the results that indicate that procedural improvements are possible. Even so, the principle effects are clear enough.

It is certainly the case that the scatter in the values of free-running speed and bearing resistance, presumed to be sensitive indicators of the state of the bearings, makes statistical analysis for trends problematic, for what appears to the eye to be a clear effect may not achieve statistical significance. Thus, one handpiece in Group C appears to presage the total bearing failure prior to the 24-month readings by a steady decline in free-running speed (although the others do not), but the slope of the fitted straight line is non-significant. It would appear that more frequent monitoring than was applied to Groups B–D would be necessary for better sensitivity and discrimination. The data for Group A would suggest that monthly is appropriate.

To date, this is the first reported attempt to monitor handpiece characteristic measures while in service over a substantial period and, as such, has provided valuable insight and advantages over work that involved static testing (Eames & others, 1979; Watanabe & others, 2000) and subjective assessment (Worthington & Martin, 1998). The fact that the handpieces were in daily use in clinical practice responds to the shortcomings identified by Dyson and Darvell (1993a,b,c, 1995a,b, 1997, 1999) with regard to the relevance of laboratory-based studies.

Leonard and Charlton (1999) had concluded that the rate of deterioration of autoclaved handpieces was very rapid after 120 to 150 cycles. The Group C handpieces, which suffered bearing failure between 21 and 23 months, had been autoclaved 560 times and the Group A, B and D handpieces were still functioning after 820 autoclave cycles. Therefore, there is no evidence that such a limit applies in general, although exact comparison may not be possible, because of differences in conditions of use and autoclaving. Thus, it can only be a speculation, but the rigorous attention to cleaning, lubrication and autoclaving conducted in the course of this study may have contributed to the substantial working life of most bearings that are still functioning at 30 months. In contrast, informal comparisons with the practice records for the purchase of replacement bearing-turbine cartridges suggested a typical lifetime of around 10 months. This observation, one of the motivations for this study, was in accordance with a survey report that some 52% of GDP respondents said their turbines needed repair within 7 to 12 months of use and that only one in five survived one year of use (Lloyd, Burke & Cheung, 1995).

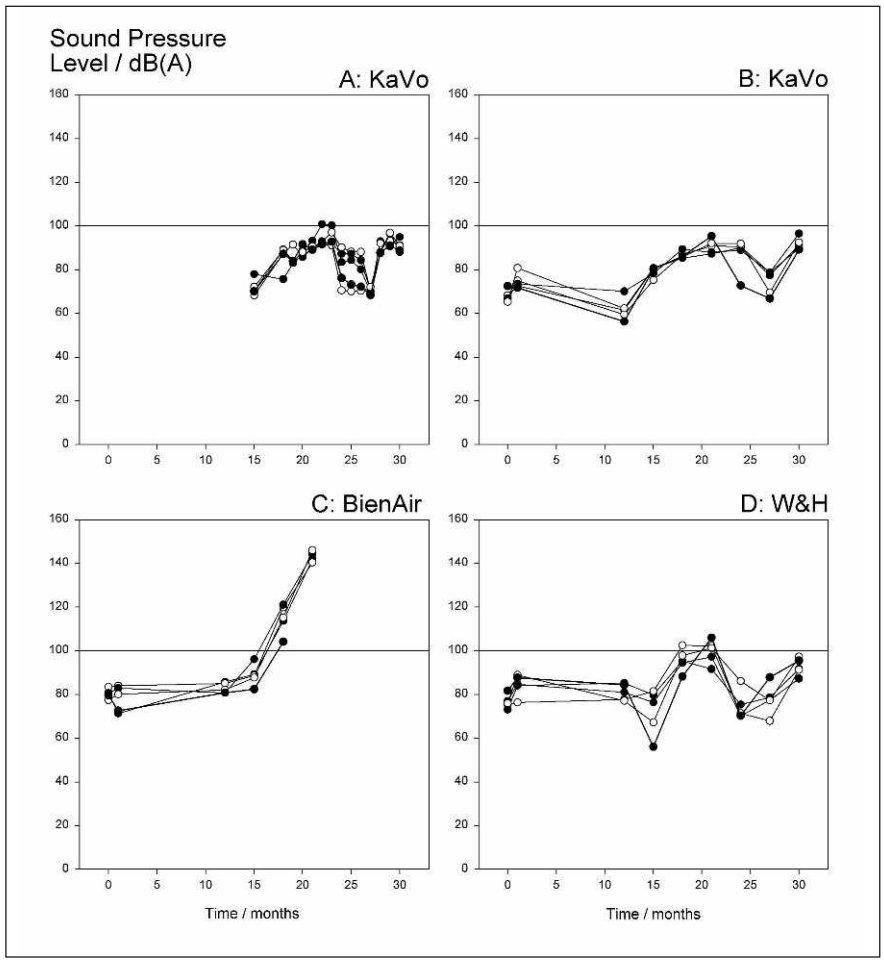

Figure 3. *Variation of sound pressure level with time of use.*

Of course, it is not possible from these results to disentangle the effects of autoclaving from the wear and tear of use. This would require a full parallel set of handpieces which were cleaned, lubricated and autoclaved but only used for testing, an option excluded here based on economic and logistical grounds but which may be of value now that various operational issues are understood, such as time-scale and frequency of testing.

Free-running Speed

The increase in free-running speed found at the second observation point over baseline in several cases, most notably with Group B, suggests three things. First, that some residue of the bearing manufacturing process or an anti-corrosion treatment prevents the full performance allowed by the design from being realized. To some extent this is supported by the corresponding bearing resistance measurements (Figure 2). Second, it might be appropriate to clean and lubricate, then gently "run in" a new bearing before it is put into full use. Third, product comparisons (including standards specification compliance testing) should allow for the possibility of such early variation. The lack of uniformity between handpieces at baseline is not, in itself, an issue, as this reflects details of design (and air pressure). It would appear that this phenomenon deserves closer study in a laboratory, rather than in service, to avoid confounding by other factors.

Bearing Resistance

Bearing resistance is affected by wear, not only of the metal surfaces, but also of the ball-cages, when roughness contributes to friction. Wear debris might accumulate in an erratic manner, as it would seem evident that a thorough cleaning of such complex spaces is difficult without running the turbine during the process. Lubricant viscosity is also a factor, and the grease used for Group C may have some effect, although from the current data, this does not appear to be large.

The abrupt increase in value found for two handpieces in Group D (Figure 2) may be indications of damage, although not yet of a magnitude to appreciably affect free-running speed. This relationship will need further investigation to determine the sensitivity of the one to the other.

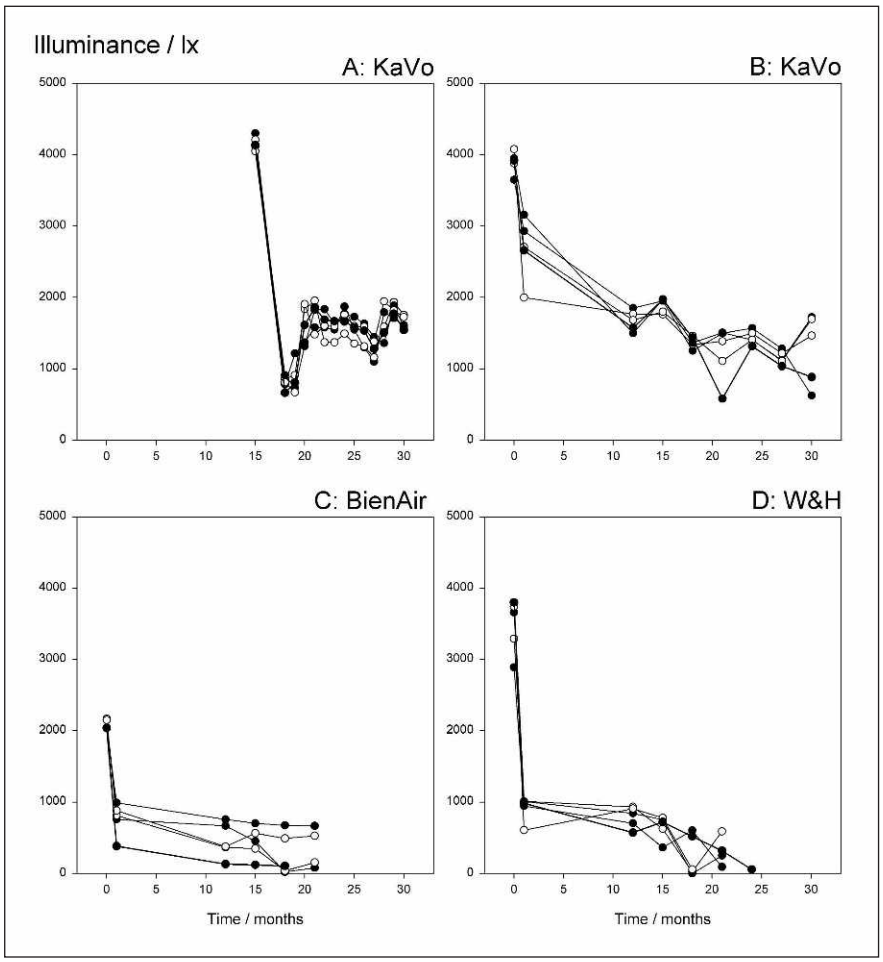

Figure 4. *Variation of illuminance with time of use.*

Sound Pressure Level

The sound pressure level data show signs of systematic variation that is correlated over time between handpieces within their groups. This suggests an instrumentation problem that will need resolution in future work but may relate to an unsuspected directionality of emission given the high frequencies involved despite the attempt to control positioning. Even so, the Group C data appears to show a very clear early warning of the impending total bearing failures suffered before 24 months, even though no sign of this is apparent in either free-running speed or bearing resistance. It should have been easily apparent to the users that there was a problem, as ordinarily values above about 120 dB(A) cause pain. This finding provides support for the clinical observation that substantial, and often sudden increase in noise heralds imminent bearing failure.

At baseline, there was a significant difference (p=0.004) in sound pressure level between groups, with Groups A and B having generally lower values than Groups C and D.

Illuminance

The general sharp decline in illuminance is as surprising as it is disappointing and no prior report of such marked deterioration seems to have been made. For Groups C and D, with values below 1000 lx, the illumination given was of little clinical use—indeed, in Group D, the fibre-optic system had failed completely (illuminance < ~50 lx) by month 25. It was ascertained that this was not due to the failure of either bulb or switch. The illuminance in Groups A and B was mostly above 1000 lx and was judged subjectively as clinically useful.

The handpiece models tested here used the same principle—a tungsten-halogen bulb with a fibre-optic to conduct the light to the head of the handpiece. The KaVo handpieces (Groups A and B) had the bulb located in the quick-fit connector, interfacing with the fibre-optic in the body of the handpiece. The BienAir (Group C) and W&H (Group D) handpieces had the bulb located within the body of the handpiece, the disadvantage being is that it is therefore autoclaved, which may have some bearing on the results for these groups. However, it seems that attention needs to be paid to the state of the adit and exit windows of the fibre-optic itself to determine whether an accumulated patina is responsible for the decline or whether the glass is etched. If the former, a specific inspection and cleaning procedure may be appropriate; evidence for this may lie in the "recovery" for Group A around 20 months. If the glass is etched, other measures will be required, noting that silica glasses are subject to hydrolysis and that presumably this will be accelerated at high temperature and partial pressure of water vapor. Handpieces of the type represented by Groups C and D may suffer more from the accumulation of surface contamination, but those of Groups A and B may be more easily damaged by careless handling. The exit window, of course, is generally more vulnerable.

Kilpatrick has observed that "outside sources of light are constantly being blocked by the lips and teeth of the patient and by the head, fingers and instruments of the operator or assistant. These light arrangements need constant adjustment during a procedure. Some mouth positions make it difficult to achieve optimum lighting conditions" (Kilpatrick, 1974). He also noted that poor illumination within the oral cavity was cited by dentists as contributing to stress and fatigue. Thus, the decline in illuminance from the handpiece is likely to con-

Table 2: *Trend Analysis of Free-running Speed Results: Straight Line Regression Excluding Points at t=0 (month 15 for A: KaVo), that is, after presumed "run-in." Slope in units of Hz/month.*

Group		r^2	n	F-ratio	*p*	slope ± se	
A: KaVo		0.814	12	43.75	6.0e-5	***	-141 ± 21
0.398		12	6.61	0.028	*	-49 ± 19	
0.236		13	3.40	0.092	NS	-35 ± 19	
0.082		13	0.98	0.343	NS	-19 ± 19	
0.003		13	0.04	0.853	NS	6 ± 31	
combined:		0.184	63	13.76	4.5e-4	***	-45 ± 12
B: KaVo		0.020	8	0.12	0.737	NS	3 ± 7
0.571		8	7.97	0.030	*	-39 ± 14	
0.861	7	31.05	2.6e-3	**	-99 ± 16		
0.704	8	14.27	9.2e-3	**	-45 ± 12		
0.429	7	3.76	0.110	NS	-31 ± 16		
combined:		0.372	38	21.37	4.7e-5	***	-36 ± 8
C: Bien Air		0.581	4	2.77	0.238	NS	-12 ± 7
0.017	5	0.05	0.834	NS	4 ± 16		
0.314	5	1.37	0.326	NS	-48 ± 41		
0.495	5	2.94	0.185	NS	-14 ± 8		
0.724	5	7.87	0.068	NS	-78 ± 28		
combined:		0.174	24	4.65	0.042	*	-34 ± 16
D: W&H		0.585	8	8.47	0.027	*	-33 ± 11
0.615	8	9.59	0.021	*	-62 ± 20		
0.491	8	5.79	0.053	NS	-40 ± 16		
0.413	8	4.23	0.086	NS	-36 ± 18		
0.587	7	7.10	0.045	*	-40 ± 15		
combined:		0.474	39	33.37	1.3e-6	***	-38 ± 7

Key: r^2: coefficient of determination; n: number of data points used; p*: significance probability; se: standard error of estimate; NS: not significant; *:0.01 <p<0.05; **: 0.001 <p<0.01; ***:* p*<0.001*

Table 3: *Trend Analysis of Bearing Resistance Results: Straight Line Regression Excluding Points at t=0 (month 15 for A: KaVo), that is, after presumed "run-in."*

Group	r^2	n	F-ratio	*p*	slope ± se	
A: KaVo	0.290	13	4.50	0.057	NS	4.34 ± 2.04
0.494	13	10.75	7.3e-3	**	2.81 ± 0.86	
0.572	13	14.68	2.8e-3	**	4.16 ± 1.09	
0.536	13	12.71	4.4e-3	**	3.81 ± 1.07	
0.702	13	25.87	3.5e-4	***	3.26 ± 0.64	
combined:0.411	65	44.00	8.7e-9	***	3.68 ± 0.55	
B: KaVo	0.528	8	6.72	0.041	*	1.22 ± 0.47
0.010	8	0.06	0.810	NS	0.12 ± 0.49	
0.633	8	10.34	0.018	*	1.07 ± 0.33	
0.481	8	5.55	0.057	NS	0.75 ± 0.32	
0.474	8	5.41	0.059	NS	1.12 ± 0.48	
combined:0.357	40	21.10	4.7e-5	***	0.86 ± 0.19	
C: Bien Air	0.283	5	1.18	0.357	NS	0.46 ± 0.43
0.302	5	1.30	0.338	NS	0.46 ± 0.40	
0.114	5	0.39	0.578	NS	0.26 ± 0.42	
0.006	5	0.02	0.902	NS	0.06 ± 0.47	
0.916	4	21.94	0.043	*	1.18 ± 0.25	
combined:0.163	24	4.30	0.050	NS	0.42 ± 0.20	
D: W&H	0.713	6	9.92	0.035	*	0.67 ± 0.21
0.718	7	12.70	0.016	*	1.57 ± 0.44	
0.726	8	15.91	7.2e-3	**	1.51 ± 0.38	
0.154	8	1.09	0.337	NS	0.29 ± 0.28	
0.394	8	3.91	0.095	NS	1.23 ± 0.62	
combined:0.420	37	25.53	1.4e-5	***	1.05 ± 0.21	

The two high points for D: W&H at 30 months (see Figure 2) were excluded. Slope in units of μN/month. Key as Table 2.

tribute appreciably to operator stress and fatigue as well as making the work itself more difficult.

CONCLUSIONS

Long-term monitoring of handpiece behavior in service is both feasible and informative about the various kinds of deterioration that may occur and, thus, potentially can distinguish between products in terms of performance longevity. This is aided by the use of specialized monitoring equipment. However, a longer sequence with larger sample sizes and more frequent measurements will be necessary to test comprehensively to failure and obtain good sensitivity and discriminatory power. Distinguishing between various lubrication and autoclave regimes, to say nothing of bearings, will require much more data. Sound output seems to be an indicator of some value that may provide a useful early warning of failure. An obvious weakness present in the handpieces tested here, and one that is presumably fairly general, lies in the fibre-optic illumination system: improvement here is essential. Ultimately, it can only be data of these kinds that will permit clinicians to make informed decisions about products

Table 4: *Trend Analysis of Sound Pressure Level Results: Straight Line Regression. Slope in units of dB(A)/month. For C: Bien Air, 0- and 1-month data excluded; "combined" row in addition excludes 12-month data.*

Group	r^2	n	F-ratio	*p*	slope ± se	
A: KaVo	0.051	14	0.64	0.439	NS	0.47 ± 0.59
	0.102	14	1.36	0.266	NS	0.59 ± 0.51
	0.078	14	1.02	0.333	NS	0.47 ± 0.47
	0.010	14	0.12	0.739	NS	0.22 ± 0.64
	0.002	14	0.03	0.868	NS	0.10 ±0.58
combined:	0.033	70	2.35	0.130	NS	0.37 ± 0.24
B: KaVo	0.519	9	7.57	0.028	*	0.70 ± 0.25
	0.449	9	5.70	0.048	*	0.70 ± 0.29
	0.651	9	13.06	8.6e-3	**	0.76 ± 0.21
	0.270	9	2.58	0.152	NS	0.58 ± 0.36
	0.126	9	1.01	0.348	NS	0.41 ± 0.40
combined:	0.355	45	23.66	1.6e-5	***	0.63 ± 0.13
C: Bien Air	0.917	4	22.14	0.042	*	6.43 ± 1.37
	0.930	4	26.53	0.036	*	6.57 ± 1.28
	0.991	4	216.66	4.6e-3	**	7.18 ± 0.49
	0.931	4	27.16	0.035	*	7.30 ± 1.40
	0.801	3	4.03	0.294	NS	3.88 ± 1.93
combined:	0.956	14	263.01	1.6e-9	***	9.04 ± 0.56
D: W&H	0.104	9	0.82	0.396	NS	0.24 ± 0.26
	0.046	9	0.34	0.579	NS	0.26 ± 0.44
	0.010	9	0.07	0.796	NS	0.08 ± 0.31
	0.112	9	0.89	0.378	NS	0.37 ±0.39
	0.032	9	0.23	0.643	NS	0.24 ± 0.50
combined:	0.049	45	2.23	0.142	NS	0.24 ± 0.16

Key as Table 2.

Table 5: *Trend Analysis of Illuminance Results: straight line regression excluding points at t=0 (but t=15 for A: KaVo) and in addition data for t=1 for B: KaVo. Slope in units of lux/month.*

Group	r^2	n	F-ratio	*p*	slope ± se	
A: KaVo	0.015	11	0.14	0.719	NS	8 ± 22
0.002	11	0.02	0.885	NS	-4 ± 28	
0.004	11	0.03	0.858	NS	-4 ± 20	
0.022	11	0.20	0.662	NS	8 ± 17	
0.182	11	2.00	0.191	NS	-19 ± 14	
combined:	0.001	55	0.06	0.804	NS	-2 ± 9
B: KaVo	0.672	7	12.27	0.017	*	-56 ± 16
0.467	7	5.25	0.071	NS	-22 ± 10	
0.078	7	0.51	0.507	NS	-12 ± 16	
0.148	7	1.04	0.354	NS	-17 ± 16	
0.456	7	5.04	0.075	NS	-48 ± 22	
combined:	0.307	35	14.59	5.6e-4	***	-31 ± 8
C: Bien Air	0.748	5	5.93	0.093	NS	-38 ± 13
0.910	5	30.50	0.012	*	-37 ± 7	
0.956	5	65.37	4.0e-3	**	-17 ± 2	
0.550	5	3.66	0.152	NS	-18 ± 10	
0.934	4	28.44	0.033	*	-17 ± 3	
combined:	0.323	24	8.112	9.4e-3	**	-24 ± 8
D: W&H	0.740	5	8.52	0.062	NS	-37 ± 13
0.476	5	2.73	0.197	NS	-35 ± 21	
0.646	5	5.47	0.101	NS	-45 ± 19	
0.114	4	0.26	0.663	NS	-17 ± 34	
0.854	6	23.41	8.4e-3	**	-36 ± 8	
combined:	0.543	25	27.37	2.6e-5	***	-35 ± 7

Key as Table 2.

and ensure that the best value is obtained by adhering closely to appropriate sterilization and maintenance protocols.

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Laboratory Research

A Testing Machine for Dental Air-turbine Handpiece Characteristics: Free-running Speed, Stall Torque, Bearing Resistance

BW Darvell • JE Dyson

Clinical Relevance

The instrument described permits the routine monitoring of the principle performance characteristics of dental air-turbine handpieces both from the point of view of design and deterioration in service.

SUMMARY

The measurement of performance characteristics of dental air turbine handpieces is of interest with respect to product comparisons, standards specifications and monitoring of bearing longevity in clinical service. Previously, however, bulky and expensive laboratory equipment was required. A portable test machine is described for determining three key characteristics of dental air-turbine handpieces: free-running speed, stall torque and bearing resistance. It relies on a special circuit design for performing a hardware integration of a force signal with respect to rotational position, independent of the rate at which the turbine is allowed to turn during both stall torque and bearing resistance measurements. Free-running speed without the introduction of any imbalance can be readily monitored. From the essential linear relationship between torque and speed, dynamic torque and, hence, power, can then be calculated. In order for these measurements to be performed routinely with the necessary precision of location on the test stage, a detailed procedure for ensuring proper gripping of the handpiece is described. The machine may be used to verify performance claims, standard compliance checks should this be established as appropriate, monitor deterioration with time and usage in the clinical environment and for laboratory investigation of design development.

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INTRODUCTION

The air-turbine handpiece is used for most of the cutting work done in dentistry, and this is likely to remain the case for some time to come. However, despite this importance, little fundamental work of value had been reported over about a 40 year period since its introduction (Dyson & Darvell, 1993b,c, 1995a). Detailed investigation showed that the behavior of these devices required several values to be determined (Dyson & Darvell, 1999b) (Dyson, 1993), foremost among which

are free-running speed and stall torque. These two values are dependent on supply pressure. In addition, bearing resistance—the torque required to turn the rotor with supply pressure at zero—is expected to provide an indication of deterioration through wear.

There are two issues in making measurements of this kind. First, the torques involved are rather small, being of the order of 0.2–0.7 mNm/bar (Dyson & Darvell, 1999b) and sensitive equipment is required. Second, free-running speeds are very high rotation rates of the order of 5–8 kHz at 2 bar (Dyson & Darvell, 1999a,b)—and non-contact methods are essential to avoid loading and erroneously low values. In a research laboratory context, these are not as important, given that specialized jigs can be built, but for more routine monitoring (such as in the clinic), a straightforward set-up would be advantageous.

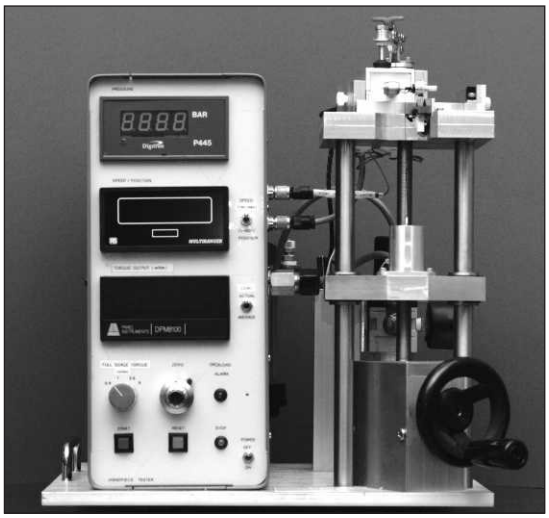

Figure 1. *General view of the front of the prototype handpiece testing machine. The load frame is on the right, with the test stations on top, and the electronics on the left, with the displays of air pressure, speed and torque.*

Table 1: *General Specifications of the Handpiece Tester*

Design maximum load	10 N, tension
Load display	Switchable, 4 ranges: 0-0.5, 1, 2.5, 5 N
External load output	0-5V FS (4 ranges)
Audible alarm setting	11 N
Torque integration	Switchable, 4 ranges: 0-2, 4, 10, 19.99 mNm
External torque output	0-5 V F.S (4 ranges)
Maximum pressure	10 bar
External pressure output	0 - 5 V FS
Maximum rotational speed	10,000/s
External speed output	0-2.5 V FS
Position measurement	500 pulses per revolution
Maximum count rate	165/s; equivalent to: 3 s/rev
Overall accuracy	1% FS range, all ranges
Cross-head travel	100 mm, minimum
Dimensions (w d h)	~330 340 330 mm^3
Weight	~12 kg

The purpose is to describe an instrument which was built for just that application: the routine precise determination of stall torque and free-running speed under standardized conditions.

Design Brief

The value limits of the test system need to accommodate safely any current handpiece, as indicated above, in addition to potential future designs. In order to conduct the torque test, several factors have to be taken into account. Reproducible positioning with the turbine axis accurately normal to the load axis is necessary, yet the irregular shapes of all the various shapes of handpiece must be accommodated. Similar considerations apply for the free-running speed test. A system of custom mounting blocks has been designed to enable these conditions to be met. Although not always recognized in earlier studies (Brockhurst & Shams, 1994; Eames & others, 1979; Walker & Morrant, 1975), the nature of most air-turbine designs is such that the stall-torque varies with turbine rotational position, depending on blade and internal nozzle details and the position of the one with respect to the other, and it is the weighted mean torque over a full revolution that is required (Dyson & Darvell, 1999b). Similarly, if bearing resistance, which is likely to be erratic, is to be measured, it is appropriate to consider a full revolution. It is necessary, then, to perform these measurements over an accurately determined full revolution. Furthermore, the measured force must be integrated over that interval. Normally, integration is performed electronically with respect to time, but to simplify the design, it is necessary to use a manually-driven device, and control of speed is then very poor. This problem then needs to be addressed.

DESCRIPTION

Mechanical

In essence, the device (Figure 1) consists of an electronic and a mechanical part; the general specifications are indicated in Table 1. The mechanical part resembles a small, screw-driven universal testing machine consisting of fixed and movable crossheads, the latter carrying a load cell, the former being the stage upon which the handpiece is mounted. For convenience, a test station for determining free-running speed is located on the same stage. The moving crosshead, manually driven by a handwheel via a 2:1 speed-reducing nylon bevel gear train (B20-2-D, Davall Stock Gears, Hatfield, Herts, UK) and a 1-mm pitch worm screw, moves 0.5 mm per drive shaft revolu-

Table 2: *Major Electronic Components*

Function		Item	Supplier	Catalogue/Part #
1	Load transducer	Load cell	RDP	AL311-1000 grams
2	Position sensor	Encoder module and wheel	Farnell	415-327
3	Speed sensor	Slotted opto-switch	Farnell	491-410
4	Speed, rotation display	Digital indicator	RS	260-094
5	Load, torque display	Digital voltmeter	Farnell	252-451
6	Pressure display	Digital pressure indicator	RS	256-758
7	Pressure transducer	Pressure transducer	RS	249-3858

Suppliers

Farnell Components (HK) Ltd, Hong Kong

RDP Electronics Ltd, Wolverhampton, England

RS Components Ltd, Hong Kong

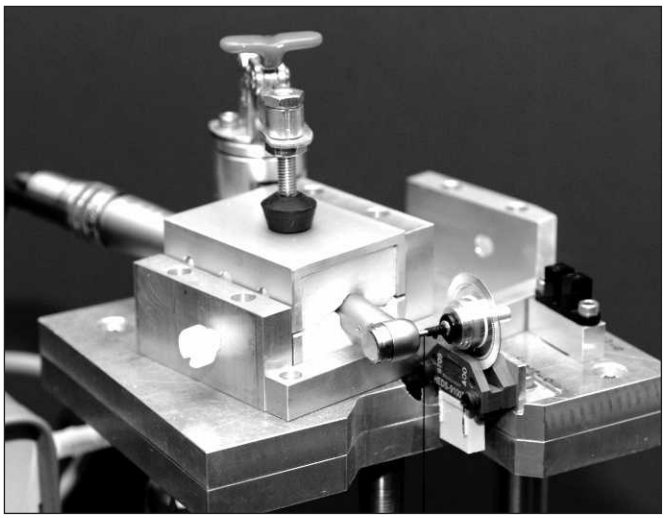

Figure 2. *View of a handpiece mounted in the torque and bearing resistance test station, with the position-sensor sector disk in place.*

tion; cross-head position does not need to be known precisely and is not instrumented. A force signal is generated by a miniature stainless steel-clad tension-compression load cell, capacity ± 110 N (Table 2[1]).

Turbine rotational position is determined from a 500-pulse per revolution sector disk code-wheel and corresponding sensor (Table 2[2]), the disk being mounted on a mandrel for mounting in the turbine chuck (Figure 2). The hub of the disk on the turbine side carries a small neoprene O-ring under which is trapped one turn at one end of a braided silk thread (3/0 suture, Mersilk, Ethicon, Edinburgh, UK) not less than about 125-mm long, the other end of which is knotted into a loop about 8-mm long (see below). The other side of the hub is extended into a 12 mm 3-mm Ø rod as a convenient handle to allow easy insertion of the mandrel into the chuck.

Free-running speed is determined opto-electronically using an infra-red LED and detector package (Table 2[3]). Beam interruption is accomplished by the rotation of a blank mandrel that has been ground both sides to a thin (0.5 mm) blade on about 8 mm of its length, yielding two pulses per revolution.

Electronic

The circuitry is in three sections: digital, analogue and power supply and an outline diagram is shown in Figure 3. The digital portion includes a quadrature decoder (Table 2[2]) which determines both position and direction of rotation, enabling integration in one sense only. Counter pulses are converted to binary-coded decimal to supply the 5-digit speed/position panel meter (Table 2[4]) for position indication on a scale of 0-500. These pulses also drive the integrator (see below) and are used to determine the integration end-point through a logic-driven switch.

The optical speed sensor also produces a pulse train. The frequency is divided by two, prior to being fed to the digital meter. A digital-to-analogue converter provides a proportional voltage signal that can be fed to a chart recorder.

The analogue portion handles the load cell signal (10 mV at 10 N maximum design load; Table 2[5]) via a two-stage voltage amplifier switched to give display ranges of 0.5, 1, 2 and 5 N full scale. An overload alarm is preset to operate at 11 N. The force signal integrator has been described elsewhere (Darvell & others, 1996), but essentially, the load signal is chopped (modulated) into fixed-width pulses (920 μs, to better suit the low rotation rate appropriate here) that are then independent of rotation rate or even pauses. The integration is thus effectively of force vs distance rather than force vs time (what is in effect calculated is work done). The start of the integration is controlled by a push-button switch and the stop point is automatic on reaching a pulse count of 500. The result of the integration can be monitored continuously on a 3 1/2-digit LED panel meter (Table 2[5]), switchable to show actual load (N); this integration is calibrated to display the final value in millinewton-meters (mNm) (these units are, of course, equivalent to newton-millimeters, which may be easier to visualize). Torque calibration is effected by dead-weight loading and trimming the time constant of the load integrator, given the radius of the shaft on which the silk thread is wound (see below) and making due allowance for the effect of thread thickness (Dyson & Darvell, 1999b). This calibration relies on the numerical identity of the average torque to rotate the turbine

and the total work done (mJ) in performing a full revolution.

The 3 1/2-digit LED panel meter (Table 2[6]) for the pressure display is fed directly from the pressure transducer (Table 2[7]) in the compressed air line, downstream of the supply valve and filter. The power supply provides multiple dc rails for the various components and incorporates filtration and stabilization to provide a clean, well-regulated supply for stability and accuracy.

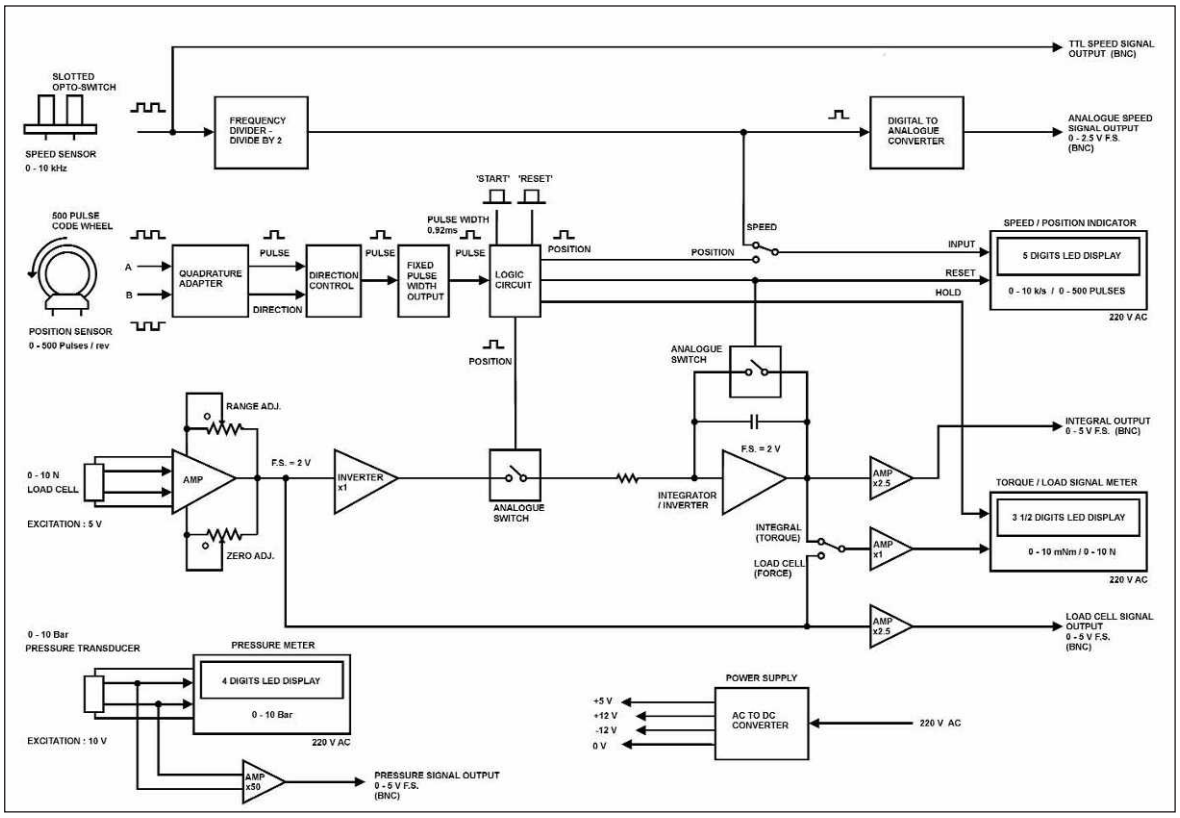

Figure 3. *Schematic diagram of the principal circuitry and components of the handpiece tester.*

OPERATION

Preparation of Clamping Blocks

For both measurements, accurate and precise repeated repositioning is essential to enable the sensors to be in the correct position, that is, deliberately obviating delicate adjustments each time. This is achieved by gripping the handpiece in a two-part clamping block, the lower half of which uses cold-cure acrylic for rigidity and the upper part a stiff, addition-polymerizing (for long-term stability) silicone rubber (for example, Exaflex silicone putty impression material, GC, Tokyo, Japan). This block is molded to suit the particular model (indeed, the exact item). This entails the use of a special jig system to ensure proper alignment.

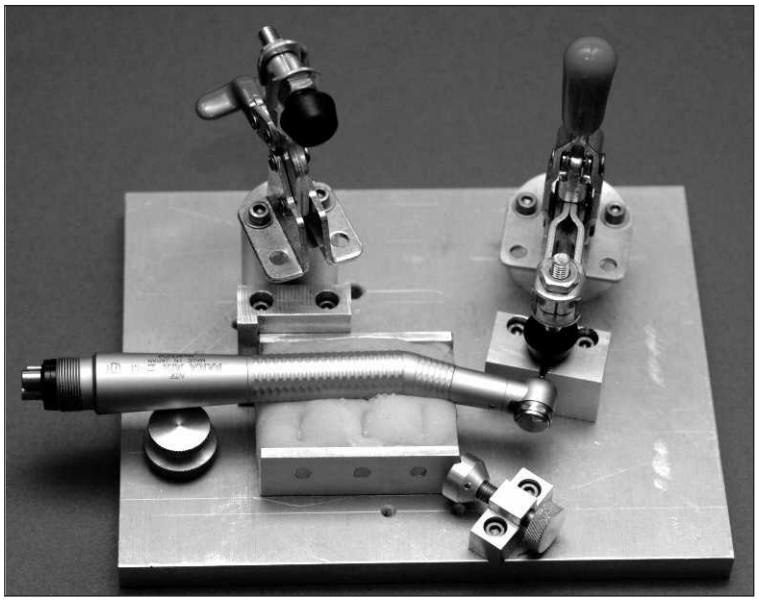

Figure 4. *The handpiece mounting block preparation jig.*

Thus, a blank mandrel (30.0 mm) is fitted to the chuck, which is then inserted to a predetermined depth in a location V-block and held in place with a hard rubber-faced toggle clamp. Allowing the body of the handpiece to rotate freely, its alignment with respect to the horizontal of the jig is then adjusted by eye (as a non-critical issue) by means of a large-headed screw rest. A section of rectangular, thick-walled aluminum channel (50 38 17 mm^3, 3-mm wall thickness), with retention holes in the side walls, is located in its place in the jig, the handpiece having been rotated clear. This, the "lower" channel, is then loaded with a portion of the mixed cold-cure acrylic that has been allowed to proceed

to a firm dough stage, and the handpiece is rotated back and gently seated on the screw rest. The acrylic dough can be adjusted to not exceed in height the midline of the body and be roughly level with the channel walls. However, an accurately level surface is not required (indeed, it is a disadvantage, as some irregularity provides extra location keying and some indentations may be made deliberately with advantage). When set, the handpiece is again rotated out of position; the use of a very thin smear of petroleum jelly ensures clean release from the acrylic. Removing the channel from the jig, the ends of the block can be trimmed up as necessary, as excess material can extrude from the retention holes. Any material on the channel wall edges is also removed, along with any gross excess on the upper surface.

After applying a very thin film of petroleum jelly to the acrylic and the handpiece, as necessary, the lower channel is replaced in the jig and the handpiece rotated back into its impression (Figure 4). A second piece of channel is then loaded with freshly mixed silicone putty and placed over the first, rubber to acrylic, to form the upper counterpart clamping block. This is held in place with a second toggle clamp to ensure that it remains undisturbed while setting. When set, the ends can be tidied up with a sharp blade, and the slight flash that will be present initially (beneficially) between the mating metal surfaces removed. This last ensures that, when reassembled and clamped, the rubber is slightly compressed and the handpiece held securely.

Bearing Resistance

The handpiece, with the sector disc mounted, is located and clamped in the appropriate stage position using the clamping blocks (Figure 2). The silk thread is then wound (in the correct sense) for several non-overlapping turns on the shaft, the free-end loop passed over a hook on the load cell, and the crosshead lowered to take up the slack. When the thread is sufficiently taut, such that the turbine has started to turn, the integration may be started and the crosshead lowered slowly, unwinding the thread until the position count has reached 500 and the integration has stopped automatically when the average torque can be recorded. The integration may be repeated while sufficient thread remains wound on the mandrel.

Stall Torque

The procedure for this measurement is essentially the same as for bearing resistance except that air is supplied to the turbine at whatever is the required pressure for the test in question once the thread has been correctly wound and the slack taken up.

Free-running Speed

For this test the handpiece with the blade mandrel fitted is mounted in the second stage position. Apart from turning on the air and adjusting the supply pressure to the target value, there is nothing more to do except note the displayed value.

APPLICATIONS

There are a number of areas of interest in characterizing dental air-turbine handpieces. The most obvious is, of course, product comparison. The figures of merit previously described provide a means of doing this (Dyson & Darvell, 1999b), and data as obtained from this system is central to that. Second, and in a similar context, the ready verification of manufacturer's claims is an important means of ensuring that practicing dentists have reliable information on which to base purchasing decisions. The effect of such checks may be judged based on the kinds of problems that might be identified (Dyson & Darvell, 1997). Likewise, standards compliance testing would be facilitated by such means.

A fourth area of some considerable interest is the monitoring of bearing quality, both by reference to the bearing resistance as such and the consequential effects on both free-running speed and stall torque and, hence, on power. This may be viewed in terms of conditions of use (duration, duty cycle, lateral loading, lubrication) and the effects of autoclaving, where corrosion is an issue of concern, for example, the possible benefits of ceramic vs steel bearings might then be elucidated.

In a more developmental direction, turbine and nozzle design might be approached more rationally. There appears to be a measure of arbitrariness evident in some designs and clarification of the relevance of certain factors or dimensions is overdue (Dyson & Darvell, 1993a). Attention might also be given to lubricant formulation in a similar manner. Indeed, any aspect of handpiece design that has a bearing on performance may now be readily investigated with such a device with respect to the accessible properties.

DISCUSSION

Determining the three values accessible by this machine is not sufficient for a complete characterization of an air-turbine handpiece. In particular, the free-air consumption rate needs to be determined for the standardized efficiency index to be calculated (Dyson & Darvell, 1999b). However, data for the determination of the handpiece pressure effectiveness, which requires a graphical treatment of transformed speed data vs pressure (Dyson & Darvell, 1999a), can be obtained with a systematic approach. The data obtainable using this device would enable work to be undertaken toward improved turbine design. Certainly, it is hard to imagine that current designs have been optimized, given the range of characteristics previously catalogued (Dyson & Darvell, 1999b).

Considerable effort has been made to simplify the measurement process, and much of the otherwise essential fine adjustments have been eliminated. The

tester has proved to be simple and easy to use on a routine basis (Monaghan, Wilson & Darvell, 2005). Thereby, the understanding gained might allow some progress in that more vexed area, the evaluation of cutting itself (Dyson & Darvell, 1995b), although adequate proxies for dental tissues remain to be identified.

It should be remembered that the user's primary concern is essentially with the efficiency of the cutting process, however, this is to be expressed. Thus, the designs of the cutting instruments attract attention, but manufacturers' claims cannot be adequately assessed in the absence of an objective measure. The difficulty has been that the behavior of the cutting device could not be disentangled from that of the handpiece, given that its performance has not been quantifiable. Further complexity is found in the sense of the users' behavior: duty cycle (pattern of cutting vs noncutting periods during a task); loading (force) patterns, both axial and lateral; use and amount of irrigation of the cutting site and tolerance of wear (how often cutters are replaced). Certainly, much information needs to be gathered about these aspects in real practice situations. However, none of this is of much help unless the capabilities of the "power plant"–the actual turbine–are documented.

The work of cutting involves the creation of a great deal of surface area, plastic deformation, other internal damage in the chips and much frictional heat (noise is usually negligible in comparison). Since only heat is, in principle, easily quantifiable (calorimetrically), the power delivery needs to be known accurately for what might be termed the true work of comminution to be evaluated, and for this to be related to cutting conditions, especially cutting device design, the development of which can only have been somewhat pragmatic rather than systematic. Thus, the kind of information now available from this test device is fundamental to progress in this area of more immediate dental interest. Another dimension to the subject often overlooked is that of patient tolerance. Reduction of cutting time and the effort involved can only increase patient acceptance of what are otherwise stressful experiences.

It is true to say that the techniques embodied in this device can be realized in any reasonably well-found research laboratory. However, what is presented here is a means of avoiding the commitment of substantially more expensive generalized test equipment so that long-term monitoring of such matters as the effects of autoclaving or other sterilization techniques, lubrication regimes and wear related to duty cycle and loading patterns becomes straightforward, if not routine. In addition, "field" use is enabled.

CONCLUSIONS

In summary, there is no aspect of the design of handpiece air-turbines and their use that cannot be addressed through the means. It may be that this entire area, in which research has rather stagnated in recent decades, can at last be opened up to fruitful investigation.

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Influence of Flowable Liner and Margin Location on Microleakage of Conventional and Packable Class II Resin Composites

CJ Tredwin • A Stokes • DR Moles

Clinical Relevance

Microleakage scores in this *in vitro* study suggest that gingival margins of resin composite restorations showed significantly less leakage in enamel than dentin, conventional and packable resin composites do not perform differently and flowable linings not only showed significant leakage for both conventional and packable resin composites, but leaked significantly more than either restorative material, alone.

SUMMARY

This *in vitro* study evaluated gingival wall microleakage in packable and microhybrid conventional composite restorations with and without a flowable composite liner. Each group was evaluated with gingival margins situated in both enamel and cementum/dentin.

Two hundred and forty Class II cavities were prepared in extracted third molars, half with gingival margins in enamel and half with margins in dentin/cementum. In groups of 30, restoration was undertaken with packable alone (3M Filtek P60), conventional alone (3M Z250), packable plus flowable liner (3M Filtek Flow) and conventional plus flowable liner. All used 37% phosphoric acid etch and Scotchbond 1 (3M) as the bonding system. After restoration, the teeth were thermocycled (between 5°C, 37°C and 60°C) 1,500 times, soaked in 0.1% methylene blue, sectioned and microleakage from the gingival margin scored. Statistical analysis was performed using Kruskal Wallis and Mann-Whitney U tests.

There was no significant difference between systems in terms of leakage scores when gingival margins were situated in enamel (p=0.70). All restorations with margins in cementum/dentin leaked significantly more than those with margins in enamel (p<0.001). There was no significant difference between leakage scores of 3M Z250 and Filtek P60 with cementum/dentin gingival margins (p=0.68). Use of a flowable composite liner (3M Filtek Flow) against cementum/dentin was associated with increased microleakage (p<0.001).

In this study, leakage scores suggest that gingival margins should be placed in enamel. The conventional and packable resin composites

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tested were not associated with differences in microleakage. Leakage data do not support the use of flowable resin composite linings in Class II resin composite restorations.

INTRODUCTION

Despite advances that have been made, many clinical and material limitations have restricted the universal use of resin composites as a posterior restorative material. Some problems confronting clinicians when placing resin composites in Class II cavities include difficulties with polymerization contraction and marginal seal, obtaining proximal contact, adhesion to placement instruments and poor adaptation (Belvedere, 1999; Leinfelder, Radz & Nash, 1998).

Recently, several manufacturers have introduced "packable" composites to the marketplace as alternatives to amalgam (Leinfelder, 1997; Leinfelder & Prasad, 1998; Adams, 1999). Packable composites use amalgam techniques for placement and have been proposed to produce acceptable interproximal contacts (Leinfelder & others, 1998). Because of the high depths of cure and low polymerization shrinkage of packable composites, a bulk-fill technique may be possible. However, these stiffer materials may not adequately adapt to internal areas and cavosurface margins, particularly at the cervical joint (Leevailoj & others, 2001). Flowable resin composites used as liners in areas of difficult access have been suggested to address this concern. This assumes that these less viscous materials flow easily onto all prepared surfaces, resulting in less leakage and postoperative sensitivity. Flowable resin composite liners may also act as a flexible intermediate layer that help relieve stresses during polymerization shrinkage of the restorative resin (Kemp-Scholte & Davidson, 1990). However, flowable resin composites shrink more than traditional composites, because they have less filler loading (Tolidis & Setcos, 1999).

This study investigated the influence of a flowable composite intermediary upon leakage between tooth tissues and both packable and conventional resin composites in Class II cavities.

METHODS AND MATERIALS

Materials

Tables 1 and 2 summarize the product profiles and batch numbers of the materials used in this study.

Restorative Methods

Tooth Selection and Cavity Preparation

One hundred and twenty recently extracted, non-carious, restoration-free human third molars were stored in 0.1% thymol solution until use, after cleaning with a

Table 1: *Product Profile of Restorative Materials Used (manufacturer's data)*

Product	Presentation	Chemical Composition	Batch #s (Reference) *(Expiry Date)*
Filtek Z250 (visible light activated anterior and posterior resin)	Single paste in syringes	Fine particle (0.01 to 3.5 microns), heavily filled (60% by volume) composite utilizing synthetic zirconia/silica filler. Resin is a mixture of Bis-GMA, UDMA and Bis-EMA	20020108 (6020A3) *(2004-11)*
Filtek P60 (visible light activated "packable" posterior resin)	Single paste in syringes	Fine particle (0.01 to 3.5 microns), heavily filled (61% by volume) composite utilizing a synthetic zirconia/silica filler. Resin is a mixture of Bis-GMA, UDMA and Bis-EMA	20011101 (4720A3) *(2004-09)*
Filtek Flow Flowable Restorative (low viscosity, visible-light activated, radiopaque flowable restorative)	Low viscosity single paste in syringes	Contains Bis-GMA and TEGDMA resins. The filler is zirconia/silica. Inorganic filler loading is 47% by volume with a particle size of 0.01 to 6.0 microns (average 1.5 μm)	20020110 (3700A3) *(2004-11)*

Table 2: *Product Profile of Etchant and Adhesive Used (manufacturer's data)*

Product	Presentation	Chemical Composition	Batch #s (Reference) *(Expiry Date)*
Scotchbond Etchant	Single liquid	Aqueous solution of 35% Phosphoric acid and a non-silica thickener.	20011126 (7423) *(2004-09)*
Scotchbond Adhesive (two step smear layer removing adhesive system)	Single liquid	Primer incorporated based on HEMA.	20020108 (4242) *(2004-10)*

pumice water slurry. Class II box-only cavity preparations were prepared on the mesial and distal surfaces of each tooth. These preparations were accomplished with diamond burs (Shofu 411, Shofu Inc, Kyoto, Japan) and carbide burs (No 330, Beavers Dental, Morrisburg, Ontario, Canada) in a high-speed handpiece with water spray. There was no occlusal connection between the preparations. In 60 teeth, 120 cavity preparations were cut, with the gingival margins placed 1-mm coronal to the cementoenamel junction (CEJ). In the other 60 teeth, 120 cavity preparations were prepared with the gingival margins placed 1 mm apical to the CEJ. All other cavity dimensions were as follows:

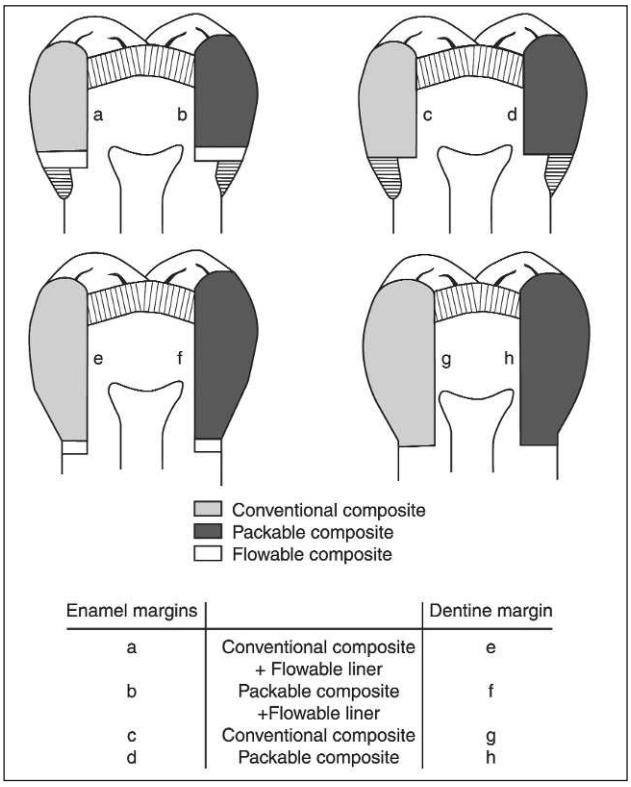
- The buccolingual width was 2 mm. The dimensions of the burs used gauged all depths.
- Buccal and lingual walls of the preparations were approximately parallel and connected to the gingival wall with rounded line angles.
- The boxes were prepared 2-mm deep axially.
- The margins were not beveled but smoothed, using hand instruments (margin trimmers).

Restoration

Following cavity preparation, each tooth was rinsed, cleaned using a pumice/water slurry in a rubber cup, rinsed once again and dried. Filling procedures recommended by the manufacturer were then followed. Scotchbond etchant was applied with a disposable brush (Microbrush, 3M Dental Products Division, St Paul, MN, USA) for 15 seconds to both enamel and dentin of the prepared cavity. The etchant was rinsed off for 10 seconds with water from a triple syringe. Excess water was then blotted with a disposable brush, leaving the tooth moist.

Two consecutive coats of Scotchbond 1 Adhesive were applied to the enamel and dentin using a fully saturated disposable brush, and the cavity was gently air dried for five seconds to leave a shiny surface. The adhesive was then light cured for 10 seconds using an SDS Kerr Optilux Demetron light-curing unit with a light intensity of 400mW/cm^2. This unit was used for all light curing procedures.

A metal Auto-Matrix band (Caulk Automate KJ Lazarus, USA) was tightened and held by finger pressure against the gingival margin of the cavity so that the preparations could not be overfilled at the gingival margin. This also allowed light to be directed only in an apical direction when curing the composite. When a flowable resin composite liner was used, the cavity was lined on the pulpal and axial walls with a 1-mm thickness at the gingival wall of the cavity preparation. This depth was judged by a groove placed on the delivery needle. The other resin composites used were built up using a diagonal incremental technique. Each 1-mm increment was light cured for 40 seconds. The matrix



Enamel margins		Dentine margin
a	Conventional composite + Flowable liner	e
b	Packable composite +Flowable liner	f
c	Conventional composite	g
d	Packable composite	h

Figure 1. *Diagram shows the design of eight groups in this study.*

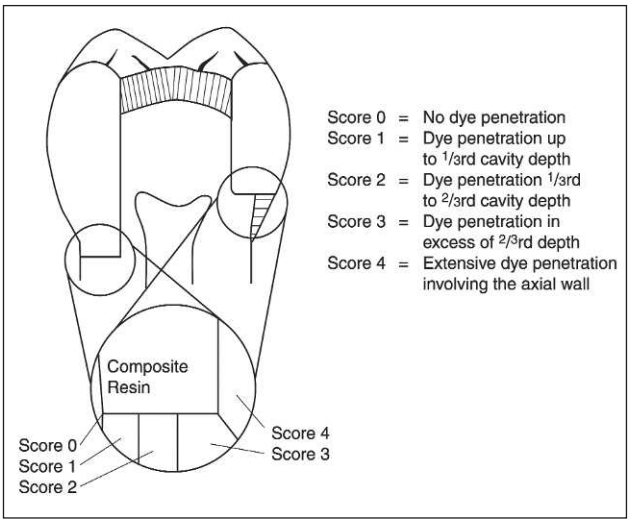


Figure 2. *Diagram shows dye penetration scoring protocol.*

was removed after both restorations were completed. A series of Sof-Lex disks (3M) were used to finish margins that would be accessible clinically. Gingival margins were not disked.

Figure 1 illustrates the eight resulting restorative variants (30 of each type).

Following restoration, the teeth from all groups were stored in deionized water in a sealed container at 37°C for 60 days.

Thermal Cycling

After 60 days, the teeth were thermally cycled between 5°C, 37°C and 60°C water baths for 1,500 cycles with a 20-second dwell time and 10-second transfer time. They were then stored in distilled water at 37°C for an additional five days.

Dye Penetration

For evaluation of leakage by dye penetration, the restored teeth were cleaned with a pumice/water slurry and dried. The apices were sealed with resin and the crown and root surfaces painted with three coats of fingernail varnish to within 1 mm of the gingival restoration margin. After allowing 15 minutes for the varnish to set after each coating, the teeth were immersed in 0.1% methylene blue dye solution at 37°C for 24 hours. On removal from the dye, the teeth were rinsed thoroughly and cleaned with a bristle brush to remove surface dye, then returned to deionized water prior to sectioning.

Sectioning

In the vertical plane, each tooth was sectioned mesiodistally across the center of the restorations using an Exakt 0.2-mm band saw with continuous water irrigation.

Evaluation of Sections

After sectioning, each specimen was stored dry in labeled containers. The sectioned teeth were examined under a light microscope at 20x magnification by two independent evaluators and scored for dye penetration on a 0 to 4 scale.

The diagram shown in Figure 2 was used for reference while scoring. Using the same protocol, scoring was repeated 24 hours later by the same examiners without reference to the previous scores.

The scores were then tabulated and a final data set compiled from each scoring of the specimens using the worst score for each interface.

Statistical Analysis

The groups were compared for differences in microleakage ratings. Inter- and intra-examiner agreement was assessed using Kappa statistics. Kappa interpretations were made using the description by Altman (1991).

Because the data are on an ordinal scale, a Kruskal-Wallis test was used to assess for differences within the enamel or dentin groups. If there was evidence of a difference from the global Kruskal-Wallis test, then Mann-Whitney U tests were used to investigate the pairwise differences between different groups of filling materials.

RESULTS

Examiner Agreement

There was close agreement between examiners—overall (Kappa=0.84) for restorations with enamel margins (Kappa=0.83) and for restorations with dentin margins (Kappa=0.89). Of the 240 restorations scored, there were only 17 disagreements between examiners based on only one category.

Reproducibility by each examiner 24 hours later was also very good. Table 3 shows the Kappa results for reproducibility by each examiner.

Comparison of Restorative Materials

The leakage scores for the different filling material and gingival margin locations are shown in Table 4 and graphically depicted in Figures 3 and 4.

Kruskal-Wallis tests showed no significant differences between the leakage scores of the restorative materials used within enamel (p=0.70); however, they showed significant differences between leakage scores

Table 3: *Intra-examiner Agreement*

Category	Kappa Score	
	Examiner 1 (ANS)	Examiner 2 (CJT)
Overall	0.91	0.89
Enamel gingival margins	0.92	0.89
Dentin gingival margins	0.89	0.90

Table 4: *Frequency of Microleakage Scores*

Situation/ Filling Type	SCORE					Total Number
	0	1	2	3	4	
Enamel Conventional (Z250)	28	2	0	0	0	30
Enamel Packable (P60)	25	5	0	0	0	30
Enamel Conventional + Flowable	27	1	2	0	0	30
Enamel Packable + Flowable	26	2	2	0	0	30
Dentin Conventional (Z250)	11	14	5	0	0	30
Dentin Packable (P60)	9	20	1	0	0	30
Dentin Conventional + Flowable	4	7	7	6	6	30
Dentin Packable + Flowable	3	5	7	8	7	30

of the different restorative materials used in dentin ($p<0.001$).

Mann-Whitney U tests for independent samples showed:

- All of the restorative modes studied leaked significantly more when located with gingival margins in dentin rather than with margins in enamel ($p<0.001$).
- There was no significant difference between the leakage scores of Z250 (Universal Resin Composite) and Filtek P60 (Packable Resin Composite) with gingival margins in dentin ($p=0.68$).
- With gingival margins in dentin, Z250 with a Filtek Flow flowable liner had significantly higher leakage scores than Z250 alone ($p<0.001$).
- With gingival margins in dentin, Filtek P60 with a Filtek Flow flowable liner had significantly higher leakage scores than Filtek P60 alone ($p<0.001$).

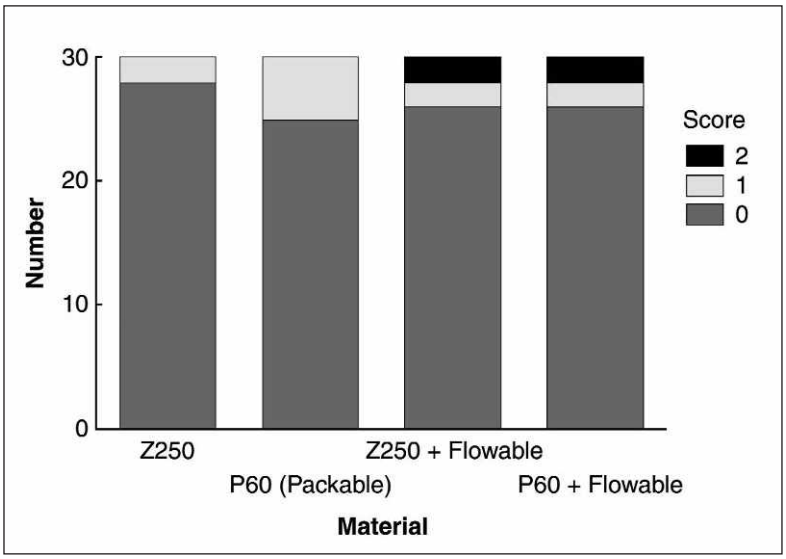


Figure 3. *Stacked bar graph shows gingival leakage scores in Enamel.*

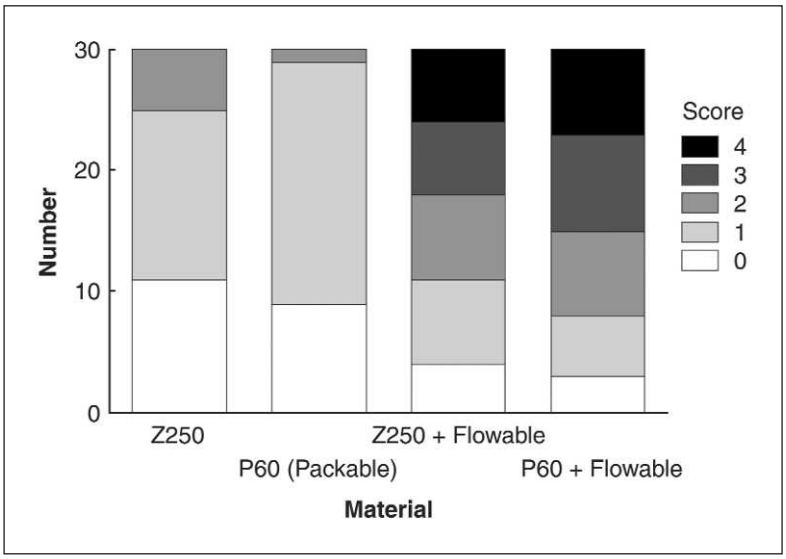


Figure 4. *Stacked bar graph shows gingival leakage scores in Dentin.*

DISCUSSION

Enamel and Cementum/Dentin Margins

Class II cavities were prepared with either enamel or cementum/dentin gingival margins. Without exception, the composite restorations with cementum/dentin gingival margins showed significantly higher leakage scores than those with gingival enamel margins. This was expected, as bond strength to enamel is usually higher than bond strength to dentin. Dentin is a less favorable bonding substrate than enamel (Nakabayashi, Ashizawa & Nakamura, 1992), and the enamel margins of composite restorations are reported to have less leakage than cementum/dentin margins (Nakabayashi & Pashley, 1998).

The Use of a Flowable Resin Composite

Flowable composites are recommended to enhance the adaptation of more viscous resin composites, particularly in proximal boxes of Class II preparations. The presumption is that use of these less viscous results in less leakage and post-operative sensitivity. It has also been suggested that flowable liners may act as a flexible intermediate layer, which helps relieve stresses during polymerization shrinkage of the restorative resin (Kemp-Scholte & Davidson, 1990).

The wear rate of flowable composites is higher than packable composites; therefore, it has been proposed that flowable composites should be used only at contact-free areas (Bayne & others, 1998). In this study, the application of approximately 1-mm thickness of flowable liner at the gingival wall was considered clinically acceptable since this is a contact-free area.

With margins situated in enamel, the use of a flowable liner did not make a significant difference to microleakage, while with margins situated in cementum/dentin, the use of a flowable liner actually made the microleakage significantly worse ($p<0.001$). This calls into question the premise for using flowable liners.

With the gingival margins in enamel, none of the combinations of materials showed any significant difference in microleakage scores. The conventional (3M Z250), packable (3M Filtek P60), conventional plus flowable (3M Filtek Flow) and packable plus flowable were 93%, 83%, 90% and 86% leakage free, respectively. It is worth noting that while not statistically signifi-

cant, only the filling combinations with flowable liners gave leakage scores of more than 0 or 1, with 6% of the conventional plus flowable and packable plus flowable, scoring 2. This could imply a potential clinical distinction.

With the gingival margins situated in dentin, there was not a significant difference between the microleakage scores of the conventional and packable resin composites when used alone. However, when comparing the conventional with and without a flowable liner and the packable with and without a flowable liner, the microleakage scores were significantly worse when a flowable liner was used. The results provided by this study thus bring into question the assumption that the use of less viscous flowable liners results in less leakage around a resin composite filling.

Flowable composites shrink more than traditional composites because they have less filler loading. Manufacturer's data available on Z250 and Filtek P60 reported a volumetric change during polymerization shrinkage of approximately 1%, while Filtek Flow shrinks by about 4%. The increased polymerization shrinkage of the flowable liner may have caused the greater leakage seen in association with this material.

There are few directly comparable studies in this area. One recent *in vitro* study proposed that the use of flowable composite or compomer liners reduced microleakage in Class II packable and microhybrid resin composite restorations with margins in dentin (Leevailoj & others, 2001). In this study, agreement between the scores of the examiners was only fair to moderate, with Kappa scores of 0.33 to 0.54, whereas, the Kappa scores in this study were 0.83 to 0.92. Furthermore, the sample sizes used were only 10 in each category compared to 30 in this study. Different materials were also used with four packable composites (Alert, Surefil, Pyramid and Solitaire) and one conventional microhybrid composite (Renew), with their respective manufacturer's bonding agents and flowable liner. This study used just one manufacturer's products (3M) strictly according to manufacturer's recommendations. Any of the previous factors could have influenced the reported results.

It has been proposed that current flowable materials can be easily syringed into the cavity but are sometimes difficult to manipulate because of their stickiness, and air is sometimes trapped in the restorations while removing the syringe tip from the cavity (Leevailoj & others, 2001). Neither of these difficulties was experienced with the Filtek Flow flowable liner used in this study. No porosities/air bubbles were observed by either examiner while examining the samples at 20x magnification.

Packable Resin Composites

The filler level in a resin composite determines its mechanical strength and physical properties. Higher filler levels dispose the resin composite to improved wear resistance (Li & others, 1985), compressive strength and fracture toughness (Ferracane, Antonio & Matsumoto, 1987) and less polymerization shrinkage (Iga & others, 1991). The sandwich technique (flowable resin composite lining with hybrid/"packable" resin composite overlay) seeks to combine these advantages with the potential for close adaptation offered by the flowable component. Compared to conventional hybrid composites, packable composites need greater force to place the material into the cavity preparation. Previous studies have reported difficulties with packable composites sticking to dental instruments, along with problems with adapting the materials to the preparations (Leevailoj & others, 2001). Filtek P60 did not exhibit these characteristics in this study.

There was no significant difference in leakage patterns between the conventional composite (Z250) and packable composite used (Filtek P60) when placed with gingival margins in enamel or dentin. When comparing like with like, both materials showed significantly more leakage when gingival margins were located in cementum/dentin.

Another study has suggested that packable resin composites may be more prone to porosities than conventional composites (Leevailoj & others, 2001). No porosities were detected in this study in either the Filtek P60 or Z250 at 20x magnification by either of the independent examiners.

CONCLUSIONS

Under the conditions of this *in vitro* study:

1) Leakage scores suggest that gingival margins of resin composite restorations should be placed in enamel.
2) The conventional and packable resin composites tested were not associated with differences in microleakage.
3) With no differences in microleakage scores for any of the groups studied in enamel and, significantly worse scores for flowable liners with margins placed in cementum/dentin, the data do not support the use of flowable resin composite in Class II resin composite restorations.

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One-day Bonding Effectiveness of New Self-etch Adhesives to Bur-cut Enamel and Dentin

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A Poitevin • P Lambrechts • B Van Meerbeek

Clinical Relevance

A trend exists toward simplified adhesive application procedures for bonding of resin composites. However, the most "advanced" one-step adhesives seem significantly less effective; whereas, some two-step self-etch adhesives approach the bonding performance of three-step etch-and-rinse adhesives.

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SUMMARY

Self-etch adhesives try to solve difficulties commonly associated with the clinical application of etch-and-rinse adhesives. Their application procedure is considered less time-consuming and, more importantly, less technique-sensitive. The main objective of this study was to determine the bonding effectiveness to and the interaction with enamel/dentin of three contemporary one- and two-step self-etch adhesives by microtensile bond strength testing (μTBS), Fe-SEM and TEM when compared to a control two-step self-etch and a three-step etch-and-rinse adhesive. The one-step self-etch adhesive, Adper Prompt (3M ESPE), scored the lowest μTBS of all experimental and control adhesives tested. Conversely, the two-step self-etch adhesives Clearfil SE (Kuraray) and OptiBond Solo Plus Self-Etch (Kerr) approached the values obtained by the three-step etch-and-rinse control (OptiBond FL, Kerr) when bonded to enamel and dentin. Ultramorphological characterization showed that interfacial morphology and the pH of the self-etch primer/adhesive are strongly associated. The interaction with dentin varied from the formation of a submicron, hydroxyapatite-containing

hybrid layer for the "mild" self-etch adhesive Clearfil SE to a 3-5 µm thick, hydroxyapatite-depleted hybrid layer for the "strong" self-etch adhesive Adper Prompt. The two-step self-etch adhesives AdheSE and OptiBond Solo Plus Self-Etch presented with a hybrid layer with a hydroxyapatite-depleted top part and a hydroxyapatite-containing base part and were therefore classified into a new group of self-etch adhesives, namely "intermediary strong" self-etch adhesives.

INTRODUCTION

A most recent innovation in dental adhesive technology involves the introduction of "self-etch" adhesives. Self-etch adhesives make use of acidic monomers that simultaneously condition and prime enamel and dentin and provide vinyl groups for co-polymerization with the resin composite. The bonding mechanism of self-etch adhesives is based upon changing the chemical composition of the substrate surface, commonly referred to as hybridization; the surface layer of enamel/dentin is partially dissolved and the resultant porosity filled by resin (Inoue & others, 2000).

These self-etch adhesives try to solve difficulties commonly associated with the clinical application of etch-and-rinse adhesives. Their application procedure is considered less time-consuming and, more importantly, less technique-sensitive, in particular, with regard to keeping the dentin surface in an adequate state of hydration. Self-etch adhesives have, for instance, been associated with less nanoleakage (Sano & others, 1995). This is most likely attributed to resin impregnation that proceeds simultaneously with dentin etching (Watanabe, Nakabayashi & Pashley, 1994). The risk of a discrepancy between the depth of dentin demineralization and hybridization is consequently limited, which is expected to be advantageous in the long term. In support of this effect, Miyazaki and others (1998) reported a larger decrease in bond strength to dentin after thermocycling for "one-bottle" (or two-step etch-and-rinse) adhesives than for self-etch adhesives. Also, Sano and others (1999) found no decrease in microtensile bond strength (µTBS) after one-year *in-vivo* functioning of restorations bonded with a self-etch adhesive. However, self-etch adhesive bonds to enamel are thought to be more susceptible to degradation, as was demonstrated by thermocycling in a shear-bond strength and SEM study (Miyazaki, Sato & Onose, 2000). This was corroborated by SEM evaluation of resin-enamel interfaces after thermal cycling; small cracks and porosities were observed between enamel and adhesive that were not present before.

The main objective of this study was to determine the bonding effectiveness to and interaction with enamel/dentin of contemporary one- and two-step self-etch adhesives by µTBS, Fe-SEM and TEM, when compared to a control, two-step self-etch and three-step etch-and-rinse adhesive.

METHODS AND MATERIALS

Thirty non-carious human third molars were stored in 0.5% chloramine solution at 4°C and used within one month after extraction. The teeth were randomly divided into three experimental and two control groups. First, all teeth were mounted in gypsum blocks to ease manipulation. The µTBS to enamel and dentin was determined following the protocol previously described (De Munck & others, 2002). All adhesives were applied according to manufacturer's instructions (Table 1). The pH of the non-diluted primer, as applied to the tooth surface, was determined at ambient temperature (20-25°C) using a digital pH meter (Inolab pH Level 2, WTW, Weilheim, Germany).

Enamel Specimen Preparation

Lingual and/or buccal enamel was flattened using a high-speed medium-grit (100 µm) diamond bur (842, Komet, Lemgo, Germany) mounted in the MicroSpecimen Former (The University of Iowa, Iowa City, IA, USA). Subsequently, the adhesives were applied (Table 1), after which the surface was built-up with a micro-hybrid resin composite Z100 (3M ESPE, St Paul, MN, USA) in three to four layers to a height of 5 to 6 mm. Because bond strength is also largely influenced by the composite used (Van Noort & others, 1989), the authors opted to use the same resin composite with all adhesives to exclude this variable. During specimen processing, care was taken to prevent dehydration of the specimens.

Dentin Specimen Preparation

The occlusal third of the molars was removed using a slow-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). Dentin surfaces were verified for the absence of enamel and/or pulp tissue using a stereomicroscope (Wild M5A, Heerbrugg, Switzerland). A standard smear layer was created by removing a thin layer of the surface using a high-speed medium-grit (100 µm) diamond bur (842, Komet) mounted in the MicroSpecimen Former. The adhesives and restorative composite were applied following the methodology described above.

µTBS Testing

The experimental set-up is schematically presented in Figure 1. After bonding procedures, specimens were stored for 24 hours in tap water at 37°C. The teeth were then sectioned perpendicular to the bonding surface using the Isomet saw to obtain rectangular specimens about 1.8x1.8 mm wide and 8- to 9-mm long. These specimens were mounted in the pin-chuck of the MicroSpecimen Former and trimmed at the tooth-biomaterial interface to a cylindrical hour-glass shape with a diameter of about 1.2 mm using a cylindrical

extrafine-grit (15 µm) diamond bur (835 KREF, Komet) in a high-speed handpiece under air/water spray coolant. The specimens were then fixed to Ciucchi's device (Pashley & others, 1999) with cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin, Ohtawara, Japan) and stressed at a crosshead speed of one mm/minute until failure in a LRX testing device (Lloyd, Hampshire, UK) using a load cell of 100 N. The µTBS was expressed in MPa, as derived from dividing the imposed force (in N) at the time of fracture by the bond area (in mm^2). When the specimens failed before actual testing, the mean µTBS was determined from the specimens that survived specimen processing with an explicit note of the number of pre-testing failures. The mode of failure was determined at a magnification of 50x using a stereo-microscope. For the dentin

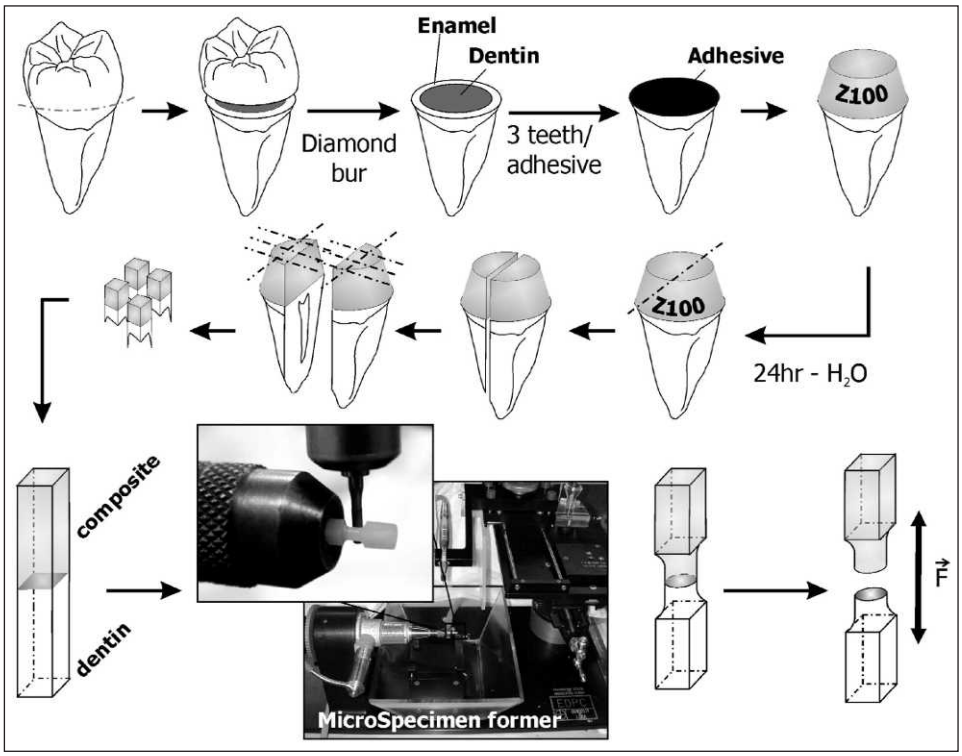

Figure 1. *Schematic presenting the experimental design of microtensile bond strength testing.*

Table 1: *Adhesives Investigated in This Study*

Product Name	Manufacturer	Composition[1] [Lot #]	Application
Adper Prompt	3M ESPE, St Paul, MN, USA	Liquid 1: Methacrylated phosphoric esters, Bis-GMA, camphorquinone, stabilizers Liquid 2: Water, HEMA, polyalkenoic acid, stabilizers	Activate blister. Apply adhesive and rub for 15 seconds. Gently air blow. Light cure for 10 seconds.
AdheSE	Ivoclar-Vivadent, Schaan, Liechtenstein	Primer: Dimethacrylate, phosphonic acid acrylate, initiators, stabilizers, water Bond: HEMA, dimethacrylate, silicon dioxide, initiators	Dry surface. Apply Self-etch primer for at least 30 seconds, from which at least 15 seconds with rubbing motion. Remove excess of primer with air. Apply bond for at least 10 seconds. Gently air blow. Light cure for 10 seconds.
OptiBond Solo Plus Self-Etch	Kerr, Orange, CA, USA	Primer: GPDM, camphorquinone, ethanol, water Bond: Bis-GMA, GDM, HEMA, GPDM, ethanol	Apply self-etch primer and rub for 15 seconds. Air thin 3 seconds. Apply OptiBond Solo Plus and rub for 15 seconds. Air thin 3 seconds. Apply OptiBond Solo Plus and rub for 15 seconds. Light cure for 20 seconds.
Clearfil SE	Kuraray, Osaka, Japan	Primer: 10-MDP, HEMA, hydrophilic dimethacrylate, photoinitiator, water Bond: 10-MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, microfiller	Apply primer for 20 seconds; Gently air blow; Apply bonding agent; Light cure for 10 seconds.
OptiBond FL	Kerr, Orange, CA, USA	Etchant: 37.5% Phosphoric acid, silica thickener Primer: HEMA, GPDM, PAMM, ethanol, water, photoinitiator Adhesive: TEGDMA, UDMA, GPDM, HEMA,Bis-GMA, filler, photoinitiator	Apply etchant for 15 seconds; Rinse for 15 seconds; Gently air-dry for 5 seconds; Scrub surface for 30 seconds with primer; Apply thin coat of bonding and light cure for 30 seconds.

[1] *Composition as provided by respective manufacturer: Bis-GMA = Bisphenol-glycidyl methacrylate; GPDM = Glycerol phosphate dimethacrylate; GDM = Glycerol dimethacrylate; HEMA = Hydroxyethylmethacrylate; 10-MDP = 10-Methacryloyloxydecyl dihydrogen phosphate; PAMM = Phthalic acid monoethyl methacrylate; TEGDMA = Triethylene glycol dimethacrylate; UDMA = Urethane dimethacrylate.*

and enamel group, one-way ANOVA and Tukey HSD multiple comparisons test were used to determine statistical differences in μTBS between the experimental and control groups at a significance level of 0.05.

Fe-SEM Evaluation

The bonding mechanism to enamel was morphologically assessed by Field-emission Scanning Electron Microscopy (Fe-SEM) following a protocol described by Perdigão and others (1997). Two additional enamel surfaces were prepared for each group in the same way as for μTBS testing. One of the self-etch adhesives was then applied and covered with resin composite (Z100). After 24 hours water storage at 37°C, the specimens were stored in 6N HCl for eight hours to completely dissolve the enamel. The remaining composite blocks were then rinsed in NaOCL solution for 10 minutes and air dried for 24 hours. After mounting on aluminum stubs, the resin replica of the enamel etch-pattern was evaluated by Fe-SEM (Philips XL30, Eindhoven, The Netherlands).

TEM Evaluation

The bonding mechanism to dentin was morphologically assessed by Transmission Electron Microscopy (TEM). Two dentin surfaces were prepared for each group in the same way as for μTBS testing. Following adhesive treatment, the resin-bonded dentin specimens were cross-sectioned perpendicular to the resin-dentin interface to obtain 1-mm wide sticks using a slow speed diamond saw. Half the specimens were then demineralized and fixed simultaneously in a 10% formaldehyde-formic acid solution (Gooding and Stewart Fluid, Prosan, Gent, Belgium) for at least 36 hours. Further TEM specimen preparation of both the demineralized and non-demineralized sections was performed in accordance with common procedures used for ultra-structural TEM examination of biological tissues (Van Meerbeek & others, 1996). Then, 70-90-nm thick sections through the resin-dentin interface were cut using a diamond knife (Diatome, Bienne, Switzerland) in an ultramicrotome (Ultracut UCT, Leica, Vienna, Austria). For evaluation of collagen, TEM sections were positively stained with 5% uranyl acetate (UA) for 20 minutes and saturated lead citrate (LC) for three minutes prior to TEM examination (Philips CM10, Eindhoven, The Netherlands). Unstained sections were evaluated as well. On each section, the minimum and maximum hybrid layer thickness was determined (in μm). The same diamond-knife cut interfaces were gold sputtered and evaluated by Fe-SEM as well.

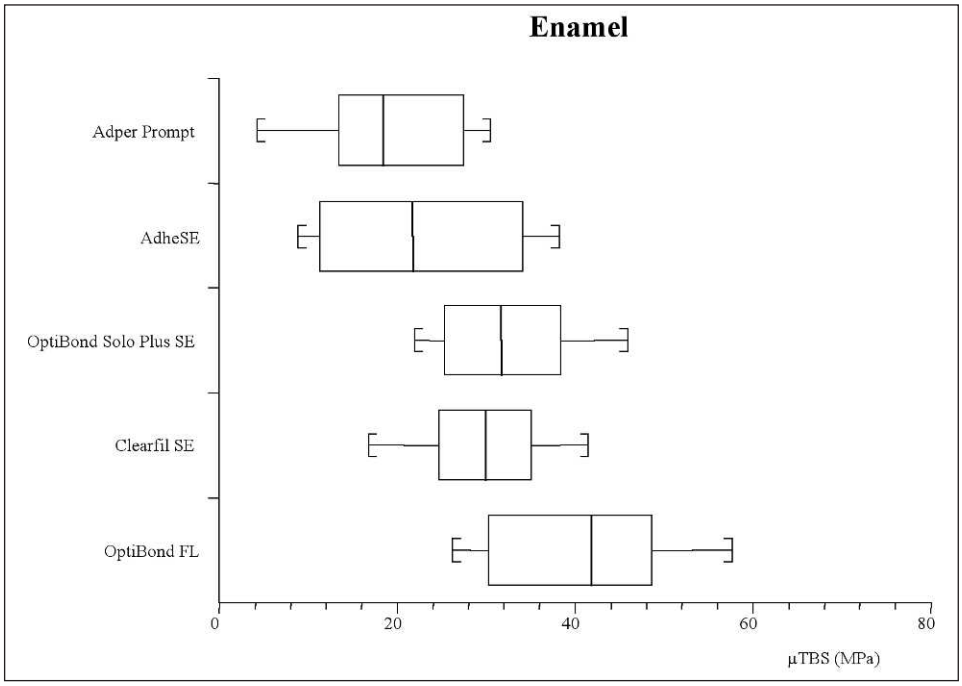

Figure 2. *Box plot of enamel μTBS results. The box represents the spreading of the data between the first and third quartile. The central vertical line represents the median. The whiskers extend to the minimum and maximum value.*

Table 2: *Results*

	pH primer	Enamel μTBS Mean	Enamel μTBS SD[1]	Enamel μTBS ptf/n[2]	Dentin μTBS Mean	Dentin μTBS SD[1]	Dentin μTBS ptf/n[2]	Hybrid Layer Thickness
Adper Prompt	0.41	18.6[c]	7.9	0/12	17.8[b]	5.2	2/12	1.7-4.5 μm[3]
AdheSE	1.40	23.2[b,c]	10.8	1/12	30.9[b]	13.1	0/12	2.1-2.4 μm
OptiBond Solo Plus SE	1.48	32.3[a,b]	7.5	0/14	52.2[a]	9.2	0/12	2.1-2.4 μm
Clearfil SE	1.92	29.8[b]	7.3	0/10	48.1[a]	11.5	0/12	0.7-1 μm[4]
OptiBond FL	1.78	41.1[a]	10.3	0/11	47.3[a]	13.1	0/12	4-5 μm[5]

Means with the same superscript are not statistically significant different (p<0.05, Tukey HSD multiple comparisons); [1]SD = Standard deviation; [2]ptf = Pre-testing failures, n = total number of specimens; [3]Hybrid layers thinner than 3.5 μm were only noticed when no thick bonding layer was present; [4]Inoue & others, 2000; [5]Van Meerbeek & others, 1996.

RESULTS

µTBS

The mean µTBS, pH and hybrid-layer thickness are summarized per experimental group in Table 2 and graphically presented in box-whisker plots in Figure 2 for enamel and in Figure 3 for dentin. When bonded to enamel, the µTBS of the control etch-and-rinse adhesive (OptiBond FL) was higher than that of any self-etch adhesive, (Figure 2, Table 2), though not significantly different from OptiBond Solo Plus Self-Etch. When bonded to dentin, no statistically significant differences were observed between the control adhesives and OptiBond Solo Plus Self-Etch. The lowest µTBS was obtained when Adper Prompt, the only one-step adhesive tested, was bonded to dentin and enamel; also, some pre-testing failures were recorded for this adhesive (Table 2). The failure patterns are summarized in Table 3. Most failures were "mixed" including "adhesive" failure between tooth and resin and "cohesive" failure of resin or tooth. Some specimens also failed entirely "adhesively" or "cohesively" within the resin part of the specimen. Only few specimens failed cohesively within the tooth surface (Table 3), a pattern associated with higher bond strengths. No difference in failure pattern could be detected between the different adhesives tested, except that the "stronger" adhesives tended to fail more cohesively in dentin than the weaker ones.

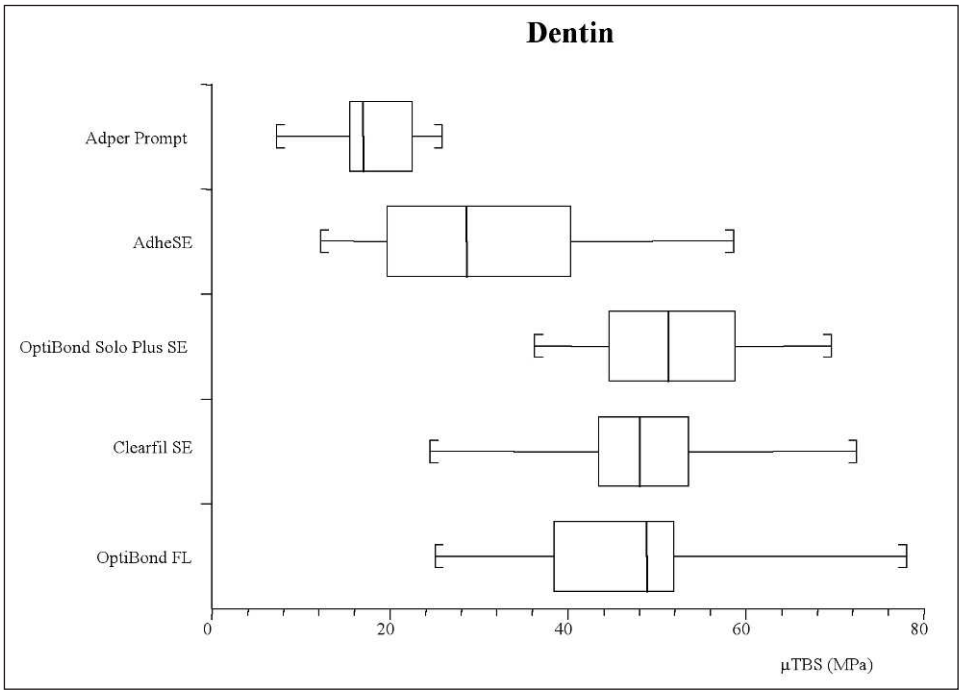

Figure 3. *Box plot of dentin µTBS results. The box represents the spreading of the data between the first and third quartile. The central vertical line represents the median. The whiskers extend to the minimum and maximum value.*

Interfacial Morphology

The enamel etching pattern, for the most acidic (Adper Prompt, pH=0.41) and least acidic self-etch adhesives (OptiBond Solo Plus Self-Etch, pH=1.48), was non-uniform and seemed dependent on the local smear layer features (Figure 4). This non-uniform pattern is probably due to the thick and irregular smear layer prepared in this study. All experimental groups were, however, able to demineralize inter-prismatic enamel and form acid-resistant resin tags into the created porosities (Figure 4). The size of the resin tags seemed relatively independent of the primer pH, although more detailed TEM observation can probably better discriminate between the different etching patterns (Pashley & Tay, 2001).

When bonded to dentin, all experimental self-etch adhesives were able to create distinct resin tags (Figures 5 through 7). The thickness of the hybrid layer

Table 3: *Failure Patterns of µTBS Specimens as Analyzed Through Stereo-Microscopy*

Enamel	Cohesive in Enamel/Dentin	Adhesive Tooth/Resin	Mixed*	Cohesive in Resin	Total
Adper Prompt			9	3	12
AdheSE		3	7	1	11
OptiBond Solo Plus Self-Etch			10	4	14
Clearfil SE	1		6	3	10
OptiBond FL			10	1	11
Dentin					
Adper Prompt			4	6	10
AdheSE			9	3	12
OptiBond Solo Plus Self-Etch	3		4	5	12
Clearfil SE	6		5	1	12
OptiBond FL	5		5	2	12

**Involving cohesive failure in resin and adhesive failure between tooth and resin*

seemed to relate well with the pH of the primer (Table 2). Hydroxyapatite crystals could only be detected in the bottom part of the hybrid layer for AdheSE and OptiBond Solo Plus Self-Etch (Figures 5 and 6). Loose collagen fibrils extending into the bonding resin, known as a "shag-carpet" appearance (Van Meerbeek & others, 1996), could be detected for AdheSE as well as OptiBond Solo Plus Self-Etch (Figures 5 and 6). Tubule-wall hybridization was very prominent for Adper Prompt, but could also be detected to a lesser extent for AdheSE and OptiBond Solo Plus Self-Etch (Figures 5 through 7). When Adper Prompt was bonded to dentin, two different zones could be detected in the bonding resin: first, a uniform layer of 10 µm that had heavily reacted with the staining solution, morphologically resembled the resin that formed the resin tags (Figures 7a, b); second, an intermediary zone between bonding resin and resin composite. On some spots, only the intermediary zone could be detected (Figures 7c, d), associated with a thinner hybrid layer (±1.5 µm, Figure 7c). In the presence of a separate bonding layer on the other hand, the bonding layer was always at least 3.5 µm thick.

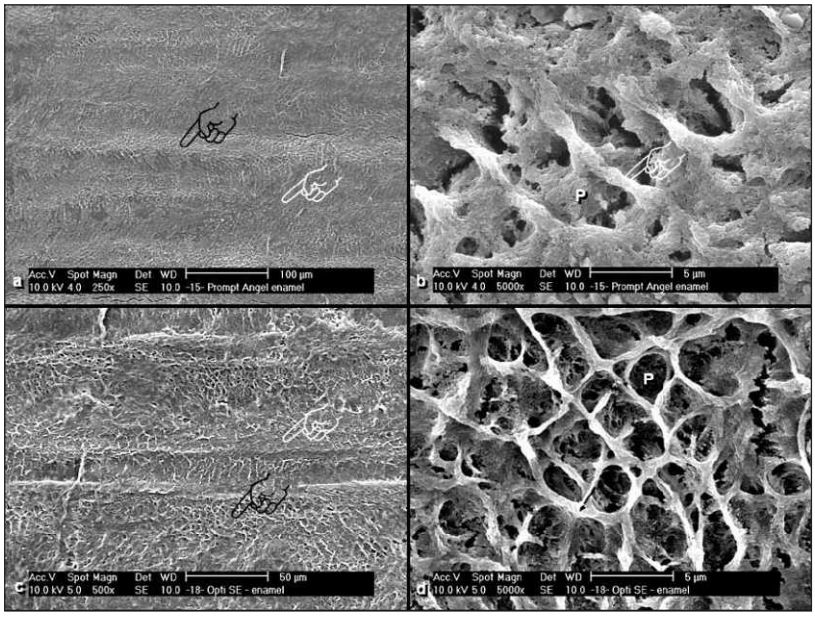

Figure 4. *Fe-SEM evaluation of the enamel etch-pattern replica after complete decalcification of enamel in HCL. (a) Adper Prompt bonded to enamel. The enamel etching pattern was non-uniform. Some zones show only very limited etching effects (white hand pointer), while on other spots the adhesive was clearly able to form acid-resistant tags (black hand pointer). (b) Higher magnification of acid-resistant tags created by Adper Prompt. Around the dissolved enamel prisms (P), resin macro-tags (white hand pointer) can be observed, while nearly no micro-tags were observable. (c) OptiBond Solo Plus Self-Etch bonded to enamel. The enamel etch-pattern was non-uniform. On some spots etching of enamel was very limited (white hand pointer), while on other spots the adhesive was clearly able to penetrate into interprismatic porosities (black hand-pointer). The interaction seems to be dependent on the local smear layer properties (horizontal scratches). (d) Higher magnification of (c) at the black pointer. Enamel was preferably dissolved around the prisms, creating resin macro-tags (arrow) that surround the enamel prisms (P).*

DISCUSSION

In this study, the bonding effectiveness and mechanism to enamel and dentin of three new self-etch adhesives was evaluated by µTBS and electron microscopy. Care was taken so that all adhesives were applied to tooth substrates prepared in a standard way and strictly according to manufacturer's instructions. The buccal/lingual enamel was flattened parallel to the tooth axis to standardize the orientation of enamel prisms. For dentin, only the central portion of the mid-coronal dentin surfaces was used in order to have all tubuli oriented perpendicular to the surface. In this way, the authors minimized any regional effects on the µTBS (Shono & others, 1997; Yoshiyama & others, 1998; Carvalho & others, 2000). Although bonding to such laboratory "model" substrates may clinically be of less relevance, it allowed the authors to determine the most optimal bonding effectiveness under ideal circumstances and enabled for comparison with previously conducted µTBS studies at Leuven BIOMAT (Inoue & others, 2001a; De Munck & others, 2002, 2003a; Van Meerbeek & others, 2003a).

All adhesives tested were applied to bur-cut smear layers prepared using the MicroSpecimen Former. This device was equipped with a high-speed dental contra-angle handpiece that held a regular-grit diamond bur, which, clinically, is commonly used for adhesive cavity preparation. This procedure resulted in a uniform, but rather thick, smear layer with a roughness comparable to a smear layer created with 60-grit SiC paper (Wahle & Wendt, 1993). A TEM study revealed that thick smear layers (up to 4.1 µm) did not hinder Clearfil SE to hybridize dentin (Tay & others, 2000a), although penetration of the primer might be compromised for weaker one-step self-etch adhesives (Inoue & others, 2001b). Despite the thick smear layer in this study, all experimental adhesives dissolved the smear layer and created a hybrid layer of at least 2-µm thick (Figures 5 through 7) within intact dentin (Tay & Pashley, 2001). Consequently, it can be assumed that the bonding effectiveness of these adhesives is relatively independent of the preparation method used (Tay & others, 2000b; Van Meerbeek & others, 2001).

Besides standardized bur-cut smear-layer formation, the basic capability of the MicroSpecimen Former is to generate specimens that (a) are not only uniform in dimension, but (b) are also rounded and (c) narrowed at the resin-tooth interface itself. An inverse relationship between enamel/dentin µTBS and cross-sectional area was found (Sano & others, 1994). This apparatus enabled the preparation of

specimens with a cylindrical bond area that closely varied around 1 mm^2, which corresponds to the bonding area size recommended by Shono and others (1997) and Phrukkanon, Burrow and Tyas (1998a). Using finite element analysis, the stress distribution at the interface was more uniformly distributed within cylindrical specimens as compared to rectangular μTBS specimens, though no significantly different μTBS between both shapes was found (Phrukkanon, Burrow & Tyas, 1998b). Another benefit is that the highest tensile stresses are imposed at the narrowest area, where the stress lines are concentrated most. As stated by Phrukkanon and others (1998b), other reasons to recommend preparation of cylindrical bond areas are the ease of specimen processing and testing, reduced risk of pre-testing failures, appropriate bonding area in compensation for dentin irregularities and potential internal interface defects.

A major drawback of μTBS-testing is that the rather aggressive specimen preparation may induce defects at the interface that may lead to lower bond strength values or even pre-testing failures. Thus, the more gentle (as compared to hand trimming) and standardized specimen preparation of the MicroSpecimen Former may be beneficial, especially taking into account that the highest stresses occur at the outer side of the beam (Phrukkanon & others, 1998b). Hence, any risk for manipulation errors can be considered minimal. Furthermore, the fact that all specimens were prepared in the same way and pre-testing failures only occurred in the weakest groups (Table 2) strongly suggests that the pre-testing failures should, rather, be attributed to less effective bonding and not solely to manipulation errors.

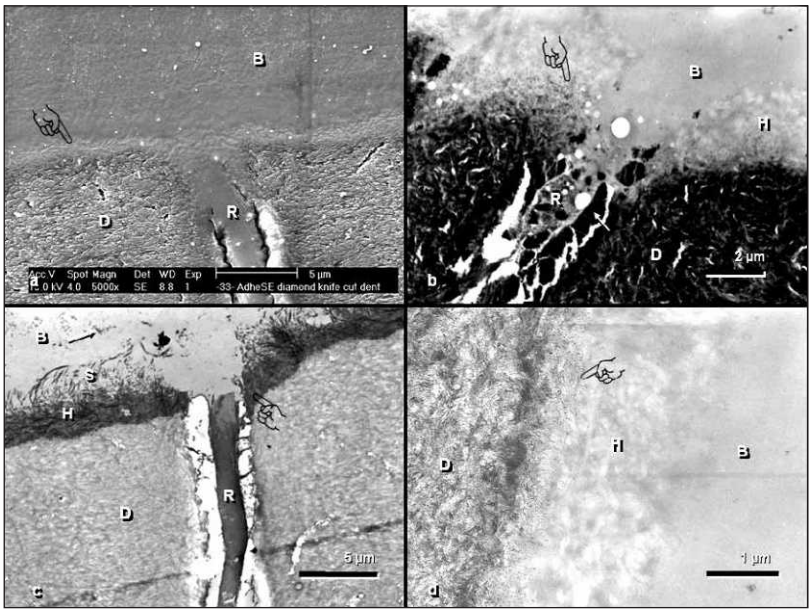

Figure 5. *Electron microscopic evaluation of the interface of AdheSE bonded to dentin. (a) Fe-SEM photomicrograph of a diamond-knife cut interface to dentin. On top of dentin (D), a hybrid layer (hand pointer) and a bonding layer (B, ±11 μm thick, not visible on this picture) can be observed. A resin tag (R) was formed in the dentinal tubulus. In the hybrid layer, collagen fibrils can be detected (hand pointer). (b) TEM photomicrograph (non-demineralized, unstained section) of the adhesive-dentin interface. A hybrid layer (H, thickness: ±2.1 μm) is formed with hydroxyapatite crystals clearly scattered within the bottom half (hand pointer). A resin tag (R) was formed, but some remnants of the smear plug were still present. (c) TEM photomicrograph (demineralized, stained section) of the interface. Hybrid layer collagen reacted heaviest with the staining solution, revealing a typical "shag-carpet" appearance (S), with collagen fibrils unraveled into their micro-fibrils and directed towards the adhesive resin. Note also some limited tubule wall hybridization (hand pointer). (d) TEM photomicrograph (non-demineralized, unstained section) of the adhesive resin-dentin interface. Hydroxyapatite crystals are clearly scattered within the bottom half of the hybrid layer (hand pointer). B = Bonding resin; D = Unaffected dentin; H = Hybrid layer (thickness: ±2.2 μm).*

The only one-step self-etch adhesive tested in this study was Adper Prompt, the successor to Prompt L-Pop, a typical "strong" self-etch adhesive (Van Meerbeek & others, 2000, 2001). The main difference with the older version is that the amount of non-acidic methacrylates has been increased to obtain a higher viscosity (technical information obtained from 3M ESPE). This may help to provide a sufficient amount of primer to the surface and obtain a thicker adhesive layer. In fact, TEM evaluation revealed the presence of a uniform layer, nearly free from phase separations, on top of the hybrid layer (Figure 7a), which was not present with its predecessor (Van Meerbeek & others, 2000). On some sites, however, only an intermediary adhesive/composite zone was present (Figure 7c). It is also noteworthy that if the primer was strongly blown out after application (less than with a correct application technique), some zones still exhibited a thick bonding layer (Figure 7d). Also worth noting is that if a distinct bonding layer was present, it was at least 10 μm thick. Due to oxygen inhibition, adhesive layers need to have a minimal thickness (Unterbrink & Liebenberg, 1999), consequently, it can be hypothesized that if the adhesive layer does not reach this minimal thickness, the adhesive may not cure adequately and may be pushed away by the resin composite. This may explain the marked difference between zones with a thick adhesive layer and zones without an adhesive layer. A thick adhesive layer, as mentioned before, is beneficial as a shock-absorber between tooth and composite (Kemp-Scholte & Davidson, 1990; Van Meerbeek & others, 1993; Uno & Finger, 1995; Choi, Condon & Ferracane, 2000) and is expected to be especially advantageous longer term (Van Meerbeek & others, 1998; Ausiello, Apicella & Davidson, 2002).

On some spots where the bonding layer was absent, the hybrid layer formed by Adper Prompt was also substantially thinner (Figure 7c). This local, insufficient adhesive penetration may be due to different causes: a)

Locally insufficient adhesive application; b) Insufficient penetration time; this is less probable, as some spots on the same surface exhibited much thicker hybrid layers; c) Penetration may have been hampered by an amorphous layer on top of the hybrid layer, formed by the polyalkenoic-acid co-polymer (a component not present in the former version of the adhesive). A similar phenomenon is known for the primers of Scotchbond Multipurpose and Scotchbond 1 that contain the same polyalkenoic-acid co-polymer (Van Meerbeek & others, 1996). This hypothetical phenomenon may be corroborated by the presence of a thin electron-dense line at the top of the hybrid layer that was only present in case of a thin hybrid layer (Figure 7c) but not when the adhesive was strongly blown out (Figure 7d); (d) Also, a lack of rubbing may have caused the reduced penetration, as this can freshen the adhesive solution at the interface and prevent local concentrations of polyalkenoic acid. Most likely, a combination of these factors caused the less optimal infiltration of the adhesive. Note, however, that the thickness of the hybrid layer is not directly related to its bonding effectiveness (Van Meerbeek & others, 2001).

The long-term durability of Adper Prompt bonded to dentin is questionable (Shirai & others, 2003). A hypothesis regarding this effect was advanced by Tay and others (2002a); the curing of a combination of acidic monomers, HEMA and water does not result in a uniform, hydrophobic resin layer, but areas of incomplete polymerization and hydrogel formation are present. These areas may then permit water fluxes within the hybrid layer and, subsequently, accelerate water sorption of the adhesive interface. Combined with degradation and extraction of resin components (Tay & others, 2002b), this process is known to be detrimental to the bond integrity (Hashimoto & others, 2000, 2002; De Munck & others, 2003a).

AdheSE is a two-step self-etch adhesive. Its self-etch capacity is based on phosphonic acid acrylates (opposite to Adper Prompt, which is based on methacrylated phosphoric esters). This particular adhesive is less acidic than Adper Prompt and subsequently results in a thinner hybrid layer (Table 2). Some residual hydroxyapatite crystals were detected at the bottom of the hybrid layer (Figure 5b), indicating that demineralization was not complete at that location. Consequently, this self-etch approach resulted in a smoother transition to the deeper intact dentin. However, the system is more aggressive than the control self-etch adhesive Clearfil SE (pH=1.92). This is reflected in the formation of a thicker hybrid layer and more pronounced resin tags instead of the hybridized smear plugs typically observed with Clearfil SE. Previously, self-etch adhesives were divided into "mild" self etch adhesives (pH ± 2.0, sub-micron hybrid layer formation) and "strong" self-etch adhesives (pH<1.0, interfacial ultra-morphology similar to etch-and-rinse adhesives) (Van Meerbeek & others, 2001, 2003b). As the pH and the interfacial ultra-morphology of AdheSE is intermediary between the structural characteristics produced by the mild and strong self-etch adhesives, it cannot be incorporated into one of two groups but should be regarded as an "intermediary strong" self-etch adhesive. The major morphologic characteristics of the different types of self-etch-adhesives are summarized in Figure 8. The application instructions provided with this adhesive included extensive rubbing of the primer. This rubbing motion loosened the collagen fibrils and must have promoted resin infiltration and adaptation of the resin to the collagen (Figure 5c). This rubbing motion morphologically resulted in a shag-carpet appearance (Van Meerbeek & others, 2000).

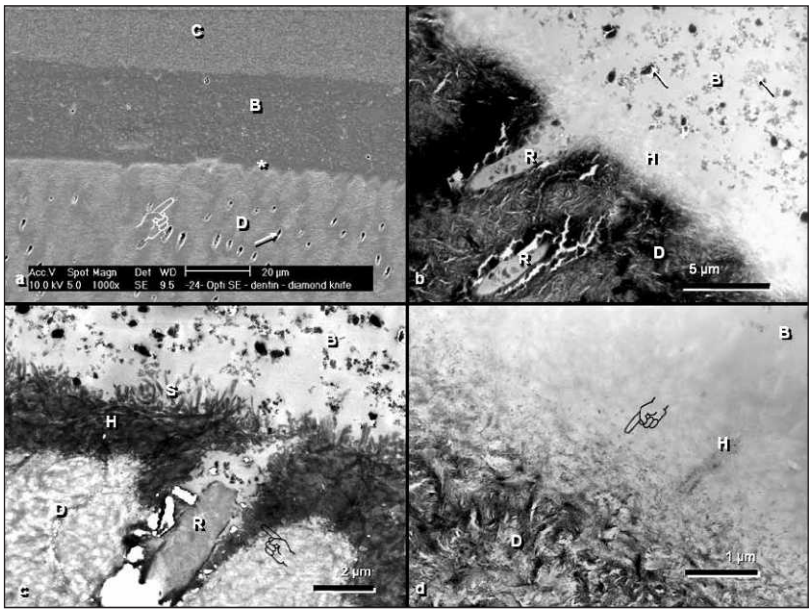

Figure 6. *Electron microscopic evaluation of the interface of OptiBond Solo Plus Self-Etch bonded to dentin. (a) Fe-SEM photomicrograph of a diamond-knife cut adhesive-dentin interface. On top of dentin (D) a hybrid layer (H) is formed (asterisk). In-between the hybrid layer and composite (C), a 27 µm thick bonding (B) layer can be observed. Near the interface, the dentinal tubuli seem to be obturated by resin tags (hand pointer), while further away the tubuli are empty (arrow). Consequently, resin tags only extend up to about 10 µm into the dentin surface. (b) TEM photomicrograph (non-demineralized, unstained section) of the adhesive-dentin interface. (c) TEM photomicrograph (demineralized, stained section) of the adhesive-dentin interface. A typical "shag-carpet" appearance (S) with collagen fibrils unraveled into their microfibrils and directed towards the adhesive resin can be observed. Note also the tubule wall hybridization (hand pointer). D = Unaffected dentin; H = Hybrid layer (thickness: 2.4 µm); R = resin tag. B = bonding resin with larger glass particles and nanofillers. (d) TEM photomicrograph (non-demineralized, unstained section) of the adhesive-dentin interface. Hydroxyapatite crystals are clearly scattered within the bottom half of the hybrid layer (hand pointer). B = Bonding resin, D = Unaffected dentin; H = Hybrid layer (thickness: ±2.2 µm); R = resin tag formed by resin and filler particles.*

The ultra-morphology of OptiBond Solo Plus Self-Etch (Figure 6) is very similar to that of AdheSE. As also the acidity of both primers is comparable (Table 2), this adhesive should also be regarded as an intermediary strong self-etch adhesive. Besides the different chemical composition (Table 1), the main difference between both adhesives is the presence of filler particles in the bonding resin. These may help stabilize the interface and obtain an adequate thickness of the bonding layer (Figures 5 and 6) in light of reduction of interfacial stresses (Van Meerbeek & others, 1993, 1998; Perdigaõ & others, 1996; Ausiello & others, 2002). In contrast to AdheSE, the bonding resin of OptiBond Solo Plus Self-Etch is not solvent-free. This may induce some technique-sensitivity, as the solvent has to be properly removed by air drying.

AdheSE and OptiBond Solo Plus Self-Etch do not remove all hydroxyapatite at the bottom of the hybrid layer (Figures 5 and 6). Some adhesive monomers are known to interact chemically with hydroxyapatite, which may be beneficial to interfacial sealing (Van Meerbeek & others, 2001; Yoshida & others, 2004). For the monomers used in this study, however, no data are yet available. Chemical bonding in this part of the hybrid layer may, however, be very beneficial, especially considering that this area is more sensitive to nanoleakage (Sano & others, 1995).

When comparing the enamel bonding effectiveness of self-etch adhesives with standard etch-and-rinse adhesives, the etch-and-rinse approach remains the "gold standard." This is especially true in clinical dentistry, because etching is relatively independent from smear layer properties and preparation methods (Van Meerbeek & others, 2003a). The etching pattern created by self-etch adhesives, on the other hand, is less uniform, dependent on the acidity of the primer (Pashley & Tay, 2001) and smear layer properties (Figure 4). Etching aggressiveness is, however, not entirely correlated with bonding effectiveness, as some mild and intermediary strong self-etch adhesives do approach the etch-and-rinse standard, in contrast to the strong self-etch adhesive tested, which clearly underscored all other adhesives (Table 2). This must probably be attributed to properties of the adhesive resin itself (Pashley & Tay, 2001).

When bonded to dentin, it is evident that two-step self-etch adhesives are able to compete with etch-and-rinse adhesives, not only in terms of early bonding effectiveness (Inoue & others, 2000, 2001a; Van Meerbeek & others, 2003a), but also in terms of durability (Miyazaki & others, 1998; Sano & others, 1999; Nikaido & others, 2002; Shirai & others, 2003). Taking into account the lower technique-sensitivity (Van Meerbeek & others, 2001), the faster application procedure and the lower risk to nanoleakage (Sano & others, 1995), the two-step self-etch approach may become the future standard of adhesion.

Up to now, no clinical data are available for the experimental adhesives tested in this study. The predecessor of Adper Prompt, which is very similar in composition, performed, however, variably in clinical Class V studies (Brackett, Covey & St Germain, 2002; Van Dijken, 2003), whereas, the control adhesives (OptiBond FL and Clearfil SE) both performed well in clinical trials (Boghosian 1996; De Munck & others, 2003b; Peumans & others, 2003; Van Meerbeek & others, 2003b). For the

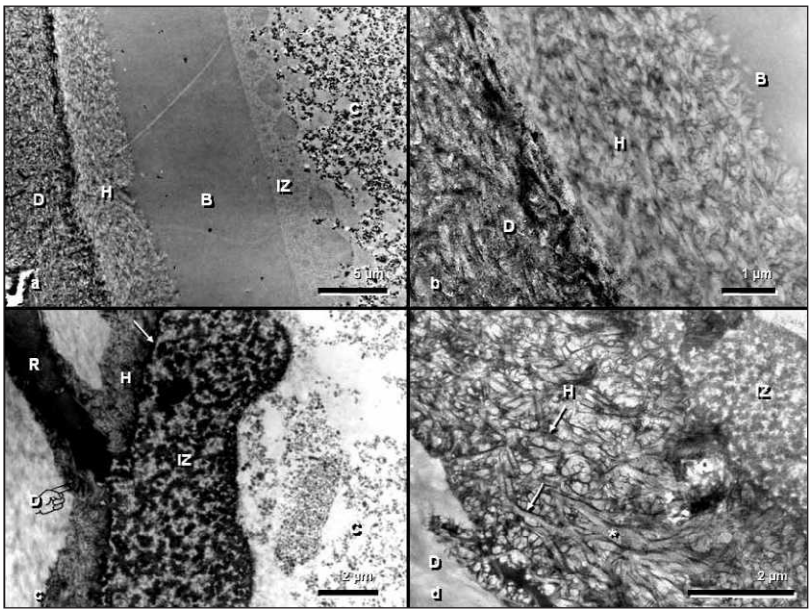

Figure 7. *Electron microscopic evaluation of the interface of the one-step self-etch adhesive Adper Prompt to dentin. (a) TEM photomicrograph (non-demineralized, unstained section) of the adhesive-dentin interface. The hybrid layer is about 4 µm-thick, the bonding resin about 9 µm. (b) Higher magnification of (a). The relatively high electron density of resin resulted in a kind of negative staining of the electron lucent collagen fibrils within the hybrid layer. Notice the abrupt transition of hybrid layer to dentin and the absence of hydroxyapatite crystals, both characteristics of etch-and-rinse and "strong" self-etch adhesives. (c) TEM photomicrograph (demineralized, stained section) of the adhesive-dentin interface. The stain was strongly picked up by the phosphate-based resin. In contrast to (a), the hybrid layer is much thinner (±1.5 µm) and no bonding layer is present. On top of the hybrid layer, a thin electron dense line can be observed (arrow). Note also the extensive tubule-wall hybridization (hand pointer). (d) TEM photomicrograph (demineralized, stained section) of the adhesive-dentin interface. Although not recommended by the manufacturer, the adhesive had been strongly blown out after application. Consequently, the bonding layer was not present any more, although in some sections of the same specimen, a ±10 µm thick bonding layer, was still observed. Hybridization, however, was not hampered, as the adhesive was able to demineralize dentin for up to about 4.5 µm. Also, no electron dense line on top of the hybrid layer (like in c) can be detected. Due to the high stainability of the resin, the pathway of resin infiltration within the exposed collagen scaffold can be clearly observed. Within an interfibrillar space, the collagen outer surface appears to be coated by a more dense line (arrows), which results in a kind of negative staining (resin instead of collagen was stained) of the hybrid layer. Cross-banding, typical of type-I collagen can be observed (asterisk). B = Bonding resin; C = Composite; D = Unaffected dentin; H = Hybrid layer; IZ = intermediary zone between adhesive and composite; R = Resin tag.*

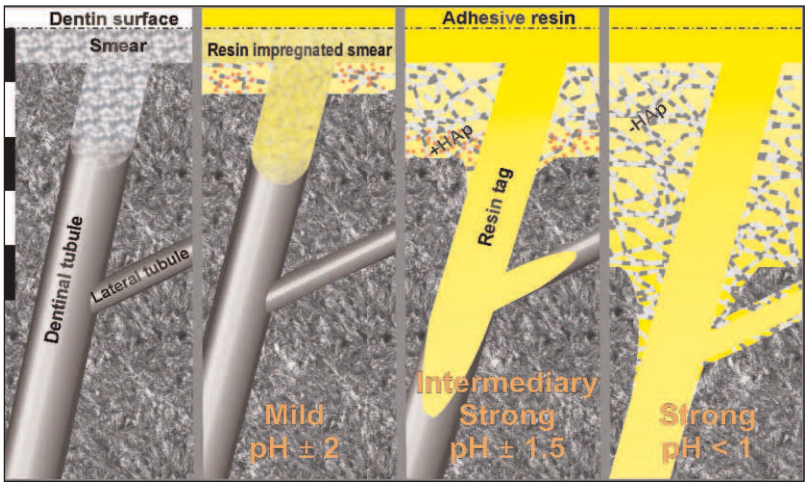

Figure 8. *Schematic overview of the interaction of different self-etch adhesives with dentin (bar at the left represents approximately 5 µm). On the left, unaffected dentin is represented with a typical smear layer and smear plug occluding a dentinal tubule. On the right, interaction of the three classes of "self-etch" adhesives with this smear layer covered dentin is represented. The "mild" self-etch adhesives do not completely remove the smear layer, but do form a submicron hybrid layer. Throughout the whole depth of the hybrid layer, residual hydroxyapatite remains attached to the exposed collagen fibrils and remains available for chemical interaction. The "intermediary strong" self-etch adhesives dissolve the smear layer and plug, forming short (±10 µm) resin tags. Some residual hydroxyapatite can be found only in the bottom third of the hybrid layer. Also, limited lateral tubule wall hybridization can be observed. The "strong" self-etch adhesives present with a morphology very alike that produced by etch-and-rinse adhesives, with a 3-5 µm thick hybrid layer, extensive resin tags, tubule-wall and lateral tubule-wall hybridization.*

intermediary strong self-etch adhesives, no data are available yet. Consequently, no predictions can be made regarding the clinical effectiveness of AdheSE and OptiBond Solo Plus Self-Etch.

CONCLUSIONS

Based upon their interfacial morphology and pH, self-etch adhesives can be subdivided into three classes: "mild," "intermediary strong" and "strong" self-etch adhesives. The early bonding effectiveness of self-etch adhesives varies substantially, depending on the type of adhesive. In particular, the strong one-step self-etch adhesive tested seemed to be less effective. The bonding effectiveness of some two-step self-etch adhesives, on the other hand, was comparable to that of the control etch-and-rinse adhesive. More elaborate studies on the long-term bonding effectiveness and clinical trials remain necessary.

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The Effect of Dentin Adhesive and Cure Mode on Film Thickness and Microtensile Bond Strength to Dentin in Indirect Restorations

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Clinical Relevance

The effect of light curing prior to resin cementation on microtensile bond strength was dependent on the specific dentinal adhesive. Light curing the adhesive prior to resin cementation increased film thickness for all the dentin adhesives tested but this effect may be tolerable if a careful technique is utilized. Any benefits of pre-curing the adhesive to the microtensile bond strength must be weighed against the risk of incomplete restoration seating.

SUMMARY

This study evaluated the influence of dentin adhesive application technique (pre-curing vs non pre-curing) on microtensile bond strength (μTBS) to dentin and adhesive layer thickness in indirect resin restorations. Seven proprietary dentin adhesives were tested, including one-step and multi-step products. Experimental groups included adhesive pre-cure (PC) with a halogen light source and no pre-cure (NPC) prior to resin cement insertion.

Thirty caries-free molars received an MO inlay preparation. Inlays made with Tetric Ceram resin composite were cemented using a dual-cured resin luting agent. Prior to inlay cementation, each tooth was treated with one dentin bonding agent, using pre-cure (PC) or no pre-cure (NPC). After storage in distilled water at 37°C for 24 hours, the teeth were sectioned along their long axis to produce serial sticks for microtensile bond strength testing at 0.5 mm/minute. The results were subjected to statistical analysis by one-way and two-way analysis of variance (ANOVA) and Tukey's multiple comparison test ($p \leq 0.05$). For the film thickness evaluation, 10 additional teeth were restored and sectioned mesiodistally. The thickness of the adhesive layer was evaluated by SEM at 1000x magnification at the pre-selected locations.

The μTBS varied from 11.7 ± 4.5 MPa to 43.4 ± 9.8 MPa. The effect of pre-curing the adhesive was material specific. No adhesive layer was visualized for the adhesives used without the pre-curing step. The thickness of the adhesive layer for the pre-cured groups varied according to the different areas analyzed.

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INTRODUCTION

The use of resin luting cements has increased considerably in recent years. Resin cements provide increased retention (Browning & others, 2002), improved esthetics and greater resistance to dissolution over conventional cements (White & others, 1992). They are essential for the cementation of esthetic inlays, onlays, veneers and the majority of all-ceramic crowns in providing strength to the bonded assembly. Resin luting cements are, however, technique sensitive and their use demands a careful implementation of a series of steps, including the application of enamel and dentin adhesives. The clinical success and longevity of the bonded esthetic restoration depends on both the adhesive and the resin luting cement forming an optimal attachment to tooth structure.

Adhesive procedures currently provide a high degree of clinical success (Van Meerbeek & others, 1998). The interlocking mechanism, or hybridization between dentin adhesive and tooth substrate is a result of resin infiltration and interpenetration into the exposed collagen framework of acid-demineralized superficial dentin (Nakabayashi, Kojima & Masuhara, 1982). Adhesive systems that use primer and adhesive either separately or in combination are available. More recently, self-etching systems have been introduced with the objective of simultaneous demineralization and resin infiltration (Van Meerbeek & others, 1998).

Differences in material composition and adhesive application technique can affect many properties such as film thickness, bond strength, radiopacity, adaptation and marginal seal (Opdam, Roeters & Verdonschot, 1997; Frankenberger & others, 1999; Hahn & others, 2000). Resin adhesives and resin cements are available in self-cure, light-cure and dual-cure formulations. The dual-cured resins provide rapid photo-irradiation capability as well as a delayed chemical redox reaction between the organic peroxide and tertiary amine to ensure complete polymerization when light penetration is inadequate (Rueggeberg & Caughman, 1993). The degree of polymerization plays a pivotal role in determining the ultimate biological, physical and mechanical properties of the material (Ferracane & Greener, 1984).

It is important to establish a strong bond of the restoration to dentin (McCabe & Rusby, 1994; Opdam & others, 1997; Walshaw & McComb, 1996; Van Meerbeek & others, 1998; Frankenberger & others, 1999; Hahn & others, 2000). A significant increase in bond strength has been demonstrated when the adhesive was cured prior to insertion of the resin composite (McCabe & Rusby, 1994) in both direct and indirect restorations (Frankenberger & others, 1999). However, if the thickness of the polymerized adhesive layer is high, either generally or in localized areas, this adhesive pre-curing step could prevent complete seating of the indirect restoration.

This study evaluated the effect of different application techniques (pre-curing vs no pre-curing) of several dentin adhesive systems on the film thickness and microtensile bond strength to dentin in indirect inlay restorations. The null hypothesis was that light curing the dentin adhesives prior to insertion of the inlays would have no effect on microtensile bond strength and adhesive layer thickness.

METHODS AND MATERIALS

Forty non-carious human third molars extracted within the past three months were used in this study. They were rinsed thoroughly in running water and the soft tissue was removed prior to storage in 4°C distilled water containing thymol crystals. The teeth were randomly divided into 10 groups of four teeth and allocated to one of the selected bonding systems according to Table 1. All bonding systems with their respective components are listed in Table 2.

Tooth Cavity Preparation and Restorative Procedures

Three teeth from each group were used for microtensile bond strength tests. An MO cavity preparation was prepared in each tooth without an axial wall to provide a greater area for the μTBS test specimens. The cavities were prepared using a conical diamond bur (Brasseler—845KR) in a high-speed handpiece with air-water spray. The bur was replaced after every four preparations. The bucco-lingual width of the preparation was maximized for each tooth in order to increase the area of the bonded interface for the microtensile test. The depth of the cavity preparations was standardized by demarcating the burs at 3 mm ± 0.1 from their tips. The pulpal floor was prepared flat and the walls were tapered approximately 15 degrees. The internal line angles were rounded and enamel margins were prepared with a butt-joint without bevels.

Composite inlays were fabricated for each prepared tooth by placing the resin composite, Tetric Ceram (Ivoclar-Vivadent, Schaan, Liechtenstein), directly into each preparation in five increments. No separating medium, except water, was used. Each increment was photocured for 40 seconds using a visible light-curing unit (Optilux 501, Kerr Corporation, intensity 760 mW/cm^2). After inlay removal, additional photopolymerization was performed for 40 seconds on the internal surface of the inlay. The internal surfaces of the inlays were then sandblasted with 50 mm Al_2O_3 powder at 2 bar pressure and treated with 35% phosphoric acid for one minute to remove surface contaminants. The procedure of fabricating inlays directly into each preparation was simple and reliable. The same procedure was previous described by Dietschi and Moor (1999).

Prior to inlay cementation, each tooth was treated using one dentin bonding agent in accordance with the manufacturer's directions (Table 1). The test materials were selected in order to compare various recommended self-cure light-cure and dual-cure bonding agents, appropriate for indirect restorations, each from of a selection of major manufacturers. Three adhesive systems (Optibond Solo Plus, Excite DSC and IntegraBond) provided a direct comparison of pre-curing (PC) and no pre-curing (NPC), since these adhesives are available as a photo and/or a dual-cured system. The PC step consisted of a 20-second light exposure using a halogen light (Optilux 501). The product Syntac Classic was added to the study, because it was used as the sole bonding agent in a clinical study in progress of indirect restorations by one of the authors (MJS). The purpose was to provide for possible future comparison of *in-vitro* and *in-vivo* performance.

The base and catalyst pastes of the dual-cured resin luting agent (Variolink II, Ivoclar-Vivadent) were mixed at 1:1 volume ratio according to manufacturer's directions and applied to the internal surface of the inlay. A 5-kg mass was applied to the occlusal surface of the inlay during cementation. The excess luting composite was removed with a probe. The resin luting cement was photopolymerized for 40 seconds from each of the buccal, lingual, mesial and occlusal aspects, for a total photopolymerization time of 160 seconds. The restored specimens were subsequently stored in distilled water at 37°C for 24 hours prior to further manipulation.

Preparation of Specimens for Microtensile Testing and Test Procedure

After 24 hours, each tooth was attached to an acrylic block with impression compound and mounted for sectioning in the Accutom low speed diamond saw (Micro Metallurgical Limited, Richmond Hill, ON) under continuous water irrigation to produce serial sticks containing the bonded interface of the pulpal floor with an approximate cross sectional area of 0.5 mm^2. The first series of cuts through the bonded interface was in a mesio-distal direction made parallel to the longitudinal surface of the tooth, such that the composite material was sectioned first, then the root portion. The second series of cuts was made perpendicular to the previous cuts in a bucco-lingual direction. The last cut was oriented transversally to the longitudinal surface in order to liberate the sticks.

Table 1: *Dentin Treatments and Materials Used for Each Group*

Group	Material	Etching	Priming	Bonding
1	Excite DSC **NPC**#	Etch 15 seconds□, rinse 20 seconds*		Apply with brush for 10 seconds, air thin for 3 seconds; do not light cure
2	Excite DSC **PC**#	Etch 15 seconds□, rinse 20 seconds*		Apply with brush for 10 seconds, air thin for 3 seconds; light cure 20 seconds
3	IntegraBond **NPC**#	Etch 15 seconds□, rinse 20 seconds*	Mix Adhesive and Auto-Cure Activator	Apply and leave undisturbed for 15 seconds; air thin 15 seconds
4	IntegraBond **PC**#	Etch 15 seconds□, rinse 20 seconds*	Mix Adhesive and Auto-Cure Activator	Apply and leave undisturbed for 15 seconds; air thin 15 seconds; light-cure for 20 seconds
5	Optibond Solo Plus **NPC**#	Etch 15 seconds□, rinse 20 seconds*	Mix adhesive with activator for 3 seconds	Apply with brush for 15 seconds, air thin for 3 seconds; do not light cure
6	Optibond Solo Plus **PC**#	Etch 15 seconds□, rinse 20 seconds* for 3 seconds	Mix adhesive with activator	Apply with brush for 15 seconds, air thin for 3 seconds; light cure for 20 seconds
7	Optibond Solo Plus Self-etch **PC**#		Apply primer etcher for 15 seconds; air thin 3 seconds	Apply adhesive for 15 seconds; air thin 3 seconds; repeat; light-cure for 20 seconds
8	Single Bond **PC**#	Etch 15 seconds□, rinse 20 seconds*		Apply 2 consecutive coats, air thin for 3 seconds; light cure for 10 seconds
9	Scotchbond MP Plus **NPC**#	Etch 15 seconds□, rinse 20 seconds*	Activator Primer Catalyst	Apply, air thin 5 seconds, Apply, air thin 5 seconds, Apply; do not light cure
10	Syntac Classic **NPC**#	Etch 15 seconds□, rinse 20 seconds*	Primer Adhesive Bond	Apply for 15 seconds; dry 5 seconds Apply for 10 seconds; dry 5 seconds Apply; do not light cure

□*35% phosphoric acid Scotchbond etching gel (3M Dental).*

**After water rinsing, excessive surface water was removed with a small piece of absorbent paper, leaving a visible moist surface.*

#*PC = Adhesive was pre-cured prior to the inlay insertion; NPC = Adhesive was not pre-cured adhesives prior to the inlay insertion.*

The specimens were glued to the microtensile device with cyanoacrylate (Zapit, Dental Ventures of America, Inc, Corona, CA, USA) and subjected to tensile forces at a crosshead speed of 0.5 mm/minute, with 50 N load cell, using the Instron universal testing machine (Model 4301, Canton, MA, USA). After testing, the cross-sectional area of each specimen was measured with a digital caliper (Mitutoyo Corp, Kawasaki, Japan). The bond strength (MPa) was calculated by dividing the maximum load (N) by adhesion area (mm^2). Ten sticks were evaluated per tooth, resulting in a total of 30 specimens per group. One-way analysis of variance ANOVA Tukey's multiple comparison test were used to statistically analyze all the microtensile (μTBS) data ($p \leq 0.05$). Two-way ANOVA was used on the groups that used both cure modes ($p \leq 0.05$) to analyze for the significance of each factor and any interaction.

SEM (Scanning Electron Microscope)

One tooth from each group was used for film thickness evaluation. The film thickness of the adhesive systems was measured using teeth restored with Class II composite inlays containing an axial wall. The restored tooth was sectioned mesio-distally producing two central slices, 1.5-mm thick, using a slow-rotating saw. Both slices were polished sequentially using 600, 1000 and 4000 grit silicon carbide paper, then etched with 5% maleic acid for 20 seconds, rinsed thoroughly with water and stored overnight in 100% ethanol. Subsequently, each specimen was critical point dried,

Table 2: *Dentin Adhesives Systems, components, batch number and manufacturers [HEMA= hydroxyethyl methacrylate; Bis-GMA= bisphenyl glycidyl methacrylate; TEGMA= triethylene glycol-dimethacrylate; GDM= glycerol dimethacrylate; GPDM= glycerol phosphate dimethacrylate; HFGA-GDM= hexafluoroglutaric anhydride – glycerodimethacrylate adduct; MEHQ= 4- methoxyphenol; ODMAB= 2- (Ethylhexyl) -4-(dimethylamino) benzoate; CQ= camphorquinone; BHT= 2,6-Di (tert-butil)-4-hydroxytoluene; TS530= Fumed Silicon Dioxide; OX50= Fumed Silicon Dioxide; A1174= gamma-Methacryloxypropyltrimethoxsilane; SP345= Barium Aluminoborosilicate; Na2SiF6= Disodium hexafluorosilicate]*

Dentin Adhesives	Components	Batch #	Manufacturer
Excite DSC (Photo and dual-cured)	Phosphoric acid acrylate, Hyhroxyethyl methacrylate, ethanol, highly dispersed silicon dioxide, catalysts and stabilizers	D61321	Ivoclar-Vivadent AG, FL-9494 Schaan/Liechtenstein
	Microbrush coated with initiators		
Integra Bond (Photo and dual-cured)	*Primer/Adhesive:* TEGMA, HEMA, Aliphatic polyester urethane acrylate, adhesive acelerator, photoiniciators, acetone	07301091	Premier Dental Products, Co King of Prussia, Pa, Israel
	Activator: acetone, ethylalcohol, benzoil peroxide	07517101	
OptiBond Solo Plus (Photo and dual-cured)	*Primer/Adhesive:* Bis-GMA, HEMA, GDM, GPDM, CQ, ODMAB, BHT, TS530, OX50, A174, SP345, Na2SiF6, ethanol	110173	Kerr Corporation, Orange, CA, USA
	Activator: Ethanol, Bis-GMA, HEMA, Benzenesulfinic acid sodium salt	112196	
OptiBond Solo Plus Self-etch (Photo-cured)	HFGA-GDM, GPDM, MEHQ, ODMAB, CQ, ethanol, water	202630	Kerr Corporation Orange, CA, USA
Scotchbond Multipurpose Plus (Self-cured)	*Activator:* Ethyl alcohol, Benzene sulfinic acid, sodium salt	9KD	3M Dental Products, St Paul, MN, USA
	Primer: water, 2-Hydroxyethyl methacrylate, Polycarboxylic acid copolymer	9XT	
	Catalyst: 2-Hydroxyethyl methacrylate, Bisphenol A Diglycidyl Ether, Benzoyl Peroxide	9BU	
Single Bond (Photo-cured)	Bis-GMA, HEMA, polyalkenoic acid copolymer, ethanol, water, photoinitiator	9CR	3M Dental Products, St Paul, MN, USA
Syntac Classic (Self-cured)	*Syntac Primer*: Polyethylene glycol dimethacrylate, maleic acid, and acetone in an aqueous solution	E52572	Ivoclar-Vivadent AG, FL-9494 Schaan/Liechtenstein
	Syntac Adhesive: Polyethylene glycol dimethacrylate, and glutaraldehyde in an aqueous solution	E52926	
	Heliobond: Bis-GMA, TEGMA	E52252	

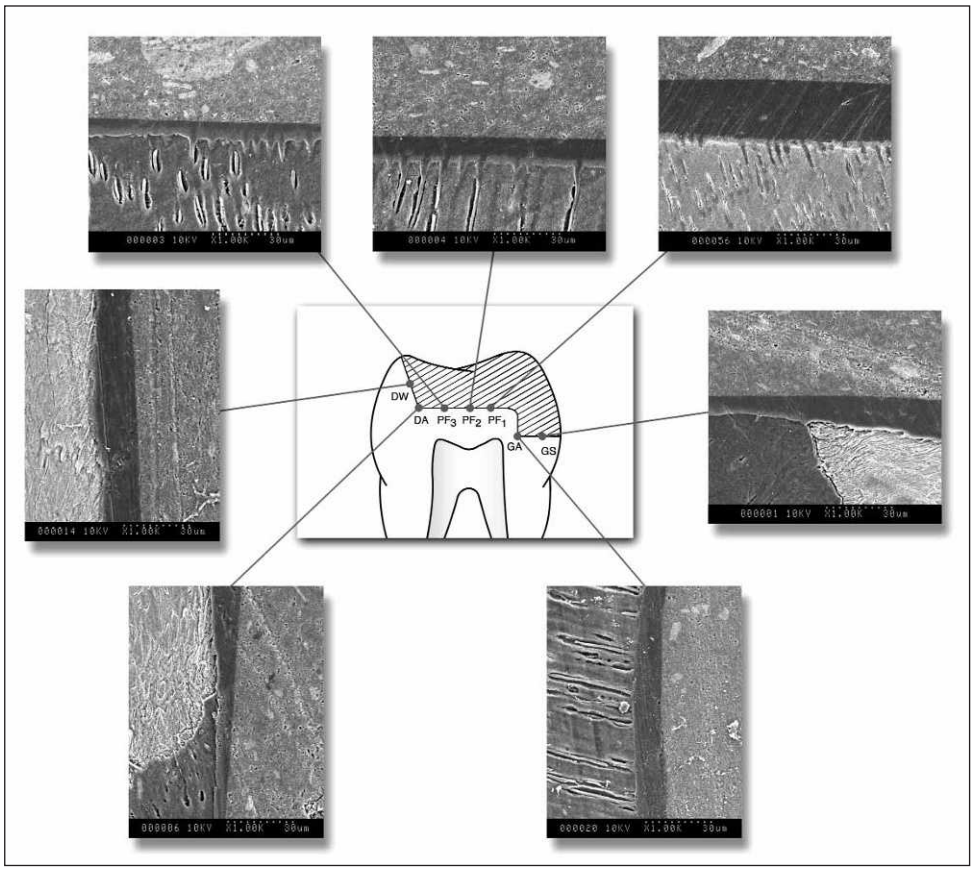

Figure 1. *Photomicrographs of the inner restoration tooth-interface for the film thickness evaluation at the pre-cured adhesive (Single Bond) at different locations.*

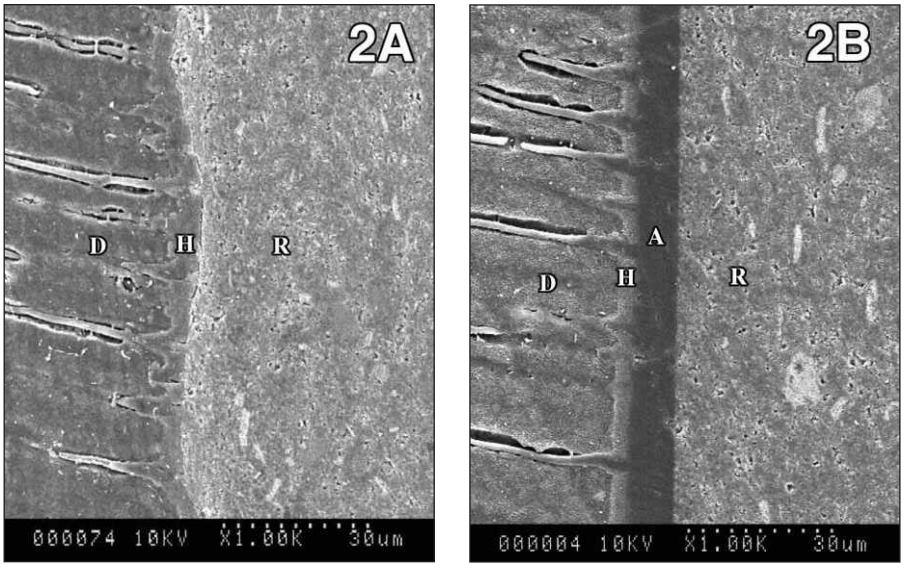

Figure 2. *Photomicrographs of a non-pre-cured adhesive version (A) and a pre-cured one (B). Note the presence (B) and absence (A) of an adhesive layer, depending on the precuring mode (OptiBond Solo Plus). R= resin composite; H = hybrid layer; D = dentin; A = adhesive.*

mounted on a 12-mm aluminum stub using a cyanoacrylate adhesive and sputter-coated with platinum. The adhesive film thickness was measured for each slice (two slices/tooth) under a scanning electron microscope (SEM) (Hitachi S-2500, Hitachi Ltd, Mito City, Japan) at 1000x magnification at the following locations: gingival seat (GS), gingivo-axial line angle (GA), pulpal floor 1 (PF_1), pulpal floor 2 (PF_2), pulpal floor 3 (PF_3), disto-pulpal angle (DA) and distal wall (DW) (Figure 1).

RESULTS

Microtensile Bond Strength (μTBS)

The μTBS data are summarized in Figure 3. The μTBS varied from 11.7 ± 4.5 MPa to 43.4 ± 9.8 MPa. Single Bond (PC) had the highest mean bond strength result ($p>0.05$). The three-step systems, Syntac Classic and Scotchbond Multipurpose Plus, along with the one-bottle adhesive Integra Bond showed the lowest μTBS values. There was a significant interaction effect between the factors of dentin bonding agent and cure mode. The pre-curing step significantly improved the μTBS of Excite DSC. The pre-curing step had no significant effect on the μTBS results for the adhesives OptiBond Solo Plus and Integra Bond. The self-etching bonding system OptiBond Solo Plus SE showed a good performance with no significant difference from OptiBond Solo Plus (PC and NPC) and Excite DSC (PC and NPC).

SEM Examination of Film Thickness

The adhesive resin layer was not discernible under the SEM in groups without the pre-curing step (Figure 2). The results of the pre-cured adhesive resin layer thickness measurements are presented in Table 3. The thickness of the adhesive layer for the pre-cured groups varied according to the different areas analyzed. The thickness of the adhesive layer was generally greatest at PF_1, DW and GA. The overall thicknesses of the adhesive layer are illustrated in Figure 1.

DISCUSSION

In this study, the microtensile bond strengths of seven adhesives with different application procedures for indirect restorations were measured. Numerous mechanical testing methods were performed to evaluate the adhesion of dentin bonding agents. The shear bond strength test is the most common test system used (Armstrong, Boyer & Keller, 1998; Sudsangiam & van Noort, 1999; Pashley & others, 1999). Its non-uniform stress distribution, however, creates areas of high stress concentration and causes a high percentage of fractures in dentin (Schreiner & others, 1998; Sudsangiam & van Noort, 1999; Pashley & others, 1999). In contrast, the diminutive size of the adhesive interface in the microtensile test reduces the degree of stress concentration and the incidence of cohesive failures. Furthermore, multiple specimens for microtensile testing can be produced from a single tooth (Sano & others, 1994; Phrukkanon, Burrow & Tyas, 1998; Pashley & others, 1999). Specimens with square cross sections of 0.25 to 1.2 mm^2 have been reported in a great number of studies (Armstrong & others, 1998; Cardoso, Braga & Carrilho, 1998; Frankenberger & others, 1999; Inoue & others, 2001a; Nunes, Swift & Perdigão, 2001).

Table 3: *Film Thickness of the Pre-cured Adhesive Layer According to Material and Area*

Areas	Film Thickness (µm)					
	Excite DSC	Integra Bond	OptiBond Solo Plus	Optibond Self-etch	Single Bond	Mean (SD)
GS	12.2	2.2	3.9	3.2	9.4	6.2(±6.9)
GA	10.0	4.6	9.8	11.3	25.5	12.2(±8.5)
PF_1	13.1	4.3	8.8	51.3	3.4	16.2(±18.9)
PF_2	12.5	8.4	6.4	10.6	6.8	8.9(±5.3)
PF_3	5.9	4.8	5.4	5.6	8.5	6.1(±3.2)
DA	5.7	4.3	13.4	5.1	7.2	7.1(±5.9)
DW	10.3	8.7	18.2	17.1	25.1	15.9(±8.7)
Mean(SD)	9.9(±6.9)	5.7(±4.2)	9.4(±6.4)	14.8(±16.3)	12.2(±10.2)	

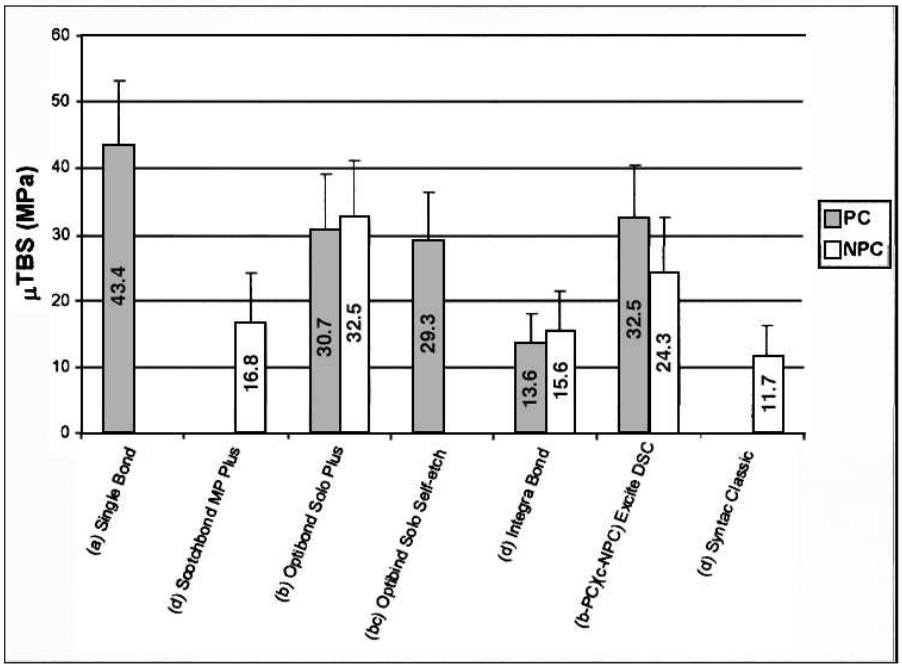

Figure 3. *Microtensile bond strength to dentin.*
PC= pre-cured adhesives; NPC= not pre-cured adhesives.
Same letters show no significant difference (p<*0.05)*

The results of this study showed that the one-bottle photo-cured system Single Bond produced significantly higher microtensile bond strengths than all the other adhesive systems tested. This is in agreement with the findings by Cardoso and others (1998), Nakajima and others (2000) and Nunes and others (2001). The one-bottle system combines the primer with the adhesive to allow a simplified application procedure. However, for indirect procedures, the shortcoming of this product is that it requires light-curing prior to resin cementation. Although the mean overall film thickness (12.2 µm) of this product might be thin enough to allow an inlay placement with no interference, some specific areas presented greater adhesive layer film thickness. Therefore, this cured adhesive layer could contribute to interference during the seating of the indirect restoration.

The one-bottle adhesive OptiBond Solo Plus was developed with an activator to provide dual-cured capability. The instructions for post cementation suggest no adhesive pre-curing to avoid the risk of seating interference during post insertion. The instructions for indirect restoration cementation, however, recommend an adhesive pre-curing step in order to prevent postoperative sensitivity in vital teeth. Chen and others (2001) found that the insufficient curing of some dentin bonding agents could have cytotoxic effects upon dental pulp tissue with potential for pulpal irritation. Adhesive pre-curing of OptiBond Solo Plus did not significantly improve microtensile bond strength. The

good results achieved by this adhesive in this study were consistent with the observations made by Duke, Platt and Rhodes (2000).

On the other hand, the adhesive Excite DSC showed significantly better performance when pre-cured. Excite DSC is a one-dose dual-curing bonding agent with initiators in the brush. The instructions for this product do not recommend light curing the adhesive prior to inlay cementation. The weak performance of Excite DSC (NPC), however, might be due to incomplete resin polymerization and suggests that this product should be pre-cured. The benefits of pre-curing Excite DSC to microtensile bond strength must be weighed against the risk of incomplete restoration seating.

The Integra Bond adhesive showed low bond strength values for both pre-cured and non pre-cured modes. Integra Bond contains acetone as its solvent and, although acetone is considered an efficient "water chaser," the acetone-based materials are more technique sensitive to the degree of residual surface wetness than ethanol-based adhesives (Kanca, 1992; Nunes & others, 2001). Dry dentin caused significantly reduced bond strength when primers with acetone solvents were used (Kanca & Sandrik, 1998). The other adhesive with acetone as an organic solvent, Syntac Classic, presented the lowest bond strength among all adhesives tested. In this study, the excess water that remained after rinsing was removed with a small piece of absorbent paper, leaving a visibly moist surface. However, it is difficult to standardize the level of moisture left on the surface. The level of residual dentin moisture might have deleteriously affected the acetone-based adhesives more than the ethanol-based adhesives.

Scotchbond Multipurpose Plus and Syntac Classic revealed relatively low bond strengths. These adhesives are designed to be used only as a self-cured system for indirect applications. The greater number of steps required by these adhesive systems can be a disadvantage for the already technique-sensitive procedure of indirect restoration cementation. Scotchbond Multi-purpose exhibited significantly higher bond strength values than some one-step and self-etching systems for direct applications (Bouillaguet & others, 2001). The better performance of Scotchbond Multipurpose in direct applications may be attributable to the light curing step of the adhesive in those applications.

The self-etching bonding system OptiBond Solo Plus SE presented a good bond strength to dentin (29.3 ±7.1 MPa). Self-etching primers are mainly an aqueous mixture of acidic monomers that have the ability to etch dentin and enamel without the need for rinsing and the inexact determination of optimal dentinal surface moisture (Toledano & others, 2001). The advantage of this system is that it combines conditioning and priming into one step, avoiding a gap between inorganic component demineralization and primer infiltration (Tanumiharja, Burrow & Tyas, 2000). The weak acidity of the self-etching primers, however, does not remove the smear plugs, resulting in greater difficulty in infiltration of acidic monomers through the smear layer into the dentin matrix (Frankenberger & others, 1999; Inoue & others, 2001a; Ogata & others, 2001). Inoue and others (2001b) found that two-step self-etch adhesives (acidic primer and adhesive) tended to have higher bond strengths than one-step self-etch (all-in-one) adhesives. Recently, some studies have concluded that the use of regular grit diamond burs decreased the bond strengths of self-etching bonding systems to dentin due to differences in the quantity and quality of the resultant smear layer (Inoue & others, 2001a; Ogata & others, 2001). They suggested that high bond strengths could be expected for self-etching systems when SiC paper #600 or steel burs were used. In this study, all cavities were prepared with regular grit diamond burs. There were no significant differences in bond strength among OptiBond Solo Plus Self-etch, OptiBond Solo Plus (PC and NPC) and Excite DSC (PC and NPC).

The reported results for the varying thicknesses of the adhesive layer are in agreement with that of other studies for light cured materials (Bouillaguet & others, 2001; Walshaw & McComb, 1996). Adhesive thickness was significantly higher when the adhesive was light-cured prior to resin inlay cementation and in regions conducive to adhesive pooling. The absence of a visible adhesive layer when the adhesive was not pre-cured could be explained by incorporation of the adhesive resin into the fluid resin cement.

Frankenberger and others (1999) and Hahn and others (2000) showed that adhesive resin pre-curing showed significantly thicker luting spaces but also the smallest dye penetration values. In this study, the alternate hypothesis that pre-curing was superior was not upheld for all materials tested. The effect of light curing prior to resin cementation on microtensile bond strength was dependent on the specific dentinal adhesive. Light-curing the adhesive prior to resin cementation increased the film thickness for all the dentin adhesives tested, but this effect may be tolerable if a careful technique is utilized. Further investigation is necessary to find out the real influence of the pre-curing and adhesive thickness on the internal and marginal adaptation of indirect restorations.

CONCLUSIONS

1) For the three dual-cure adhesives tested with and without pre-curing, two adhesives provided similar bond strengths with and without pre-curing and one adhesive showed higher bond strengths with pre-curing. The effect of pre-curing on bond strength was material specific.
2) Single-bottle primer/adhesives used for indirect restorations are capable of as high or higher

bond strengths than materials with separate primer and adhesive. The single-bottle adhesive Single Bond produced the highest microtensile bond strengths. The multi-step adhesives, Syntac Classic and Scotchbond Multipurpose Plus, each with four application steps, showed lower bond strengths.

3) Pre-curing the adhesive produced thicker adhesive film thickness. The resulting mean overall film thickness ranged from 5.7 to 14.8 microns. Film thickness, however, varied widely along the internal border of the indirect restoration. For adhesives used without pre-curing, no adhesive film could be distinguished from the resin-luting layer.

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Approximal Carious Lesion Depth Assessment with Insight and Ultraspeed Films

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Clinical Relevance

The new film, Insight, performed equally well as Ultraspeed at approximal carious lesions depth assessment.

SUMMARY

This study evaluated the efficiency of a new E/F-speed film, Insight, at the determination of approximal carious lesion depths compared with Ultraspeed. Radiographs of 80 extracted human molars and premolars were taken with both films under standardized conditions. The presence or absence of caries and depth of lesions was determined by three observers using a predetermined scale. The actual status of each surface was determined histologically. Observer responses were assessed with the Gamma measure of association test. Differences between the observers' agreement levels were not significant.

The efficiency of Insight and Ultraspeed at true depth diagnosis was found to be 54.9%; 55.8% and Gamma values were found to be 0.883 and 0.922, respectively, at $p<0.001$. The difference between the two films was not statistically significant ($p=0.852$). This study suggested that there was no statistically significant difference between the two films at detecting the depths of approximal carious lesions.

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INTRODUCTION

The use of faster films today may be the most important factor in reducing patient exposure in conventional radiographic techniques; therefore, manufacturers are developing films with increased sensitivity to radiation.

Until 1980, the fastest film available was D-speed film (Thunthy & Weinberg, 1995b). In 1981, Eastman Kodak (Rochester, NY, USA) introduced a faster dental film, Ektaspeed, an E-speed film capable of reducing the amount of radiation to approximately 50% (Thunthy & Weinberg, 1982; Hashimoto & others, 1992). However, this film was found to have decreased contrast,

increased fog and was more grainy than D-speed film (Kleier, Hicks & Flaitz, 1987; Kaffe, Littner & Kuspet, 1984). In the '90s, new E-speed films were introduced to the market, for example, Ektaspeed Plus (Eastman Kodak) and Dentus M2 (Agfa-Gevaret, Mortsel, Belgium) (Syriopoulos & others, 2001). Ektaspeed Plus was manufactured with T-Grain Emulsion Technology. This emulsion uses flat shaped, light-sensitive silver halide grains oriented to face the x-ray beam in a perpendicular fashion (Thunthy & Weinberg, 1995a; Ludlow & Platin, 1995).

Recently, prototypes of F-speed films have been introduced by Flow X-ray (West Hempstead, NY, USA). This film was found to have similar latitude and contrast characteristics and was 50% faster than E-speed films (Farman & Farman, 2000).

In April 2000, a new F-speed film, Insight, was introduced (Eastman Kodak). This film is described as an F-speed film when processed in a roller transport automatic processor and as an E-speed film in other manual processing conditions. According to the manufacturer, this film reduces radiation exposure dose up to 20% compared with Ektaspeed Plus and up to 60% compared to Ultraspeed films. Ludlow, Platin and Mol (2001b) reported that under ISO conditions, Insight required only 77% of the exposure of Ektaspeed Plus and 44% of the exposure of Ultraspeed films and its performance was not statistically different from the E- or D-speed films at caries detection (Ludlow, Abreu & Mol, 2001a).

This study compared the efficiency of Insight and Ultraspeed films at approximal carious lesion depth assessments.

METHODS AND MATERIALS

Eighty extracted human teeth, including 40 molars and 40 premolars were used in this study. The teeth had been stored in a 10% formalin solution since their extraction and their history was unknown. Some of the teeth were visually caries free and some had interproximal caries ranging from small white spot lesions to surfaces with small cavitations. The roots of the teeth were embedded in sticky wax and mounted in dental stone, simulating the anatomical contacts of two premolars and two molars. An arrangement was used that allowed the x-ray source, teeth blocks and films to be fixed at the same position at each exposure. The teeth blocks were imaged with size 2 Ultraspeed (USA Eastman Kodak, finished in France) and Insight films (Kodak Co, Rochester, NY, USA) in a buccolingual direction tangential to the approximal surfaces. Soft tissue simulation was made by using a 1-cm acrylic block (Ludlow & others, 2001a). The films were exposed with a Trophy (Trophy IRIX 708 France 77437) x-ray machine operating at 70 kVp, 8 mA, with a filtration of 2.5 mm Al and 0.8 x 0.8-mm focal spot 2 at 16-inch focal spot film distances. Exposure times were chosen from the guideline for Eastman Kodak intraoral dental films at a machine operating at 70kVp, 8mA, 16-inch focal spot film distance with automatic processing. Insight films were exposed for 0.16 seconds (40% of the dose required for Ultraspeed films) and Ultraspeed films were exposed for 0.40 seconds. The films were processed in an automatic roller transport machine (Velopex, Extra-X, Medivance Instruments Limited and England NW107A) with fresh chemicals.

Each observer was given instructions and a demonstration was conducted regarding how to code the radiographs. The radiographs were arranged randomly and cross-referencing of the radiographs was not permitted. The mesial surfaces of the teeth were rated according to a five-point scale:

1 - Caries definitely not present

2 - Caries probably not present

3 - Unsure (Equal chance of caries being present or absent)

4 - Caries probably present

5 - Caries definitely present as determined by three oral and maxillofacial radiologists (Ludlow & others, 2001a)

When a score of four or five was made, the depth of caries was rated according to the following scale:

1 - Radiolucency in the outer half of enamel

2 - Radiolucency in the inner half of the enamel

3 - Radiolucency in the external one-third of dentin

4 - Radiolucency in the middle one-third of dentin

5 - Radiolucency in the internal one-third of dentin, approaching the pulp

All radiographs were viewed on an x-ray viewbox without magnification and a period of two weeks separated the rating sessions between the two films (Svanaes, Møystad & Larheim, 2000). About four weeks later, the radiographs were assessed again for intra-rater reliability.

To record the true extent of caries, the mesial surface of the teeth was marked with waterproof ink and was hemi-sectioned in the mesiodistal direction. If an approximal lesion was clinically visible, the teeth were cut centrally (Hintze & Wenzel, 2003). The sections were evaluated under a stereomicroscope with 10x magnification (SZ-PT Olympus, Japan) and the actual status was determined by two observers having experience in histological evaluation. Any disagreement was solved by forced consensus.

After viewing the radiographs and evaluating the histological sections for the true extent of caries, the

Gamma measure of association test from the SPSS statistical package program (Release 7.5.1, standard version, SPSS Inc) was used to asses the inter-intra observer reliability and performance of two films at detecting depths of approximal carious lesions. (For radiographic evaluation, the first set of readings was used).

This test was used between two ordinal categorical variables, with +1.0 representing perfect agreement, 0.0 representing no agreement and –1.0 representing perfect disagreement.

RESULTS

In the study, from the 80 surfaces, three were destroyed during the histological preparation and only 77 were available for both histological and radiographic examination. From the histological examination, 21 surfaces were found to be caries free (score 0), 46 had lesions localized at enamel and dentin (scores 1, 2, 3, 4) and 10 had lesions in the inner half of dentin approaching the pulp.

Observer scores for each surface were evaluated to find the degree of agreement. The results showed there was a positive correlation between raters and the agreement level was almost perfect for both films. Table 1 shows the inter-observer agreement values. A high-positive intra-observer correlation level between true approximal caries depth diagnosis was also found between Ultraspeed and Insight (Gamma values ranged between 0.825-0.990 for Ultraspeed and 0.921-0.931 for Insight at $p<0.001$).

Table 1: *Inter-agreement Levels Between Observers*

Observers	Ultraspeed (Gamma value)	Insight (Gamma value)
1-2	0.953	0.940
1-3	0.908	0.875
2-3	0.893	0.899

p<0.01

Table 2: *Cross tabulations illustrating three observers correct diagnosed, under estimated, overestimated depth and false positive values of approximal carious lesions rated according to Ultraspeed films. Numbers written in italics define the cases which agreement existed between histology and films.*

	Actual Status Scores						
Film Scores	**0**	**1**	**2**	**3**	**4**	**5**	**Sum**
0	*54*	15	3	2			74
1	8	*11*		9			28
2	1	4	*1*	7	3		16
3			1	*11*	16	1	29
4			1	7	*26*	6	40
5					18	*26*	44
Sum	63	30	6	36	63	33	231

(Note: The agreement levels of the three observers were almost perfect, so that the sum of their scores were tabulated). Gamma value: 0.922, p<0.001

Table 3: *Cross tabulations illustrating three observers correct diagnosed, under estimated, overestimated depth and false positive values of approximal carious lesions rated according to Insight films. The numbers in italics define the cases where agreement existed between histology and films.*

	Actual Status Scores						
Film Scores	**0**	**1**	**2**	**3**	**4**	**5**	**Sum**
0	*54*	15	4	9	1		83
1	5	*9*		7	5		26
2	1	6	*1*	4	4		16
3	3			*12*	12		27
4			1	4	*21*	3	29
5					20	*30*	50
Sum	63	30	6	36	63	33	231

(Note: The agreement levels of the three observers were almost perfect, so that the sum of their scores were tabulated). Gamma value: 0.883, p<0.001

The correct depth diagnosis for Ultraspeed films was 55.8% (Gamma value 0.922, Table 2) and 54.9% for Insight films (Gamma value 0.883, Table 3) at $p<0.001$. These results suggest that although the Gamma value for Insight films was lower than Ultraspeed films, the difference was not significant ($p=0.852$). The underestimation of lesion depth (observer rated the depth seen on radiographs as being more shallow than the actual status) and overestimation of lesion depth percentage (observer rated the depth seen on radiographs as being deeper than the actual status) was calculated also. On the Ultraspeed films, 26.8% and on the Insight films, 27.7% of the lesions depth was underestimated. On the other hand, both films overestimated 13.5% of lesion depth. From the examined sample, 3.9% of the sound surfaces were scored as caries, with various depths by both films.

With the depth assessment of carious lesions localized at enamel and showing minimal involvement of dentin (scores 1, 2, 3), correct diagnosis performance for Ultraspeed was 31.9% (Gamma value 0.641) and 30.5% for Insight (Gamma value 0.657). The difference between these values was also found to be not significant ($p=0.857$).

DISCUSSION

From the results of this study, no significant difference between the two films was found regarding detecting the depths of approximal carious

lesions. In addition, the overestimation of depths and sound surface percentages scored as carious were also the same. Insight performed equally well as Ultraspeed, with a 20% lower radiation dose.

Insight was reported to be equal to Ektaspeed Plus and CMOS-APS digital sensor (Nair & Nair, 2001). Ludlow and others (2001a) reported that the contrast performance of the Insight film was between the 0.5-0.8 density range, which is typical for coronal density in posterior teeth, which was an indication of potential performance for incipient caries diagnosis.

This film has been classified as an E/F-speed film, depending on processing conditions. It is correctly classified in the ISO F-speed group when processed in a roller transport automatic processor machine with fresh solutions (Ludlow & others, 2001b). The film is said to achieve F-speed in an automatic processor using a roller transporting system. The gentle squeezing and flexing of the film in this type of automatic processor serves to activate the emulsion, thus increasing the rate of development. The reduction in mechanical activation in a dip-tank and automatic processors that do not use roller transport moves the emulsion speed into the high end of the E-speed range (Ludlow & others, 2001b).

The films were processed in a roller-transport automatic processor machine (Velopex Medivance Instruments) with fresh solutions (Velopex chemicals) used in the study so that the film was in the F-speed range.

According to logistic regression, mechanical defects prepared in approximal enamel are easier to detect, even when the depths are equal, compared to natural caries (Kang & others, 1996). In addition, mechanical defects created with round burs have sharper borders and contain air; whereas, natural caries have gradual boundaries and often contain varying amounts of calcified salts within the cavity (Newburn, 1989) so that there could be an overestimation of the efficiency of techniques (Kang & others, 1996). Therefore, approximal surfaces of premolars and molars with and without natural caries were used in this study to test the performance of Insight and Ultraspeed films, providing the best *in vitro* standards.

With the hemi-sectioning technique, it is difficult to centrally cut each approximal surface located on the opposite sides of the same tooth. To solve this problem, only the mesial surfaces of teeth were assessed both radiographically and histologically according to other studies (White & Yoon, 1997; Syriopoulos & others, 2000).

Gamma measure of association test was proposed by Goodman and Kruskal and correlation between two ordinal categorical variables could be made with this test (Agresti, 1990). Caries depths are ordinal variables, so that this could be used in studies whose aim is to compare the efficiency of films at detecting lesion depths seen on the radiograph and its actual status. The presence or absence of any radiolucency seen on radiographs was coded according to a five-point rating system, a comparison of the scores was rated on both radiographs and the actual status of the lesions was made.

It could be concluded that the correct determination rate of depths of lesions located at enamel and the outer third of dentin is lower than those located at the middle and inner third of dentin. When an analysis was made for the efficiency of Insight at detecting these lesions (scores 1, 2, 3), although there was a decrease in correct diagnosis number, no significant difference was found compared with Ultraspeed.

Dental practitioners who use x-ray equipment have a responsibility to produce radiographs that are taken with the least amount of radiation and produce high diagnostic quality at minimal cost (Diehl, Gratt & Gould, 1986). The diagnosis performance of Insight film for approximal carious lesions did not differ when processed in a roller transport automatic machine and required a reasonably lower radiation dose compared to Ultraspeed film, while maintaining the quality.

CONCLUSIONS

Insight was equally successful as Ultraspeed in determining approximal carious lesion depths with a 60% dose reduction.

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Dentin Bond Strength of Self-etching Primers/Adhesives

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Clinical Relevance

Not all the self-etching systems tested were capable of producing predictable bond strengths to that achieved by the total-etch system. SBS to dentin with self-etching systems may depend on the specific composition of those systems.

SUMMARY

This study compared the shear bond strengths (SBS) to dentin achieved with six self-etching systems and one total-etch one-bottle adhesive system. Seventy freshly extracted bovine incisors were mounted in acrylic molds and the facial surfaces ground to expose middle dentin, which was polished by 600-grit sand paper. The incisors were randomly assigned to groups (n=10): Adper Prompt Self-Etch Adhesive, 3M-ESPE (ADP) and One-Up Bond F, Tokuyama (OU) as self-etching adhesives; AdheSE, Ivoclar-Vivadent (ADH), Clearfil SE Bond, Kuraray (SE), Optibond Solo Plus–Self-Etch, Kerr (OP) as self-etching primers, Tyrian SPE, BISCO (TY) as a self-priming etchant and Single Bond, 3M-ESPE (SB), a total-etch one-bottle adhesive served as a control. All adhesives were applied according to the manufacturers' instructions with the respective hybrid composites. The specimens were thermocycled for 500 cycles (5°C to 55°C), then loaded to failure in an Instron Universal Testing Machine at a crosshead speed of 0.5 mm/minute. Mean bond strengths were analyzed with one-way ANOVA, followed by a Duncan's post hoc test. SBS (mean ±SD) were: ADH = 13.2 (±5.3)^{b}; ADP = 6.8 (±4.4)^{c}; OP = 18.2 (±3.8)^{a}; OU = 3.5 (±1.5)^{c}; SB = 12.2 (±4.2)^{b}; SE = 12.4 (±4.0)^{b}; TY = 5.5 (±1.4)^{c}. Superscript letters indicate Duncan's homogeneous subsets. The self-etching adhesives OU and ADP and the self-priming etchant TY resulted in lower dentin SBS. OP resulted in the highest mean dentin SBS, while the other materials tested in this study (SE and ADH) presented similar dentin SBS to a total-etch one-bottle bonding system (SB).

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INTRODUCTION

Bonding to dentin has been known as one of the major challenges in adhesive dentistry mainly because of the inherent characteristics of this substrate (Perdigão, 2002; Lopes & others, 2002; Perdigão & Lopes, 1999). Current adhesion strategies involve two trends: the total-etch bonding technique characterized by the complexity of its components and bonding procedures, and self-etching systems, following a trend toward simplification. The self-etching systems have recently become available and combine the functions of primer and adhesive components, not requiring a separate acid etch step and, thus, eliminating the need for rinsing. The self-etching systems are capable of etching the tooth surface and simultaneously preparing it for adhesion. This was made possible by the use of primers that contain non-rinsing polymerizable monomers. These monomers include an acidic group that dissolves or converts the smear layer, penetrating the aqueous channels formed between its particles and subsequently interacting at the top of the underlying dentin (Chigira & others, 1994; Watanabe, Nakabayashi & Pashley, 1994). Thus, the smear layer will be incorporated into the adhesive layer (Pashley & Carvalho, 1997). As acidic monomers are responsible for etching and bonding, the depth of demineralization is equal to the depth of penetration of the monomers and, clinically, this corresponds to a reduced chance of postoperative sensitivity (Opdam & others, 1998). In addition to simplifying the bonding procedure and reducing working time, eliminating rinsing and drying and the need for an "ideal" dentin wetness reduces the negative influences of these steps on establishing adhesion (Kanca, 1992; Tay, Gwinnet & Wei, 1996). Currently, there are several self-etching systems available but little is known about their capacity to adhere to dental tissues. This *in vitro* investigation compared the shear bond strengths (SBS) to dentin achieved with several self-etching systems. The null hypothesis was that self-etching adhesives and primers present different shear bond strengths to a total-etch adhesive system.

METHODS AND MATERIALS

Seventy defect-free maxillary bovine incisors were obtained just after extraction. The teeth were debrided, cleaned and kept in a 0.1% thymol aqueous solution until utilization. After the root portions were grounded to the cervical third, the crowns were mounted in phenolic rings and embedded with self-curing acrylic resin. The labial surface of each tooth was ground to remove enamel and expose 5- to 6-mm areas of middle-depth dentin with a model trimmer. The exposed dentin was then polished for 30 seconds with 240, 400 and 600 grit silicon carbide abrasive sandpaper under water. The specimens were randomly assigned to one of the seven experimental groups of 10 specimens each (n=10). Table 1 lists the materials investigated in this study. Bonding agents were applied and light cured according to the manufacturers' instructions under standardized conditions as follows:

AdheSE (Ivoclar Vivadent, North America, Amherst, NY, USA): the primer was applied for 30 seconds and gently air dried, then the bonding agent was applied and light cured for 10 seconds.

Adper Prompt Self-Etch Adhesive (3M-ESPE, St Paul, MN, USA): after mixing the two solutions (A and B) for five seconds, the mixture was applied for 15 seconds, gently air dried and light cured for 10 seconds.

Clearfil SE Bond (Kuraray Co, Osaka, Japan): the primer was applied for 20 seconds, gently air dried, then the bonding resin was applied, gently air dried and light cured for 10 seconds.

One-Up Bond F (Tokuyama Co, Tokyo, Japan): bonding agents A and B were mixed until they turned homogeneously pink. They were then applied for 20 seconds, gently air dried and light cured for 10 seconds.

OptiBond Solo Plus–Self-Etch Primer (Sybron Kerr Dental Specialties Inc, Orange, CA, USA): the self-etching primer was applied for 15 seconds and gently air thinned for three seconds, then the bonding agent was applied for 15 seconds and gently air thinned

Table 1: *Materials Used in This Study*

Group	Adhesive System	Composite	Manufacturer	Batch #
ADH	AdheSE	Tetric Ceram	Vivadent	E35150/ D15352
ADP	Adper Prompt Self-Etch	Filtek Z250	3M-ESPE	EXM618/2NX
OU	One-Up Bond F	Palfique Estelite	Tokuyama	531/E6081
OP	OptiBond Solo Plus– Self Etch Prime	Point 4	Kerr	203D20/ 204D42/ 201308
SB	Single Bond	Filtek Z-250	3M-ESPE	2GR/2NX
SE	Clearfil SE Bond	Clearfil APX	Kuraray	137/00476B
TY	Tyrian–One Step Plus	Renew	BISCO	0200002694/ 0200003755/ 0200001887

for three seconds, reapplied for 15 seconds, gently air thinned and light cured for 20 seconds.

Single Bond (3M-ESPE): dentin was etched for 15 seconds with 35% phosphoric acid (Scotchbond Etchant Gel, 3M-ESPE) and rinsed with water for 10 seconds. Excess water was removed by blotting with a cotton pellet, leaving the surface moist. The adhesive was applied in two consecutive coats and gently air dried and light cured for 10 seconds.

Tyrian SPE (BISCO Dental Products, Schaumburg, IL, USA): Tyrian was applied for 10 seconds and the preparation was dried with a foam pellet, then two coats of One-Step Plus were applied, air dried for 10 seconds and light cured for 10 seconds.

A hybrid composite, Filtek Z-250 (3M-ESPE) was condensed into #5 gelatin capsules with a 4.3-mm diameter (Torpac Inc, Fairfield, NJ, USA), to fill approximately two-thirds of the capsule and light cured in a triad oven for 120 seconds. After the application of the bonding systems, a final increment of a composite respective to the brand of each adhesive was added to fill the capsules, which were seated securely and perpendicular against the flattened dentin surfaces. Excess material was carefully removed with 4x magnification to prevent any residual composite flash, then the composite was light cured for a total of 80 seconds (20 seconds from each perpendicular direction). All light curing was performed using an XL 2500 conventional light curing unit (3M-ESPE) at 450 mW/cm^2. The specimens were subjected to thermocycling for 500 cycles between water baths held at 5°C and 55°C, with a dwell time in each bath of 30 seconds and a transfer time of three seconds. After thermocycling, SBS was tested with an Instron Universal Testing Machine Model 4444 (Instron Corporation, Canton, MA, USA), using the Series IX Software System to record the data. Each specimen was mounted in a shear testing apparatus and a chisel-shaped shearing rod with a crosshead speed of 0.5 mm/minute was used to load the specimens flush to the dentin-composite interface, delivering a parallel force until fracture. After testing, the specimens were observed under an optical microscope to evaluate the type of failure. The data were subjected to one-way analysis of variance (independent variable: adhesive, outcome variable: SBS). A Duncan's *post hoc* test was used to identify statistical differences between pairs of means at a confidence level of 95% for each data set. All statistical analysis was carried out with the SPSS 10.0 software package (SPSS Inc, Chicago, IL, USA).

RESULTS

Mean SBS of the self-etching adhesives ranged from 3.5 MPa for One-Up Bond to 6.8 MPa for Adper Prompt Self-Etch Adhesive. Mean shear bond strengths of the self-etching primers ranged from 5.5 MPa for Tyrian to 18.2 MPa for OptiBond Solo Plus–Self-Etch Primer. Mean SBS of the control group, Single Bond, was 12.2 MPa. The results are summarized in Table 2 and Figure 1. One-way ANOVA revealed a significant difference at $p<.0001$. Duncan's *post hoc* test ranked these differences in three subsets at a confidence level of 95%

Table 2: *Mean Shear Bond Strengths, Duncan's Ranking and Predominant Types of Failure*

Group	Mean SBS ± SD (MPa)*	Type of Fracture**
ADH	13.2(±5.3)^b	5A-7C
ADP	6.8(±4.4)^c	12A
OU	3.5(±1.5)^c	11A 1M
OP	18.2(±3.8)^a	1A 9C 2M
SB	12.2(±4.2)^b	7A 2C 3M
SE	12.4(±4.0)^b	4A 3C 5M
TY	5.5(±1.4)^c	10A 1C 1M

**Superscript letters indicate Duncan's homogeneous subsets.*
**A = Adhesive; C = Cohesive in dentin; M = Mixed.

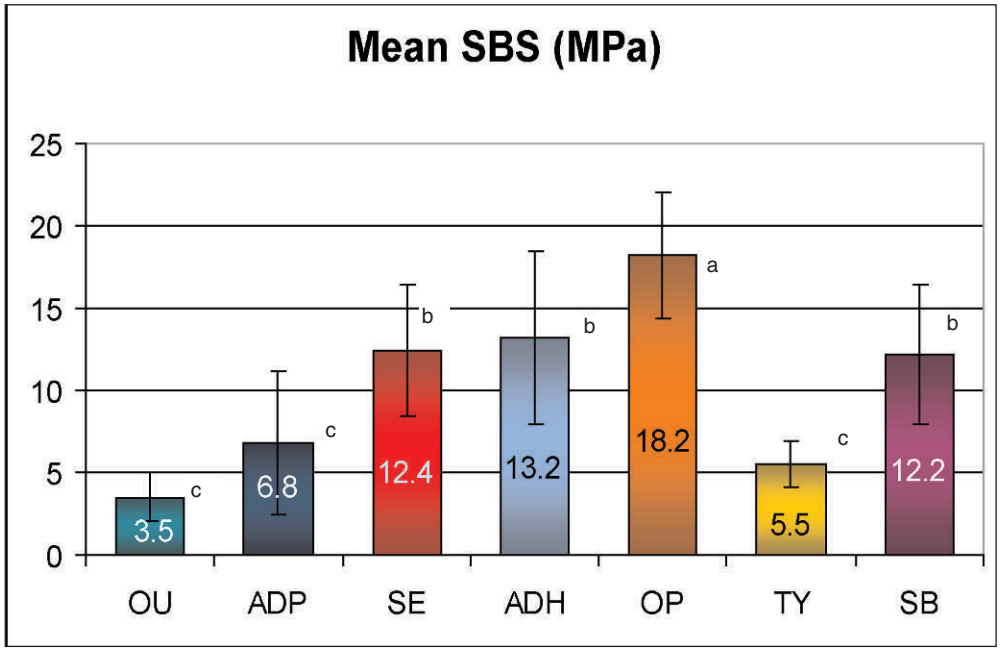

Figure 1: *Mean shear bond strengths to dentin (same superscript letters indicate Duncan's homogeneous subsets).*

(Table 2). OP had the highest mean dentin SBS. The ADH, SE and SB specimens were ranked in the intermediate Duncan's subset, while ADP, OU and TY had the lowest SBS.

DISCUSSION

The effectiveness of self-etching primers and adhesives was compared within the same parameters in this investigation. Shear bond strength test is a simple evaluation procedure used to test the adhesion of dental adhesives (Barkmeier & Cooley, 1992). *In vitro* bond strength tests are useful and essential for predicting the performance of new adhesive systems and possible correlation with clinical issues; however, *in vitro* investigations are not capable of predicting clinical success.

Due to the difficulty in acquiring adequate quantities of human teeth, bovine teeth are considered an acceptable substitute for laboratory studies (Nakamichi, Iwaku & Fusayama, 1983; Saunders, 1988; Reeves & others, 1995; Schilke & others, 1999; Miyazaki & others, 1999).

The use of self-etching primers and adhesives is a recent approach towards the simplification of bonding techniques. This approach does not require rinsing and can be done in a two-step method, combining the etching and priming functions, or in a one-step method, combining etching, priming and bonding functions. The rationale behind the use of the self-etching systems is the formation of a continuity between tooth surfaces and adhesive material, which is accomplished by the simultaneous demineralization and penetration of its agents (Rosa & Perdigão, 2000). This could be an advantage compared to the claimed technique sensitivity of conventional total-etch dentin bonding agents.

The lowest means SBS were obtained by One Up Bond F, a self-etching adhesive composed of methacryloyloxyalkyl acid phosphate (phosphoric acid monomer), MAC 10, multifunctional methacrylic monomer, HEMA, fluoroaluminosilicate glass filler, water and initiators, probably as a consequence of its relatively high pH of 2.57 (Tay & others, 2001). Despite the methodological differences, this result disagrees with similar studies (Kimishima & others, 2003; Lopes & others, 2003b; Miyazaki, Iwasaki & Onose, 2002a; Miyazaki & others, 2002b). Similar low bond strengths were achieved with Tyrian and Adper Prompt Self-Etch.

Tyrian is a self-priming etchant composed of 2-acrylamido-2-methyl propanesulfonic acid Bis(2-(methacryloyloxy)ethyl) phosphate and ethanol and is used with the fifth generation one-bottle adhesive One-Step Plus. Other recent studies have reported higher bond strengths with this system (Cardoso & Sadek, 2003; Strukowska & others, 2003; Fuentes & others, 2003; Vuu & others, 2003; Naughton, Latta & Barkmeier, 2003), which could possibly be related to differences in the testing methodology.

Adper Prompt Self-Etch is based on the original Prompt-L-Pop adhesive and is composed of methacrylated phosphoric esters, Bis-GMA, initiators, stabilizers, water, HEMA and polyalkenoic acid. Its results disagree with some similar recent studies (Kimishima & others, 2003; Lopes & others, 2003a; Naughton & others, 2003; De Munck & others, 2003). The low bond strengths obtained with this system may be due to an incomplete infiltration of the acidic monomers and subsequent partial dissolution of the smear layer, suggesting inconsistent performance in terms of achieving a quality bond (Ibarra & others, 2002).

The adhesive Single Bond was selected as a control because it has been extensively documented (Swift & Bayne, 1997; Perdigão, Swift & Lopes, 1999; Ritter & others, 2000). The AdheSE and Clearfil SE Bond systems have achieved similar bond strengths to that of the control group.

While Clearfil SE Bond is a well established and documented self-etching primer (Perdigão & Geraldeli, 2003; Nikaido & others, 2002; Ibarra & others, 2002; Cardoso & others, 2002; Pashley & Tay, 2001; Hayakawa, Kikutake & Nemoto, 1998), AdheSE is a relatively new self-etching system containing a primer composed of phosphonic acid acrylate, bis-acrylamide, water, initiators and stabilizers and a bonding component composed of dimethacrylate, hydroxyethyl methacrylate and highly dispersed silicon dioxide, initiators and stabilizers. Data supplied by the manufacturer suggest that higher bond strengths could be achieved (Ivoclar-Vivadent Scientific Documentation). Despite the methodological differences, recent studies reported higher bond strengths with this system for both dentin (De Munck & others, 2003) and enamel (Duarte & others, 2003; Lopes & others, 2003a).

The highest mean of shear bond strengths were obtained with OptiBond Solo Plus Self-Etch, a self-etching primer composed of ethyl alcohol, water, alkyl dimethacrylate resins, stabilizers and activators, used with the fifth generation one-bottle adhesive OptiBond Solo Plus. These results may be related to its low pH (1.2–1.5), assuring a better monomer penetration to the critical mode of application of this system and by the fillers in the adhesive composition, enhancing the bond strength to dentin (Cardoso & Sadek, 2003; Lopes & others, 2003b; De Munck & others, 2003).

The wide range of values and discrepancies comparing this investigation to others may be related to differences between the experimental conditions and investigators. The relatively low bond strengths encountered may be related to the nature of the shear bond test, the use of larger bonded areas and also differences between bovine and human dentin. The high number

of cohesive failures achieved by some systems may not be taken in consideration and could also be explained either by the thinner dentin substrate of bovine teeth and the methodology used, since the bonded areas in shear tests are probably more resistant than tensile tests (Sano & others, 1999).

CONCLUSIONS

The hypothesis tested was partially accepted, since the self-etching systems AdheSE, Clearfil SE Bond and OptiBond Solo Plus Self-Etch achieved similar shear bond strengths to a total-etch system (Single Bond), while Adper Prompt Self-Etch, One-Up Bond F and Tyrian–One Step Plus showed different shear bond strengths. Clinical evaluations are obviously necessary to confirm these observations.

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Barcoll Hardness of Different Resin-based Composites Cured by Halogen or Light Emitting Diode (LED)

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Clinical Relevance

Barcoll hardness of resin-based composites cured by LED LCU was statistically equivalent to those cured by a halogen LCU. With its inherent advantages, such as a constant power output over the lifetime of the diodes, LED LCUs have great potential for achieving a clinically consistent quality of resin composite cure.

SUMMARY

The clinical performance of light curing resin composites is greatly influenced by the quality of the light-curing unit (LCU). Halogen LCUs are commonly used for curing composite materials. However, they have some drawbacks. The development of new, blue, super bright light emitting diodes (LED LCU) of 470-nm wavelength with high light irradiance comes as an alternative to standard halogen LCUs of 450-470-nm wavelengths.

This study evaluated the surface hardness of the different resin-based composites (flowable, hybrid and packable resin composites) cured by LED LCU or halogen LCU. A Teflon mold 10-mm in diameter and 2-mm in depth was made to obtain five disk-shaped specimens for each experimental group. Then, the specimens were cured by an LED LCU or halogen LCU for 40 seconds. The hardness of the upper and lower surfaces was measured with a Barcoll hardness-measuring instrument. The statistical analysis was performed using one-way analysis of variance (ANOVA) and Duncan test at a p=0.05 significance level.

The results of the hardness test indicated that the hardness of resin composites cured by an LED LCU were greater than those cured by a halogen LCU. Additionally, for all resin-based composites, the hardness values for the upper surfaces were higher than the lower surfaces. However, for both results no statistically significant differences were observed (p>0.05).

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INTRODUCTION

Light-curing resin-based composites have revolutionized clinical dentistry by maximizing working time and minimizing setting time. One of the limitations of these materials is that a hard-upper surface is not an indica-

tion of adequate polymerization throughout the depth of the restoration (Pilo & Cardash, 1992). Incomplete polymerization of resin results in an increase in water sorption, a decrease in hardness and strength and deterioration of the mechanical properties of the material through softening of the polymer matrix by unreacted monomer and decreasing wear resistance (Ferracane & others, 1997). This can also lead to undesirable consequences such as gap formation, marginal leakage, recurrent caries, adverse pulpal effects and the ultimate failure of the restoration.

The effectiveness of resin-based composite polymerization may be assessed directly or indirectly. Direct methods such as laser Raman spectroscopy (Louden & Roberts, 1983) and infrared spectroscopy (Asmussen, 1982a) have not been accepted for routine use, because the methods are complex, expensive and time-consuming (Rueggeberg & Craig, 1988). Indirect methods have included scraping (Cook, 1980), visual inspection (Murray, Yates & Newman, 1981) and surface hardness (Asmussen, 1982b). Surface hardness has been shown to be an indicator of the degree of polymerization, and hardness testing appears to be the most popular method for investigating polymerization due to the relative simplicity of the method (Yap, 2000).

The early light-cured, resin-based composites were cured by UV light, later versions by visible light. A halogen lamp is routinely used as the dental light activation unit. Halogen lamps produce light by incandescence, whereby a filament is heated and causes the excitation of atoms over a wide range of energy levels, producing a very broad spectrum of light. Filters are therefore needed to restrict the emitted light to the blue region of the spectrum for the polymerization of resin-based composites. However, halogen light curing units used to polymerize dental materials have several drawbacks. Halogen bulbs have a limited effectiveness of approximately 100 hours. In addition, the LCU bulb, reflector and filter can degrade over time, because of high operating temperatures and the large quantity of heat produced during operating cycles. This results in a reduction in the LCUs curing effectiveness over time, insufficient physical properties and an increased risk of premature failure of restoration (Martin, 1998; Pilo, Oelgiesser & Cardash, 1999).

Solid-state light emitting diode (LED) technology has been proposed for curing as a means of overcoming the problems inherent to halogen LCUs (Mills, 1995). Rather than a hot filament, as used in halogen bulbs, LEDs use junctions of doped semiconductors (p-n junctions) to generate light (Nakamura, Mukai & Senoh, 1994). Under proper forward biased conditions, electrons and holes recombine at the LED p-n junction, leading, in the case of gallium nitride LEDs, to the emission of blue light. A small polymer lens in front of the p-n junction partially collimates the light. The spectral output of gallium nitride blue LEDs fall conveniently within the absorption spectrum of the camphorquinone photoinitiator (400-500 nm) present in light activated dental materials, so that no filters are required in LED LCUs. Furthermore, LED LCUs have an expected lifetime of several thousand hours without significant degradation of light flux over time (Haitz, Craford & Weissman, 1995).

Several studies have addressed the application of blue LED technology to cure resin-based composite (Mills, 1995; Mills, Jandt & Ashworth, 1999a; Stahl & others, 2000; Uctasli, Shortall & Burke, 2002). In recent studies, LED LCU prototypes have been used for depth of cure (Jandt & others, 2000), compressive strengths (Jandt & others, 2000) and flexural strengths (Stahl & others, 2000) that are not statistically significantly different from the values obtained with a halogen LCU with an irradiance of 755 mW/cm^{-2} in many cases. This was surprising, since the LED LCU irradiance was less than half that of the halogen LCU.

Since publication of these studies, LED technology has advanced significantly (Mills & others, 1999b). The development of new LED LCUs of 470-nm wavelengths and the construction of LED LCUs with high power light sources with similar irradiance to conventional LCUs are now available (Dunn & Bush, 2002; Mills & others, 2002; Yoon & others, 2002). Therefore, additional information about the mechanical properties of resin composites cured with new LED LCUs are required to judge the LEDs clinical potential.

This study investigated the Barcoll hardness of five different resin-based composites (flowable, hybrid and packable resin composites) cured with LED LCUs or halogen LCUs.

METHODS AND MATERIALS

Five light-cured resin-based composites were used in this study. The list of composites, including their type and composition (organic matrix, filler size, filler type and volume amount), are shown in Table 1. A3 shade was used for all the resin-based composites.

For each material, five disc-shaped specimens (10-mm diameter and 2-mm thickness) were prepared using Teflon molds. These molds were placed on flat glass plates on top of acetate strips (Hawe-Neos Dental, Bioggio, Switzerland) and than filled with resin-based composites. The resin was covered with an acetate strip and gently pressed with another glass plate against the mold to extrude excess material. The specimens were then irradiated from the top through the glass slide and acetate strip with an LED LCU (Elipar Freelight, 3M-ESPE Dental Products, St Paul, MN, USA) or halogen LCU (Hilux Ultra Plus, Benlioglu Dental Inc, Ankara, Turkey).

Table 1: *List of Tested Resin-based Composites Including Type and Compositions*

Materials	Type	Organic Matrix	Inorganic Filler	Filler Content (% by volume)	Filler Size (μm)
Filtek Flow (3M, St Paul, MN, USA)	Flowable resin composite	Bis-GMA, TEGDMA	Zirconia/Silica	47	0.01-6.0 (mean-1.5)
Esthet-X (Dentsply DeTrey GmBH, Konstanz, Germany)	Hybrid resin composite	BIS-GMA-adduct, ethoxylated Bisphenol-A-dimethacrylate, TEGDMA	Barium alumino fluoroborosilicate glass with nano-sized silicon dioxide	60	0.02-2.5 (mean-0.6-0.8)
Filtek Z 250 (3M, St Paul, MN, USA)	Hybrid resin composite	BIS-GMA, UEDMA, Bis-EMA	Zirconia/Silica	60	0.01-3.5 (mean-0.6)
Filtek P 60 (3M, St Paul, MN, USA)	Packable resin composite	BIS-GMA, UEDMA, Bis-EMA	Zirconia/Silica	61	0.01-3.5 (mean-0.6)
Surefil (Dentsply DeTrey GmBH, Konstanz, Germany)	Packable resin composite	Urethane modified BIS-GMA	Boro-silicate-aluminum	66	0.04-9.0 (mean-0.8)

Table 2: *Mean Hardness Values and Standard Deviations of Upper Surfaces and Lower Surfaces of the Tested Resin-based Composites Cured with LED LCU or Halogen LCU (same letter are not statistically different between resin-based composites at p=0.05)*

	LED LCU		Halogen LCU	
Materials	**Upper Surface**	**Lower Surface**	**Upper Surface**	**Lower Surface**
Filtek Flow	74.00 (2.64)^{cA}	73.10 (1.34)^{eA}	73.33 (0.88)^{cA}	71.77 (1.57)^{dA}
Filtek Z 250	89.21 (0.50)^{aA}	87.66 (0.33)^{cA}	87.99 (2.90)^{aA}	87.22 (0.19)^{bA}
Esthet-X	90.44 (1.64)^{bA}	89.88 (0.19)^{dA}	90.00 (1.73)^{bA}	81.44 (0.50)^{cB}
Filtek P60	84.55 (1.16)^{aA}	83.11 (0.84)^{aA}	84.22 (0.84)^{aA}	88.66 (0.88)^{abA}
Surefil	90.10 (1.38)^{aA}	88.55 (0.19)^{bA}	88.88 (1.38)^{abA}	88.20 (0.72)^{aA}

The LED LCU used in the study contained 19 blue LEDs. The light from the LED LCU was concentrated using polymer optical tapers with a measured output diameter of 8 mm. The irradiance of LED LCU was 400 mWcm^2, with a wavelength of 440-490 nm. Light intensity was measured by means of a radiometer (Elipar Free Light, 3M-ESPE). LED LCU was used in a standard mode (full light intensity during the complete exposure cycle) for polymerization of resin-based composite. A halogen LCU had a light guide tip diameter of 8 mm, with an irradiance of 600 mWcm^2. Its wavelength was 450-520 nm. Light intensity was measured by a radiometer (Hilux Curing Light Meter). Both LCUs were used at full power for 40 seconds.

Immediately after light curing, the cover glasses were removed from the mold and the top and bottom surfaces of each specimen were polished with a Sof-Lex disk (3M-ESPE). Then, the specimens were removed from the mold and the lower surfaces were marked with a pen and stored in the dark in distilled water at 37°C for 24 hours. Five specimens were prepared for each experimental group.

The hardness of the upper and lower surfaces was measured with a Barcoll hardness-measuring instrument (Barcoll surface tester, Barber-Collman Comp, Loves Park, IL, USA). Three readings were taken at three points within each surface (upper and lower). The mean of the Barcoll hardness and ratio of the five specimens were calculated and tabulated using the formula:

Hardness ratio = Barcoll hardness of lower surface/Barcoll hardness of upper surface

The statistical analysis was performed using one-way analysis of variance (ANOVA) and Duncan tests at a p=0.05 significance level.

RESULTS

The means and standard deviations and statistical comparison of Barcoll hardness of the upper and lower surfaces of resin specimens cured with the LED LCU or halogen LCU are shown Table 2.

For all resin-based composites, Barcoll hardness of specimens cured with LED LCU was greater than the Barcoll hardness of specimens cured with halogen

Table 3: *Mean Hardness Ratio and Standard Deviations of the Tested Resin-based Composites Cured with LED LCU or Halogen LCU*

	LED LCU	Halogen LCU
Materials	**Hardness Ratio**	**Hardness Ratio**
Filtek Flow	0.96 (0.005)	0.97 (0.02)
Filtek Z 250	0.97 (0.03)	0.99 (0.01)
Esthet-X	0.98 (0.00)	0.97 (0.01)
Filtek P60	0.97 (0.01)	0.98 (0.01)
Surefil	0.96 (0.02)	0.98 (0.005)

LCU at upper and lower surfaces. However, there were no statistically significant differences in the Barcoll hardness at both the upper and lower surfaces among LED LCUs or halogen LCU of all resin-based composites ($p>0.05$), except Esthet-X's lower surface ($p<0.05$).

For both LCU and hybrid composite, Esthet-X showed the highest values and flowable composite, whereas, Filtek Flow showed the lowest hardness values for upper and lower surfaces.

The upper surfaces of the specimens cured with LED LCUs and halogen LCUs showed higher hardness values than the lower surfaces of the specimens ($p<0.01$). However, no statistically significant difference was observed between the upper and lower surface for each material ($p>0.05$).

Table 3 shows the statistical analysis of hardness ratio of the resin composite materials cured with the different LCU. The hardness ratio of the tested resin-based composite materials ranged from 0.96 to 0.99. However, no statistically significant difference was observed between the hardness ratio for each material ($p>0.05$).

DISCUSSION

In this study, the Barcoll hardness of resin-based composites cured by different LCUs was measured as an indirect method for evaluating the relative degree of conversion (Mehl, Hickel & Kunzelmann, 1997). The effective cure of resin-based composite is vital, not only to ensure optimum physical-mechanical properties (Asmussen, 1982a), 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).

In this study, the total irradiance of tested LED LCU and halogen LCU was 400 $mWcm^2$ and 600 $mWcm^2$, respectively. However, it remains surprising that the Barcoll hardness results of the resin-based composites cured by LED LCU were higher than the Barcoll hardness results of resin-based composites cured by halogen LCU. To understand this phenomenon, one has to look at the total irradiance of LED LCU and halogen LCU. The total irradiance of the halogen LCU was 0.66 times higher than the irradiance of the LED LCU. One might therefore expect the hardness of resin-based composites cured by the halogen LCU to be superior to those cured by LED LCU, but the situation is, in fact, not so simple. The rate of light-induced chain polymerization is proportional to the square root of the light intensity. In other words, the rate of polymerization increases only 1.44 times when the intensity is doubled (Kloosterboer & Litjen, 1990). The effectiveness of polymerization not only depends on the total irradiance, but also upon the chemistry of the material. In addition, polymerization depends on the exposure time, position, diameter of the light cure tip and depth and shade of resin composite (Harrington, Wilson & Shortall, 1996). Thus, A3 shade was selected to minimize the effects of colorants on light polymerization in this study. The composite material investigated and the distance of the light-cure tip from the composite was standardized 1 mm via usage glass slide. Two-mm thick composite specimens were used, as they ensured uniform and maximum polymerization (Yap, 2000). Also, in both light-curing methods, 40 seconds exposure time was applied. All these factors were kept constant during the curing of both LCUs in this study, but there was a difference in hardness results obtained by the cure LED LCU or halogen LCU. Therefore, an assessment of the LED LCU and halogen LCU performance-only based on a comparison of irradiances may not be straightforward.

The wavelength of emitted light is another important factor in determining the efficiency of the light source in composite curing. Nomoto (1997) found that in the 450-490-nm wavelength range, the degree of conversion was only weakly sensitive to wavelength and the light intensity within this range was more important than the peak wavelength. Outside this range, however, the wavelength dependence was much stronger and the degree of conversion dropped rapidly. The development of new blue superbright LEDs of 470-nm wavelengths comes as an alternative to standard halogen LCUs of 450-470-nm wavelengths (Stahl & others, 2000; Knezevic & others, 2001). In this study, the wavelength of LED LCU was 440-490 nm and the hardness values of LED LCUs were better than halogen LCU values at 2-mm depth. The important advantage of LED LCUs was the possibility for choosing the most efficient wavelength, justifying the very narrow wavelength preference to camphorquinone.

Jandt and others (2000) found that LED LCU containing 27 blue LEDs had the ability to polymerize resin-based composite materials. Fusibayashi and others (1996) demonstrated effective curing depth and degree of conversion using an LCU consisting of 61 blue

LEDs. Dunn and Bush (2002) claimed that poorer performance of an LED LCU would be its lower blue LED, so this LCU, containing seven LEDs, may not adequately cure 2-mm increments of the resin-based composite. The 19 blue LEDs used in this study may be the result of the higher hardness obtained by LED LCU rather than halogen LCU. Therefore, increasing the number of LEDs has improved power output.

The Barcoll hardness for the upper surface of all resin-based composite specimens cured by both LED LCU and halogen LCU was greater than the lower surface of resin-based composite specimens used in this study. The amount of light reaching decreased and setting at the lower surface may not be sufficient to explain this result (Pires & others, 1993; Hansen & Asmussen, 1997). The degree to which light activated resin-based composite polymerizes is proportional to the amount of light to which the material is exposed (Rueggeberg, Caughman & Curtis, 1994). In the instance of the upper surface of the resin-based composites, where no overlying composites interfere with light transmission, it has been found that even a curing source with relatively low intensity can cure the resin matrix to an extent almost equal to that when high-intensity lights are used (Rueggeberg & Jordan, 1993). This result may be due to the sufficient light energy obtained for the upper surface.

In the specimens cured by both LED LCU and halogen LCU, the lowest hardness value was obtained by flowable composite, Filtek Flow, while Esthet-X had the highest hardness values. This can be primarily explained by the difference in the composition of resin matrix, filler size, filler volume and filler type of the resin composites. An increase in filler volume and polymerization of resin leads to increased hardness (O'Brien, 1997).

Comparing packable and hybrid composites, the highest hardness values were obtained from both Surefil packable and Esthet-X hybrid composites utilizing LED LCU. That result can be explained by reason that the hardness could be specific to a given composite due to its unique combination of filler, resin characteristics and formulation.

Although Filtek Z250 and Filtek P60 have the same molecules in their organic matrices, they showed different hardness values, especially at lower surfaces. This difference in resin-based composites based on urethane dimethacrylates is due to the fact that these materials differ in many other aspects, for example, amount of filler, initiators and silanation of the filler particles.

In the ideal situation, the degree of polymerization of the resin-based composite should be the same throughout its depth, and the hardness ratio should be equal to 1:1 or very close to it. As light passes through the bulk of the composite, the light intensity is greatly reduced due to light scattering, thus decreasing the effectiveness of polymerization (Ruyter & Oysaed, 1982). This scattering of light accounts for the minor differences in hardness between the upper and lower surfaces of resin-based composites evaluated when specimens were 2-mm deep. It has been suggested that the upper-to-lower hardness gradient should not exceed 10-20% (hardness ratio should be greater than 0.8) for adequately polymerized light activated resin-based composite (Yearn, 1985; Pilo & Cardash, 1992). In this study, the mean hardness ratio of all materials cured with LED or halogen LCU was 0.96 and 0.98. These findings have demonstrated that adequate polymerization using LED or halogen LCU was obtained at 2-mm depths for all resin-based composites.

Consequently, this study has also shown that LED LCU should have sufficient irradiance to cure resin-based composites (hybrid, packable, ormocer) with curing times of 40 seconds at 2-mm depth. LED LCU polymerization was superior to halogen LCU in that the adherent advantages of LED LCU technology offers a good alternative to conventional techniques. However, further testing, such as measurement of the depth of cure or material structure and properties of dentin-resin interfaces cured with LED technology or material shrinkage, are required.

CONCLUSIONS

1. Comparing LED LCU and halogen LCU curing regimes, the Barcoll hardness of all tested resin-based composite specimens did not show statistically significant differences between the upper and lower surfaces.
2. Flowable composite showed the lowest hardness values compared to tested resin-based composites without being dependent upon the difference in LCUs.

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The Antimicrobial Activity of a Dentin Conditioner Combined with Antibacterial Agents

MG Botelho

Clinical Relevance

The use of a cetrimide containing glass ionomer cement dentin conditioner may be useful in eliminating residual cariogenic bacteria.

SUMMARY

Dental hand instruments are not efficient in removing all infected dentin when performing carious removal for minimal intervention techniques. The use of an antibacterial dentin conditioner may therefore be useful when restoring cavities that have residual carious dentin. Antibacterial agents—chlorhexidine hydrochloride, cetylpyridinium chloride, cetrimide, benzalkonium chloride and sodium hypochlorite, were added either to a dentin conditioner used for glass ionomer cements or distilled water at 1% concentration. Dentin conditioning solutions at pH 2.5, 4.9 and 7.7 were also prepared, along with 1% aqueous thymol. Using an agar diffusion test, 25 µl aliquots were examined for their inhibitory effects on three cariogenic bacteria. After 24 hours, an agar pellet was extracted adjacent to the agar well and placed on a second inoculated agar plate to observe sustained inhibitory effects, after which this procedure was repeated one more time. Antibacterial dentin conditioners showed significant inhibitory effect compared to the control over the three test periods ($p<0.016$). The combination of dentin conditioners with antibacterial agents significantly reduced the inhibitory effect compared to the antibacterial aqueous solutions ($p<0.016$). One-percent aqueous thymol showed no inhibitory effect against the test bacteria. The cetrimide-dentin conditioner showed the greatest inhibitory effect against all three test bacteria over the three experimental periods ($p<0.016$). The inhibitory effect of antibacterial agents was significantly reduced when combined with a dentin conditioner. Only the cetrimide-dentin conditioner combination produced significant inhibitory effects against all three test organisms.

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INTRODUCTION

The atraumatic restorative technique (ART) utilizes dental hand instruments for the treatment of caries and restoration with glass ionomer cement (GIC); this is advocated for environments with limited dental resources. However, dental hand instruments have been shown to be less effective in the removal of dentin caries when compared with rotary burs (Terashima & others,

1969), and it is almost inevitable that residual bacteria will be present prior to placement of the restoration.

Some studies have shown resin-based restorative materials to be effective at sealing in dental caries (Handelman & others, 1987; Mertz-Fairhurst & others, 1998); however, the effectiveness of GICs in this regard may be questioned given that cariogenic organisms may remain viable under GIC for up to two years (Weerheijm & others, 1993; Weerheijm & Groen, 1999). In addition, a number of recent studies have shown secondary caries to be one of the common causes of failure of GIC restorations (Burke & others, 1999; Mjör, 1997), despite GICs being considered anticariogenic materials (Pereira, Inokoshi & Tagami, 1998; Qvist & others, 1997). Therapeutic benefit may therefore be gained in using an antibacterial component in GIC restorative procedures whenever the possibility of residual infected dentin exists, in order to prevent secondary caries.

While GIC materials have been shown to have an inherent antibacterial activity when freshly mixed (Coogan & Creaven, 1993; McComb & Ericson, 1987; Tobias, Browne & Wilson, 1985), when set, these materials demonstrate no apparent antibacterial action (Botelho, 2003; Yap, Khor & Foo, 1999). Antibacterial agents have also been added to GICs and have been shown to have significant antibacterial action (Botelho, 2003; Jedrychowski, Caputo & Kerper, 1983); however, it appears that the incorporation of these agents significantly affects the material's physical properties (Botelho, 1998). To overcome such adverse effects, the use of an antibacterial conditioning agent would allow targeting of the antimicrobial to infected dentin while unlikely affecting the physical properties of the GIC. The antimicrobial effect of antibacterial conditioners for resin composite systems has been reported (Chan & Hui, 1992; Chan & Lo, 1994; Grobler, Basson & Rossouw, 1996); however, no studies have reported on the use of antibacterial GIC conditioners.

Cationic disinfectants have been shown to be highly effective in inhibiting oral micro-organisms in both *in vitro* and *in vivo* studies (Gjermo, Baastad & Rölla, 1970; Hennessey, 1977). Sodium hypochlorite has been used widely as a surface disinfectant (Rutala & Weber, 1999) and a root canal irrigant (Kuruvilla & Kamath, 1998). Thymol has been recommended for use as a mouthwash (Fine, Letizia & Mandel, 1985), and more recently as an anti-caries varnish when combined with chlorhexidine (Petersson & others, 2000; Twetman & Petersson, 1999).

It is known that the antimicrobial activity of cationic antiplaque agents observed *in vitro* does not correlate well with clinical activity (Gjermo & others, 1970). This is related to the pharmacodynamics of adsorption and the release of antibacterial agents (Bonesvoll & Gjermo, 1978) and the modifying effects that bacteria growing in biofilms is known to have on antibacterial agents (Costerton & others, 1994). However, as there appears to be no accepted carious tooth model at present, it may be worthwhile to perform simple *in vitro* tests to help determine whether further investigations are warranted.

This study investigated the effects of incorporating six different antibacterial agents with a GIC dentin conditioner on three bacteria associated with dental caries using an agar diffusion test. One percent aqueous thymol was used as one of the antibacterial agents; previous research having shown that thymol had no observable antibacterial effect below 256 µg/ml concentration (Botelho, 2000).

METHODS AND MATERIALS

Six antibacterial agents used in clinical dentistry were investigated in this study. They are: chlorhexidine hydrochloride (CX), cetylpyridinium chloride (CP), cetrimide (CT), benzalkonium chloride (BC) (Sigma Chemical Co, St Louis, MO, USA), sodium hypochlorite (SH) and thymol (TY) (BDH Lab Supplies, Poole, England). These agents were prepared to standard 5% (w/v) aqueous solutions, and from these, 1% (w/v) aqueous solutions were prepared and 1% (w/v) in dentin conditioner (Dentin Conditioner, GC Corporation, Tokyo, Japan), a 10% polyacrylic acid solution. However, thymol formed a supersaturated aqueous solution at 1%, and, as preliminary investigations showed that even in this state thymol showed no antibacterial activity, it was excluded from further investigation.

To investigate the possibility that the pH of polyacrylic acid dentin conditioner would have an inhibitory effect on test bacteria, the dentin conditioner was prepared to different pH values between pH 4.5 and pH 7.5 using sodium hydroxide. An equivalent volume of sterilized distilled water was also added to a control conditioner to take into account the dilution factor produced by adding the sodium hydroxide. This diluted dentin conditioner produced a pH of 2.5. The pH values of the conditioners were measured with an electronic pH meter (Orion Combination pH Electrode Model 91, Orion Research, Incorp, MA, USA) and recorded on a digital pH meter (Orion Model 501, Orion Research). Prior to the pH, the electrode was calibrated with a pH 4 buffer solution (Sigma Chemical Co).

Genera of bacteria that have been commonly associated with active caries were selected (Edwardsson, 1974; Hoshino, 1985) including *Actinomyces naeslundi*, *Lactobacillius casei subsp casei* and *Streptococci mutans* (gifts from Professors S Edwardsson, G Bowden and J Hardie, respectively).

Bacterial inocula were prepared from a 24-hour blood agar culture in a candle jar. Organisms were harvested and re-suspended in sterile brain-heart infusion media (Oxoid, Unipath Ltd, Bedford, UK) to produce a MacFarland 0.5 optical density (equivalent to 10^8 colony forming units). From this, 20 µl was dispersed

onto TSA agar (Oxoid, Unipath Ltd) plates using the Autoplate 4000 Spiral Plater (Spiral System Inc, Cincinnati, OH, USA). A 5-mm diameter stainless steel punch was used to cut wells in the inoculated agar surface, which was later removed using a sterile glass pipette under vacuum.

Each antibacterial agent and pH adjusted dentin conditioner was tested in four wells on individual agar plates for each bacterium under investigation. The plates were then incubated in a candle jar for 24 hours. After the first experimental period (T1), the diameter of the inhibition zone was measured using a dial caliper (±0.1 mm) and the surface area of inhibition determined. Twenty-four hours after the first inoculation, the stainless steel punch was used to cut a plug of agar directly adjacent to the well into which the antibacterial agents had diffused. The plug was then transferred onto a fresh TSA agar plate that had been inoculated with an identical test organism. This was incubated again in a candle jar for 24 hours (T2), after which the zone of inhibition was measured and its area calculated. The stainless steel punch was used for the third and final time to remove a plug of agar from beneath the previous agar plug. This was then transferred to a final TSA agar plate, again inoculated with the same organism and incubated for 24 hours. After this period (T3), any area of inhibition was measured. Figure 1 shows the summary of this experimental procedure.

The cumulative data for the three bacteria over the three test intervals were analyzed to determine differences between antibacterial agents. Statistical analysis was performed using SPSS software (SPSS Inc, Chicago, IL, USA), applying a one-way analysis of variance (ANOVA) and Tukey's studentized test with an adjusted level of significance set at $p<0.016$ due to multiple testing (Bonferroni's correction) for the three added time intervals (T1 to T3). A paired *t*-test was also performed to determine differences between the antibacterial solutions and conditioners with significance also set at $p<0.016$.

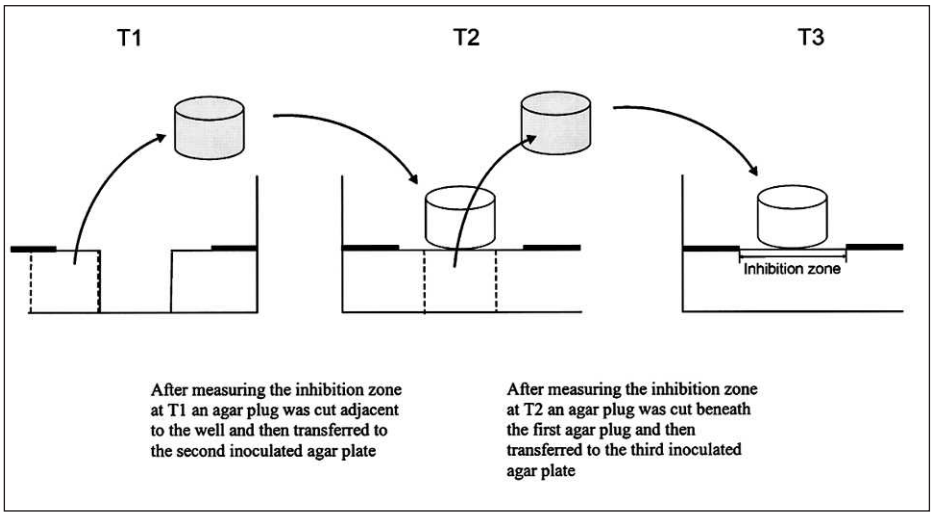

Figure 1. *Diagrammatic representation of experimental procedure.*

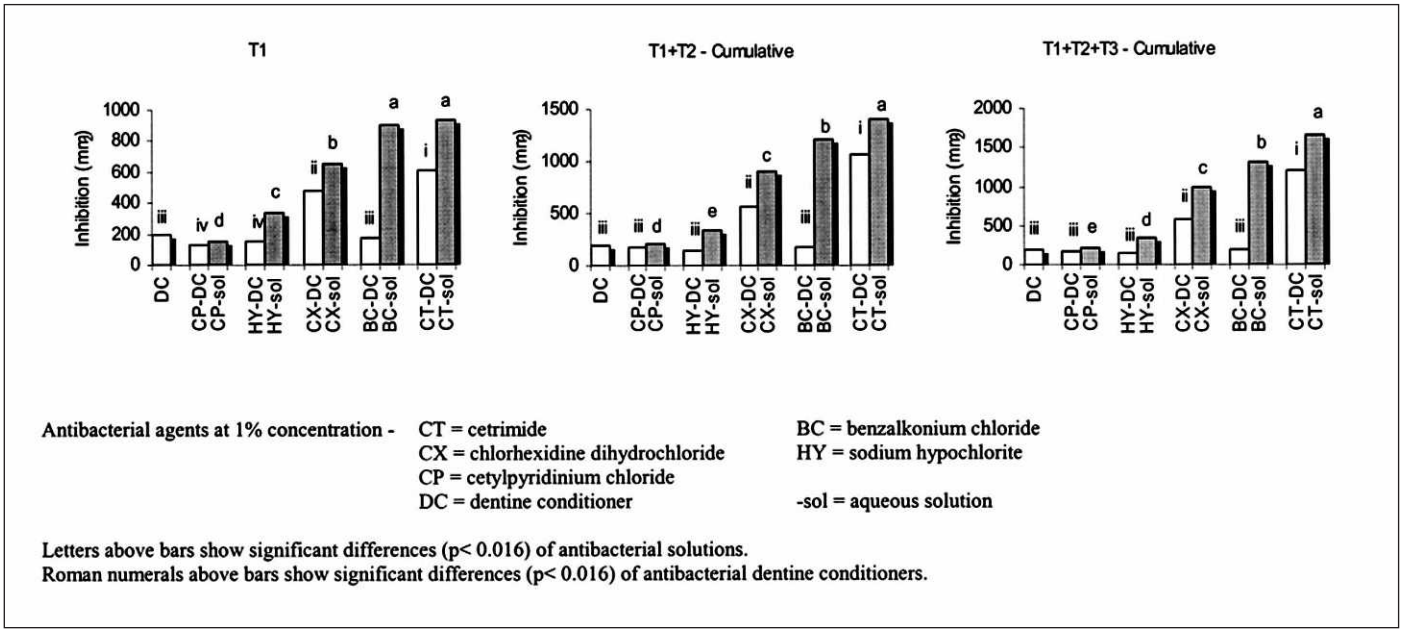

Figure 2. *Inhibition (mm_2) of A. naeslundii by test agents at three time intervals.*

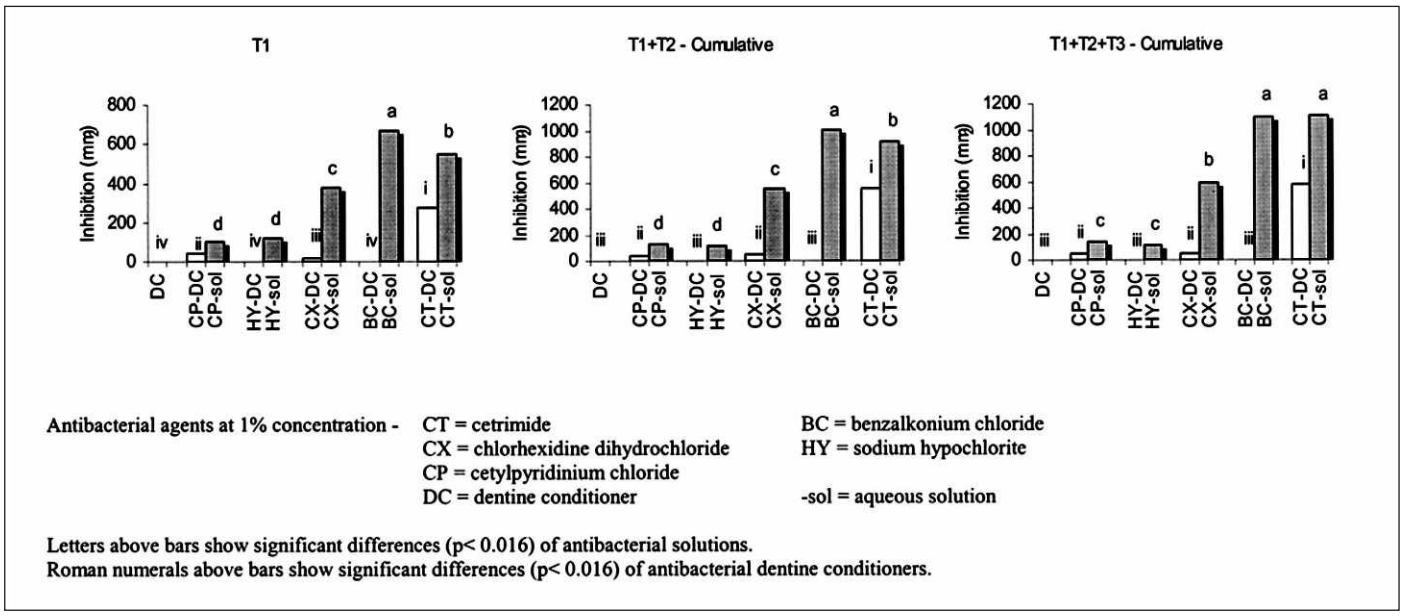

Figure 3. *Inhibition (mm_2) of* L casei subsp casei *by test agents at three time intervals.*

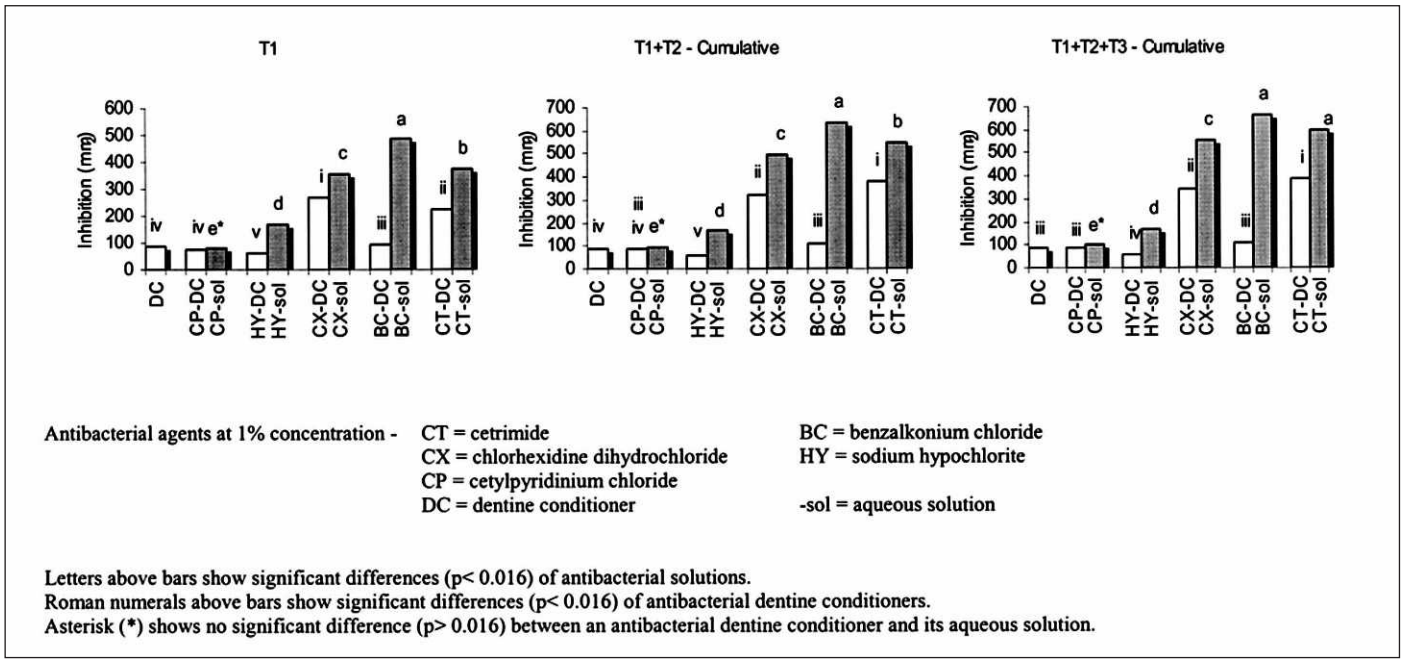

Figure 4. *Inhibition (mm_2) of* S mutans *by test agents in an ADT at three time intervals.*

Effectiveness of the antibacterial dentin conditioner was also determined relative to its aqueous solution to ascertain the extent to which the dentin conditioner had affected the action of the antibacterial agent.

RESULTS

One-way ANOVA showed that there were significant differences among the antibacterial test groups for all three experimental time periods ($p<0.016$). The inhibitory effects of the test solutions over the three time periods are shown in Figures 2 through 4. All antibacterial solutions showed significantly greater inhibitory effects than their respective antibacterial-dentin conditioners at time intervals ($p<0.016$) apart from CP-solution when tested against the *S mutans* (Figure 4).

Table 1: *Mean area of bacterial inhibition in mm² (SD) for the antibacterial solutions (-sol) and antibacterial dentin conditioners (-DC) for each of the three experimental periods (T1-T3). The data for pH-adjusted conditioners and control only include T1, as there was no further inhibitory effect after this time interval.*

Bacteria	Agent	T1		T2		T3		Agent	T1		T2		T3		Agent	T1	
A	BC-sol	904	(14)	307	(14)	93	(7)	BC-DC	176	(4)	7	(8)	0		DC-	245	(8)
L		674	(10)	335	(7)	90	(19)		0	0	0		0		pH2.5	25	(2)
S		489	(2)	143	(3)	29	(12)		96	(6)	10	(7)	0			148	(9)
A	CP-sol	158	(7)	47	(4)	14	(2)	CP-DC	133	(4)	40	(21)	3	(3)	DC-	108	(7)
L		100	(2)	30	(1)	10	(2)		41	(2)	13	(12)	0		pH4.9	0	
S		78	(3)	18	(3)	5	(1)		78	(3)	12	(3)	0			39	(1)
A	CT-sol	934	(14)	462	(28)	254	(8)	CT-DC	608	(14)	462	(20)	132	(62)	DC-	11	(2)
L		554	(11)	361	(10)	198	(16)		273	(6)	276	(19)	34	(13)	pH7.7	157	(10)
S		376	(7)	169	(14)	57	(4)		228	(6)	149	(7)	4	(6)		14	(1)
A	CX-sol	654	(11)	247	(23)	81	(7)	CX-DC	483	(17)	86	(5)	17	(8)	DC-	192	(9)
L		380	(11)	169	(28)	48	(16)		20	(1)	36	(3)	0		Control	0	
S		357	(3)	138	(3)	57	(2)			266	(1)	57	(6)	20	(7)	86	(6)
A	HY-sol	341	(19)	1	(3)	0		HY-DC	147	(2)	3	(4)	0				
L		118	(3)	0		0			0	0	0		0				
S		166	(7)	0		0			61	(7)	0		0				
A								DC	192	(9)	0		0				
L									0	0	0		0				
S									86	(6)	0		0				

A = A naeslundii *L* = L. casei subsp casei *S* = S mutans. *CT = cetrimide, BC = benzalkonium chloride, CX = chlorhexidine dihydrochloride, HY = sodium hypochlorite, CP = cetylpyridinium chloride.*

Table 2: *Inhibitory Effect of the Antibacterial-dentin Conditioner When Compared to Its Antibacterial Solution When Tested Against Three Genera of Bacteria at T1 Investigatory Period*

Bacteria	Antibacterial	Inhibition mm² Aqueous	Antibacterial Dentin Solution	Inhibition mm² Conditioner	Inhibitory Effect %
A	BC-sol	904	BC-DC	176	19.5
L	674		0	0.0	
S	489		96	19.6	
A	CP-sol	158	CP-DC	133	84.2
L	100		41	41.0	
S	78		78	100.0	
A	CT-sol	934	CT-DC	608	65.1
L	554		273	49.3	
S	376		228	60.7	
A	CX-sol	654	CX-DC	483	73.9
L	380		20	5.3	
S	357		266	74.5	
A	HY-sol	341	HY-DC	147	43.1
L	118		0	0.00	
S	166		61	36.75	

A = A naeslundii *L* = L casei *subsp* casei S = S mutans *-sol = aqueous solution, -DC = dentin conditioner*

Of the aqueous solutions, CT and BC solutions showed the greatest ($p<0.016$) cumulative inhibitory effects against the test bacteria over the test intervals. CP solution had the least inhibitory effect ($p<0.016$) of all the antibacterial solutions against *S mutans* and *A naeslundii*. However, with the lactobacillus organism, it was not significantly different from the HY solution. The CX solution only showed the third highest ($p<0.016$) inhibitory effects against all three bacteria.

The CT and CX dentin conditioners showed the greatest and second greatest ($p<0.016$) inhibitory effects against the three test bacteria over the three test periods. At all

three test intervals, CP, HY and BC antibacterial dentin conditioners showed no significantly greater inhibitory effects ($p>0.016$) than the control dentin conditioner against either *S mutans* or *A naeslundii*. Against *L casei subsp casei*, BC-dentin conditioner, HY-dentin conditioner and the control showed no inhibitory effects over the three test intervals. The CX-dentin conditioner showed minimal inhibitory effect.

None of the dentin conditioners showed any inhibitory effect after T1. Both *A naeslundii* and *S mutans* were increasingly inhibited with increasing acidity of the dentin conditioner (Table 1). *L casei subsp casei* showed no inhibition with respect to the control and pH 4.9 dentin conditioner, however, the greatest inhibitory effect was observed with the pH 7.7 dentin conditioner ($p<0.016$) (Table 1).

A comparison of the efficiency of the antibacterial dentin conditioners when compared to its aqueous antibacterial solution is shown in Table 2. The inhibitory effects of the antibacterial agents against the *actinomyces* and *streptococcus* species showed similar effects for all the antibacterial agents. In the case of the *lactobacillus* species, this showed a much lower inhibitory effect of the antibacterials when the dentin conditioner was compared to its aqueous solution. The benzalkonium chloride dentin conditioner showed the greatest reduction in inhibitory effect when compared to its aqueous solution.

DISCUSSION

The combination of antibacterial agents with a dentin conditioner shows marked inhibitory effects; however, these were significantly less than the corresponding aqueous solutions. This is likely to be due to either a pH effect caused by the acidic Dentin Conditioner or a possible interaction effect between the polyacrylic acid and antibacterial agents affecting the antibacterial efficacy. The effects of reduced pH have been shown to reduce bacterial activity (Kostenbauder, 1991) and the antibacterial action of cationic antimicrobials (Cleghorn & Bowden, 1989; Martindale, 1999; Turesky & others, 1973). This is supported by previous reported observations that chlorhexidine is most inhibitory at higher (pH 6-10) than lower pH values (Turesky & others, 1973). However, against *lactobacillus* strains, chlorhexidine was observed to be less inhibitory, as the pH was lowered from pH 7.4 to 5.0 (Cleghorn & Bowden, 1989). Cetrimide was least affected by combination with the dentin conditioner, with it showing significant antimicrobial effect over the three test periods; this may be related to a greater stability of the agent at lower pH. Adverse interactions between chlorhexidine and a phosphoric acid etchant have been observed to form a white precipitate "over time" (Chan & Lo, 1994), suggesting a salt formation between the chlorhexidine and phosphate groups of the acid.

A cavity disinfectant with 2% chlorhexidine gel is available under the name of Consepsis (Ultradent, South Jordan, UT, USA). Although not extensive, what literature there is on this product has shown it to have good wetting properties and increased bond strength (Cao & others, 1995) and reduces bacterial ingress into dentinal tubules (Brännström, 1987); however, its clinical effect on cariogenic organisms is not known.

Studies investigating the antibacterial effects of freshly mixed GICs on oral bacteria *in vitro* have reported that *Lactobacilli* strains appear to be among the most resistant oral bacteria, in particular *L casei* (Herrera & others, 1999; Palenik & others, 1992). The *L casei subsp casei* species in this study was also resistant to the inhibitory effects of antibacterial dentin conditioners tested apart from the CT-dentin conditioner. This resistance may be related to the acidophilic nature of the bacteria, as it is known to lower pH *in vitro* (van Houte, Lopman & Kent, 1996) and is associated with the advancing demineralizing front in carious lesions. This observation appears to be supported by the observation that the least acidic dentin conditioner (pH 7.7) had the greatest inhibitory effect on the *Lactobacilli* species.

The use of cetrimide in a GIC dentin conditioner may therefore prove beneficial in eliminating bacteria associated with residual caries, particularly as *lactobacilli* are associated with primary caries (Edwardsson, 1974) and have the ability to survive under GIC restorations (Weerheijm & others, 1999).

The control dentin conditioner and pH adjusted dentin conditioners showed no inhibitory effect after T1. This most likely reflects a neutralization of its inhibitory pH effect after diffusing into the surrounding agar that then had no sustained antibacterial action when transferred to the second agar plate.

The action of the pH inhibitory effect is supported by the observation that the lower the pH of the dentin conditioner, the greater the bacterial inhibitory effect seen, except in the case of the acidophilic *lactobacillus* species.

While *in vitro* investigations have shown thymol or its derivatives to have antibacterial activity (Kulevanova & others, 2000; Shapiro, Meier & Guggenheim, 1994), other studies have reported thymol to have little or no inhibitory effect (Botelho, 2000). Furthermore, on some bacteria, they have been reported to stimulate growth and plaque formation (Evans & others, 1977). Under the limitations of the test conditions, low concentrations of aqueous thymol appear to have little or no observable bacterial inhibitory effect. The use of a sodium hypochlorite in a dentin conditioner, while a proven surface disinfectant, may not be a viable consideration given its known deterioration over time, particularly at low pH (Cotter & others, 1985).

CONCLUSIONS

Cetrimide had the greatest inhibitory effect when combined with Dentin Conditioner. It also showed a significant and sustained effect against the *lactobacillus* species, which was not observed with other antibacterial agents. As *lactobacilli* are known to be associated with the advancing carious front and survive under GIC restorations for prolonged periods of time (Weerheijm & others, 1993), the use of a CT-dentin conditioner for GICs may be beneficial. However, it is not known whether CT would be effective *in situ* against a complex ecology of cariogenic organisms. Furthermore, *in vitro* investigations are needed before clinical use.

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The Relationship Between the Color of Carious Dentin Stained with a Caries Detector Dye and Bacterial Infection

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Clinical Relevance

From anassessment of an *in vitro* objective method for the clinical evaluation of carious dentin using colorimetry, the rates of bacterial detection in caries were inversely related to the lightness of the carious dentin stained with a caries detector dye.

SUMMARY

This *in vitro* study aimed to design a method for the objective evaluation of carious dentin using numerical values. This study also investigated the relationship between the color of carious dentin stained with a caries detector dye using this objective method and the rate of bacterial detection as detected by a polymerase chain reaction (PCR). In 15 molars with occlusal dentin caries and three extracted sound molars, dentin was removed in multiple steps with 300 µm removed each step. Before and after every removal, images of a color-matching sticker and carious surfaces stained with a caries detector dye were acquired simultaneously using a CCD camera and dentinal tissue samples were removed with a round bur. Next, corrected L*, a* and b* values of the carious surfaces (CIE 1976 L*a*b* color system) were calculated from the color changes of the stickers in the images. In addition, bacterial DNA in the dentinal tissue was detected by PCR. From evaluations of the receiver operating characteristic curves for the L*, a* and b* values, the L* value was determined to be a more useful parameter than a* or b* for detecting bacterial infection using the caries detector dye. The bacterial detection rates of carious dentin decreased as the L* values of carious dentin stained with the dye increased. When the L* values were more than 60, the dentin had no bacterial infection. This study clarified the relationship between the colors of lesions stained with a caries detector dye and the rates of bacterial detection.

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INTRODUCTION

The color of caries evaluated by visual inspection and the hardness of the lesions detected with an excavator has been used for the diagnosis of dentin caries in clinical situations (Sturdevant & others, 1994). As these diagnostic methods are quite subjective, the reproducibility of the diagnostic results is low.

On the other hand, the use of a caries detector dye has sometimes been recommended when caries is removed (Anderson, Loesche & Charbeneau, 1985; Van de Rijke, 1991; Thomas & others, 2000), since staining with such dyes allows for easy clinical discrimination of caries in comparison to a visual inspection without staining. Generally, the components of such dyes include 1% acid red in propylene glycol (Fusayama, 1988). However, when a caries detector dye was used to remove caries, excessive removal of dentin (Yip, Stevenson & Beeley, 1994) or bacteria present in the remaining dentin (Boston & Graver, 1989) was reported. It has been suggested that, although most bacterial infection in caries is detected by caries detector dyes, the detection is not absolute (Boston & Graver, 1989), that is, caries detector dyes do not stain some cariogenic bacteria; instead, they stain the dentinal structure destroyed and demineralized by bacterial metabolites (Anderson & others, 1985). Therefore, Fukushima (1981) and Sano (1987) investigated the evaluation conditions and recommended that if dentin was stained light pink, due to the absence of bacteria, it should not be removed in order to prevent excessive dentin removal. However, the use of a caries detector dye does not provide a completely objective method, especially for the removal of deep caries layers close to the pulp, since the degree of light pink staining of dentin in the deep layers is difficult to evaluate objectively by visual inspection. In addition, there is some doubt regarding the relationship between the results using a caries detector dye and the extent of bacterial infection (McComb, 2000).

Several additional developments have involved dental adhesion (Moll, Gärtner & Haller, 2002) and remineralization of tooth surfaces prior to cavitation (Artun & Thylstrup, 1989; Johansen & others, 1987). As a result, when caries is removed and the cavity is restored, the volume of enamel and dentin removed should be minimized through control of the cariogenic bacteria and treatment to stimulate remineralization. Based on this information, the concept of minimal intervention dentistry has been advocated (Tyas & others, 2000; Mount & Ngo, 2000). Thus, for minimal removal of caries during clinical restorative treatment, more objective diagnostic methods using numerical values and objective clinical standards are required. In addition, investigation of the relationship between the results of the objective diagnostic method and bacterial infection is required.

The objectives of this *in vitro* study were (1) to design a method for objective quantitative colorimetric evaluation of carious dentin stained with a caries detector dye and (2) to investigate the relationship between the numerical values obtained and the rates of bacterial detection assessed using polymerase chain reaction (PCR).

METHODS AND MATERIALS

Color Image of the Dentin Surface and Dentinal Tissue Sampling

In this study, the authors selected 15 carious and three sound extracted human molars, with the sound molars serving as controls. The carious molars had dentin caries on the occlusal surface. The degree of dentin caries varied and included both cases with and without a carious cavity. The dentin caries were clearly stained with a caries detector dye (1% acid red in propylene glycol, Caries Detector, Kuraray Medical Inc, Okayama, Japan) and were without exposed pulp tissue. Molars that had natural staining and those with hypoplasia were excluded. Following extraction, the molars were stored in physiological saline at 4°C and used within 24 hours. All molars were ground from the occlusal surface with a polishing machine (Ecomet III, Buehler Ltd, Lake Bluff, IL, USA) until the dentin caries was exposed. The root of each molar was fixed in a handmade silicone mold with self-curing acrylic resin (Uni-Fast II, GC Corporation, Tokyo, Japan). After the resin had hardened, the molars were removed from the molds.

Each molar was placed on a standardized cavity preparation device (Itoh Engineering Co, Kyoto, Japan)

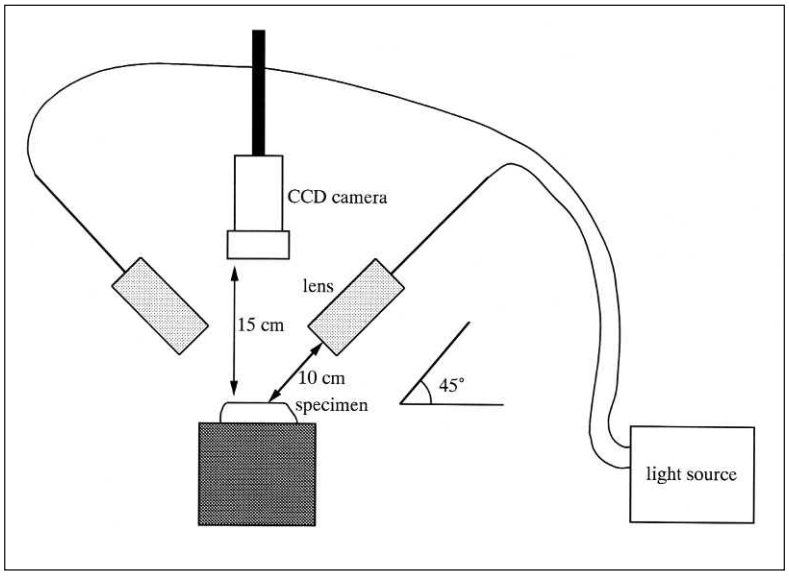

Figure 1. *Schema of image acquisition with a CCD camera.*

(Iwami, Yamamoto & Ebisu, 2000) that had three micrometers controlling the movement of a handpiece with a round bur. The caries detector dye was applied to the ground carious dentin surface of the molar for 10 seconds. The surface was rinsed with water and air dried using a three-way syringe (Fukushima, 1981; Sano, 1987). Next, for color evaluation, the image of the molar surface together with a color-matching sticker (Casmatch, Dai Nippon Printing Co Ltd, Tokyo, Japan; details of these stickers are described in the next section) was acquired using an operation microscope (Universal S3, Carl Zeiss, Oberkochen, Germany) with a CCD camera (Hitachi, Tokyo, Japan). The magnification of the images was 15x. The distance between the two standard light sources (Cold Spot, PCS-HRX-150, Nippon PI Corp, Tokyo, Japan; color temperature: 3100 K) and the molar surface was 10 cm, and the angle between the light sources and the molar surface was 45° (Figure 1). The angle was based on CIE Technical Report No 15.2 (1986). The illumination of the test site was approximately 1,000 lx as measured using an illuminometer (T-10M, Minolta, Osaka, Japan). After collecting an image of the molar surface, the dentinal tissue of the test area was reduced in thickness by 300 µm from the molar surface with a #5 round bur set using the standardized cavity preparation device, and the readings of the micrometers of the device were recorded. From the readings taken before and after thinning, the relative portion of the test site was determined for each molar. In addition, the dentinal tissue on the round bur was used for detection of bacterial infection using a PCR technique. The sequence of grinding down the surface by 300 µm in the direction of the pulp chamber, taking the readings of the micrometers and collecting an image of the molar surface was repeated until the sound dentin of the carious molars appeared or the total removal of tissue reached 3 mm for the sound molars. The round bur was changed to a new one after each removal of 300 µm of dentin.

Dentin Color Correction

The images of the molar surfaces were transferred to a personal computer (iBook Special Edition, Apple Computer Inc, Cupertino, CA, USA). The color of the caries or sound dentin in the image was corrected by referencing the color changes of the associated stickers that had known color values. The values of L*, a*, b*, as defined by the CIE 1976 L*a*b* color system (CIE Technical Report No 15.2, 1986), were used for the color evaluation. L* is the psychometric lightness and shows the lightness of an object with a range from 0 to 100. a* and b* are the psychometric chromaticness or color coordinates, where a* shows the coordinate of green - red and b* showed that of blue - yellow. The ranges of a* and b* are both from -128 to 127.

Color correction was undertaken on a computer via a series of steps. The color-matching stickers consisted of nine colors—red, green, blue, yellow, magenta, cyan, black, gray and white. The real L*, a* and b* values of these colors were measured six times with a colorimeter (CR-100, Minolta, Tokyo, Japan) and their mean values (L, a, b) were used for the next step. After the digital image of each test surface together with its color-matching sticker had been transferred to the computer, the image files were opened with a color retouching software program (Adobe Photoshop version 5.0, Adobe Systems, San Jose, CA, USA) and the L*, a* and b* values of the test sites and color-matching stickers were measured six times using the software. The mean values of the test sites (L_{t1}, a_{t1}, b_{t1}) and those of the color-matching stickers (L_1, a_1, b_1) were then calculated.

The relationships between L, a, b and L_1, a_1, b_1 using an approximate polynomial formula with a 3 x 3 matrix were defined by Ohta (1997). The matrix of the formula was calculated by a least squared method. Therefore, the corrected color values after color reproduction (L_{t2}, a_{t2}, b_{t2}) were calculated using the approximate polynomial formula (1) with this matrix (g_{1L} ... g_{3b}: elements of the matrix) and the color values of the test sites (L_{t1}, a_{t1}, b_{t1}).

$$\begin{pmatrix} L_{t2} \\ a_{t2} \\ b_{t2} \end{pmatrix} = \begin{pmatrix} g_{1L} & g_{1a} & g_{1b} \\ g_{2L} & g_{2a} & g_{2b} \\ g_{3L} & g_{3a} & g_{3b} \end{pmatrix} \begin{pmatrix} L_{t1} \\ a_{t1} \\ b_{t1} \end{pmatrix} \quad \text{.....(1)}$$

In addition, for evaluation of the accuracy of this color correction, the color differences (ΔE) were calculated in the following manner. The corrected color values of the color-matching sticker (L_2, a_2, b_2) were calculated by substituting the mean color values of the color-matching sticker (L_1, a_1, b_1) for the values (L_{t1}, a_{t1}, b_{t1}) in formula (1). The color differences (ΔE), L* differences (ΔL*), a* differences (Δa*) and b* differences (Δb*) of the color-matching sticker were calculated using formula (2).

$$\Delta L^* = |L - L_2|,\ \Delta a^* = |a - a_2|,\ \Delta b^* = |b - b_2|,$$

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad \text{.....(2)}$$

The mean color differences (ΔE_{ave}), mean L* differences (ΔL^*_{ave}), mean a* differences (Δa^*_{ave}) and mean b* differences (Δb^*_{ave}) of the nine colors were calculated for all teeth.

Detection of Bacterial Infection Using a PCR Technique

The dentinal tissue on the round bur was suspended in 20 µl of DNA isolating buffer (Lyse-N-Go PCR Reagent, Pierce, Rockford, IL, USA) and chromosomal DNA in the tissue was isolated. The dentin suspension was vortexed for 10 seconds in a sealed tube, then heated for 10 minutes at 98°C, followed by centrifugation at 15,000 rpm for 10 minutes at 4°C. Supernatant was used as

the DNA suspension. The primers for the PCR were designed based on the nucleotide sequence of a conserved region of the 16S rDNA (Nadkarni & others, 2002). One microliter of the DNA suspension and DNA polymerase (TaKaRa Ex Taq, TaKaRa Biomedicals, Otsu, 520-2193, Japan) were placed in a new tube in a final volume of 20 µl. Standard bacterial suspensions (*Streptococcus mutans* MT8148 at concentrations of 1.0×10^{-6}) were used as positive controls. Next, the tubes containing DNA suspension and those for the negative (distilled water) and positive controls were placed in a thermal cycler (Gene Amp PCR System 9700, PE Applied Biosystems, Foster, CA, USA). The initial stage consisted of 94°C for two minutes, and amplification consisted of 35 cycles of 94°C for 30 seconds, 60°C for 30 seconds and 72°C for one minute. The reaction was terminated at 72°C for five minutes. The PCR amplification products were analyzed by electrophoresis in a 1.5% agarose gel. DNA was visualized with ethylene bromide and UV light produced by a transilluminator (Dinco & Rhenium Industries Ltd, Jerusalem, Israel).

Statistical Analysis

A receiver operating characteristic (ROC) curve was plotted for each color value (L*, a* and b* values). The microbial test by PCR was used as the gold standard for drawing the ROC curve. The area under each ROC curve (Az value) was calculated for each color value and used for comparing the accuracy of the caries diagnosis using the three color values. These

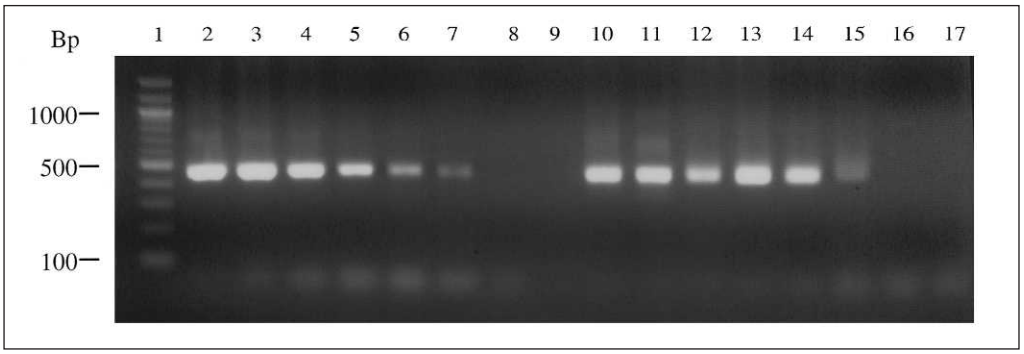

Figure 2. *An example of the electrophoresis results after PCR. A band of 466 bp indicates the presence of the PCR product of the 16S rRNA gene sequence. Lane 1: DNA ladder (100 bp Ladder NEB); lanes 2-8: positive controls (1.0 X 10^6, 10^5, 10^4, 10^3, 10^2, 10^1 and 10^0 cells of* S mutans *MT8148, respectively); lane 9: negative control (sterile distilled water); lanes 10-17: results for each 300 µm of caries dentinal tissue, starting from the dentin surface (for example: lane 10: surface to 300 µm; lane 11: 300 µm from the surface to 600 µm).*

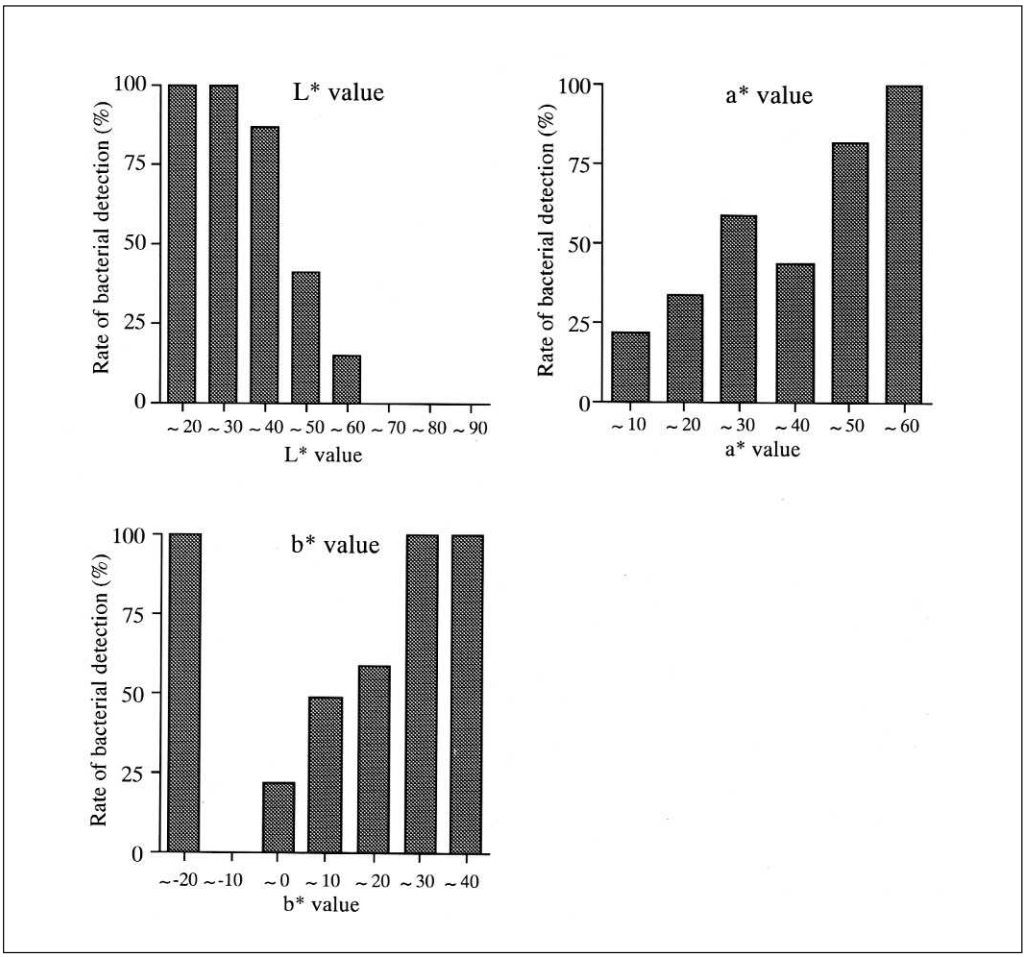

Figure 3. *Relationship between the color values (L*, a*, b*) of the dentin stained with the caries detector dye after color correction, and the rates of bacterial detection using PCR. The L* values increase as the rates of bacterial detection decrease when the L* values are between about 30 to 60. When the L* values are more than about 60, the rates are 0%. The rates tend to increase as the a* values increase. There is no consistent relationship between the rates of bacterial infection and the b* values.*

Az values were analyzed by a univariate Z score test. Statistical significance was set at the 1% probability level.

RESULTS

The mean color difference (ΔE_{ave}), mean L* difference (ΔL^*_{ave}), mean a* difference (Δa^*_{ave}) and mean b* difference (Δb^*_{ave}) of the color-matching stickers in all images between the real L*, a*, b* values of these colors measured by a colorimeter and the corrected color values were calculated. From the results, the range of ΔE_{ave} for the different colors was from 2.3±1.3 (gray) to 10.3±2.4 (black) (mean±SD) and the mean value was 6.5±3.3. The range of ΔL^*_{ave} was from 1.2±1.1 (gray) to 8.2±2.7 (black) and the mean value was 2.4±2.8. The range of Δa^*_{ave} was from 1.2±0.9 (gray) to 4.5±2.7 (yellow) and the mean value was 3.2±2.2. The range of Δb^*_{ave} was from 1.2±0.9 (gray) to 7.3±3.4 (yellow) and the mean value was 4.0±2.9. Therefore, the color differences for black and yellow were greater than for the other colors.

An example of the electrophoretic results after PCR is shown in Figure 2. The PCR product of the 16 S rDNA sequence gave a band of 466 bp. From the electrophoretic results of the positive controls, 1×10^1 or 1×10^2 bacterial cells could be detected by the PCR method used in this study.

In the carious molars, the L* values after color correction (L_{t2}) increased as the distance increased; however, the a* and b* values after color correction (a_{t2}, b_{t2}) showed no correlation with an increase in distance. The L*, a* and b* values in the 15 carious molars ranged from 19.5 to 89.2, from -3.0 to 37.0 and from -26.7 to 23.5 (minimal value to maximal value), respectively. The amount of dentin caries in the caries molars before reduction by round bur varied, because their original L*, a* and b* values were different. On the other hand, the L*, a* and b* values of the three sound molars ranged from 61.5 to 78.8, from -2.8 to 19.5 and from 4.2 to 17.3 (minimal value to maximal value), respectively. The L*, a* and b* values of the sound molars were more consistent than the carious molars. In particular, the L* values (mean ± SD: 68.6±4.1) were very similar.

Figure 3 shows the relationship between the color values of the carious lesions stained with caries detector dye after color correction (L_{t2}, a_{t2}, b_{t2}) and the rates of bacterial detection by PCR (15 carious molars). When the L* values were between approximately 30 to 60, the rates of bacterial detection decreased as the L* values increased. When the L* values were approximately 60 and less than 30, the rates were 0% and 100%, respectively. The rates of bacterial detection tended to increase with increasing a* values. There was no relationship between the b* values and the rates of bacterial detection.

The ROC curves for the three-color values (L*, a* and b* values) for detection of bacterial infection are shown in Figure 4. The closer the ROC curve was to the upper left-hand corner of the graph, the more useful the value. In addition, from the results of a univariate Z score test, the Az value of L* was significantly larger than a* or b* ($p<0.01$). Therefore, L* values are a more useful parameter than a* or b* values for detecting bacterial infection.

DISCUSSION

Carious molars clearly stained with caries detector dye were used in this study to simplify the experimental conditions and thus obtain clear results. In addition, when the dye stains the tooth surface, the color contrast of the caries surface is very evident (Fusayama & Terashima, 1972). Fusayama (1979) reported that typical acute (or active) caries was stained with the caries detector dye, but typical chronic (or arrested) caries was only stained very weakly. Therefore, it is presumed that only molars with typical active caries were used in this study. A future examination using typical arrested caries needs to be carried out.

Streptococcus mutans is thought to be important in the formation of enamel caries (Loesche & Straffon, 1979; Schwartz, Summit & Robbins, 1996). However, for deep dentin caries, many species of bacteria, other facultative anaerobes or some obligate anaerobes have been detected at higher rates than for surface caries, and the rate of *S. mutans* is thought to be lower than in surface caries (Bjorndal & Larsen, 2000; Hoshino, 1985). The role of many species in the formation of deep caries has not been clarified; however, by working together, most probably play some role in the formation of deep caries (Hojo, Takahashi & Yamada, 1991). The evaluation of deep dentin caries is an important target for this study investigating diagnostic aids for removing carious dentin. The PCR method for bacterial detection is suitable for detecting bacterial infection in deep dentin caries, since obligate anaerobes and non-living bacteria can be detected if the bacterial DNAs remain (Becker & others, 2002). Therefore, a universal primer based on the nucleotide sequence in a conserved region of the 16S rDNA was used for the PCR in this study.

Since the conditions for taking images did not follow an ideal Luther condition, which indicates that the spectral sensitivity of an image input unit is a linear equation of the color matching function (Ohta, 1997), color correction was used to calculate approximate corrected values. ΔE^*_{ave} was 6.5±3.3 and ΔL^*_{ave} was 2.4±2.8. However, since the color differences for black and yellow were greater than for the other colors, the selection of a color-matching sticker and the formula used for color correction needs further investigation.

Figure 4 shows that the L* value is a more useful parameter for the evaluation of dentin caries than a*

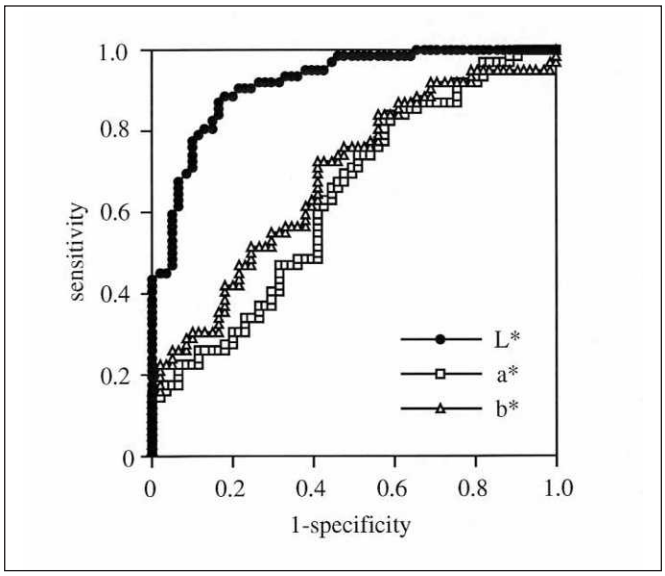

Figure 4. *Receiver operating characteristic curves for the L*, a* and b* values. The areas under each ROC curve (Az value) for the L*, a* and b* values are 0.93, 0.61 and 0.69, respectively.*

and b* values, since the Az value of L* was significantly larger. In this study, since the caries detector dye was used to obtain a high contrast, it was thought that the degree of staining with dye was shown only by the intensity of the red staining. However, for diagnosis of chronic (or arrested) caries or diagnostic discrimination between acute (or active) and chronic caries, a* or b* values might be useful parameters. Therefore, these aspects will be the subjects of future investigations.

From the results shown in Figure 3, lightness (L* values) was related to the intensity of staining with the caries detector dye. The degree of staining of carious dentin correlated with the amount of destruction of dentin by cariogenic bacterial acid or collagenase, since acid red appears to stain demineralized organic matrix rather than the bacteria, themselves (Kuboki, Liu & Fusayama, 1983; Boston & Graver, 1989). To ensure clear results in the experiments in this study, molars with natural staining were not used. It was previously reported that sound circumpulpal dentin and sound dentin at the enamel-dentin junction were stained with caries detector dye due to the higher proportion of organic matrix normally present in these sites (Yip & others, 1994). In this study, sound circumpulpal dentin and sound dentin at the enamel-dentin junction were not intentionally used. Since the L* values of sound molars (controls) and the sound dentin of carious molars were more constant than that of the carious portions, the condition of the dentin specimens used in this study was considered to be quite constant. Therefore, since destruction of the dentin structure in the carious molars used in this study was considered to be primarily caused by bacterial acid and collagenase, bacteria are required for the destruction of dentin in caries and the lightness of the stained dentin was indirectly related to the rate of bacterial detection. However, stained circumpulpal dentin and dentin at the enamel-dentin junction are not removed if the dentin at these sites is sound. Therefore, it is necessary to pay attention to the staining at these sites, and the dentin at these sites has to be evaluated synthetically from the results of the hardness and other conditions of the dentin.

From the results shown in Figure 3, the 0% point of bacterial detection was an L* value of more than 60. From the results of previous studies (Fukushima, 1981; Sano, 1987), dentin stained light pink by a caries detector dye had no bacterial infection. When the L* values of the light pink areas of the photographs reported in one of these previous studies (Sano, 1987) were measured by the objective method proposed in this study for color evaluation, the values were found to be approximately 60-64. Therefore, the 0% point of bacterial detection in this study was almost the same as in previous studies. However, the rates of bacterial detection decreased as the L* values increased when the L* values were between about 30 to 60 (Figure 3). Thus, the lightness of the stained dentin does not correlate completely or directly with the existence of bacteria. Recently, it was reported that a few bacteria might remain in dentinal tubules for caries treatment, for example, ART (Smales & Fang, 1999; Ho, Smales & Fang, 1999) or sealed restoration (Mertz-Fairhurst & others, 1998). Therefore, the optimal cut-off point for removal of caries in clinical situations needs to be ascertained for investigating the relationship between the prognosis of clinical restoratives and the color values or the degree of bacterial infection. In addition, new color-matching stickers or materials and clinical light sources for use in clinical situations need to be investigated. However, as a first step towards the establishment of an objective clinical diagnosis, this study provides the principles for an objective method for color evaluation of carious dentin for future clinical use, instead of the subjective visual inspection method currently used for caries evaluation.

CONCLUSIONS

In this *in vitro* study, an objective method for color evaluation of carious dentin was designed. The color of carious lesions was evaluated using the objective values, L*, a* and b*. From an evaluation of the receiver operating characteristic curves for bacterial infection investigated using a PCR technique and the L*, a* and b* values, the authors found that L* values are a more useful parameter than a* or b* values for detecting bacterial infection. The rates of bacterial detection decreased as the L* values increased when the L* values were between approximately 30 and 60. The 0% point of bacterial infection was an L* value of more than 60. The results of this study clarified the relation-

ship between the colors of lesions stained with a caries detector dye and the rates of bacterial detection.

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Bonding to Sound vs Caries-affected Dentin Using Photo- and Dual-cure Adhesives

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Clinical Relevance

Bond strengths to sound and caries-affected dentin were compromised when dual-cure adhesive was used.

SUMMARY

This study aimed to evaluate the microtensile bond strength (μTBS) of photo- and dual-cure adhesives to sound and caries-affected dentin using total- and self-etch techniques. Human third molars with occlusal caries were prepared as previously described by Nakajima and others (1995). Dentin surfaces were bonded with Optibond Solo Plus (Kerr; photo-cure adhesive) or Optibond Solo Plus + Dual-cure activator (Kerr; dual-cure adhesive) with total- and self-etch technique. Clearfil AP-X (Kuraray) was used for composite buildups. Following storage in distilled water at 37°C for 24 hours, the teeth were sectioned into 0.7-mm thick slices to obtain sound and caries-affected dentin slabs, then trimmed to form hour glass shapes with a 1 mm² cross-sectional area. The specimens were subjected to microtensile testing using EZ-test (Shimadzu) at 1 mm/minute. Data were analyzed using three-way ANOVA and Student's *t*-Test ($p<0.05$). Bond strengths to sound dentin with photo- and dual-cure adhesives using total- and self-etch techniques were significantly higher than those to caries-affected dentin. Dual-cure adhesive significantly decreased bond strengths both to sound and caries-affected dentin. The total-etch technique showed no beneficial effect on caries-affected dentin compared with the self-etch technique. Scanning electron microscopic observation of the resin-dentin interfaces revealed that hybrid layers in caries-affected dentin were thicker than those observed in sound dentin with photo- and dual-cure adhesives. Resin infiltration into dentinal tubules of caries-affected dentin was hampered by the presence of mineral deposits.

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INTRODUCTION

The primary aim of this minimally invasive restorative treatment is to remove the irreversibly denaturated, highly infected carious dentin (infected dentin) stained by caries detector dyes and to preserve uninfected, remineralizable caries-affected dentin (Fusayama, 1979).

Staining via dyes is not the result of loss of mineral or bacterial presence but the denaturation of collagen fibers (Kuboki, Liu & Fusayama, 1983). As the fundamental principle of adhesion to dentin is to expose a microporous network of collagen, followed by the infiltration and subsequent *in situ* polymerization of resin within the created surface microporosities (Van Meerbeek & others, 2003), removal of the denaturated collagen is important in achieving good adhesion. In fact, bond strength to caries-infected dentin was reported to be very low (Yoshiyama & others, 2002). Although micromechanical interlocking is believed to be a prerequisite to achieving good bonding within clinical circumstances, the potential benefit of additional chemical interactions between functional monomers and tooth substrate components has recently gained new attention (Van Meerbeek & others, 2003). If caries-affected dentin has normal collagen, hydrogen bonding between resins and collagen may contribute to bond strengths similar to sound dentin (Yoshiyama & others, 2002). However, due to the cyclic demineralization-remineralization process, changing the mineral phase of caries-affected dentin may influence hybrid layer formation (Nakajima & others, 2002) and chemical interaction with carboxylic or phosphate derivatives of methacrylates (Yoshiyama & others, 2002).

Contemporary self-etch systems, regardless of regional differences in dentin, bond well to sound dentin (Yoshikawa & others, 1999; Yoshiyama & others, 1998), though their bond strengths were compromised when applied to caries-affected dentin (Nakajima & others, 1995, 1999a; Yoshiyama & others, 2002, 2003); whereas, some total-etch systems showed better results on caries-affected dentin (Ceballos & others, 2003). It has been shown that acidic single-bottle total-etch and single-step self-etch adhesives are incompatible with self/dual-cure composites (Swift & others, 2001; Tay & others, 2003a), caused by the interaction between the nucleophilic tertiary amine accelerator in the composite and uncured acidic resin monomers in the oxygen inhibition layer of the adhesives (Yamauchi, 1986).

Recently, many manufacturers introduced dual-cure adhesives in combination with total- and self-etch adhesives for improving the bond strength at the adhesive–composite interface through an interfacial reaction between the dual-cure activator and components of the composites (Braga, Cesar & Gonzaga, 2000). One of these adhesives, Optibond Solo Plus Dual-Cure Activator (Kerr, Orange, CA, USA) contains sodium salt of aryl sulphinic acid, one of the common co-initiators used to overcome the incompatibility between acidic adhesives and self/dual-cured composites (Nyunt & Imai, 1996). Polymerization of these adhesives is slower, which might be beneficial for the infiltration of resins into porous intertubular dentin of caries-affected dentin. However, the addition of a dual-cure activator into the adhesives also might weaken the mechanical properties of the adhesives, which might affect the bond strength to dentin as contraction stresses of composites generally concentrate at the bonding interface during its polymerization (Kemp-Scholte & Davidson, 1990).

The objectives of this study were to evaluate the microtensile bond strength and interfacial ultrastructure on bonding of photo- and dual-cure adhesives to sound and caries-affected dentin using total- and self-etch technique. The hypotheses tested were that 1) dual-cure adhesive performed good adhesion to caries-affected dentin and sound dentin; and 2) bonding of the total-etch system to caries-affected dentin was better than the self-etch system.

METHODS AND MATERIALS

Twenty-four extracted human third molars with occlusal caries, stored in isotonic saline with thymol crystals at 4°C, were used in this study. The occlusal surface of each tooth was ground under running water using 600 grit silicon carbide paper to expose sound and caries dentin. The carious dentin was removed by means of 600 grit silicon carbide paper according to the combined criteria of visual examination and a caries detector solution (Kuraray Co, Ltd, Osaka Japan) as previously described (Nakajima & others, 1995). The discolored hard dentin that stained pink was classified as caries-affected dentin; whereas, the surrounding yellow hard dentin was classified as sound dentin. The teeth were then randomly divided into four groups according to bonding agent (Optibond Solo Plus photo-cure; Optibond Solo Plus dual-cure) and technique (total-etch; self-etch).

Optibond Solo Plus Total-Etch Photo-cure (OSP total-etch photo-cure): Kerr etchant was applied to the dentin surface for 15 seconds, rinsed for 15 seconds and gently air dried, leaving the surface visibly moist. Optibond Solo Plus was applied to the dentin surface for 15 seconds using a light brushing motion and was air-thinned for three seconds and light cured for 20 seconds.

Optibond Solo Plus Self-Etch Photo-cure (OSP self-etch photo-cure): Optibond Solo Plus self-etch primer was applied to the dentin surface with a light brushing motion for 15 seconds and air-thinned for three seconds. Optibond Solo Plus was applied to the dentin surface for 15 seconds and air-thinned for three seconds, the procedure was repeated and light cured for 20 seconds.

Optibond Solo Plus Total-Etch Dual-cure (OSP total-etch dual-cure): Kerr etchant was applied to the dentin surface for 15 seconds, rinsed for 15 seconds and gently air dried, leaving the surface visibly moist. One drop each of Optibond Solo Plus and Optibond Solo

Plus Activator was mixed for three seconds into a disposable mixing well and applied to the dentin surface for 15 seconds, air-thinned for three seconds and light cured for 20 seconds.

Optibond Solo Plus Self-Etch Dual-cure (OSP self-etch dual-cure): Optibond Solo Plus self-etch was applied to the dentin surface with a light brushing motion for 15 seconds and air-thinned for three seconds. Dual-cure adhesive was applied to the dentin surface as described in Optibond Solo Plus total-etch dual-cure.

Composite buildup was performed using Clearfil AP-X (Kuraray Co). Each of the three 2-mm resin composite increments was light cured for 20 seconds using a light-curing unit (XL 3000, 3M ESPE, St Paul MN, USA) with the intensity at 600 mW/cm^2. Following storage in distilled water at 37°C, the teeth were sectioned into 0.7-mm thick slices using a low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water lubrication to obtain caries-affected and sound dentin slabs. Each slice was examined under a dissecting microscope to determine the type of dentin (sound; caries-affected). Then, the adhesive-dentin interface was hand-trimmed with a fine diamond bur to form hour-glass shapes with approximately 1 mm^2 cross-sectional areas using a digital micrometer. The specimens were attached to a table top material tester (EZ-test, Shimadzu Co, Kyoto, Japan) with a cyanoacrylate adhesive (Zapit, DVA, Anaheim, CA, USA) and subjected to microtensile testing at a crosshead speed of 1-mm/minute until they fractured (Figure 1). All specimens were capable of being tested. All fractured specimens were fixed in 10% neutral formalin for 24 hours, then gold sputter-coated and observed with a scanning electron microscopy (JXA-5400, JEOL, Tokyo, Japan). Failure modes were classified as adhesive if debonding occurred between resin and dentin, as mixed if partial adhesive was exhibited, partial cohesive failure in bonding resin or the hybrid layer, as cohesive in resin or cohesive in dentin.

Bond strength data were analyzed by three-way ANOVA (sound vs caries-affected dentin; total-etch vs self-etch and photo-vs dual-cure) and multiple comparisons were performed by Student's *t*-Test at a significance level of 0.05 using computer software (SPSS 11, SPSS Inc, Chicago, IL, USA).

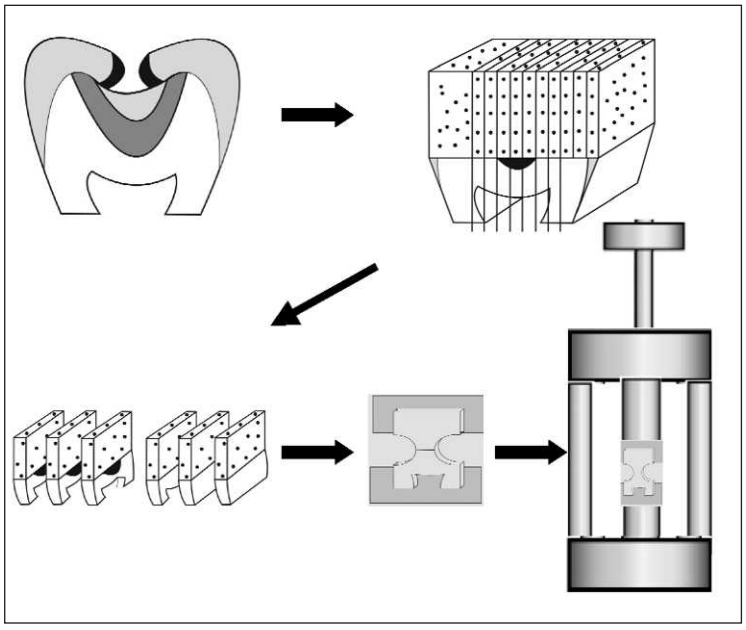

Figure 1. *Schematic used for illustrating sample preparation and microtensile bond strength testing.*

Scanning Electron Microscopy

Four additional carious third molars were used for SEM observations of the resin-dentin interfaces. The teeth were prepared in the same manner as the bonding procedure, then sectioned longitudinally to the bonding surface under running water using a low-speed diamond saw (Isomet) and stored for 24 hours in neutral formalin. The specimens were then embedded in epoxy resin (Epon 815, NISSIN EM Co Ltd, Tokyo, Japan) and polished using wet silicon carbide abrasive papers and diamond pastes of decreasing abrasiveness to 0.25 µm (DP-Paste,P,Streuers A/S, Copenhagen, Denmark). The specimens were then subjected to argon ion etching (EIS- 1E, Elionix Ltd, Tokyo, Japan) for five minutes with a constant voltage of 1 kV and ion current density of 0.2 mA/cm^2, with the ion beam directed 90° to the specimen surface (Inokoshi & others, 1993). The specimens were then gold-sputter coated and observed by a scanning electron microscope (JSM 5400; JEOL Ltd, Tokyo, Japan).

RESULTS

Table 2 shows the mean microtensile bond strengths and standard deviations. Optibond Solo Plus photo- and dual-cure adhesives, both with self-etch and total-etch techniques, showed significantly higher bond strength values to sound dentin than to caries-affected dentin (p<0.05). Using photo-

Table 2: *Mean Microtensile Bond Strength Values to Sound and Caries-Affected Dentin (MPa)*

Bonding System	Sound Dentin	Caries Affected Dentin
OSP total-etch photo-cure	38.7±8.9^a (20)	28.5±5.0^d (10)
OSP self-etch photo-cure	44.2±7.7^b (20)	29.2±4.3^d (10)
OSP total-etch dual-cure	17.2±4.7^c (20)	10.5±3.9^e (10)
OSP self-etch dual-cure	18.3±6.1^c (20)	13.5±3.3^e (10)

Figures in brackets are numbers of the specimens; Means identified by different superscript letters are significantly different (p<0.05) by three-way ANOVA and Student's t-Test

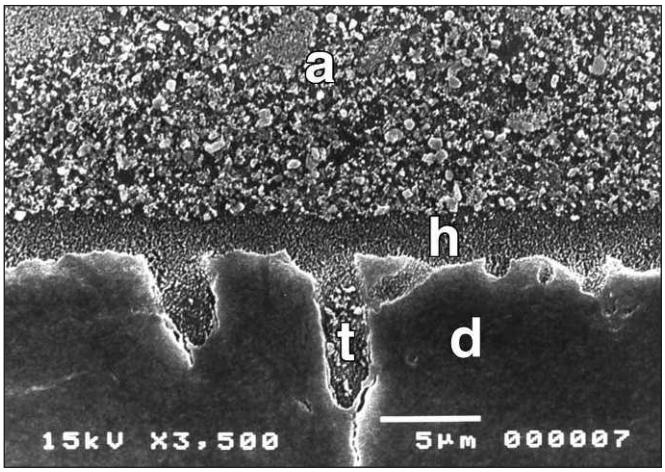

Figure 2A. *SEM of the resin-dentin interface bonded with Optibond Solo Plus total-etch photo-cure to sound dentin after argon ion etching. Fillers of the adhesive were penetrated into the dentinal tubule (t), (h) hybrid layer, (a) filled adhesive, (d) dentin (magnification 3500x).*

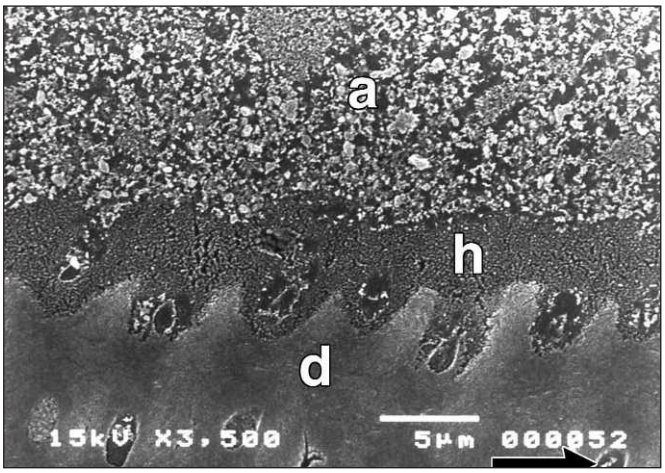

Figure 2B. *SEM of the resin-dentin interface bonded with Optibond Solo Plus total-etch photo-cure to caries-affected dentin after argon ion etching. The hybrid layer (h) in caries-affected dentin was thicker than that in sound dentin (compare Figure 2A). Dentinal tubules were often obliterated with mineral deposits (black arrow). (a) filled adhesive, (d) dentin (magnification 3500x).*

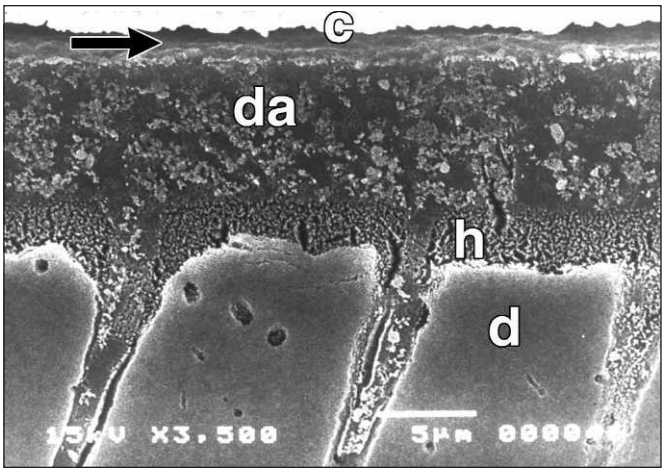

Figure 3A. *SEM of the resin-dentin interface bonded with Optibond Solo Plus total-etch dual-cure to sound dentin after argon ion etching. Black arrow shows separation between the dual-cure adhesive and composite resin, (h) hybrid layer, (da) dual-cure adhesive, (c) composite, (d) dentin (magnification 3500x).*

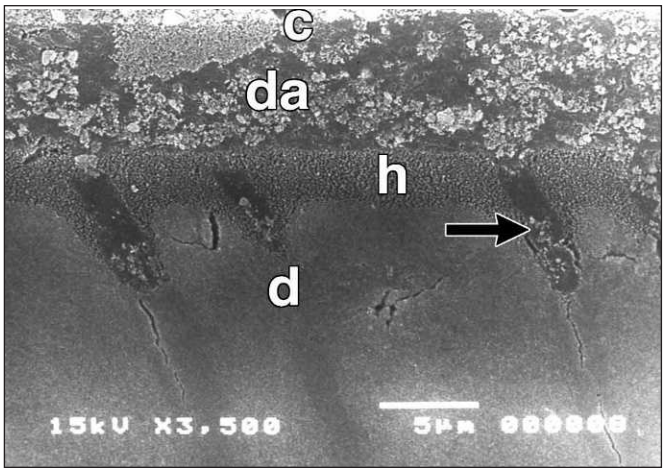

Figure 3B. *SEM of the resin-dentin interface bonded with Optibond Solo Plus total-etch dual-cure to caries-affected dentin after argon ion etching. The hybrid layer (h) in caries-affected dentin was thicker than that in sound dentin. Resin infiltration into dentinal tubules were hampered by the presence of the mineral deposits (black arrow); (da) dual-cure adhesive, (c) composite, (d) dentin (magnification 3500x).*

cure adhesive, the µTBS to sound dentin applied with self-etch was significantly higher than total-etch ($p<0.05$), whereas, there were no significant differences between self-etch and total-etch in µTBS to caries-affected dentin ($p>0.05$). The µTBS of the dual-cure adhesive was significantly lower than the photo-cure adhesive in both sound and caries-affected dentin ($p<0.05$).

Using total-etch, SEM observation of the resin-sound dentin interfaces with photo- and dual-cure adhesives revealed 2.7-µm thick hybrid layers with resin tags that exhibited characteristic funnel cone shapes (Figures 2A and 3A, respectively). For both adhesives, the hybrid layers in caries-affected dentin were thicker than those observed in sound dentin and their dentinal tubules were often obliterated with mineral deposits (Figures 2B and 3B, respectively). The hybrid layers created with Optibond Solo Plus self-etch with photo- and dual-cure adhesives to sound dentin were 2 µm-thick, with long, funnel-cone shape resin tags (Figures 4A and 5A, respectively). When caries-affected dentin was bonded with Optibond Solo Plus self-etch with photo- and dual-cure adhesives, the hybrid layers were thicker than those formed in sound dentin (Figures 4B and 5B).

The adhesive layers above the hybrid layer in the Optibond Solo Plus total-etch and self-etch photo-cure

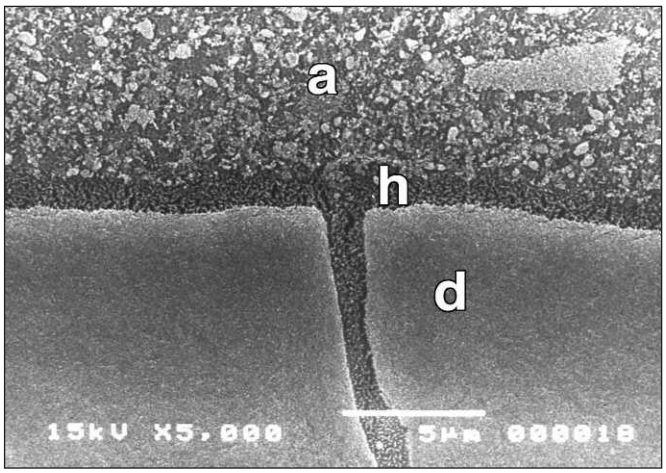

Figure 4A. *SEM of the resin-dentin interface bonded with Optibond Solo Plus self-etch photo-cure to sound dentin after argon ion etching. (h) hybrid layer, (a) filled adhesive, (d) dentin (magnification 5000x).*

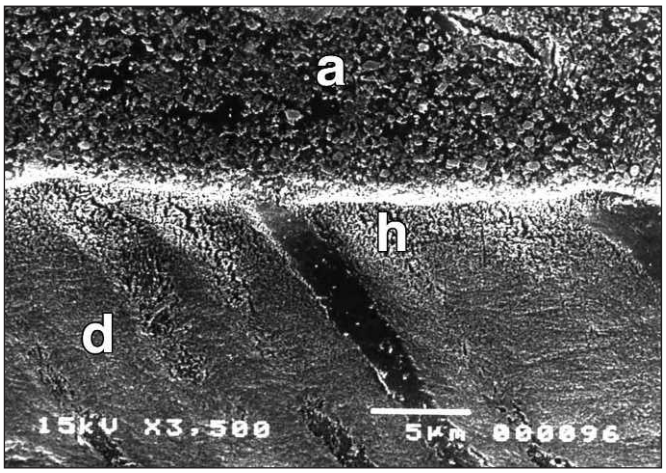

Figure 4B. *SEM of the resin-dentin interface bonded with Optibond Solo Plus self-etch photo-cure to caries-affected dentin after argon ion etching. (h) hybrid layer, (a) filled adhesive, (d) dentin (magnification 3500x).*

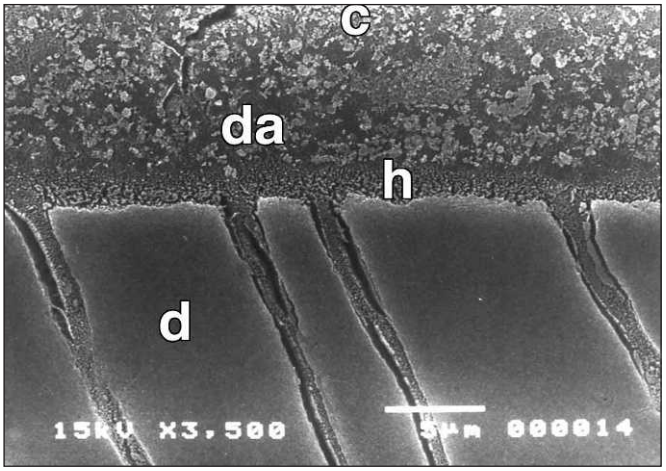

Figure 5A. *SEM of the resin-dentin interface bonded with Optibond Solo Plus self-etch dual-cure to sound dentin after argon ion etching. (h) hybrid layer, (da) dual-cure adhesive, (c) composite (d) dentin (magnification 3500x).*

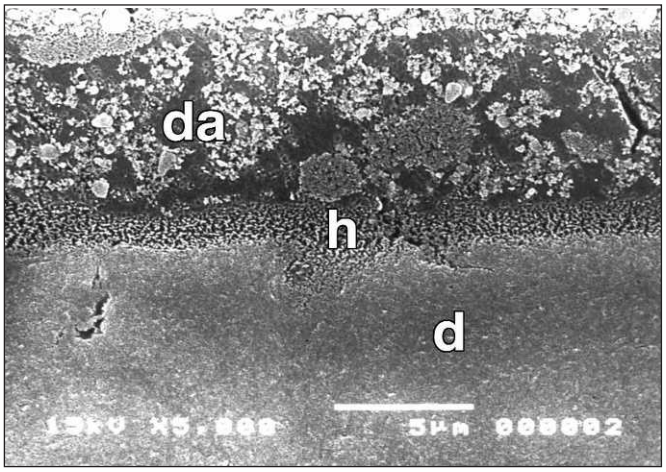

Figure 5B. *SEM of the resin-dentin interface bonded with Optibond Solo Plus self-etch dual-cure to caries-affected dentin after argon ion etching. (h) hybrid layer, (da) dual-cure adhesive, (d) dentin (magnification 5000x).*

groups were homogeneous with filler particles (Figures 2A, 2B, 4A and 4B); whereas, they were diluted with unfilled resinous components derived from the activator solution in the dual-cure groups (Figures 3A, 3B, 5A and 5B).

Table 3 shows the failure modes of the specimens in various groups. The majority of the failures in sound dentin with Optibond Solo Plus total- and self-etch adhesives with photo-cure were adhesive between resin and dentin; whereas, many of the specimens bonded to caries-affected dentin failed cohesively in dentin. Most of the specimens bonded to sound and caries-affected dentin with Optibond Solo Plus total- and self-etch adhesives with dual-cure showing mixed failure patterns, partially adhesive and partially cohesive, in bonding resin or hybrid layers.

DISCUSSION

Previously, it was believed that caries-affected dentin was sclerotic or harder than sound dentin, because of hypermineralization due to occlusion of the tubules with mineral. However, filling the tubule lumen with mineral does not enhance the mechanical properties of caries-affected dentin and this finding is in agreement with the microhardness data on both permanent (Fusayama, Okuse & Hosoda, 1966; Nakajima & others, 1995, 1999a,b, 2000) and primary dentin (Hosoya & others, 2000). This suggests that peritubular dentin does not induce significant changes in overall mechanical properties of dentin, and dentin properties are largely dependent on the properties of the intertubular dentin (Kinney & others, 1999). The loss of mineral from intertubular dentin causes a higher degree of

porosity, making caries-affected dentin softer (Fusayama & others, 1966; Nakajima & others, 1995) and susceptible to phosphoric acid or acidic primers, although mineral crystals occluding dentinal tubules of caries-affected dentin are acid resistant (Ogawa & others, 1983). This may lead to deeper penetration of adhesive resins during bonding, resulting in thicker hybrid layers compared to sound dentin, which does not correlate with bond strength values (Nakajima & others, 1999a, 2000; Yoshiyama & others, 2000).

Our ultrastructural results are consistent with previous studies, that hybrid layers in caries-affected dentin were thicker than those created in sound dentin and resin infiltration into dentinal tubules was hampered by the presence of acid resistant mineral deposits (Figures 2B and 3B). The length of resin tags does not contribute to bond strengths (Swift, Perdigão & Heymann, 1995); however, resin-tag hybridization to peritubular dentin is considered important for reliable bonding (Van Meerbeek & others, 1996a). Therefore, the primary causes for lower tensile bond strengths to caries-affected dentin using photo- and dual-cure adhesives both with total- and self-etch techniques compared to sound dentin are probably due to the obliteration of dentinal tubules with acid resistant mineral deposits (Ogawa & others, 1993) that prevent optimal resin tag formation, decreases in the modulus of elasticity (Marshall & others, 2001) and in the cohesive strength of caries-affected dentin (Yoshiyama & others, 2002). In this study, most specimens bonded to caries-affected dentin with photo-cure adhesives failed cohesively in dentin, most probably because of the weakness of caries-affected dentin compared to adhesive. Hence, this was not the case, as many of the specimens bonded to sound dentin with photo-cure adhesives failed adhesively (Table 3). Besides, the partially demineralized intertubular dentin may elongate more than sound dentin during testing, producing higher local stress concentrations that result in bond failure at lower apparent forces (Nakabayashi & Pashley, 1998).

The etching component of Optibond Solo Plus self-etch is glycerophosphoric acid dimethacrylate (GPDM), a phosphate ester. GPDM is an adhesion promoter that is also used in Optibond Solo Plus adhesive. Previous studies indicated that the acidity of some contemporary self-etch systems may not be sufficient for bonding to abnormal dentin substrates (Yoshiyama & others, 2000). However, Optibond Solo Plus self-etch was indicated as an "intermediary strong" two-step self-etch adhesive, because of its pH (1.5), hybrid layer dimension and morphology (Van Meerbeek & others, 2003), which probably results in equal tensile bond strengths as the total-etch system to caries-affected dentin. On the other hand, Optibond Solo Plus self-etch created significantly higher bond strengths to sound dentin than total-etch. One possible explanation might be that self-etch systems are less technique sensitive compared with total-etch systems.

Using total-etch systems, it is difficult to standardize the level of moisture left on the surface, which might affect the alcohol-based adhesive. Moreover, using self-etch, the susceptibility to moisture contamination of the adhesive through transudation of dentinal wetness might be reduced compared with total-etch (Itthagarun & Tay, 2000). However, this contamination would not occur within caries-affected dentin surfaces, because of less permeability due to occlusion of dentinal tubules of caries-affected dentin (Tagami & others, 1992). The chemical (water, mineral and collagen) conditions of caries-affected dentin subsurface after etching would be very different from sound dentin.

If superficial dentin is completely demineralized and resin monomer infiltration is complete, the hybrid layer consists of approximately 70 vol % of resin and 30 vol % of collagen fibrils (Marshall, 1993). The degree of polymerization of the adhesive resin inside the hybrid layer depends on the mode of polymerization (light cured and/or chemically cured), the site of initial polymerization (interfacial or originating in the adhesive resin) and the degree of *in situ* available double-carbon bonds. Water that is already present in the hybrid layer or introduced when a wet bonding technique is used and oxygen in dentin may also affect resin polymerization inside the hybrid layer (Van Meerbeek & others, 1996b). In this study, the addition of a dual-cure activator to Optibond Solo Plus both with the self-etch and total-etch technique caused significant decreases in microtensile

Table 3: *Fracture Modes of the Specimens After the Microtensile Bond Test*

Groups	Adhesive	Mixed	Cohesive in Composite	Cohesive in Dentin
OSP total-etch photo-cure (SD)	13	5	2	-
OSP total-etch dual-cure (SD)	4	16	-	-
OSP total-etch photo-cure (CA)	3	1	-	6
OSP total-etch dual-cure (CA)	1	6	-	3
OSP self-etch photo-cure (SD)	14	6	-	-
OSP self-etch dual-cure (SD)	7	13	-	-
OSP self-etch photo-cure (CA)	1	4	-	5
OSP self-etch dual-cure (CA)	1	7	-	2

SD: Sound dentin; CA: Caries-affected dentin
Adhesive: between resin and dentin,
Mixed: partially adhesive, partially cohesive failure in bonding resin or hybrid layer,
Cohesive in resin or Cohesive in dentin.

Table 1: *Materials Used in the Study*

Materials	Composition	Manufacturer
Optibond Solo Plus	Ethanol, Bis-GMA, GPDM, HEMA, fumed silica, barium aluminoborosilicate glass, sodium hexafluorosilicate	Kerr Corporation, Orange, CA, USA
Optibond Solo Plus Self-Etch	Ethyl alcohol, water, GPDM, stabilizers and activators	Kerr Corporation, Orange, CA, USA
Optibond Solo Plus Dual-Cure Activator	Ethanol, Bis-GMA, HEMA, BSA	Kerr Corporation, Orange, CA, USA
Kerr Etchant	Phosphoric Acid 37. 5 % with benzalkonium chloride	Kerr Corporation, Orange, CA, USA
Clearfil AP-X	Bis- GMA, TEGDMA, filler (Barium, SiO_2)	Kuraray Co, Osaka, Japan

bond strength values. Reaction between acidic resin monomer GPDM in Optibond Solo Plus adhesive and Optibond Solo Plus self-etch with binary peroxide amine catalytic components in the dual-cure activator may be the reason for lower bond strength values. It has been indicated that acidic resin monomers polymerized poorly in the presence of peroxide-amine redox systems, as the tertiary amines were neutralized by the acidic resin monomers and lost their ability as reducing agents in redox reactions (Yamauchi, 1986). Chemical co-initiator BSA (benzene sulphinic acid sodium salt; Table 1) in the dual-cure activator may overcome this incompatibility and react with acidic resin monomers. However, sodium salts of aryl sulphinic acid are good oxygen scavengers, and it has been suggested when it consumes oxygen, it becomes oxidized and should not be able to function as a chemical co-initiator in a redox reaction for the generation of free radicals. It is possible that resin monomers included in the activator undergo only partial polymerization in the presence of BSA (Tay & others, 2003b). If polymerization of adhesive is not complete, hydrophilic monomers or small oligomers might be extracted or hydrolyzed by the presence of nanoleakage (Sano & others, 1995). Residual resin monomers may act as plasticizers to alter the mechanical properties of adhesive resins and the hybrid layer, leading to lower dentin bond strength (Miyazaki & others, 2003).

The influx of pores in the adhesive, which had been produced during mixing of the dual-cure activator with the adhesive, might also be the reason for the low bond strength values (Braga & others, 2000), as oxygen becomes trapped in the resin mixture, thereby inducing polymerization inhibition to a higher degree than in the light cure ones. The presence of pores also increases the elasticity of the material and the area available for flow during polymerization shrinkage of the composites that ultimately reduce the risk of adhesive failure (Van Meerbeek & others, 1993). In addition, polymerization of the dual-cure adhesive is slower, which also facilitates adhesive flow under stress (Perdigão & others, 1996). Another possible explanation may be the dilution of the filler, since the dual-cure activator is unfilled (Braga & others, 2000). The mixture of the filled adhesive with the dual-cure activator in a 1:1 ratio resulted in a more sparsely filled adhesive layer with areas devoid of fillers (Figures 3A, 3B, 5A and 5B). Fillers increase the viscosity of the adhesives so that they can be applied in a thick layer (Labella & others, 1999).

Adhesive systems that produce relatively thick adhesive resin layers could better resist polymerization shrinkage stress of the restorative composites (Van Meerbeek & others, 1993). The predominant failure modes of Optibond Solo Plus dual-cure adhesive with total- and self-etch technique to sound and caries-affected dentin were mixed; partially adhesive and partially cohesive in bonding resin or in the hybrid layer (Table 3). This suggests that the cohesive strength of the adhesive was similar and/or less than the bond strength to sound and caries-affected dentin. Residual amounts of solvents such as ethanol and acetone in the adhesive resin, if not completely evaporated, may affect resin polymerization inside the hybrid layer or at least occupy space that optimally should have been filled with resin (Jacobsen, Ma & Söderholm, 1994). Since dual-cure activator and Optibond Solo Plus adhesive contain ethanol, the amount of ethanol that has to evaporate in adhesive resin probably increased, which might affect polymerization and lead to mixed failures.

The tested hypotheses that 1) dual-cure adhesive performed good adhesion to caries-affected and sound dentin and 2) bonding of the total-etch system to caries-affected dentin was better than the self-etch system must be rejected.

CONCLUSIONS

Based on the results of this *in vitro* study, Optibond Solo Plus photo- and dual-cure adhesives with total- and self-etch techniques showed significantly higher bond strength values to sound dentin than to caries-affected dentin. Total-etch technique did not show any beneficial effect on caries-affected dentin compared with the self-etch technique. The lower tensile bond strengths to caries-affected dentin are probably due to the lack of resin tag hybridization to peritubular dentin, because of acid resistant mineral deposits, decreases in the modulus of elasticity and cohesive strength of caries-affected dentin. However, more research on the effect of phosphoric acid or acidic primers on caries-affected collagen is needed. The addition of a dual-cure activator to Optibond Solo Plus caused a significant decrease in bond strength values both to sound and caries-affected dentin.

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Effect of Hygiene Maintenance Procedures on Surface Roughness of Composite Restoratives

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Clinical Relevance

Composite restorations may require re-polishing after exposure to some hygiene maintenance procedures. The surface finish of composite restoratives is least affected by the use of prophylaxis gels.

SUMMARY

This study investigated the effect of various hygiene maintenance procedures on the surface finish of minifill (Filtek A110 [AO], 3M-ESPE), flowable (Filtek Flow [FF], 3M-ESPE) and polyacid-modified (F2000 [FT], 3M-ESPE) composites. Procedures included pumice-water slurry with rotating brush (PB), pumice-water slurry with rotating rubber cup (PC), prophylaxis paste with rubber cup (ZC), prophylaxis gel with rubber cup (GC) and air-powder polishing (AP). Specimens not exposed to these procedures were used as the control group. For each material, 48 specimens (3-mm long x 3-mm wide x 2-mm deep) were made and stored in distilled water at 37°C for one month. The specimens were then treated with 1200 grit sandpaper using a lapping device, stored for an additional two months in distilled water at 37°C and randomly divided into six groups (n=8). The mean surface roughness (Ra, µm) of the specimens after exposure to the various hygiene procedures was determined using a surface profilometer. Data was subjected to ANOVA/Scheffe's test at significance level 0.05. Mean Ra values ranged from 0.09 to 2.17, 0.06 to 1.38 and 0.38 to 1.25 for AO, FF and FT, respectively. The effect of hygiene procedures on surface roughness was material dependent. Among the various procedures, the smoothest surface was observed after treatment with prophylaxis gel and the roughest with air-powder polishing. For all materials, the use of pumice-water slurry with brush also caused significant roughening. Composite restorations may require re-polishing after exposure to some hygiene maintenance procedures, as Ra values exceeded the critical threshold surface roughness for bacterial adhesion (0.2 µm).

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INTRODUCTION

Maintenance therapy is an integral part of restorative and periodontal treatment. The removal of stains and

plaque from all accessible tooth surfaces is a routine part of the maintenance appointment (Shick, 1981; Schallhorn & Snider, 1981). Stain removal is usually performed using a variety of prophylaxis pastes and rotary rubber cups or brushes as carriers. Air powered devices have also been introduced into clinical practice. With these devices, sodium bicarbonate particles are propelled by air jet and are combined with a small stream of water, creating a slurry that is directed onto the tooth surface (Reel & others, 1989). An ideal prophylaxis agent should combine good cleaning ability with simultaneous polishing (morphological smoothing of dentin and enamel surfaces). In addition, the agent should cause minimal abrasion of dental hard tissues (Lutz & others, 1993; Barbakow, Lutz & Imfeld, 1987; Roulet & Roulet-Mehrens, 1982). Good cleaning efficiency is, however, associated with high surface roughness and tooth tissue abrasion. Conversely, pure polishing agents, though tooth tissue friendly, cannot efficiently remove plaque or stains (Barbakow & others, 1987). Although patients present with a wide range of maintenance requirements from cleaning to polishing, most clinicians use only one prophylaxis agent. Medium- and coarse-grade prophylaxis agents are generally more popular due to their stain removing potential (Neme & others, 2002).

As stains and plaque deposits are often heaviest in the cervical and interpromixal areas of teeth, restorative materials (if present) are inadvertently exposed to hygiene maintenance procedures. The surface finish of restorative materials can be damaged or even destroyed by hygiene procedures. Surface roughness of restorative materials can contribute to staining, plaque retention, gingival irritation and recurrent caries (Shintani & others, 1985; Dunkin & Chambers, 1983; Chan, Fuller & Hormati, 1980; Weitman & Eames, 1975; Larato, 1972). Surface roughness can also influence the appearance of the color and gloss of tooth-colored materials (Stanford & others, 1985). Although the effect of hygiene procedures on enamel and dentin has been well investigated (Gerbo & others, 1993; Lutz & others, 1993; Leknes & Lie, 1991; Roulet & Roulet-Mehrens, 1982), few studies have looked at its effect on the newer generations of composite restorative materials (Warren & others, 2002; Carr & others 2002). This study investigated the effect of five clinically employed hygiene maintenance procedures on the surface finish of minifill (Filtek A110 [AO]), flowable (Filtek Flow [FF]) and polyacid-modified (F2000 [FT]) composites. While polyacid-modified composites or compomers are polymer-based materials that contain either or both of the essential components of glass ionomer cements (but at levels insufficient to promote the acid-base cure reaction in the dark), flowable composites are low viscosity mixtures with reduced filler content.

METHODS AND MATERIALS

Technical profiles of the materials evaluated are shown in Table 1. The composite materials were placed into the square recesses (3-mm long x 3-mm wide x 2-mm deep) of customized acrylic molds and covered with matrix strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide was placed over this and pressure was applied to extrude excess material. The composite materials were then polymerized according to manufacturer's cure times (Table 1) through the glass slide using a halogen light curing unit (Polylux II, Kavo Dental, Warthausen, Germany) with an intensity of 540 mW/cm^2. Forty-eight specimens were made for each material. The specimens were stored in distilled water at 37°C for one month and treated with ANSI (American National Standard Institute) 1200 grit sandpaper (Carbimet disks; Wirtz-Buehler, Dusseldorf, Germany) using a lapping device (Phoenix Beta; Wirtz-Buehler, Dusseldorf, Germany) at 2,000 rpm for four minutes. The roughness of this grit of sandpaper (Ra=0.23 µm) was similar to "Fine" Sof-Lex finishing/polishing disks (3M-ESPE, St Paul, MN, USA). The specimens were then returned to distilled water at 37°C and stored for an additional two months.

After aging, the specimens were randomly divided into six equal groups and treated as follows: Group 1–No treatment (Control); Group 2–Pumice-water slurry (Casone,

Table 1: *Technical Profiles of the Materials Evaluated*

Material (Lot #)	Manufacturer	Cure Time (seconds)	Resin	Filler	Filler Size (mm)	Filler Content (% by volume)
Filtek A110 (lot 1720A2D)	3M Dental Products St Paul, MN, USA	40	BisGMA TEGDMA	Colloidal Silica	0.01 – 0.09	40
Filtek Flow (lot 1400A2)	3M Dental Products St Paul, MN, USA	20	BisGMA TEGDMA	Zirconia/ Silica	0.01 – 6	47
F2000 (lot 2020A2)	3M Dental Products St Paul, MN, USA	40	CMDA GDMA	Fluoro-alumino-silicate Glass, Silica	3 – 10	67

BisGMA = Bisphenol-A-glycidyl methacrylate
CDMA = Dimethacrylate functional oligomer derived from citric acid
GDMA = Glyceryl methacrylate
TEDGMA = triethylene glycol dimethacrylate

Noceto-parma, Italy) with rotating brush [PB]; Group 3–Pumice-water slurry (Casone, Casone, Noceto-parma, Italy) with rotating rubber cup [PC]; Group 4–Prophylaxis paste (Zircon F; Henry Schein, Middlesex, UK) with rubber cup [ZC]; Group 5–Prophylaxis gel (PTC Paste Regular and Fine); GC Corporation, Tokyo, Japan) with rubber cup [GC] and Group 6–Air-powder polishing (AirFlow S1; Electronic Medical Systems, Nyon, Switzerland) [AP]. With the exception of the control group, treatment time was fixed at 12 seconds for all groups. For Groups 2 through 5, a single, contra-angle slow speed handpiece was used at 2,000 rpm. Prophylaxis agents were replaced every six seconds and a new carrier (rubber cup or brush) was used for each material-treatment combination. For Group 5, Regular PTC Paste was replaced with Fine paste after the first six seconds of treatment. To minimize the effects of operator variability, all hygiene maintenance procedures were carried out by a single person.

A surface profilometer (Surftest SV-400, Mitutoyo, Kanagawa, Japan) was used to determine the mean surface roughness (Ra, µm) of the specimens after exposure to the various hygiene procedures. Readings were taken at the center of each specimen and four sampling lengths of 0.25 mm were used, giving a total evaluation length of 1 mm. All statistical analysis was carried out at significant level 0.05. Two–way ANOVA was used to determine interaction between materials and treatment groups. One-way ANOVA and Scheffe's post-hoc tests were used to compare the mean surface roughness between procedures for each material. The results were contrasted against the critical threshold surface roughness for bacterial adhesion of 0.2 µm reported by Bollen, Lambrechts and Quirynen (1997).

RESULTS

The mean surface roughness observed for the different materials and procedures is shown in Table 2 and Figure 1. The results of statistical analyses are shown in Tables 3 and 4.

Mean Ra values ranged from 0.09 to 2.17, 0.06 to 1.38 and 0.38 to 1.25 µm for Filtek A110, Filtek Flow and F2000, respectively. Specimens in the control group generally had the lowest Ra values. Among the various hygiene procedures, the smoothest surface was observed after treatment with prophylaxis gel and the roughest with air-powder polishing. The results of two-way ANOVA revealed significant interaction (p=0.0001) between materials and treatment groups. For all materials, procedures involving the use of air-powder polishing and pumice-water slurry with brush resulted in significant roughening when compared to the control group. For Filtek Flow and F2000, the use of prophylaxis paste with cup also caused significant roughening. The surface finish of F2000 was also significantly affected by the use of pumice-water slurry with rubber cup. With the exception of Group 6 (air-powder polishing), F2000 was significantly rougher than Filtek Flow.

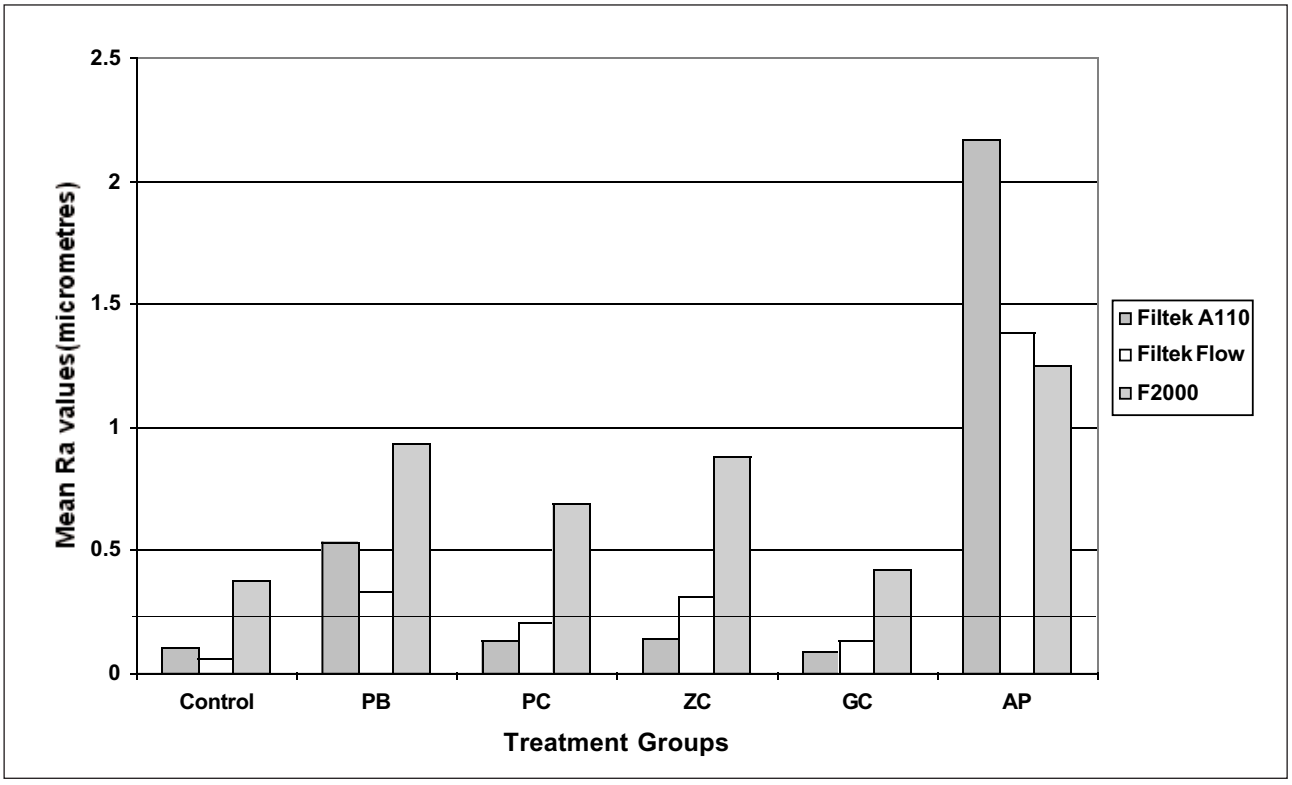

Figure 1. *Mean surface roughness of the materials with reference to the critical threshold surface roughness of bacterial adhesion of 0.2 micrometers.*

For Groups 1, 3, 4 and 5, Ra values for Filtek Flow were significantly greater than Filtek A110. Filtek A110 specimens that were air-powder polished were significantly rougher than Filtek Flow and F2000 specimens treated similarly.

Table 2: *Mean Surface Roughness Observed with the Various Hygiene Procedures*

Treatment Groups	Filtek A110 [AO]	Filtek Flow [FF]	F2000 [FT]
Group 1 (Control) No treatment	0.10 (0.02)	0.06 (0.01)	0.38 (0.04)
Group 2 (PB) Pumice with brush	0.53 (0.55)	0.33 (0.02)	0.93 (0.10)
Group 3 (PC) Pumice with rubber cup	0.13 (0.03)	0.21 (0.04)	0.69 (0.05)
Group 4 (ZC) Paste with rubber cup	0.14 (0.05)	0.31 (0.04)	0.88 (0.11)
Group 5 (GC) Gel with rubber cup	0.09 (0.02)	0.13 (0.01)	0.42 (0.04)
Group 6 (AP) Air-powder polishing	2.17 (0.09)	1.38 (0.28)	1.25 (0.26)

DISCUSSION

The smoothest surface finish possible for composite materials is obtained when they are cured against a matrix (Marigo & others, 2001; Yap, Lye & Sau, 1997). Despite careful placement of matrix strips, some degree of finishing and polishing of restorations will be required clinically. Finishing refers to the gross contouring of restorations to obtain the desired contour, while polishing refers to the reduction of roughness and scratches created by the finishing/polishing instruments. Treatment with 1200 grit sandpaper was used to simulate the surface finish obtained with "Fine" composite finishing/polishing disks. This was carried out at one month to allow for resin post cure (Watts, Amer & Combe, 1987) and the acid-base reaction of the polyacid-modified composite to occur fully (Yap & others, 2002; Eliades, Kakaboura & Palaghias, 1998). A lapping device was used instead of abrasive disks mounted on slow handpieces, as this approach was less operator dependent and resulted in more consistent surface finish/uniform baseline readings (Roulet & Roulet-Mehrens, 1982). A total aging period of three months was necessary to replicate a practical recall period employed for maintenance therapy. This aging period also allowed for the plasticizing effect of water to occur (Hansen, 1983) prior to exposure to the various hygiene procedures.

Table 3: *Comparison of Ra Values Between Different Treatment Groups*

Materials	Differences
Filtek A110	AP > PB, ZC, PC, GC, Control PB > GC & Control
Filtek Flow	AP > PB, ZC, PC, GC, Control PB, ZC > Control
F2000	AP > PB, ZC, PC, GC, Control PB > PC, GC, Control ZC, PC > GC, Control

PB = Pumice with brush; PC = Pumice with rubber cup; ZC = Paste with rubber cup; GC = Gel with rubber cup; AP = Air-powder polishing. > indicates statistical significant differences in Ra values (Results of one-way ANOVA/Scheffe's test [p<0.05]).

Table 4: *Comparison of Ra Values Between Materials*

Treatment Groups	Differences
Group 1 (Control) No treatment	F2000 > Filtek Flow > Filtek A110
Group 2 (PB) Pumice with brush	F2000 > Filtek Flow
Group 3 (PC) Pumice with rubber cup	F2000 > Filtek Flow > Filtek A110
Group 4 (ZC) Paste with rubber cup	F2000 > Filtek Flow > Filtek A110
Group 5 (GC) Gel with rubber cup	F2000 > Filtek Flow > Filtek A110
Group 6 (AP) Air-powder polishing	Filtek A110 > Filtek Flow, F2000

> indicates statistical significant differences in Ra values (Results of one-way ANOVA/Scheffe's test [p<0.05]).

The effect of hygiene procedures on surface roughness was material dependent. Composites are biphasic, with fillers embedded in a resin/polymer matrix (Yap & others, 2001). During hygiene procedures, the matrix phase is preferentially removed (Gutmann, Marker & Gutmann, 1993; Reel & others, 1989; Roulet & Roulet-Mehrens, 1982) as the abrasives employed in prophylaxis agents are harder than the resin matrix. These abrasives could even be similar in hardness to the fillers of some composite materials (Serio & others, 1988). As the resin matrix is selectively removed, filler particles are exposed, resulting in a rough surface (Roulet & Roulet-Mehrens, 1982). Filler exfoliation or displacement may occur with continued matrix abrasion ensuing in even rougher surfaces. Up to 8 μm of material can be removed from composite surfaces with some hygiene procedures (Barnes, Hayes & Leinfelder, 1987). As the filler particles in F2000 are relatively large (3 to 10 μm), the significantly higher Ra values of F2000, as compared to Filtek Flow and Filtek A110 for most treatment groups, are expected. The large filler size range of F2000 can be attributed to the use of fluoroaluminosilicate glass, which was incorporated for fluoride release. For the air-powder

polishing group (Group 6), Filtek A110 was significantly rougher than Filtek Flow and F2000. These air-powered devices require air inlet pressures of 50 to 100 psi and a water inlet pressure of 10 to 50 psi (Reel & others, 1989). These high pressures may be sufficient to degrade the interface between pre-polymerized resin fillers and the polymer matrix of microfill composites. Pre-polymerized resin fillers were introduced to increase filler loading. These resin fillers are heat-cured and do not form covalent chemical bonds with the polymer matrix. They are therefore not well bonded to the polymer matrix and may become debonded or dislodged under high pressure. This may also account for the significant standard deviation observed with Group 2 (pumice slurry and brush) for Filtek A110.

One 12-second exposure to air-powder polishing significantly roughened the surface of microfill, flowable and polyacid-modified composite restoratives. The results were in agreement with those conducted on older generations of composites (Reel & others, 1989; Serio & others, 1988). Gutmann and others (1993), however, found that air-powder polishing does not always roughen the surface of composites. Using visual observations of scanning electron micrographs, they concluded that composite surface changes were generally dependent on pre-treatment conditions, with smooth surfaces becoming rougher and surfaces that are extremely rough becoming smoother. As the pre-treatment finish was that of commonly employed "Fine" abrasive disks, any significant change in surface roughness may be clinically relevant. Air-Flow prophylaxis powder is a blend of specially selected sodium bicarbonate and hydrophobe modified silica. The powder particles are less than 100 µm, which is large compared to the particle size of the composites evaluated (0.01 to 10 µm). While the primary mechanism of surface degradation is via polymer matrix abrasion (Gutmann & others, 1993; Reel & others, 1989), the powder particles could also abrade the filler phase of composites. Composites with smaller filler particles may be more susceptible (Reel & other, 1989).

For all materials, the use of pumice-water slurry with brush also resulted in significant roughening. These findings corroborated those of Roulet and Roulet-Mehrens (1982), who found that a second polishing step with rotary brushes after the use of rubber cups significantly increased Ra values. The significantly higher Ra values may be attributed to a combination of two-body composite wear due to brush bristles and slurry wear by pumice (Mair & others, 1996). The use of Zircon F (prophylaxis paste) with rubber cup significantly increased the surface roughness of Filtek Flow and F2000. Results cannot be generalized, as the abrasiveness of prophylaxis pastes is not standardized and varies for different products (Serio & others, 1988). Zirconium silicate, the abrasive used in Zircon F, is also employed as fillers for composite restorative materials. The significant roughening of Filtek Flow and F2000 after exposure to Zircon F can be attributed to the use of these very hard abrasives and the presence of large filler particles in these two materials.

Among the various hygiene procedures, the use of PTC paste (Regular and Fine) and cup resulted in the lowest Ra values. PTC Regular paste is a silica-based prophylaxis gel. It consists of 20 weight % 19 µm porous silica and 3 weight % 0.01 µm silica. The Fine paste also comes in gel form and consists of 20 weight % 0.01 µm silica. Due to its very small abrasive particle size, the Fine paste could provide a polishing effect on composite materials. Their restoration friendliness may be at the expense of its cleaning efficiency, and this requires validation. Ra values observed after exposure to this treatment were under 0.2 µm for both Filtek A110 and Filtek Flow. While no further reduction in bacterial accumulation is expected below the threshold value of 0.2 µm, any increase in surface roughness after this critical threshold value results in a simultaneous increase in plaque accumulation and increases the risk for caries and periodontal inflammation (Bollen & others, 1997). Since the surface roughness of the materials was greater than 0.2 µm after exposure to several hygiene maintenance procedures (Figure 1), re-polishing of the restorations might be necessary to maintain periodontal health, restoration longevity and aesthetics in the clinical setting.

CONCLUSIONS

Under the conditions of this *in vitro* study:

- The effect of hygiene procedures on surface roughness was material dependent.
- In the presence of multiple microfill and flowable composite restorations, the use of air-powder polishing and pumice slurry with brush should be avoided during maintenance therapy.
- When multiple polyacid-modified composite restorations are present, the use of prophylaxis gel with rubber cup is recommended. Use of other hygiene procedures results in significant roughening.
- Composite restorations may require re-polishing after exposure to some hygiene maintenance procedures.

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The Influence of Various Conditioner Agents on the Interdiffusion Zone and Microleakage of a Glass Ionomer Cement with a High Viscosity in Primary Teeth

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Clinical Relevance

The process of applying a conditioner agent to hard tooth tissues decreases the microleakage of GIC (glass ionomer cement) and establishes a good bond between GIC and enamel and dentin.

SUMMARY

The smear layer, which occurs during cavity preparation procedures, does not constitute a stable substructure in the bond of restorative material to dental hard tissues. Depending on the dissolution of this material in the course of time, microleakage occurs between the tooth and restorative material.

This study evaluated the effects of different conditioner agents (Fuji Cavity Conditioner, 10% maleic acid, 35% phosphoric acid and 3% hydrogen peroxide) on Fuji IX microleakage, Fuji IX-enamel and Fuji IX-dentin combination in Class I cavities prepared to standards having the dimensions of 4x3x2 mm^3 in extracted primary molars. The restorations were then subjected to thermocycling procedures and soaked in the 0.5% basic-fuchsin dye for 24 hours. Some sections were taken, parallel to the long axis of the tooth in a mesio-distal direction, and evaluated under a stereo-microscope for leakage. Also, two samples of Fuji IX-enamel and dentin combinations were chosen randomly from each group for evaluation in scanning electron microscopy (SEM).

The distribution of microleakage occurred as follows: Control Group > Hydrogen Peroxide > 10% Maleic acid > Fuji Cavity Conditioner = Phosphoric acid. The difference between microleakage scores obtained from the groups was statistically significant ($p<0.05$). The SEM evaluation revealed a close interface connection in all groups except for the control and hydrogen peroxide groups in the Fuji IX-enamel combination. In the Fuji IX-dentin combination, however, a close interface connection was observed except in the control group.

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In conclusion, the application of conditioner agents to Class I cavities restored with glass ionomer cement with a high viscosity *in vitro* either diminishes or completely eliminates microleakage.

INTRODUCTION

One important feature of glass-ionomer cements (GIC) is their physico-chemical bond both to enamel and dentin (Hotz & others, 1977; Lacefield, Reindl & Retief, 1985). Some researchers (Hotz & others, 1977; Shalabi, Asmussen & Jorgensen, 1981; Powis & others, 1982; Joynt & others, 1990; Charlton & Haveman, 1994; Nitta, 1992) reported that conditioning of the tooth surface increases the bond strength of GICs to bond better to dental hard tissues. The objective of conditioning is to remove surface contaminants and the smear layer, which may limit the bond of GIC to the tooth surface, particularly dentin (Gwinnett, 1984; Pashley, 1984; White, Beech & Tyas, 1989). It was shown that the concentrations of conditioners, application methods and time periods could have an influence on the removal of the smear layer (Joynt & others, 1990; Hinoura, Moore & Phillips, 1986; Aboush & Jenkins, 1987; Hewlett, Caputo & Wrobel, 1991; Bourke, Walls & McCabe, 1994; Tanumiharja, Burrow & Tyas, 2000). Also, it was stated that the smear layer could be partly dissolved with the influence of the acidic nature of GICs placed in cavities (Ferrari & Davidson, 1997; Nakabayashi & Pashley, 1998).

The smear layer was initially preserved, because it was thought to serve as a barrier that protected pulp from noxious stimuli and reduced outward tubular fluid flow (Blunck, 2000; Nakabayashi & Pashley, 1998). However, it was emphasized that the occurring smear layer does not constitute a stable substrate for the restorative materials to bond to tooth enamel, and this layer under restorative materials may gradually dissolve over time by hydrolysis and may allow for bacterial penetration. It was also stressed that this bacterial microleakage was the most prevalent cause of pulp inflammation (Blunck, 2000; Nakabayashi & Pashley, 1998; Douglas, 1989). Blunck (2000) reported that the smear layer, being in close contact with dental hard tissue, should be modified or completely dissolved. Yilmaz and Kirzioglu (1998) restored Class I cavities using a metal-added GIC and reported some microleakage in all samples. In a study in which the primary molars with and without a conditioner agent application were restored with Fuji-IX without any conditioner agent, it was found that cavities that had a conditioner agent application presented with less microleakage (Castro & Feigal, 2002).

Some authors (Ferrari & Davidson, 1997; Tanumiharja & others, 2001; Ngo, Mount & Peters, 1997) who carried out their research on bovine and human permanent teeth showed the presence of an interdiffusion zone, also called the "ion change layer," "hybrid-like layer" and "acid-base resistant (ABR) layer," which is indicative of an intimate adaptation between GIC and dental hard tissue in the cavities to which a conditioner agent is applied. Moreover, Ngo and others (1997) stated that the same zone contributed to the high resistance of GIC restorations to microleakage.

The objective of this study was to compare the influence of the different conditioner agents applied to Class I cavities prepared in primary molars on the microleakage of Fuji IX (a GCI with a high viscosity), Fuji IX-enamel and Fuji IX-dentin interface morphologies.

METHODS AND MATERIALS

Fifty freshly extracted and caries free primary molars, without any restoration and with a root resorption lower than two-thirds, were chosen for this study. Tissue remnants were removed from the root surfaces, the roots were covered with nail polish and the teeth embedded in an auto-polymerizing acrylic resin, up to the enamel-cement junction. Then, Class I cavities 4x3x2mm^3 were prepared on the occlusal face of the teeth using a number 014 diamond fissure bur (Al Amazonas, 580 Barueni-SP, Industria Brasileria). Next, five experimental groups of 10 randomly chosen primary molars were arranged. Removal of the smear layer, which occurred during the cavity preparation process using different conditioner agents and restorative material applications, was carried out as follows:

Group 1: This was the control group in which no conditioning agents were applied to the prepared cavities and Fuji IX GC capsule (GC Corp 76-1 Hasunuma-cho, Itabashi-ku, Tokyo) was mixed and placed in the cavities in accordance with the manufacturers' recommendations. The restorations were covered with a GIC protective varnish (Art #1124, Voco-Cuxhaven-Germany).

Group 2: Fuji Cavity Conditioner (GC Corp 76-1) was applied to the prepared cavities for 10 seconds in accordance with the manufacturers' recommendations. The conditioner was then removed from the cavities by means of air-water spray and the cavities were dried with cotton pellets. The cavities were restored with Fuji IX GC capsule. The restorations were covered with a GIC protective varnish.

Group 3: In this group, 10% maleic acid already prepared under laboratory conditions was applied to the prepared cavities for 30 seconds. The acid was removed from the cavities by means of the air-water spray and the cavities were dried with cotton pellets. The restorative processes were carried out as described above.

Group 4: 35% phosphoric acid gel (Voco GmbH, Post Pach 767, 27457 Cuxhaven, Germany) was applied to

the prepared cavities for 30 seconds. Then, the acid was removed from the cavities by means of air-water spray and the cavities were dried with cotton pellets. The conditioned cavities were restored and the process completed in a similar manner to that of the other groups.

Group 5: 3% H_2O_2 (AOSEPTR, Novartis) was applied to the prepared cavities for 30 seconds. Then, hydrogen peroxide was removed from the cavities by means of air-water spray and the cavities were dried with cotton pellets. The restorative process was carried out as described above.

Teeth that had completed restorations were kept in pure water at room temperature for 24 hours and exposed to thermocycling 200 times at a range of 4°C to 55°C. Then, all the enamel faces were covered with two layers of nail polish, 1-mm away from the cavity edges in case there were some cracks in the crowns of the teeth and dye leakage occur, which would affect the results. The teeth were soaked in 0.5% basic-fuchsin dye for 24 hours. They were then taken out of the dye and their crownal parts immersed in auto-polymerizing acrylic resin. The specimens were sectioned in a mesio-distal direction along their long axis using a low-speed saw. Both halves of each sectioned tooth were polished with Sof-Lex (3M Dental Products, St Paul, MN, USA) paper discs. Microleakage in both the mesial and distal parts of the cavities were evaluated under stereomicroscope (Nicon SMZ-V multi-point-sensor system, Japan) at magnifications of 60x for the leakage-grading procedure. The presence of microleakage was graded as follows:

0: No dye penetration

1: Dye penetration up to the DEJ

2: Beyond the DEJ and up to two-thirds the full length of cavity wall

3: Beyond two-thirds the full length of cavity wall but not involving the cavity base

4: Extending to the cavity floor

In order to determine whether there was a significant difference between the groups in terms of microleakage, Kruskal Wallis variance analysis was used and Mann-Whitney U-test was applied to find out which group and/or groups led to this difference.

In addition, two samples randomly chosen from each group were taken in order to evaluate in SEM (Jeal-6400-Japan) the Fuji IX-enamel and Fuji IX-dentin relation. The samples were processed with 10% orthophosphoric acid for 10 seconds and 5% sodium hypochlorite for five minutes, respectively, in order to gain better insight into the relationship between the dental hard tissues and Fuji IX and to remove remnants from the polished section surfaces. The surfaces of the samples were covered using Au-Pd by means of a POLARON Equipment Limited SEM Coating Unit E 500 (Comercial Assens-Llofrin SA, Barcelona, Spain). After the process of covering, the samples were displayed in SEM at 10.0 kV accelerating potential.

Table 1: *Microleakage Scores as a Result of the Methods Applied to the Samples*

	Microleakage Scores				
Methods	**0**	**1**	**2**	**3**	**4**
Control	10	8	2	--	--
Fuji Cavity Conditioner	20	--	--	--	--
Maleic Acid	17	2	1	--	--
Phopshoric Acid	20	--	--	--	--
H_2O_2	*14*	*5*	*1*	--	--

Table 2: *Results of Mann Whitney U-test*

Methods	**Difference**
Control	*a*
Fuji Cavity Conditioner	*b*
Maleic Acid	*b*
Phopshoric Acid	*b*
H_2O_2	*a,b*

The difference between the groups marked by the same letter or letters is statistically insignificant (p>0.05).

RESULTS

Table 1 presents the scores obtained by microleakage evaluation of the mesio-distal sections under a microscope taken from the teeth in which Fuji IX GC restorative material was placed after applying different conditioner agents to the cavities.

As seen in Table 1, the authors observed microleakage scores 1 and 2 in the control, maleic acid and hydrogen peroxide groups. However, none of the samples in any group showed microleakage scores of 3 and 4. Microleakage was seen in 50% of the samples in the control group, in 30% of those in the hydrogen peroxide group and in 15% of those in the maleic acid group; whereas, there was no microleakage in the Fuji Cavity conditioner group and the 35% phosphoric acid group.

In order to determine whether there was a statistically significant difference between the microleakage scores obtained from the groups, Kruskal-Wallis variance analysis was applied, with the result that there was a difference between the groups ($p<0.05$). To understand the source of this significant difference, Mann Whitney U-test was applied. The results are given in Table 2.

While there were significant differences between the control group and the Fuji Cavity Conditioner group, maleic acid group and phosphoric acid group in terms

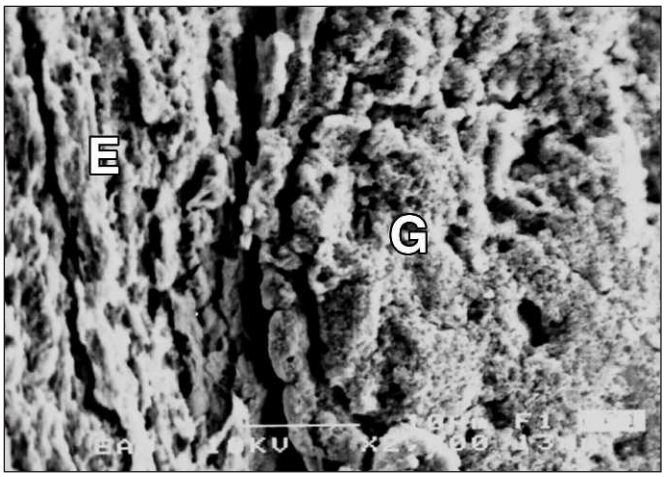

Figure 1. *There is a gap between Fuji IX and enamel. Also, cohesive failure of the Fuji IX adjacent to the interface (original magnification 2000x) (E: Enamel; G: Glass ionomer cement).*

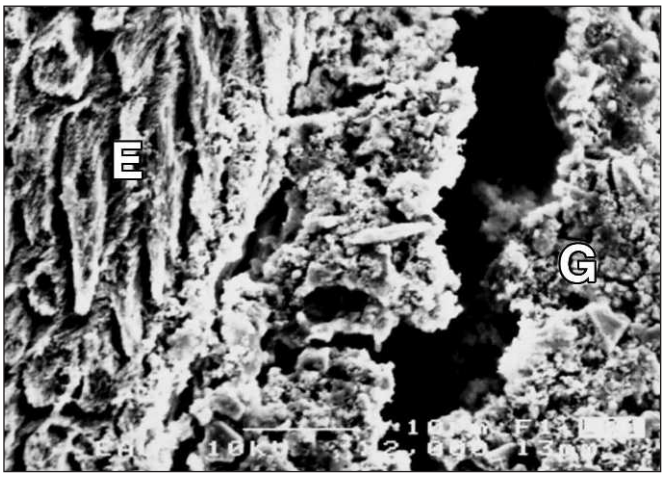

Figure 2. *There is a gap between Fuji IX and enamel. Also, cohesive failure of the Fuji IX adjacent to the interface (original magnification 2000x) (E: Enamel; G: Glass ionomer cement).*

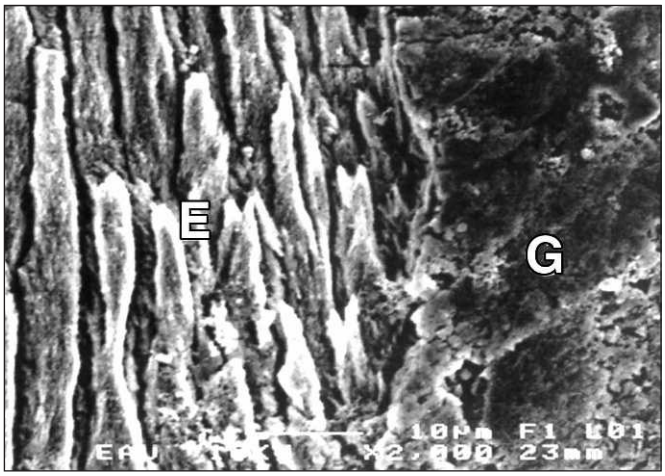

Figure 3. *There is an intimate relationship between Fuji IX and enamel (original magnification 2000x) (E: Enamel; G: Glass ionomer cement).*

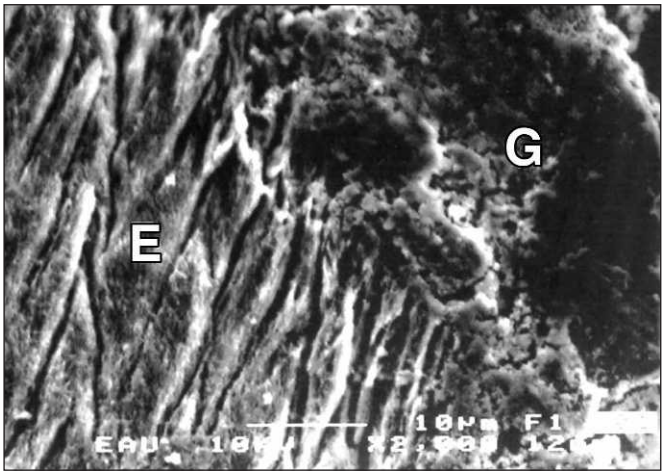

Figure 4. *There is a close relationship between Fuji IX and enamel (original magnification 2000x). Also, cohesive failure of the Fuji IX adjacent to the interface (original magnification 2000x) (E: Enamel; G: Glass ionomer cement).*

of microleakage ($p<0.05$), no significant difference was observed among the other groups ($p>0.05$).

According to SEM micrographs taken from the chosen samples, the presence of an interdiffusion zone between the enamel and Fuji IX was not observed in either the control group or the study groups (Figures 1-5). The maleic acid, phosphoric acid and Fuji Cavity Conditioner groups, on the other hand, showed intimate adaptation of Fuji IX to enamel (Figures 3-5). In addition, cohesive failures of Fuji IX were observed in parts close to the Fuji IX-enamel interface (Figures 1, 2, 4 and 5).

All of the conditioned specimens, excluding the control group, showed intimate adaptation of Fuji IX to the underlying dentin (Figures 8-11). In the control group, it was observed that the dentin tubule orifices were covered by GIC in some parts, while they were completely open in other parts. In addition, in the control group, the cement matrix-dentin interdiffusion zone was not observed (Figures 6 and 7). ABR layers of different thicknesses were observed in the hydrogen peroxide, maleic acid, Fuji Cavity Conditioner and phosphoric acid groups (Figures 8-11).

DISCUSSION

It was reported that in order for the GICs commonly used in pedodontics to be better bonded to dental hard tissues physico-chemically, the smear layer occurring during cavity preparation procedures should be removed with the use of proper conditioner agents that have different densities and application periods (Castro & Feigal, 2002; Watson, 1999). In this study, a statistically significant difference was found between the conditioner agents used to reduce microleakage ($p<0.05$).

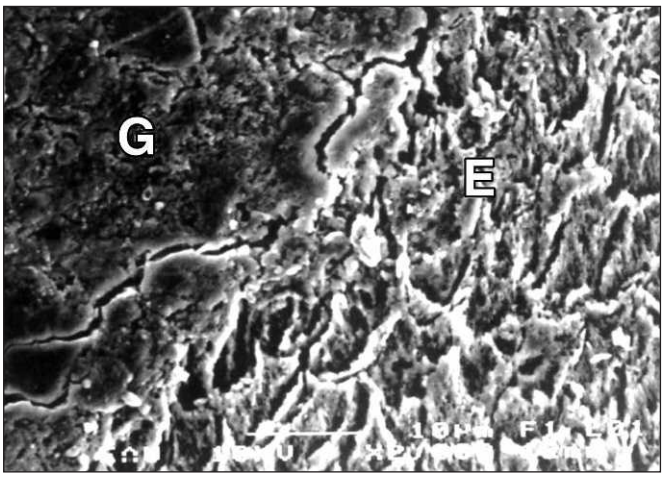

Figure 5. *There is a close relationship between Fuji IX and enamel (original magnification 2000x) (E: Enamel; G: Glass ionomer cement).*

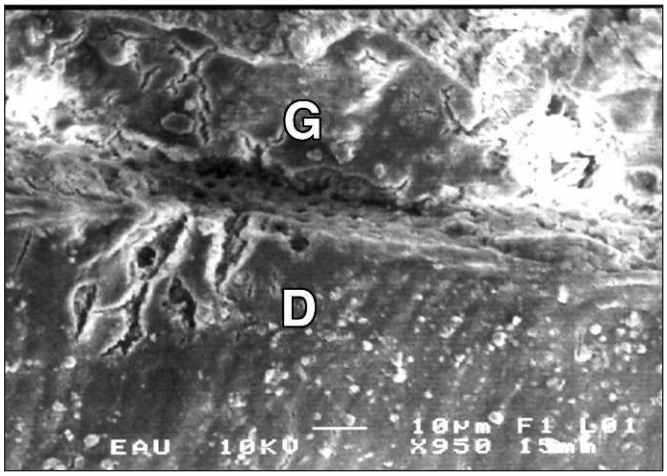

Figure 6. *Presence of a gap at the interface of Fuji IX-dentin (original magnification 950x) (D: Dentin; G: Glass ionomer cement).*

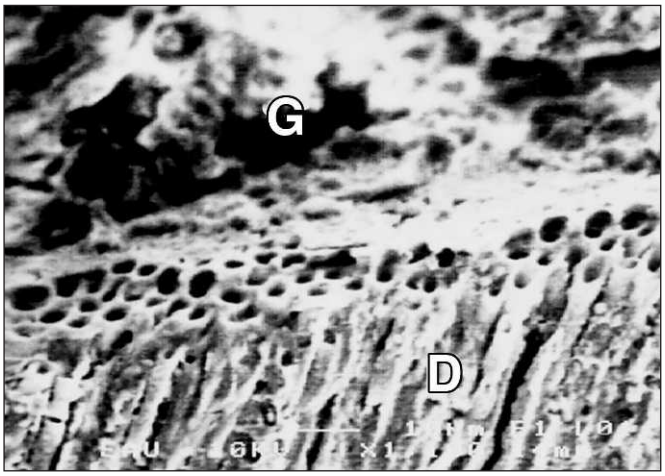

Figure 7. *While Fuji IX covers the tubule orifices in some areas of dentin, the tubule orifices in some other areas appear to be completely open (original magnification 1100x) (D: Dentin; G: Glass ionomer cement).*

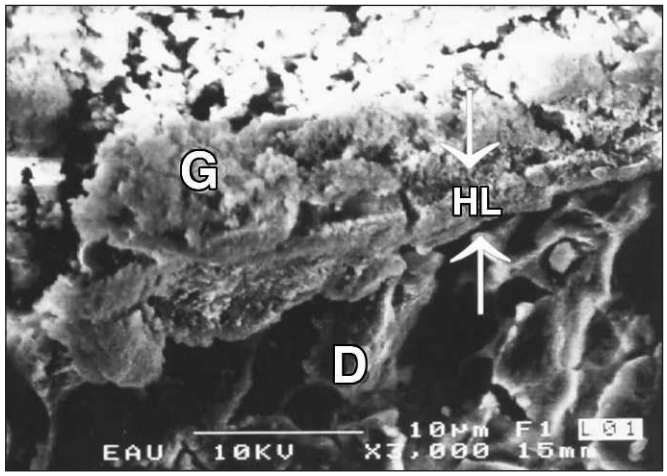

Figure 8. *The interactions zone between Fuji IX and dentin (original magnification 3000x) (D: Dentin; G: Glass ionomer cement; HL: Interdiffusion zone).*

Statistically significant differences appeared when the control group was compared with other groups except the H_2O_2 group. Microleakage scores of 3 and 4 were not observed in any of the groups.

Microleakage was observed in 50% of the control group samples, a percentage higher than in groups where different conditioner agents were used. These findings are in agreement with those of Castro and Feigal (2002). The reason why microleakage was high in this group could be understood by evaluating the SEM micrographs of dentin and enamel. This is because of the presence of a gap between Fuji IX and enamel (Figure 1). The ABR layer observed in other groups was not observed in this group (Figures 6 and 7). Between Fuji IX and dentin, the tubule orifices in some parts of the dentin were covered with Fuji IX, while the tubule orifices in others were completely open (Figures 6 and 7). In addition, no smear layer was observed in those areas (Figure 7). These findings disagree with reports by some researchers who reported that the demineralizing local effect of GIC could not dissolve the smear layer completely both because of the buffering effect of the tooth hard tissue mineral and the increase in initial pH of GIC over time (Blunck, 2000; Ben-Amar & others, 1999; Glasspoole, Erickson & Davidson, 2002).

Some manufacturers recommend the application of 3% H_2O_2 to cavities prior to placement of GIC restorative materials in the cavities. The authors found that the application of H_2O_2 to cavities before placement of Fuji IX decreased the probability of microleakage when compared with the control group. However, the difference between the microleakage scores in both groups was not statistically significant ($p>0.05$). No study was found that evaluated the influence of H_2O_2 application to the cavity on the microleakage of GIC. In light of the

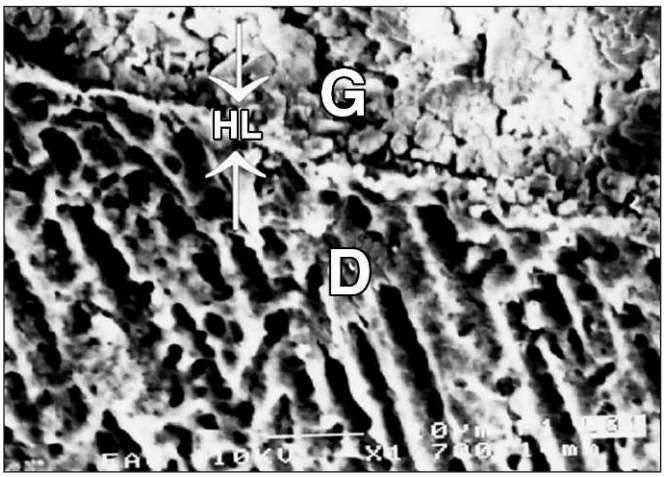

Figure 9. *The interactions zone between Fuji IX and dentin (original magnification 1700x) (D: Dentin; G: Glass ionomer cement; HL: Interdiffusion zone).*

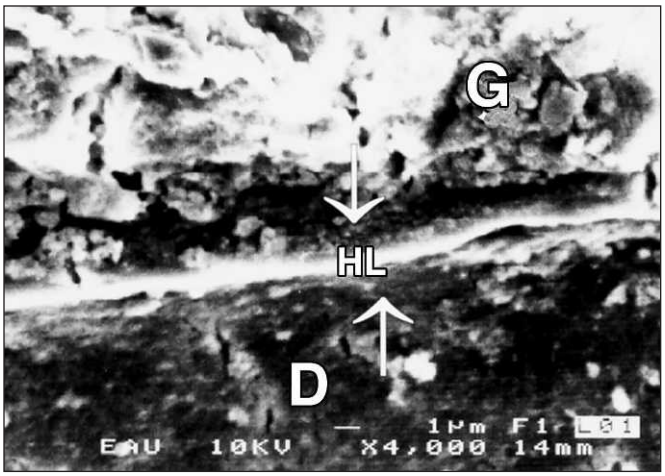

Figure 10. *A continous transition between the interdiffusion zone and the phosphoric acid conditioned dentin is shown. No gap was detectable at the interface between the two substrates (original magnification 4000x) (D: Dentin; G: Glass ionomer cement; HL: Interdiffusion zone).*

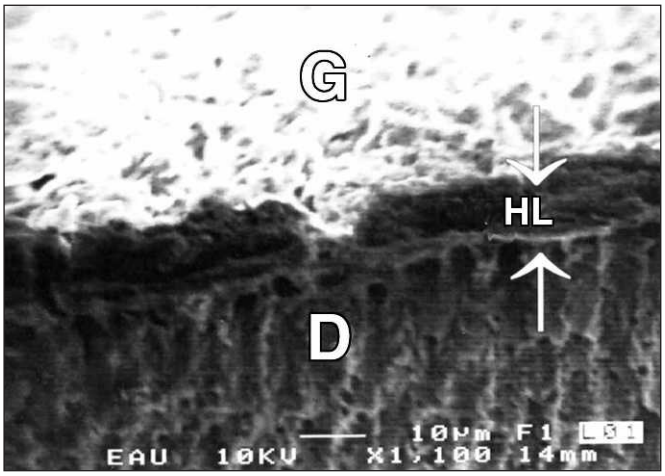

Figure 11. *A continous transition between the interdiffusion zone and the Fuji Cavity Conditioner conditioned dentin is shown. No gap was detectable at the interface between the two substrates (original magnification 1100x) (D: Dentin; G: Glass ionomer cement; HL: Interdiffusion zone).*

scores in the control group, it is believed that the decrease in microleakage is associated with the pH of H_2O_2 (pH=2 being calculated in a laboratory medium).

In the SEM micrographs taken from samples in the H_2O_2 group, a gap in the Fuji IX-enamel interface was observed, which was similar to the one in the control group (Figure 2). The presence of an ABR layer was observed in the Fuji IX-dentin interface (Figure 8). It was reported that the ABR layer that occurred between the GIC and dental hard tissues would lead to a decrease in the occurrence of microleakage in GIC restorations (Ngo & others, 1997). The microleakage scores in this group agree with this explanation.

Some microleakage was observed in 15% of the samples in which 10% maleic acid was used as a conditioner agent prior to placement of Fuji IX. When the microleakage scores observed in this group were compared with those in the control and H_2O_2 groups, some decrease was found. However, microleakage was not eliminated. The probable reasons for this might be that the maleic acid application failed to remove the smear layer completely in the samples with microleakage and, depending on the use of this acid, acid remnants affected the hardening reaction of the cement. Matos and others (1997) and Goes and others (1998) also showed that the 10% maleic acid application failed to completely remove the smear layer. On the other hand, this study was not concerned with comparing the capacity and efficiency of the conditioner agent applications for removing the smear layer.

Evaluation of the SEM micrographs of samples in the maleic acid group let the authors observe an intimate connection in the Fuji IX-enamel interface (Figure 3). Evaluation of the Fuji IX-dentin interface revealed a distinct ABR layer (Figure 9). These views might account for a decrease in microleakage scores in this group.

No observation of microleakage was noted in the 35% phosphoric acid gel and Fuji Cavity Conditioner groups in this study. Saito, Tosaki and Hirota (1999) reported that treatment of the enamel surface by phosphoric acid would not lead to a decrease in bonding strength of GICs and that bonding would be stabilized. They also stated that polyacrylic acid–aluminum chloride ($AlCl_3$) contained in Fuji Cavity Conditioner used in this study was influential on increasing the cement's adhesiveness. $AlCl_3$ was also reported to decrease the pH of polyacrylic acid to a larger extent (Tanumiharja & others, 2000). Accordingly, Glasspoole and others (2002) stated that the conditioners might also produce microporosity

in the enamel surface, which could contribute to either increased surface area for chemical bonding or micromechanical bonding. The fact that no microleakage, even score 1, was observed in any of samples in both groups and that an intimate Fuji IX-enamel interface was observed in the SEM micrographs, supports these ideas (Figures 4 and 5).

Watson (1999) reported that the use of strong acids would lead to emergence of the collagen network in dentin, thus decreasing bonding strength. It was also reported that polyacrylic acid has a minor effect on dentin, removing the smear layer and surface contaminants without opening the dentin tubules too widely (Powis & others, 1982). Tanumiharja and others (2001) showed that there was no smear layer in the dentin tubules and dentin surfaces when Fuji Cavity Conditioner agent was applied, and that Fuji IX cement matrix penetrated into the demineralized dentin, which occurred as a result of the application. In this study, a visible ABR layer in the Fuji IX-dentin interface was observed in SEM micrographs from both groups (Figures 10 and 11). This ABR layer, observed in the Fuji Cavity Conditioner group, is similar to the findings of Ferrari and Davidson (1997).

CONCLUSIONS

1. This *in vitro* study proved that applying conditioner agent to the cavity before applying high-viscosity GICs to primary teeth cavities could decrease or eliminate microleakage.
2. Although recommended by some manufacturers as the proper conditioner agent, H_2O_2 was not found to be influential in decreasing microleakage.
3. As the proper conditioner agents similar to those of traditional GICs, agents with polyacrylic acid contents would be suitable for high-viscosity GICs used in cavities requiring a minimal cavity preparation and in the ART technique.

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Microleakage at the Composite-repair Interface: Effect of Different Surface Treatment Methods

AN Cavalcanti • MM Lobo • CM Fontes
P Liporoni • P Mathias

Clinical Relevance

Surface preparation and the use of an adhesive system promote an adequate bonding, able to prevent microleakage at the repair interface.

SUMMARY

This study evaluated microleakage at the composite-repair interface after using different methods of surface treatment. Eighty resin composite specimens (Filtek Z250, 3M Dental Products) aged in artificial saliva for three months were divided into four groups (n=20) according to the following surface treatment methods: untreated control–no roughening or abrasion of the surface; roughening with diamond burs; jet prophylaxis with sodium bicarbonate particles and air abrasion with 50 µm aluminum oxide particles. Each method was examined using scanning electron microscopy (SEM) to evaluate changes in surface topography. All groups were then etched with 37% phosphoric acid, coated with a bonding agent (Single Bond, 3M Dental Products) and received new resin applications. The samples were then thermocycled (800 cycles/5°C to 55°C [±2]) and immersed in 2% methylene blue buffered dye solution (7.0 pH) for four hours. Three examiners measured the extent of microleakage in a stereoscope microscope using four representative scores. For all experimental groups, no significant difference in repair microleakage was identified by the Kruskal-Wallis test ($p>0.05$). Therefore, different testing methods of surface treatment showed the same effect on dye penetration along the repair interface.

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INTRODUCTION

Resin composites have been largely used in direct restorations. Since their introduction, several studies

have focused on their physical properties, improving esthetics and longevity (Anusavice, 1998). However, current composites still exhibit problems with material fractures (Denehy, Bouschlicher & Vargas, 1998), discolored or worn areas (Brosh & others, 1997), poor anatomic form, secondary caries and tooth fracture and pain/sensitivity (Mjör, Dahl & Moorhead, 2000).

Complete removal of defective composite restorations may lead to larger cavities with further loss of tooth structure (Denehy & others, 1998; Lewis & others, 1998). Such treatment involves difficulties such as recognizing the composite-tooth interface and the need for removing previously etched enamel to enable a new bonded restoration to be made (Söderholm & Roberts, 1991; Shahdad & Kennedy, 1998). In addition, total replacement increases pulpal trauma and the cost of the procedure (Denehy & others, 1998).

For this reason, reparability is now recognized as a highly desirable property of a restorative material (Lewis & others, 1998). Repair requires less work time, is cost-effective and represents the most conservative procedure for defective restorations (Brosh & others, 1997). According to Turner and Meiers (1993), repair situations occur regardless of the type of resin or technique used, whether macrofill, hybrid, microfill, chemical cure, light cure, heat cure, direct or indirect.

However, such an approach can also result in weaker restorations (Söderholm & Roberts, 1991; Shahdad & Kennedy, 1998). Therefore, successful resin repair requires the development of an adequate interfacial bond between the old and new resins. Several composite repair studies have shown that a surface treatment and the use of an intermediate bonding agent enhances the repair bond significantly (Söderholm, 1986; Pounder, Gregory & Powers, 1987; Swift Jr, LeValley & Boyer, 1992; Brosh & others, 1997; Lucena-Martín, González-López & Navajas-Rodrígues de Mondelo, 2001; Frankenberger & others, 2003; Öztas, Alaçam & Bardakcy, 2003). While surface roughness promotes mechanical interlocking, the bonding agent advances surface wetting and chemical bondings with the new composite (Brosh & others, 1997).

Various methods have been indicated for preparation of the surface to be repaired: acid etching, air abrasion, roughening with diamond burs and other abrasive papers (Brosh & others, 1997; Lucena-Martín & others, 2001; Öztas & others, 2003). Indication for the use of any one or more of these combinations can depend on the substrate surface to which it is to be bonded (Denehy & others, 1998). Hydrofluoric acid seems to have little effect on microfilled resins (Swift Jr, Cloe & Boyer, 1994), although other surface treatments seem to work well on the different types of composites (Kupiec & Barkmeier, 1996; Brosh & others, 1997; Lucena-Martín & others, 2001; Öztas & others, 2003).

According to Lewis and others (1998), efficiency of the repair procedure is related to the magnitude of the bond strength obtained at that interface. However, a clinically adequate bonding should also be able to prevent microleakage at its interface (Hadavi & others, 1993). This leakage leads to deterioration of the bond and accelerates failure (Chalkley & Chan, 1986; Saunders, 1990). In addition, interfacial staining could compromise esthetics, especially in anterior teeth and, as a result, require the entire restoration to be replaced (Denehy & others, 1998).

Therefore, this study aimed to evaluate the effects of different surface treatments on microleakage at the composite repair interface using a qualitative dye penetration analysis. Also, each method was examined using scanning electron microscopy (SEM) to evaluate changes in surface topography.

METHODS AND MATERIALS

Eighty specimens were made in a quadrangular acrylic resin mold with an internal space of 6 x 6 x 2 mm. A removable acrylic resin spacer was inserted to make half-length specimens (3 x 6 x 2 mm) in the first part of this study. Initially, half the mold was filled with resin composite Filtek Z250 (3M Dental Products, St Paul, MN, USA) in a single increment. A glass microscope slide was placed over the mold and pressed to remove excess material. Each specimen was light cured through the slide for 10 seconds using a visible light-curing unit (Optilight 600, Gnatus, Riberão Preto-SP, Brazil). The tip of the curing light was kept at a 90-degree angle to the top surface, in contact with the glass to achieve maximum curing depth. After the top surface had been cured, the specimens were carefully removed from the mold and another light exposure of 40 seconds was applied to specimen surfaces.

Eighty half-length specimens were then stored in artificial saliva (Shinkai, Cury & Cury, 2001) at 37°C for three months. They were then randomly allocated into four groups according to the following surface treatment methods:

Group C: Control–untreated surface

Group DB: Roughening with a diamond bur (n.1092, KG Sorensen Ind, Barueri-SP, Brazil) with a high-speed handpiece and copious water spray to simulate clinical removal of a thin layer of the old restoration

Group JP: Jet prophylaxis with sodium bicarbonate particles (Profi II, Dabi Atlante SA, Riberão Preto-SP, Brazil) for 15 seconds at 2.2 atmospheric pressure and 10-mm distance

Group AO: Sandblasting with a microetcher (Microetch, Bioart, São Carlos-SP, Brazil) for 10 seconds with 50 µm aluminum oxide particles

Each surface was rinsed with distilled water for 20 seconds and dried with compressed air for 10 seconds. The samples were cleaned with 37% phosphoric acid gel (3M Dental Products) for 60 seconds, rinsed vigorously with tap water and dried with oil-free compressed air. A bonding agent (Single Bond, 3M Dental Products) was applied according to the manufacturer's instructions and cured by 10 seconds of exposure to visible light. Each specimen was then placed back into the mold with no spacer inserted. The repair composite was applied and cured as described above, completing the repair procedure. A dark shade was selected as the repair material (C4) and a lighter one as the substrate (A1)—so there could be better assessment of the repair interface. Finishing procedures were made after 24 hours, with a decreasing abrasive sequence of aluminum oxide disks (Sof-Lex, 3M Dental Products).

Table 1: *Distribution of Dye Penetration Scores*

Groups	Score 0	Score 1	Score 2	Score 3	*Median Score*
Control (C)	14	5	-	-	0
Diamond bur (DB)	16	4	-	-	0
Jet Prophylaxis (JP)	16	4	-	-	0
Aluminum oxide (AO)	16	4	-	-	0

**Kruskal-Wallis/p>0.05 (non significant)*

The repaired specimens were removed from the mold and stored in distilled water for 24 hours. The external surfaces of each sample were coated with two layers of nail varnish, with the exception of the side directly exposed to the curing light. They were then thermocycled for 800 cycles between 5°C (±2) and 55°C (±2) with a one-minute dwell time at each temperature, then immersed for four hours in a 2% methylene blue buffered dye solution (pH 7.0).

Microleakage

The samples were transversely sectioned with a double-faced diamond disk (n.7020, KG Sorensen Ind, Barueri-SP, Brazil). Three independent examiners measured the extent of microleakage with a stereoscope microscope (40x) according to the following scores:

0 = absence of dye penetration

1 = up to 1/2 of repair interface

2 = more than 1/2 of repair interface, without total involvement

3 = complete repair interface involvement

The three evaluators were pre-calibrated before the onset of this project. The inter-rater reliability of the scores was expressed as Cohen's Kappa (Pett, 1997). Data were analyzed statistically using a non-parametric Kruskal-Wallis test at a significance level of 5% (Hollander & Wolfe, 1973; Conover, 1980).

Scanning Electron Microscopy (SEM)

Additional composite specimens were made, treated (except the control) and examined using scanning electron microscopy (JEOL, JMS, 5310) to evaluate changes in surface topography. The samples were gold sputter coated and examined using an acceleration voltage of 10kV (LAS/IMPE, São José dos Campos—SP, Brazil).

RESULTS

The overall values of the inter-rater reliability were excellent and expressed in a range of 0.82 to 0.90 Kappa values.

The median scores of all treatment groups were not significantly different from one another (Kruskal-Wallis ANOVA; $p>0.05$). Results showed little or no dye penetration at the repair interface—including the control group (Score variation from 0 to 1) (Table 1).

SEM images showed morphologic differences in surfaces after the different treatments compared with the control (Figures 1A-1D). The air abrasion method with 50 µm aluminum oxide particles and jet prophylaxis with sodium bicarbonate particles (Figures 1C and 1D) created rougher surfaces than the control group and diamond bur technique (Figures 1A and 1B). However, as described previously, this alteration did not result in significant differences between the groups.

DISCUSSION

This study was conducted to determine the effects of different surface treatment methods on composite-repair microleakage. To restrict the bond mechanism to micromechanical retention, the half-length specimens were aged for three months in artificial saliva, thus avoiding chemical bonding between methacrylate radicals from substrate resin and repair resin. This period was used because, according to Turner and Meiers (1993), the likelihood of achieving covalent bonding between resins appears to be converse to the age of the substrate resin.

Despite the differences in surface topography observed among the experimental groups (SEM images), results indicated that the surface treatments used (DB, JP, AO) did not influence dye penetration along the repair interface ($p>0.05$). All groups showed a score range from 0 to 1, including the control group, which received no roughening or abrasion of the surface.

From this data it is not possible to consider the kind of surface treatment as being responsible for sealing

the repair, because the control group showed the same score range as the other experimental groups. Considering that bond application (Single Bond) was standardized for all groups, it is reasonable to assume that it may have been the main reason for the groups' behavior, by decreasing dye penetration in the control group as well. Thus, any possible surface treatment effects may have been masked by the use of a bonding agent and those effects probably could be better evaluated if negative control groups (without bonding agent) were added to the study.

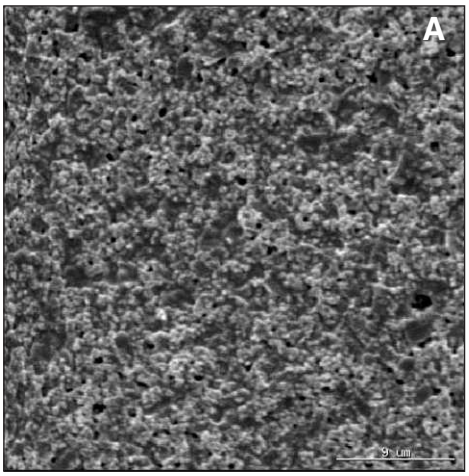

Figure 1A. *Scanning electron micrograph of the treated surfaces and control (3.500x magnification). Morphologic differences are seen after surface treatments, compared with control group (1A (original magnification 1100x) (D: Dentin; G: Glass ionomer cement).*

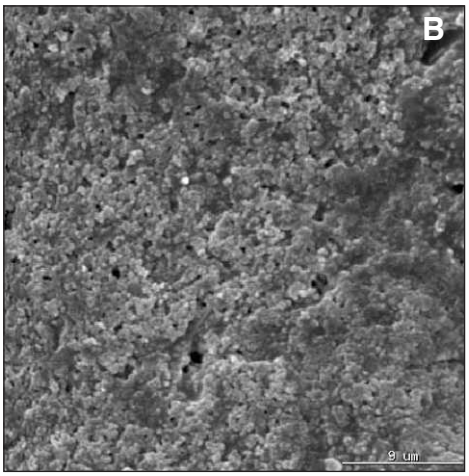

Figure 1B. *Roughening with diamond burs.*

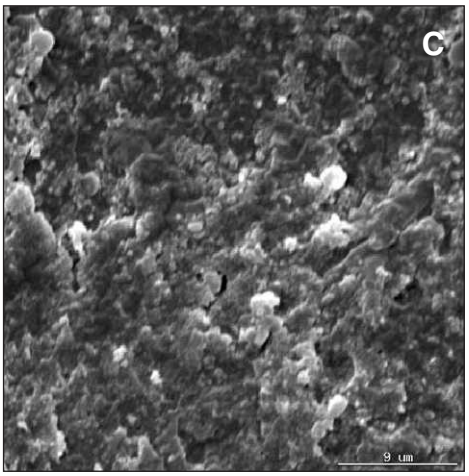

Figure 1C. *Jet prophylaxis with sodium bicarbonate particles.*

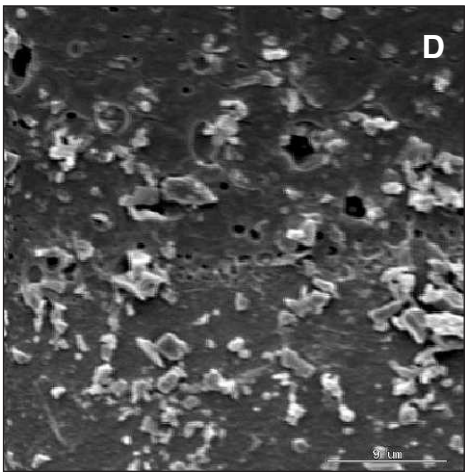

Figure 1D. *Air abrasion with 50 µm aluminum oxide particles.*

Brosh and others (1997) found that unfilled bis-GMA resin, alone or combined with silane, was the most effective procedure for enhancing the shear bond strength of the repaired composite specimens, irrespective of the surface texture created by different surface treatments. This finding is in agreement with this study, in which the control group–untreated—showed no significant difference from the other experimental groups.

Apart from the good interfacial sealing provided by the adhesive system in this study, it is uncertain whether its use, alone, without any kind of surface treatment, could maintain high repair bond strength. Most composite repair studies have indicated that surface roughness has a greater influence on repair strength than using a bonding agent (Swift Jr & others, 1992, 1994; Kupiec & Barkmeier, 1996; Turner & Meiers, 1993; Lucena-Martín & others, 2001; Öztas & others, 2003). Also, it has been shown that bond strength values of composite repairs were slightly higher when using an unfilled bonding agent but were not significantly different from treated surfaces (Kupiec & Barkmeier, 1996). Thus, it seems reasonable to suggest that efficient bonding agents and a surface treatment should be used to optimize repair procedure.

Otherwise, the results of this study are for repaired specimens stored for 24 hours in water and aged by 800 thermal cycles. Longer storage or increased thermocycling may present other results (Azarbal, Boyer & Chan, 1986; Kupiec & Barkmeier, 1996), which may also be influenced by different bonding agents. Therefore, further studies should be conducted to investigate the effect of these variables on repair microleakage.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions may be drawn:

1. Microleakage at the composite-repair interface was not influenced by surface treatments;
2. Bond application may have been the main reason for the groups' behavior, masking possible effects of surface treatments.

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Polymerization Shrinkage of Flowable Resin-based Restorative Materials

MM Stavridakis • D Dietschi • I Krejci

Clinical Relevance

Polymerization shrinkage is a critical limitation of flowable resin-based restorative materials. The dental community, although aware of the problem, should place more emphasis on this property. In this study, a wide range of values was measured for both polymerization shrinkage properties that were studied. Dentists should be aware of these differences in order to choose the more suitable material for each clinical use.

SUMMARY

This study measured the linear polymerization displacement and polymerization forces induced by polymerization shrinkage of a series of flowable resin-based restorative materials.

The materials tested were 22 flowable resin-based restorative materials (Admira Flow, Aelite Flow, Aeliteflow LV, Aria, Crystal Essence, Definite Flow, Dyract Flow, Filtek Flow, FloRestore, Flow-it, Flow-Line, Freedom, Glacier, OmegaFlo, PermaFlo, Photo SC, Revolution 2, Star Flow, Synergy Flow, Tetric Flow, Ultraseal XT and Wave). Measurements for linear polymerization displacement and polymerization forces were performed using custom made measuring devices. Polymerization of the test materials was carried out for 60 seconds by means of a light curing unit, and each property was measured for 180 seconds from the start of curing in eight specimens for each material. Statistical evaluation of the data was performed with one-way analysis of variance (ANOVA), Tukey's Studentized Range (HSD) test (p=0.05) and simple linear regression.

A wide range of values was recorded for linear polymerization displacement (26.61 to 80.74 microns) and polymerization forces (3.23 to 7.48 kilograms). Statistically significant differences among materials were found for both properties

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studied. Very few materials (Freedom, Glacier, and Photo SC) presented low values of linear polymerization displacement and polymerization forces (similar to hybrid resin composites), while the majority of materials presented very high values in both properties studied. Study of the shrinkage kinetics revealed the exponential growth process of both properties. The polymerization forces development exhibited a few seconds delay over linear polymerization displacement. Simple linear regression showed that the two polymerization shrinkage properties that were studied were not highly correlated (r^2=0.59).

INTRODUCTION

Flowable composites have been available to the dental market for a number of years. Lately, their popularity has increased to the point where most of the dental manufacturers have at least one flowable composite in their list of products. Manufacturers are proposing these materials for a variety of clinical procedures, most often without the necessary research being performed to back up the claims.

It is true that flowable composites present two desirable clinical handling characteristics: non-stickiness and fluid injectability (Bayne & others, 1998). Flowable composites are said to be easy to use due to their flow characteristics, which sometimes seem to promote the ease of placement as the material "flows" and "self-adapts" to cavity walls. This property seems advantageous for placement of the restorative material in micro-conservative occlusal cavities with very limited width but may cause problems such as marginal overhangs in cervical margins of beveled proximal boxes (Frankenberger & others, 1999).

As the "flowability" of flowable composites is mainly achieved by lowering filler loading, since their early evaluation, these materials were speculated to present increased polymerization shrinkage (Bayne & others, 1998). This was confirmed in a study where polymerization shrinkage of a series of materials, including a few flowable composites, was measured and high polymerization shrinkage values (3.65 to 6.0%) were reported for flowable composites (Labella & others, 1999). Another study demonstrated that flowable composites exhibited higher polymerization shrinkage than other resin-based restorative materials, such as ultrafine midway-filled resin composites (mainly used in anterior restorations), ultrafine compact-filled resin composites (proposed by manufacturers as "amalgam substitutes" for posterior restorations) and poly-acid-modified resin composites (compomers) (Stavridakis, Kakaboura & Krejci, 2000).

Lately, a number of new products and improved versions of previous materials have been introduced to the dental market with little, if any, data concerning the polymerization shrinkage behavior of these materials. It is well known that flowable composites comprise a rather inhomogeneous group of materials that exhibit variable radiopacity (Murchison, Charlton & Moore, 1999; Bouschlicher, Cobb & Boyer, 1999) and flow characteristics and film thickness formation (Moon, Tabassian & Culbreath, 2002). Therefore, it was the aim of this study to investigate the variability of polymerization shrinkage of flowable resin-based restorative materials. To this purpose, the linear polymerization displacement and polymerization forces induced by polymerization shrinkage of a series of flowable resin composites were measured.

METHODS AND MATERIALS

Measurements of linear polymerization displacement induced by polymerization shrinkage were performed with a custom made measuring device (Figure 1) that was developed following de Gee, Feilzer and Davidson (1993). It consisted of a stable metal frame upon which a thin aluminum platelet (13 x 20 mm) weighing 0.6 gr was loosely placed. On the underside of the platelet, a diaphragm 8 x 8 mm was glued centrally at right angles to the surface, the diaphragm consisting of a strip of photographic film impervious to infrared light (Film DX 135, ISO 50/18°, Panf Plus, Ilford, United Kingdom). The edge of the diaphragm extended into a recess in the infrared measuring sensor. A standardized amount of the material to be investigated was placed on the aluminum platelet with the aid of a cylindrical Teflon mold. The material was then carefully flattened by means of a glass plate, dimensions 41 x 18 x 1 mm, to a test height of 1.5 mm. Polymerization of the test materials was carried out for 60 seconds by means of a light-curing unit (Translux EC, Kulzer & Co GmbH, Wehrheim, Germany) with a relative intensity of 500 mW/cm^2 (Curing Radiometer, Demetron Research, Danbury, CT, USA) from a 1 mm distance through the glass plate. The vertical movement of the

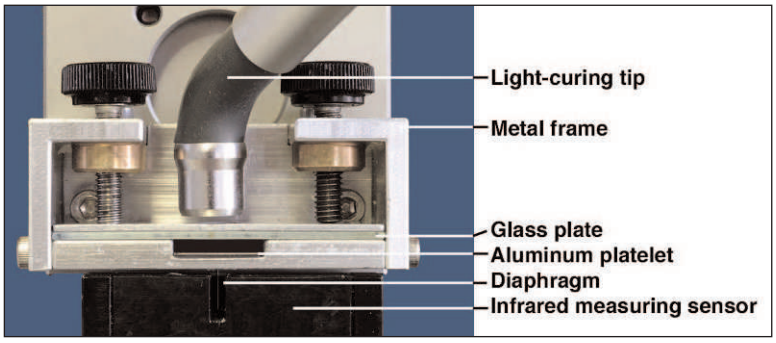

Figure 1. *Detailed image of the custom made measuring device that was used for the measurement of linear polymerization displacement.*

diaphragm caused by polymerization shrinkage of the test material was detected for 180 seconds by the infrared sensor with a 100-nm accuracy and a sampling frequency of 5 Hz. The data were fed online by means of an A/D converter and custom made software to a personal computer (Macintosh II fx, Apple Computer, Cupertino, CA, USA) and were stored on its hard disc. Eight measurements were carried out on each test material and their mean values were plotted.

Measurements for polymerization forces induced by polymerization shrinkage were performed with a custom made measuring device (Figure 2) that was developed following Feilzer, de Gee and Davidson (1987). In contrast to Feilzer's set-up, the upper part consisted of a semi-rigid load cell (PM 11-K, Mettler, Greifensee, Switzerland) to which a metal cylinder 8 mm in diameter was screwed. The load cell was axially displaced 4 µm for each kilogram of measured force. In this way, the natural deformation of the cavity walls was mimicked. The cylinder was coated with the test material that was compressed at a distance of 1.5 mm onto a glass plate attached to the base of the device. To improve adhesion, the surfaces of the metal cylinder and the glass plate were sandblasted with 50 µm Al_2O_3 (Microetcher, Danville Engineering, Danville, CA, USA) and silanized (Monobond S, Vivadent Ets, Schaan, Liechtenstein). Light curing (Translux EC, Kulzer & Co GmbH, Wehrheim, Germany) with a relative intensity of 500 mW/cm^2 (Curing Radiometer, Demetron Research, Danbury, CT, USA) was carried out for 60 seconds via a recess in the lower frame through the glass plate at a distance of 1 mm from the test material. The forces that built up during polymerization shrinkage were detected for 180 seconds by means of the load cell at a sampling frequency of 5 Hz. The data were fed online into the computer via an A/D converter using custom made software. Eight measurements were carried out for each material and their mean values plotted.

Twenty-two flowable resin composites were tested in this research (Table 1). Statistical evaluation of the data at the end of the 180-second observation period

Table 1: *Manufacturers, Colors, Batch Numbers and Expiration Dates of the Materials Studied*

	Manufacturer	Batch #
Admira Flow	VOCO GmbH, PO Box 767 Cuxhaven, Germany	005180
Aelite Flow	BISCO, Inc, 1100 W Irving Park Rd Schaumburg, IL, USA	100400
Aeliteflow LV	BISCO, Inc, 1100 W Irving Park Rd Schaumburg, IL, USA	0100001004
Aria	Danville Materials, 2021 Omega Rd San Ramon, CA, USA	505255
Crystal Essence	Confi-Dental Products Co, 416 S Taylor Avenue Louisville, CO, USA	111400
Definite Flow	Degussa Dental GmbH & Co, Postfach 1364 Hanau, Germany	30000497
Dyract Flow	DENTSPLY Caulk, 38 West Clarke Avenue PO Box 359, Milford, DE, USA	98110000554
Filtek Flow	3M ESPE Dental Products, Building 275-2SE-03 3M Center, St Paul, MN, USA	200090047
FloRestore	Den-Mat, 2727 Skyway Drive Santa Maria, CA, USA	301014
Flow-it	Jeneric/Pentron Inc, PO Box 724 Wallingford, CT, USA	24882
FlowLine	Heraeus Kulzer GmbH & Co KG Grüner Weg 11, Hanau, Germany	030021
Freedom	SDI Inc, 729 N Route 83, Suite 315 Bensenville, IL, USA	9910-20
Glacier	SDI Inc, 729 N Route 83, Suite 315 Bensenville, IL, USA	000847
OmegaFlo	Sterngold, PO Box 2967, 23 Frank Mossberg Drive Attleboro, MA, USA	00090047
PermaFlo	Ultradent Products, Inc, 505 West, 10200 South South Jordan, UT, USA	AO11
Photo SC	Kuraray Medical Inc, 1621 Sakazu, Kurashik Okyama, Japan	0017
Revolution 2	Kerr Dental, 1717 West Collins Orange, CA, USA	908866
Star Flow	Danville Materials, 2021 Omega Rd San Ramon, CA, USA	505621
Synergy Flow	Coltène AG, Feldwiesenstrasse 20 Altstätten, Switzerland	JJ01
Tetric Flow	Ivoclar Vivadent AG, Bendererstrasse 2 Schaan, Lichtenstein	B00036
Ultraseal XT	Ultradent Products, Inc, 505 West, 10200 South South Jordan, UT, USA	34101
Wave	SDI Inc, 729 N Route 83, Suite 315 Bensenville, IL, USA	000536

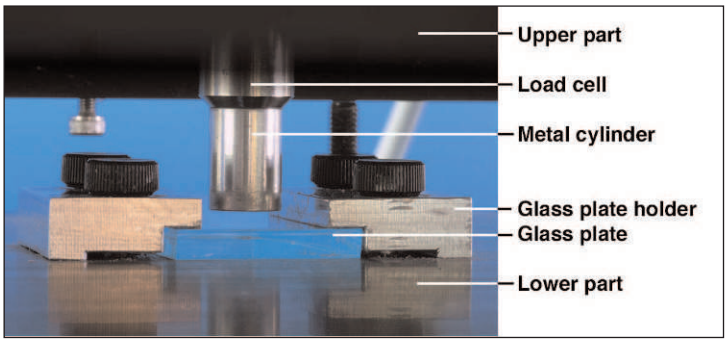

Figure 2. *Detailed image of the custom made measuring device that was used for the measurement of polymerization forces.*

was performed with one-way analysis of variance (ANOVA) at the 0.05 level of statistical significance. Upon identification of a significant difference, Tukey's Studentized Range (HSD) Test (p=0.05) was used to identify any pairwise differences. Simple linear regression was also performed between the two polymerization shrinkage properties that were studied.

The difference in evolution of the two polymerization shrinkage properties over time was also calculated using the following equation:

Equation 1.

$$A_{180} = \frac{\left| \sum_{i=0}^{180} \frac{y_{2i}}{y_{2_{180}}} - \sum_{i=0}^{180} \frac{y_{1i}}{y_{1_{180}}} \right|}{\sum_{i=0}^{180} \frac{y_{1i}}{y_{1_{180}}}} \times 100\%$$

where:

$A_{180}\rightarrow$ is the result, expressed as a percentage, from deduction of the two development curves of percentage values of the two properties.

$y_{1i}\rightarrow$ are the percentage values coming from the actual measurement of the linear polymerization displacement.

$y_{1_{180}}\rightarrow$ is the maximum percentage value of the linear polymerization displacement.

$y_{2i}\rightarrow$ are the percentage values coming from the actual measurement of the polymerization forces.

$y_{2_{180}}\rightarrow$ is the maximum percentage value of the polymerization forces.

This effort tried to depict the area (difference) coming from the deduction of the two curves: the development curve of percentage values of linear polymerization displacement over time and the development curve of percentage values of polymerization forces over time. Therefore, Equation 1 was able to show the area that expresses the result of the deduction of the two aforementioned curves and expresses it in percentage points in relation to the larger curve (that of linear polymerization displacement).

RESULTS

Table 2 compiles the linear polymerization displacement results at the end of the observation period. A wide range of values was observed (26.61 to 80.74 microns). The statistical analysis showed statistically significant differences among materials. The numerous subgroups of different statistical significance noted were due to the small standard deviations that were observed. Figure 3 displays the progression of mean values of linear polymerization displacement of each material over time. Observation of the linear polymerization displacement vs time curves of all the materials tested supports the exponential growth process of this property. Even though all materials did not display identical time-dependence patterns, the majority of the materials exhibited a near-linear growth pattern after the first one to two seconds, with the exception of Glacier and Freedom, which exhibited a more marked S-shape curve and a less steep inclination during the first 10 seconds.

Table 3 compiles the results of the polymerization forces at the end of the observation period. A wide range of values was also observed (3.23 to 7.48 kilograms). The statistical analysis, once again, showed statistically significant differences among materials, and the numerous subgroups of different statistical significance that were noted were attributed to the small standard deviations that were observed. These small standard deviations noted in both polymerization properties reflected the high consistency of the measuring techniques used in this study. Figure 4 displays the progression of mean values of polymerization force of each material over time. Observation of the polymerization force vs time curves of all the materials tested also supports the exponential growth process of this property. Even though, once again, all materials did not display identical time-dependence patterns, the majority of the materials exhibited a near-linear growth pattern after a three-to-nine second delay, except for Ultraseal XT, Crystal Essence and Glacier, which exhibited a more marked S-shape curve and a less steep inclination during the first 15 seconds.

The r-square value (0.59) of the simple linear regression (r=0.77) that was performed revealed that the two polymerization shrinkage properties studied were cor-

Table 2: *Linear Polymerization Displacement Results (in microns)*

Material	N	Mean	SD	Statistical Significance					
Freedom	8	27.95	2.21	A					
Glacier	8	29.61	1.60	A					
Photo SC	8	31.69	1.40	A					
Definite Flow	8	40.55	1.91		B				
Tetric Flow	8	45.53	1.08			C			
Crystal Essence	8	46.04	1.84			C			
Aria	8	46.61	1.57			C			
Wave	8	47.31	3.72			C	D		
FloRestore	8	50.73	1.63				D	E	
Filtek Flow	8	52.84	1.71					E	
Synergy Flow	8	53.43	2.16					E	
Ultraseal XT	8	54.25	1.73					E	F
Flow-it	8	58.03	1.63	G					F
Admira Flow	8	60.49	3.37	G	H				
Flow-Line	8	61.09	1.20	G	H	I			
OmegaFlo	8	61.64	1.78	G	H	I			
Revolution 2	8	63.01	1.63		H	I	J		
Star Flow	8	63.21	1.36		H	I	J		
Aelite Flow	8	64.38	2.15			I	J		
Aeliteflow LV	8	64.71	2.29			I	J		
Dyract Flow	8	66.52	1.99				J		
PermaFlo	8	74.19	3.70					K	

Materials with same letters did not exhibit statistical difference at the 0.05 level of statistical significance.

Table 3: *Polymerization Forces Results (in kilograms)*

Material	N	Mean	SD	Statistical Significance					
Photo SC	8	3.43	0.07	A					
Glacier	8	3.44	0.14	A					
Freedom	8	3.89	0.17		B				
Wave	8	4.25	0.21		B	C			
Tetric Flow	8	4.31	0.13			C	D		
Ultraseal XT	8	4.49	0.18			C	D		
Synergy Flow	8	4.57	0.23			C	D		
Crystal Essence	8	4.66	0.29				D		
FloRestore	8	4.69	0.21				D		
Revolution 2	8	5.16	0.21					E	
Flow-it	8	5.26	0.21					E	F
Definite Flow	8	5.48	0.19	G				E	F
PermaFlo	8	5.59	0.25	G	H				F
Admira Flow	8	5.60	0.39	G	H	I			F
Flow-Line	8	5.67	0.11	G	H	I			F
Aelite Flow	8	5.80	0.22	G	H	I			
Aria	8	5.90	0.19		H	I			
Filtek Flow	8	6.00	0.24			I	J		
OmegaFlo	8	6.34	0.35				J	K	
Star Flow	8	6.41	0.30				J	K	
Dyract Flow	8	6.60	0.24					K	
Aeliteflow LV	8	7.18	0.23						L

Materials with same letters did not exhibit statistical difference at the 0.05 level of statistical significance.

related, but not very strongly. Figure 5 illustrates the correlation between the two properties studied in this research protocol. In this graph, materials (such as Filtek Flow, Aria and Definite Flow), located on top of the fitted regression trendline, exhibited more polymerization forces than expected according to the regression's equation. On the contrary, materials located below the fitted regression's trendline (like PermaFlo, Revolution 2 and Ultraseal XT) exhibited less polymerization forces than calculated by the regression's equation.

Table 4 illustrates the difference in evolution of the two properties over time as measured with Equation 1 and the changes observed in the ranking of materials in the two properties that were studied. As mentioned before, Equation 1 expressed numerically the difference coming from the deduction of the development curves of percent values of the two properties over time (Figures 6 and 7). All values were negative, as the area below the development curve of percent values of polymerization forces is smaller than the area below the development curve of percent values of linear polymerization displacement. This signified the slower kinetic behavior of polymerization forces.

DISCUSSION

Lately, many dental manufacturers have introduced various alternative resin-based restorative materials, such as compomers, flowable composites, condensable or packable composites, and other materials with reportedly anti-cariogenic, remineralizing and superior esthetic properties. As a result, resin composite classification is becoming more complicated, and even researchers face occasional difficulties in explaining results as manufacturers often do not reveal the proprietary formulas of their products. Daily, the dental clinician faces the dilemma of product selection, with very few means in his decision making process, as many dental manufacturers may not present all the properties of their products, but only emphasize the

advantageous ones that help the promotion of their sales. One cannot argue that research is focused on modification of the formulation of resin-based restorative materials in order to reduce polymerization shrinkage, enhance degree of cure and mechanical properties, improve durability as well as wear resistance and make dental composites more esthetic, cariostatic and biologically safe. Nevertheless, polymerization shrinkage is occasionally overlooked when it comes to the production of new dental composites. Therefore, it was the purpose of this study to measure the linear displacement and forces induced by polymerization shrinkage of numerous flowable resin-based composites, as very few data concerning polymerization shrinkage of these materials may be found in the dental literature.

Table 4: *Results of Equation 1, and the Changes Observed in the Ranking of Materials in the Two Properties That Were Studied*

Material	LPD†	PF Material‡	CR¥	DDDC§
PermaFlo	74.19	7.18 Aeliteflow LV	+2	-4.04%
Dyract Flow	66.52	6.60 Dyract Flow	0	-5.12%
Aeliteflow LV	64.71	6.41 Star Flow	+2	-5.03%
Aelite Flow	64.38	6.34 OmegaFlo	+3	-5.50%
Star Flow	63.21	6.00 Filtek Flow	+8	-5.78%
Revolution Z	63.01	5.90 Aria	+10	-4.74%
OmegaFlo	61.64	5.80 Aelite Flow	-3	-5.15%
Flow-Line	61.09	5.67 Flow-Line	0	-6.24%
Admira Flow	60.49	5.60 Admira Flow	0	-6.64%
Flow-it	58.03	5.59 PermaFlo	-9	-6.29%
Ultaseal XT	54.25	5.48 Definite Flow	+8	-2.66%
Synergy Flow	53.43	5.26 Flow-it	-2	-5.46%
Filtek Flow	52.84	5.16 Revolution 2	-7	-7.42%
FloRestore	50.73	4.69 FloRestore	0	-5.07%
Wave	47.31	4.66 Crystal Essence	+2	-8.35%
Aria	46.61	4.57 Synergy Flow	-4	-8.45%
Crystal Essence	46.04	4.49 Ultraseal XT	-6	-12.15%
Tetric Flow	45.53	4.31 Tetric Flow	0	-6.16%
Definite Flow	40.5	4.25 Wave	-4	-8.73
Photo SC	31.69	3.89 Freedom	+2	-6.04
Glacier	29.61	3.44 Glacier	0	-5.91%
Freedom	27.95	3.43 Photo SC	-2	-2.88%

†LPD: Linear Polymerization Displacement (in microns).

‡PF: Polymerization Forces (in kilograms).

¥CR: Change of Ranking of materials between the two properties.

§DDDC: Results of Equation 1, showing the Difference coming from the Deduction of the Development Curves of percentage values of the two properties over time.

Unfortunately, no further investigation of the data presented in relation to the type, amount and size of the different monomers and fillers used in the fabrication of these products was feasible, as some of the manufacturers did not reveal the exact composition of the products tested. Additionally, taking into consideration the variation in composition of products that are sometimes present between different batches, one can easily understand that in order to investigate the aforementioned relations, all these parameters should have been measured for all materials tested. This laborious task was not part of the intent of this research protocol. In addition, the literature is abundant with information explaining the possible interaction of the properties that were studied with different parameters of the composition of resin-based restorative materials. Therefore, taking into consideration previous studies in this field of dental research, the results of this research could be explained.

Another parameter that would have added to the scientific value of this research protocol would have been investigating the possible relation between the two measured properties and various mechanical properties, such as the modulus of elasticity, Vickers hardness, compressive strength, fracture strength, resistance to wear and the degree of conversion. This was not attempted, as one of the main purposes of this research project was to investigate the kinetic behavior of the two properties throughout the observation period. This was accomplished with the help of the recording devices that were able to obtain five measurements every second, even though only one measurement each second was actually recorded. Neither of the aforementioned mechanical properties could be measured with the desired frequency (one measurement each second) with any of the various techniques reported in the literature. Feilzer, de Gee and Davidson (1990) used an experimental setup that allowed them to record the evolution of Young's modulus simultaneously with polymerization shrinkage in chemically cured resin composites. Nevertheless, the time needed in order to obtain each Young's modulus measurement was 2.4 seconds. This may have been suitable for chemically-cured composites, where the velocity of the polymerization process is relatively slow (measurements were made in minute intervals), but not rapid enough for light-cured composites, where the progression of the polymerization process is extremely fast, especially during the first few seconds. The relation between polymerization shrinkage and mechanical properties (Labella & others, 1999; Koran & Kurschner, 1998) or degree of conversion (Braga & Ferracane, 2002; Rueggeberg & Tamareselvy, 1995; Patel, Braden & Davy, 1987) at certain points in time is well documented, and it was not the intention of this research to confirm it.

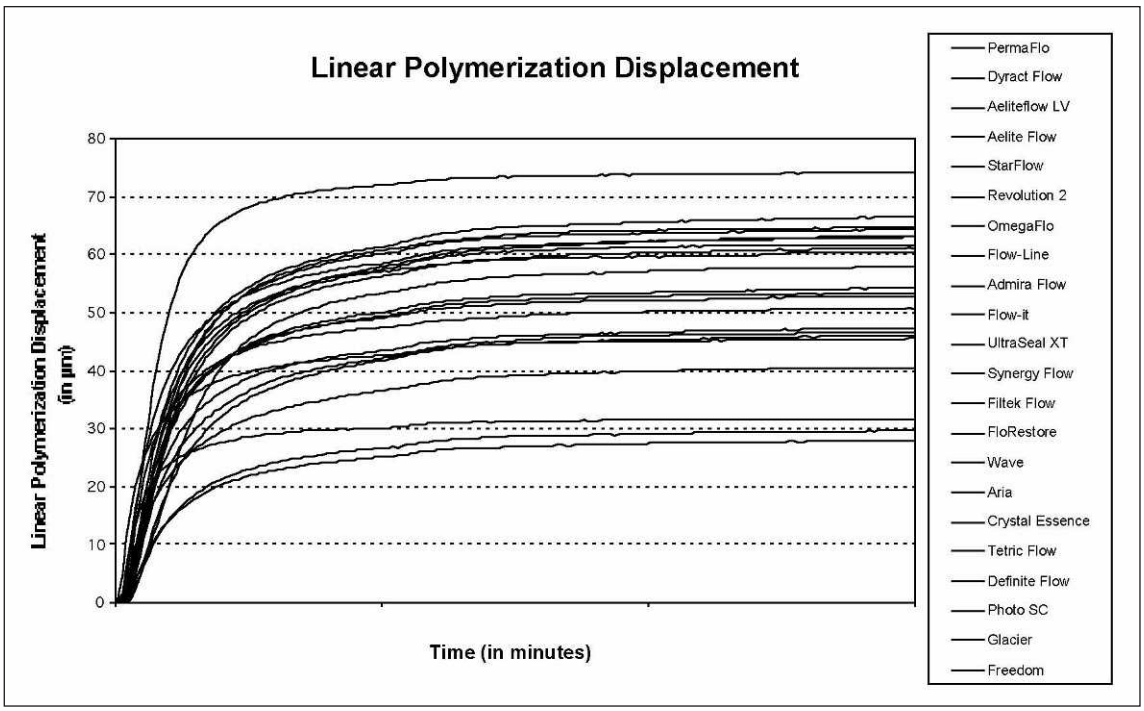

Figure 3. *The progression of the mean values of linear polymerization displacement. Materials in the legend are ordered in descending order of their values at the end of the observation period.*

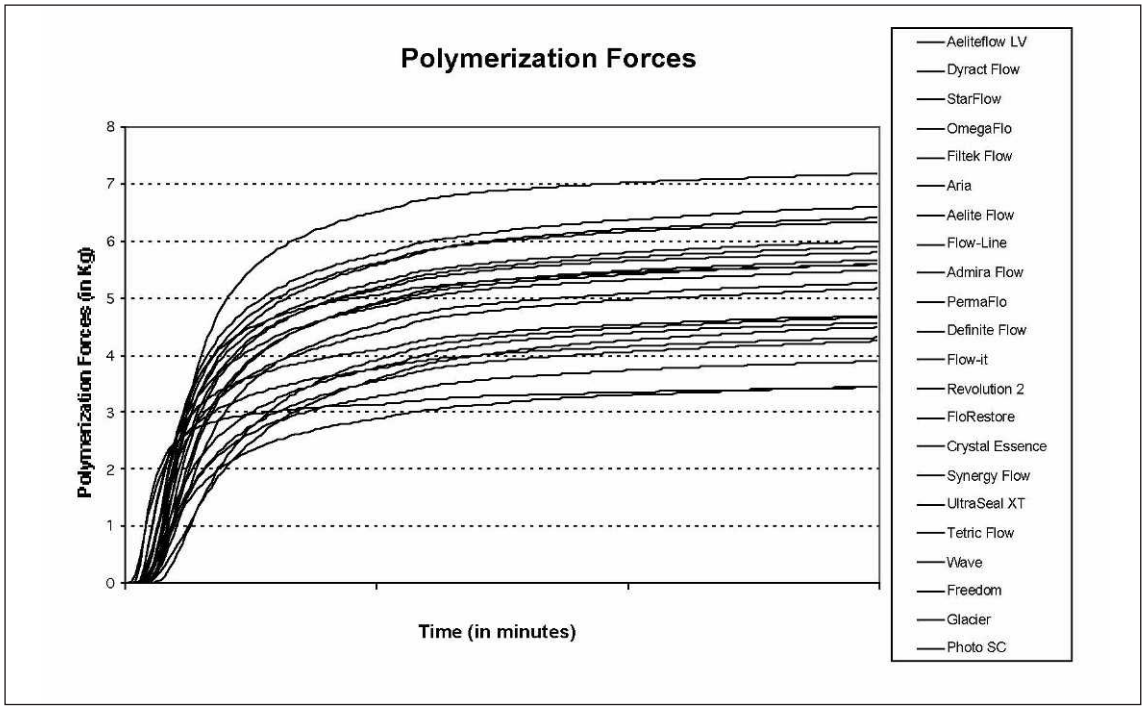

Figure 4. *The progression of the mean values of polymerization forces. Materials in the legend are ordered in descending order of their values at the end of the observation period.*

The linear polymerization displacement measured in this research is reported in microns, as this was the recording unit of the infrared sensor used for measuring the vertical movement of the diaphragm, which was caused by polymerization shrinkage of the test material. Linear polymerization shrinkage may be calculated by:

$$lin\% = \frac{\Delta L}{L + \Delta L} \times 100\%$$

Equation 2

where:

lin% is the linear polymerization shrinkage

ΔL is the recorded displacement and is the thickness of the sample after polymerization.

Using Equation 2 (de Gee & others, 1993), with the modification of using the original height of the specimen (1.5 mm) as the denominator, the data recorded in this study corresponded to a range of values of approximately 1.71% to 5.38% for linear polymerization shrinkage, which is well within the range reported by other researchers. Even though these values are an approximation, as the original height of each specimen was not measured, they do provide an estimation of the linear polymerization shrinkage of the materials tested.

When compared to linear polymerization displacement values of other resin composites, few materials

(Freedom, Glacier and Photo SC) presented low values which were comparable to linear polymerization displacement values of hybrid resin composites, such as Z100 (30.7 µm). On the contrary, the majority of materials presented very large linear polymerization displacement values. This was expected, as flowable composites are not highly filled, and the increased percentage of resin matrix may justify the high linear polymerization displacement values that were measured. Nevertheless, the vast range of values that were recorded (25.61 to 80.74 µm) raises significant questions about consistency in the behavior of materials characterized by the manufacturers as flowable composites.

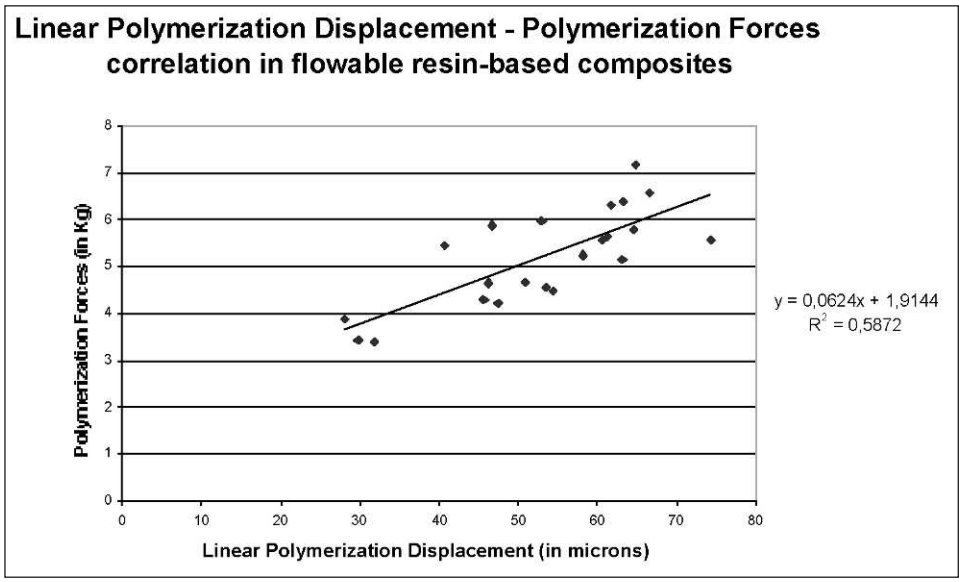

Figure 5. *The correlation of linear polymerization displacement and polymerization forces.*

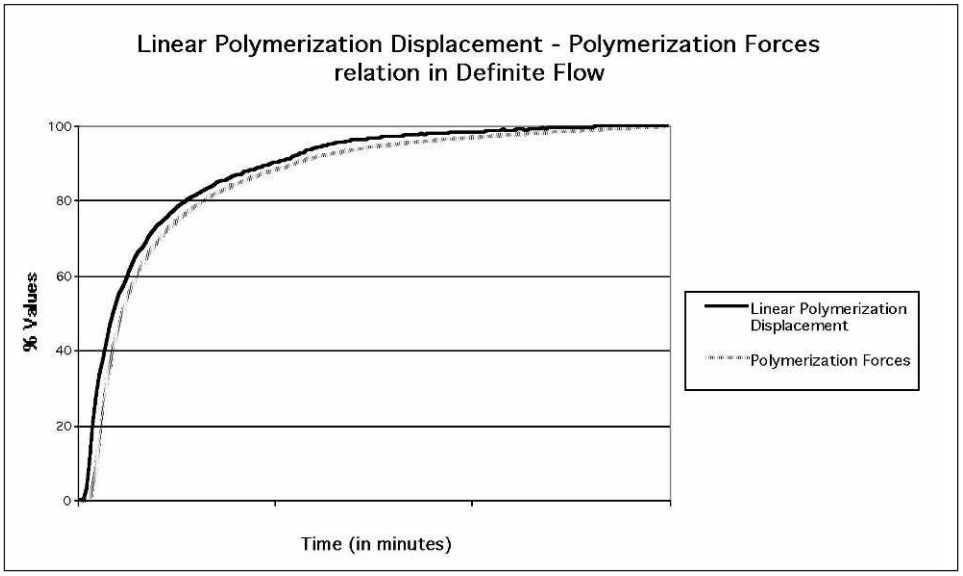

Figure 6. *Development curves of percentage mean values of linear polymerization displacement and polymerization forces in Definite Flow. The result from the deduction of the areas below the curves that was calculated with Equation 1 is -2.66%.*

Taking into consideration a study that divided flowable composites into three categories (low, medium and high) according to their flow characteristics (Moon & others, 2002) and comparing materials that were analyzed in both studies, one may observe some similarities between flow characteristics and linear polymerization displacement. Materials in the "low flow" group (like Tetric Flow) had linear polymerization displacement values below 50 µm; materials in the "medium flow" group (such as Flow-it and FloRestore) had linear polymerization displacement values between 50 and 60 µm and materials in the "high flow" group (such as Revolution 2 and Star Flow) had linear polymerization displacement values above 60 µm. Nevertheless, these similarities were not found to be absolute, as Aelite Flow, which was reported in that study to belong to the "low flow" group, presented linear polymerization displacement of great magnitude (64.38 µm).

The polymerization forces measured in this research are reported in kilograms, not as stress in megapascals (MPa), to avoid confusion with literature data with completely rigid experimental set-ups that reported higher polymerization stresses (Alster & others, 1992; Condon & Ferracane, 1998; Feilzer & others, 1987; de Gee & others, 1993). When transformed to megapascals, the measured stresses ranged from 0.65 to 1.60 MPa; values inferior to the ones reported in the dental literature. The difference is mainly based on the experimental setup used is this research, which permitted a small axial deformation of the specimen, which was only partially restricted, in contrast to the aforementioned study that measured the stresses created by "rigid" contraction, where the original height of specimens was maintained throughout the experiment, thus resulting in greater stresses. As the load cell was axially displaced 4 µm for each kilogram of measured

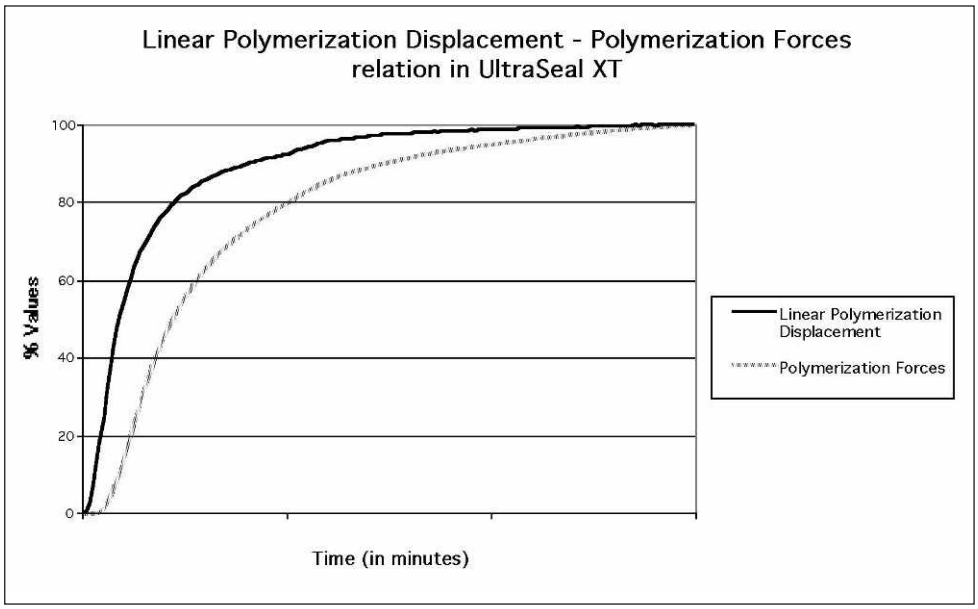

Figure 7. *Development curves of percentage mean values of linear polymerization displacement and polymerization forces in UltraSeal XT. The result from the deduction of the areas below the curves that was calculated with Equation 1 is -12,15%.*

force, a maximum 30 µm deformation of the specimen was feasible. In this way, a semi-rigid configuration of a cavity with a C-factor of 2.67 was simulated. Several studies have demonstrated that cusps of molars and premolars deflect from 18 to 45 µm (Smith & Caughman, 1989; Causton, Miller & Sefton, 1985; Lutz, Krejci & Barbakow, 1991), thus, justifying the experimental set-up used in this research.

When compared to polymerization forces values of other resin composites, few materials (Photo SC, Glacier, and Freedom) presented low values comparable to polymerization force values of hybrid resin composites, such as Z100 (3.8 Kg). On the contrary, the majority of materials presented large polymerization forces values. This may once again be attributed to the low filler loading of flowable composites. A vast range of values was also measured (3.23 to 7.48 Kg), illustrating the high variability in the behavior of flowable composites. Comparison of the polymerization forces recorded with the flow characteristics of flowable composites, according to the aforementioned study (Moon & others, 2002), was not evident. This was due to the fact that there were vast changes in the ranking of materials between the two polymerization properties studied (Table 4). These changes may also explain the fact that linear polymerization displacement and polymerization forces were not found to be highly correlated (r^2=0.59) as in previous studies (Stavridakis & others, 2000, 2004), a point which once again illustrates the non-homogenous behavior of flowable composites.

Polymerization stresses are well documented as being influenced by the degree of polymerization shrinkage (Hegdahl & Gjerdet, 1977; Davidson & Feilzer, 1997). Nevertheless, there are also other parameters that influence the development of polymerization stresses, mainly the velocity of polymerization shrinkage (Braga & Ferracane, 2002) and the elastic modulus of the material (Labella & others, 1999). Slow progression of the polymerization process is claimed to allow for the molecules to slip into new positions and orientations in the earliest stage of setting. Davidson, de Gee and Feilzer (1984) hypothesized that, at the early stages of polymerization, only chain formation occurs, and cross-linking is not at full reaction, enabling the material to flow and compensate for polymerization shrinkage. Additionally, during the early stage of curing, the resin network is still weak and, therefore, the elastic modulus is also low. Plastic yielding to the stress at this stage of setting can be achieved without damage to the internal structure of the resin composite, since the molecules can still slip into new positions and orientations. This kind of deformation is characterized as flow, which may be responsible for the discrepancy observed in the kinetic behavior of linear plastic displacement and polymerization force. Flow is influenced by the velocity of the polymerization process, since the slower the progression of the curing procedure, the more time is given for stresses to be relieved before the resin-based restorative material acquires an increased stiffness and stresses start to accumulate. Feilzer and others (1990) demonstrated that quicker polymerization rate of resin composite material inhibits material flow.

The results of Equation 1 tried to express the differences in kinetic development of linear polymerization displacement and polymerization forces. In materials such as Filtek Flow, Aria and Definite Flow, which gained numerous places (exhibited more polymerization shrinkage than expected), Equation 1 resulted in small negative values (Table 4). In those materials, the kinetic behavior of polymerization forces presented with a relatively small time delay when compared to linear polymerization displacement (Figure 6). On the contrary, in materials such as PermaFlo, Revolution 2 and Ultraseal XT, which lost many places (exhibited less polymerization shrinkage than expected), Equation 1 resulted in greater negative values (Table 4). Observation of the evolution of the two polymeriza-

tion properties in those cases revealed greater time delays in the development of polymerization forces (Figure 7). The delay in development of polymerization forces in these materials may be attributed to their low modulus of elasticity (less filled flowable composites), as linear polymerization displacement evolved with the expected exponential growth pattern.

These calculations confirm the general opinion of stress reduction by flow of low-elastic, flowable composites. However, this reduction is not necessarily beneficial for the clinician. Even if the relative stress development in flowable composites is smaller than in highly filled composites, because of the higher linear shrinkage associated with flowable composites, the absolute stress values most often exceed that of highly filled composites.

CONCLUSIONS

Flowable composites were found to have a greater range of linear polymerization displacement and polymerization forces. From a clinical standpoint, the polymerization shrinkage behavior of these materials should be taken into consideration, in combination with the volume and C-factor (bonded/unbonded surface ratio) of the cavity where they are placed (Feilzer & others, 1987; Davidson & others, 1984). Highly fluid flowable composites which exhibit substantial polymerization shrinkage might be successfully used in micro-conservative occlusal cavities, as the limited volume of the material used, in combination with the favorable C-factor of the cavity, minimizes the negative consequences of polymerization shrinkage. On the other hand, as the volume of restorative material increases, especially in cavities with an unfavorable C-factor, the clinician should reconsider the use of flowable composites and, if ever, use products with the least amount of polymerization shrinkage.

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Influence of Dietary Solvents on Strength of Nanofill and Ormocer Composites

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Clinical Relevance

The strength of nanofill and ormocer composites is generally not affected by dietary solvents. These materials are weaker than minifill composites but stronger than compomers and highly viscous glass ionomer cements.

SUMMARY

The objective of this study was to determine the influence of dietary solvents on the shear punch strength of nanofill (Filtek Supreme [FS], 3M-ESPE) and ormocer (Admira [AM], Voco) composites. The strength of these materials was also compared to a minifill composite (Z250 [ZT], 3M-ESPE), a compomer (F2000 [FT], 3M-ESPE) and a highly viscous glass ionomer cement (Ketac Molar Quick [KM], 3M-ESPE). Thirty-two specimens (8.7 mm diameter and 1-mm thick) of each material were made, randomly divided into four groups of eight and conditioned for one week as follows–Group 1 (control): distilled water at 37°C; Group 2: 0.02M citric acid at 37°C; Group 3: 50% ethanol-water solution at 37°C and Group 4: heptane at 37°C. After conditioning, the specimens were restrained with a torque of 2.5 Nm and subjected to shear punch strength testing using a 2-mm diameter punch at a crosshead speed of 0.5 mm/minute. The shear punch strength of the specimens was computed and data subjected to ANOVA/Scheffe's tests at significance level 0.05. With the exception of AM, the strength of all materials was not significantly influenced by dietary solvents. For AM, conditioning in heptane resulted in significantly higher shear strength values. The strength of the nanofill and ormocer composites was lower than the minifill composite but higher than the compomer and highly viscous glass ionomer cement investigated.

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INTRODUCTION

Despite improvements in mechanical properties and wear resistance, the use of composites for direct posterior restorations in still limited. Clinical data on amalgam and composite restorations generally indicate that amalgam restorations have longer survival times (Smales, Webster & Leppard, 1991; Mjör & Jokstad, 1993; Mjör, Moorhead & Dahl, 2000). The most common reasons for failure of composite restorations are secondary caries and bulk fracture (Mjör &

Jokstad, 1993; Mjör & others, 2000). Sarrett, Coletti and Peluso (2000) highlighted the fact that a composite material can perform well in one patient but may degrade, wear and fracture prematurely in another. They attributed the differences among patients to a variety of factors including occlusal bite forces, parafunctional habits such as clenching/bruxism, diet, salivary and plaque compositions (Sarrett & others, 2000). Microdefect analysis of clinically worn composites revealed extensively damaged layers on both occlusal contact and contact-free areas (Wu & others, 1984). The result stipulates that the intra-oral degradation of composites cannot be attributed to mechanical factors alone, but involves chemical degradation as well. Subsurface material damage was attributed to the softening and possible removal of portions of the polymer matrix by certain chemicals present in the oral environment (Wu & others, 1984; Kao, 1989; Yap, Low & Ong, 2000a). Although the softening will, by itself, degrade the restoratives, material strength may be considerably decreased by the softening process, leading to early restoration fracture.

Dental composites based on nanofill and ormocer (organically modified ceramics) technologies were recently introduced. Filtek Supreme (3M-ESPE), the first nanofill commercial product, contains a unique combination of nanofillers (5-75 nm) and nanoclusters embedded in an organic polymer matrix. The nanofillers (nanomers) are discrete non-agglomerated and non-aggregated particles of 20-75 nm in size. Nanocluster fillers are loosely bound agglomerates of nano-sized particles. These nano-sized filler particles allow the polish and polish retention typical of a microfill in addition to good handling, strength and wear properties. The technology used in the ormocer materials is different from that of conventional composites. While the latter are based on a purely organic polymer matrix, the ormocer consists of an inorganic-organic (inorganic backbone based on SiO_2 functionalized with polymerizable organic units) network matrix formed through polycondensation. The filler particles are imbedded in this cross-linked inorganic and organic network matrix. The average particle size is 0.7 μm, which is comparable to most minifill composites.

The effect of dietary solvents on the mechanical properties of nanofill and ormocer composites has not been reported. The objective of this study was to determine the influence of dietary solvents on the strength of nanofill and ormocer composites. The strength of these new materials was also compared to other direct aesthetic restorative materials.

METHODS AND MATERIALS

In addition to nanofill (Filtek Supreme [FS]) and ormocer (Admira [AM]) composites, three other kinds of direct aesthetic restorative material were selected for the study. They included a minifill composite (Z250 [ZT]), a compomer (F2000 [FT]) and a highly viscous glass ionomer cement (Ketac Molar [KM]). The materials evaluated, their manufacturers, lot numbers and cure times are shown in Table 1. All materials were of the A2 shade. Shear punch specimens were made by placing the restorative materials into stainless steel washers (17.7-mm outer diameter, 8.7-mm inner diameter and 1-mm thick) which were supported by glass slides. A second glass slide was placed on top of the washers and gentle pressure was applied to extrude excess material. The top surface of composite and compomer specimens was cured using a Max polymerization unit (Dentsply/Caulk, Milford DE, USA) with a light exit window of 13 mm and mean intensity greater than 400 mW/cm^2 according to manufacturers' cure times. The glass ionomer cement was allowed to set for five minutes with the glass slides in place.

Thirty-two specimens of each material were made and stored in distilled water at 37°C for one week. The specimens, together with their washers, were then randomly divided into four groups of eight and conditioned for one week as follows–Group 1 (control): distilled water at 37°C; Group 2: 0.02M citric acid (pH 2.4) at 37°C; Group 3: 50% ethanol-water solution at 37°C and Group 4: heptane at 37°C. At the end of the conditioning period, the specimens were washed and blotted dry with filter paper. Shear strength testing was then conducted using custom designed shear punch apparatus (Figure 1) mounted on an Instron Micro-tester (Model 5848, Instron Corp, Canton, MA, USA). The thickness of each specimen was measured with a digital vernier calliper prior to placement in the shear punch apparatus. Specimens were positioned in the apparatus by means

Table 1: *The Direct Aesthetic Restorative Materials Evaluated*

Material	Category	Manufacturer	Batch #	Shade Cure Time
Filtek Supreme [FS]	Nanofill composite	3M-ESPE St Paul, MN, USA	EXM#612	A2 20 seconds
Admira [AM]	Ormocer composite	Voco, Cuxhaven, Germany	300500	A2 40 seconds
Filtek Z250 [ZT]	Minifill composite	3M-ESPE St Paul, MN, USA	20010402	A2 20 seconds
F2000 [FT]	Compomer	3M-ESPE St Paul, MN, USA	02070062	A2 40 seconds
Ketac Molar Quick Aplicap [KM]	Highly viscous glass ionomer cement	3M-ESPE St Paul, MN, USA	139202	A2 2.5 minutes

of a self-locating recess that provided a snug-fit, with the washers holding the specimens. The specimens were restrained by tightening a screw clamp to a torque of 2.5 Nm using a torque wrench. A tool steel punch with a flat end 2-mm in diameter was used to create shear force by sliding through a punch hole with a radial clearance of 0.01 mm. Testing was done at a crosshead speed of 0.5 mm/minute and the maximum load was recorded. Shear strength was subsequently computed using the following formula:

$$\text{Shear strength (MPa)} = \frac{\text{Force (N)}}{\pi \times \text{Punch diameter (mm)} \times \text{Thickness of specimen (mm)}}$$

All statistical analysis was carried out at significance level 0.05. The interaction between materials and conditioning mediums was evaluated using two-way ANOVA. One-way ANOVA and Scheffe's post-hoc tests were used to determine inter-medium and inter-material differences.

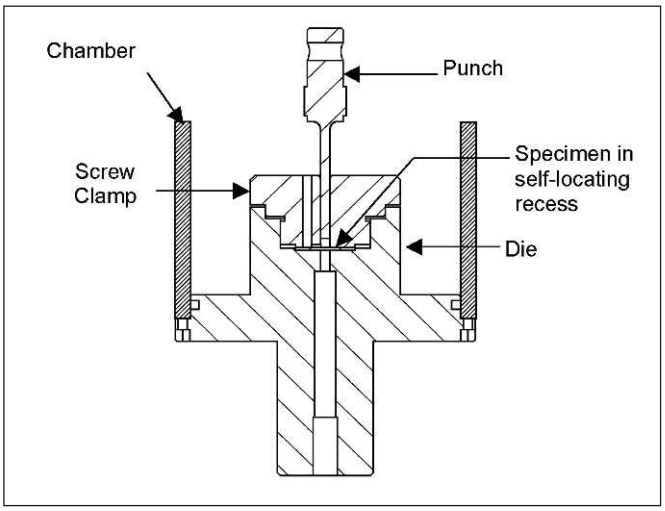

Figure 1. *Diagrammatic representation of the shear punch apparatus.*

RESULTS

The mean shear strength of the materials after conditioning in the various dietary solvents is shown in Table 2 and Figure 2. Results of statistical analyses are shown in Tables 3 and 4.

Two-way ANOVA revealed significant interaction between materials and conditioning mediums. The strength of FS, ZT, FT and KM was not significantly affected by conditioning in the various dietary solvents. For AM, exposure to heptane resulted in significantly higher shear strength values. No significant difference in strength was observed between AM specimens in the control group and those conditioned in citric acid and 50% ethanol-water solution. Regardless of conditioning medium, the strength of ZT was the highest and KM the lowest. Shear strength values ranged from 155.95 to 166.81 MPa and 45.94 to 56.62 MPa for ZT and KM, respectively. For all conditioning mediums, the strength of ZT, AM and FS

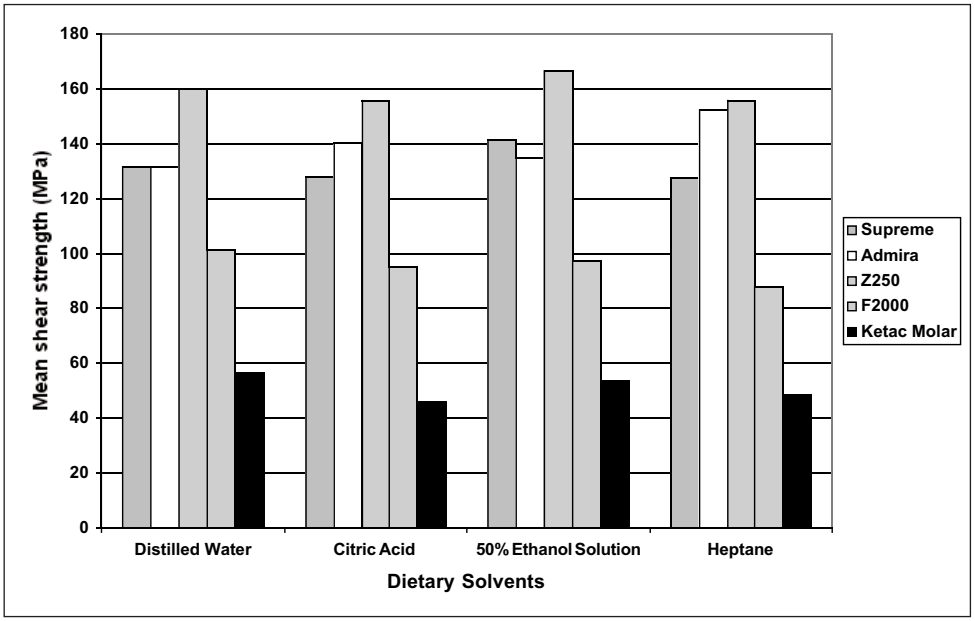

Figure 2. *Comparison of mean shear strength between materials.*

Table 2: *Mean Shear Strength (MPa) of the Materials After Conditioning in the Various Dietary Solvents*

Materials	Distilled Water	Citric Acid	50% Ethanol Solution	Heptane
Filtek Supreme [FS]	131.52 (11.68)	127.87 (8.77)	141.55 (16.14)	127.48 (16.34)
Admira [AM]	131.65 (7.19)	140.30 (17.16)	134.96 (9.05)	152.00 (11.65)
Filtek Z250 [ZT]	159.69 (7.30)	155.95 (20.80)	166.81 (6.70)	156.02 (6.67)
F2000 [FT]	101.21 (13.09)	95.12 (10.76)	97.14 (6.60)	88.01 (10.59)
Ketac Molar [KM]	56.62 (10.27)	45.94 (5.52)	53.54 (4.81)	48.25 (7.47)

Standard deviations in parentheses.

Table 3: *Results of Statistical Analysis Based on Materials*

Material	Differences
Filtek Supreme [FS]	NS
Admira [AM]	Heptane > Distilled Water
Filtek Z250 [ZT]	NS
F2000 [FT]	NS
Ketac Molar [KM]	NS

Results of one-way ANOVA and Scheffe's test (p<0.05); > indicates statistical significance while NS indicated no statistical significance.

Table 4: *Results of Statistical Analysis Based on Dietary Solvents*

Conditioning Medium	Differences
Distilled Water	Z250 > Admira, Supreme > F2000 > Ketac Molar
Citric Acid	Z250, Admira, Supreme > F2000 > Ketac Molar Z250 > Supreme
50% Ethanol-water Solution	Z250 > Supreme, Admira > F2000 > Ketac Molar
Heptane	Z250, Admira > Supreme > F2000 > Ketac Molar

Results of one-way ANOVA and Scheffe's test (p<0.05); > indicates statistical significance while NS indicated no statistical significance.

was significantly higher than FT and KM. In addition, ZT was significantly stronger than FS. AM was significantly weaker than ZT when the materials were conditioned in water and 50% ethanol solution. The strength of these two materials was, however, comparable when conditioned in citric acid and heptane.

DISCUSSION

As composites/compomers are evaluated in flexural (International Organization for Standardization [ISO] 2000) and highly viscous glass ionomer in compression (ISO 2000), the strength of these materials cannot be directly compared despite some similarities in clinical applications. Nomoto, Carrick and McCabe (2001) proposed the use of the shear punch test for standardized strength testing of all direct tooth-colored restoratives. Their approach, which was supported by a recent study by Yap and others (2003a), was selected for the current experiment. Specimens were constrained with a 2.5 Nm torque during shear punch testing, as Nomoto and others (2001) reported significantly lower strength values for specimens not restrained with a screw clamp. They hypothesized that unrestrained specimens are able to bend on application of the punch, creating localized stress concentration leading to premature failure. As shear stresses are induced in teeth and restorations during mastication and parafunction, the shear punch test reflects qualities of clinical significance (Roydhouse, 1970).

The dietary solvents used for conditioning the materials were among those recommended in the Food and Drug Administration (USA) Guidelines (FDA, 1976). Heptane simulates butter, fatty meats and vegetable oils, while ethanol solution simulates certain beverages, including alcohol, vegetables, fruits, candy and syrup. Fifty percent ethanol solution was used to simulate accelerated aging, as its solubility parameter matches that of the dimethacrylate resins used in composites (Kao, 1989). Distilled water was included to simulate the wet oral environment provided by saliva and water. A one-week hiatus prior to conditioning was incorporated to allow for composite post-cure and establishment of the acid-base reaction in the compomer and glass ionomer (Watts, Amer & Combe, 1987; Eliades, Kakaboura & Palaghias, 1998; Yap, Pek & Cheang, 2003b).

With the exception of the ormocer, the strength of all the materials was not significantly influenced by conditioning in the various dietary solvents. The results appear to contradict previous work that reported chemical degradation of direct aesthetic restoratives by food simulating solvents, especially ethanol solutions (McKinney & Wu, 1985; Kao, 1989; Ferracane & Marker, 1992; Lee & others, 1998; Yap & others, 2003a). This disparity may be explained by differences in materials, the mechanical properties evaluated, testing methods, conditioning mediums and time. The findings of this study were consistent with those of Yap and others (2000b), which used the ISO flexural test (ISO 2000), the same conditioning mediums and time. The strength of the ormocer material Admira was significantly increased by conditioning in heptane. This phenomenon was also observed for several other commercial composite and compomer materials in an earlier study (Yap & others, 2000b). The higher strength values may be attributed to the fact that heptane eliminates the leaching out of silica and combined metals in fillers, which occurs from conditioning in aqueous solutions, including dietary solvents (Söderholm, 1983).

Regardless of conditioning medium, strength ranking was consistent with the clinical performance of the different material types. The composites had significantly higher strength than the compomer, which, in turn, was significantly stronger than the highly viscous glass ionomer cement. For this reason, highly viscous glass ionomers should never be used in stress-bearing situations. However, significant differences in strength between the composite materials were conditioning medium dependent. The strength of the minifill composite Z250 was significantly greater than the nanofill (Supreme) and ormocer (Admira) composites when conditioned in distilled water and 50% ethanol solution. In addition, the minifill composite was also stronger than the nanofill composite after conditioning in citric acid and heptane. As the polymer and filler content (approximately 60% volume) between the minifill and nanofill composites were similar, the significant difference in

strength may be attributed to the differentiation in filler size. The interface between the loosely bound nanocluster fillers in the nanofill composite may also serve as possible pathways for crack propagation during shear strength testing. Differences in strength between the minifill and ormocer composites could be attributed to the lower filler content (56% volume) of Admira and the possible hydrolytic effect of water on the SiO_2 inorganic backbone of the inorganic-organic network matrix (Söderholm & Roberts, 1990).

CONCLUSIONS

Under the conditions of this *in vitro* study, the following conclusions can be made:

1. With the exception of the ormocer composite, the strength of the materials evaluated was not significantly influenced by dietary solvents.
2. For the ormocer composite, conditioning in heptane significantly increased shear strength.
3. Regardless of conditioning medium, the composite materials were significantly stronger than the compomer, which, in turn, was significantly stronger than the highly viscous glass ionomer cement.

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Awards

American Academy of Gold Foil Operators
Distinguished Member Award

Dr Warren Johnson

Warren Johnson

It is my distinct pleasure and singular honor to introduce the honoree of the highest award of the American Academy of Gold Foil Operators, the Distinguished Member Award. This year's award goes to Dr Warren Johnson from Seattle, Washington. When Dr Johnson asked me to give his introduction, I was deeply touched and thought a lot about what I was going to say. After all, we all know Warren and I did not know whether to make this into a roast or keep it as a serious introduction. For those of you who know Warren and me, I do not think I can keep it all serious but will do my best.

Warren is a retired Army Colonel and served his country in Germany and in Seattle for years in the Reserves. He worked as a dentist in the service and gained much clinical experience. He also met his lovely wife Margot while in Germany and she stole his heart—more about the family later.

After finishing active duty, Warren bought a dental practice in Seattle and began his private practice career. One day, he went to a lecture given by Dr Richard V Tucker and saw the most beautiful cast gold restorations he had ever seen. He was hooked. Since he was not able to join the study club across the lake, he decided to start his own. The rest is history. Warren has become one of the finest gold operators in the world—both cast and direct. He operates at meetings often and we have all seen his clinical skills. He is now a member of two study clubs, mentors three clubs in California, three more in New Jersey (one of those is a gold foil club) and, in addition, together with other members of his cast club, travels to Germany to mentor. Including travel time, that adds up to more than two months of time spreading the knowledge about gold—that is called commitment to teaching.

He is a member of the American Academy of Restorative Dentistry, has gone through the chairs of the Academy of Operative Dentistry and is a Past President, Past President of The RV Tucker Academy of Study Clubs and the immediate Past President of the American Academy of Gold Foil Operators. Rather than fade away after serving his time as an officer in an organization, he continues to actively contribute to both the Tucker and Gold Foil academies. That is called commitment to organized dentistry.

His family has grown recently. He and Margot now have two grandchildren from his son Torston and daughter-in-law Carla. He and Margot regularly visit Casey and Calen in Germany and just spent a month with them all here in Seattle. They will all be together in South Africa for a month next year, and he is considering helping his son buy a home in Germany large enough for them to stay in when visiting. That is called commitment to family.

But how can you talk about Warren and not mention any of his Warrenisms—some repeatable in mixed company and some not. We have all heard them—many times—Warrren sometimes forgets that he already told us that one but we laugh again anyway—especially when he is your mentor. Speaking of mentors, Warren has the utmost respect for his gold foil mentors, Dr Gerry Stibbs and Dr Bruce Smith, as well, of course, his cast mentor Dr Dick Tucker.

Warren is my mentor for both cast and foil; and he teaches respect for the classic techniques we use, he teaches respect for the great clinicians who have gone before us and he teaches respect for a high level of professionalism and code of conduct within our profession.

During study club, he is all business and becomes the excellent teacher that he has been taught to be by his mentors. His teaching goal is to help the members of his study clubs become better operators. It is my great honor to introduce the 2004 Distinguished Member of the American Academy of Gold Foil Operators, Dr Warren K Johnson.

Bruce W Small, DMD

American Academy of Gold Foil Operators
Outstanding Clinician Award
Dr David Bridgeman

David Bridgeman

This year's recipient of the American Academy of Gold Foil Operators' Outstanding Clinician Award is my good friend and colleague, Dr David Bridgeman. David is a third-generation dentist serving the community of New Martinsville, West Virginia. He is fortunate enough to be carrying on a practice started by his grandfather, continuing to treat many of his grandfather's patients and their families. David comes from a true dental family, having a grandfather, father, brother and sister all serving the profession as dentists. One brother decided to break away and become a mechanical engineer, but he is an engineer with very good teeth.

Those of us who know David closely were taken completely by surprise a few years ago when he showed up at a study club meeting with a new bride, Cathy. The fact that he did so without much fanfare was no surprise to most of us, as we have come to know that Dave is a pretty laid back sort of guy. What we did not realize was that this marriage was all part of a much grander scheme. Immediately after getting married, Cathy insisted that they look for a new home. Trust me, I had visited Dave's apartment on many occasions and I could feel her pain. New Martinsville had plenty of available homes, but Dave chose to move directly across the Ohio River to Hannibal, Ohio, just so he could contribute to the 136,000 vote Republican margin in the 2004 presidential election.

David graduated from West Liberty State College in 1977 and West Virginia University School of Dentistry in 1980. He then began his dental carrier by joining his father in the family dental practice in New Martinsville. It would be easy to say Dave followed in his grandfather and father's footsteps, but I think it would be more appropriate to say his footsteps followed the same path. He tried to maintain a low profile, but that was a short-lived attempt, as he was soon called upon to serve the profession through leadership roles in organized dentistry. David held leadership roles in the Marshall-Wetzel-Tyler Dental Society and the West Virginia Dental Association, eventually serving as president of the state association in 1993. As fortune or fate would have it, the state legislature enacted a very unpopular dental provider tax during in 1993 that West Virginia dentists still fondly refer to as the "Bridgeman Tax," so his legacy lives on. David then went on to serve as an alternate delegate to the ADA House of Delegates in 1993 and as delegate in 1994. He has also followed the family tradition of being very active in study clubs and academies. Dave became a member of the George M Hollenback Seminar in 1983, the AAGFO and Operative Academy in 1986 and the International College of Dentists in 1994. He has served as the Secretary-Treasurer of the Hollenback Seminar for many years and is currently a member of the Executive Council of the AAGFO.

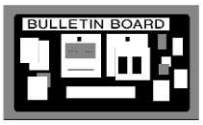
Besides service, this Award recognizes David's outstanding clinical abilities, which most of us have been fortunate to observe the many times he as operated before this Academy. I have watched David operate for the past 18 years in his office, at study club meetings and in this Academy, and there are several things that continue to amaze me. The first is that he seems to be infinitely adaptable to the situation at-hand. This was evident last year at the AAGFO clinical session in Nebraska, when what was scheduled to be a moderately large occlusal gold foil rapidly turned into a casting. Faced with what he determined would be a restoration too large for direct gold, David proceeded to prepare, wax, invest, cast and seat a beautiful inlay before most of the foil restorations had been completed. Another thing that always amazed me about Dave is that he never seems to get rattled. Even when things are not going perfectly, I have never seen him sweat. When faced with a difficult clinical task or decision, Dave just shrugs his shoulders, mumbles a bit and gets on with what needs to be done. But probably my most sincere endorsement of his clinical skills occurred several years ago when I personally broke a cusp on an olive pit at a small café overlooking the beach in Nice, France. At the time I was totally immersed in my studies of comparative anatomy and had not paid enough attention to what I was eating. I could have had anyone in this Academy restore that tooth, but there was never a question in my mind who would do it, and I could not afford his brother's fees.

Lastly, I want to speak to David as a friend. When I moved 18 years ago from a small town in Minnesota to the Washington, DC area, the culture shock I experienced could be described much like the proverbial "deer in the headlights." David and his father quickly made me feel at home in their offices, homes and community. Our friendship has led to many adventures; the time we tried to drown Dr Richard Hoard in the swamps of North Dakota, pheasant hunting on the prairies of South Dakota and turkey and deer hunting in the mountains of West Virginia. I cannot think of any individual more deserving of this Award. I am honored to call David my peer and colleague, but more so, I am honored to call him my friend. On behalf of the American Academy of Gold Foil Operators, I am pleased to present this Award for Outstanding Clinician for 2004 to Dr David Bridgeman.

Dr Fred Eichmiller

Departments

Announcements



34th Annual Meeting of the Academy of Operative Dentistry

23-25 February 2005
Fairmont Hotel, Chicago, IL

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

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

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

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

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

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

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

Classifieds: Faculty Positions

NYU College of Dentistry seeks candidates for the full-time, tenure-track position of Chair, Department of Cariology & Operative Dentistry. The Chair will lead a diverse group of faculty, students and staff in their mission to achieve programs of excellence in the areas of teaching, scholarly activity and service. The Department has major initiatives in the integration of the medical and surgical management of caries into the curriculum and in translating research from "laboratory to the operatory." The strong research atmosphere at the College supports these initiatives.

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

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

NYU is an Equal Opportunity/Affirmative Action Employer.

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