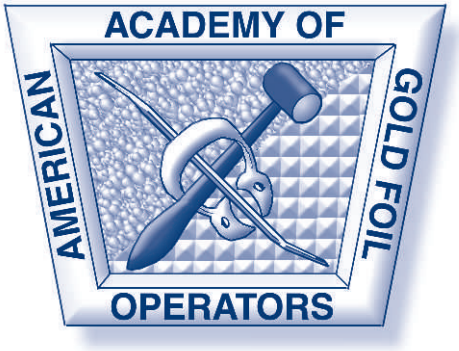
OPERATIVE DENTISTRY



september/october 2004 • volume 29 • number 5 • 481-600

(ISSN 0361-7734)

OPERATIVE DENTISTRY

SEPTEMBER/OCTOBER 2004 • VOLUME 29 • NUMBER 5 • 481-600

Aim and Scope

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.

Subscriptions: Fax (317) 852-3162

Operative Dentistry (ISSN 0361-7734) is published bimonthly by *Operative Dentistry*, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186. Periodicals postage paid at Indianapolis, IN, and additional mailing offices. **Postmaster:** Send address changes to: *Operative Dentistry*, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186.

Current pricing for individual, institutional and dental student subscriptions (both USA and all other countries) can be found at our website: www.jopdent.org, or by contacting our subscription manager:

Fax 317/852-3162
E-mail: jmatis@indy.rr.com

Information on single copies, back issues and reprints is also available. Make remittances payable (in US dollars only) to *Operative Dentistry* and send to the above address. Credit card payment (Visa, MasterCard) is also accepted by providing card type, card number, expiration date, and name as it appears on the card.

Contributions

Contributors should study the instructions for their guidance printed in this journal and should follow them carefully.

Permission

For permission to reproduce material from *Operative Dentistry* please apply to *Operative Dentistry* at the above address.

The views expressed in *Operative Dentistry* do not necessarily represent those of the Academies or of the Editors.

Editorial Office

Operative Dentistry
Indiana University School of Dentistry, Room S411
1121 West Michigan Street, Indianapolis, IN 46202-5186
Telephone: (317) 278-4800, Fax: (317) 278-4900
URL: http://www.jopdent.org/

Editorial Staff

Editorial Board

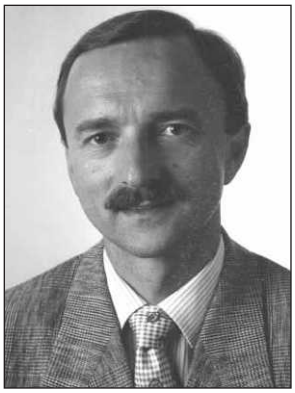
©Operative Dentistry, 2004, **29-5**, 481-508

Michael Buonocore

Buonocore Memorial Lecture

Review of the Clinical Survival of Direct and Indirect Restorations in Posterior Teeth of the Permanent Dentition

J Manhart • HY Chen
G Hamm • R Hickel



Reinhard Hickel

SUMMARY

This review provides a survey on the longevity of restorations in stress-bearing posterior cavities and assesses possible reasons for clinical failure. The dental literature, predominantly since 1990, was reviewed for longitudinal, controlled clinical studies and retrospective cross-sectional studies of posterior restorations. Only studies investigating the clinical performance of restorations in permanent teeth were included. Longevity and annual failure rates of amalgam, direct composite restorations, compomers, glass ionomers and derivative products, composite and ceramic inlays and cast gold restorations were determined for Class I and II cavities. Mean (SD) annual failure rates in posterior stress-bearing cavities are: 3.0% (1.9) for amalgam restorations, 2.2% (2.0) for direct composites, 3.6% (4.2) for direct composites with inserts, 1.1% (1.2) for compomer restorations, 7.2% (5.6) for regular glass ionomer restorations, 7.1% (2.8) for tunnel glass ionomers, 6.0% (4.6) for ART glass ionomers, 2.9% (2.6) for composite inlays, 1.9% (1.8) for ceramic restorations, 1.7% (1.6) for CAD/CAM ceramic restorations and 1.4% (1.4) for cast gold inlays and onlays. Publications from 1990 forward showed better results. Indirect restorations exhibited a significantly lower mean annual failure rate than direct techniques (p=0.0031). Longevity of dental restorations is dependent upon many different factors, including material, patient- and dentist-related. Principal reasons for failure were secondary caries, fracture, marginal deficiencies, wear and postoperative sensitivity. We need to learn to distinguish between reasons that cause early failures and those that are responsible for restoration loss after several years of service.

Juergen Manhart, DDS, MS, PhD, associate professor, Department of Restorative Dentistry and Periodontology, Ludwig-Maximilians-University, Munich, Germany

Hongyan Chen, DDS, PhD, assistant professor, Department of Restorative Dentistry and Periodontology, Ludwig-Maximilians-University, Munich, Germany

Gerald Hamm, PhD, physicist and statistician, Department of Restorative Dentistry and Periodontology, Ludwig-Maximilians-University, Munich, Germany

*Reinhard Hickel, DDS, MS, PhD, professor and chairman, Department of Restorative Dentistry and Periodontology, Ludwig-Maximilians-University, Munich, Germany

*Reprint request: Goethe Street 70, 80336 Munich, Germany; e-mail: hickel@dent.med.uni-muenchen.de

INTRODUCTION

Changes in restorative treatment patterns, the introduction of new and improved restorative materials and techniques, effective preventive programs, enhanced dental care and growing interest in caries-free teeth have greatly influenced the longevity of dental restorations (Krejci & Lutz, 1991; Mjör, 1997b). Marked changes in the use of restorative materials have occurred during the past 10 to 20 years (Hickel, 1997; Hickel & others, 1998; Mjör, 1997a) and esthetic considerations are growing in importance for the restoration of posterior teeth (Burke & Qualtrough, 1994; Scheibenbogen-Fuchsbrunner & others, 1999). Alleged adverse health effects and environmental concerns in respect to the release of mercury gave rise to controversial discussions about the use of amalgam in several countries (Hickel & others, 1998; Peters, Roeters & Frankenmolen, 1996; Roulet, 1997a). There is a growing concern about the use of metallic alloys, in general. Besides cast gold inlays, esthetic alternatives to amalgam restorations include glass ionomers, resin-modified glass-ionomers, compomers, direct composite restorations, composite inlays and ceramic inlays. The latter can be produced by a dental technician in the laboratory or by means of CAD/CAM-machining (Cerec, Sirona, Bensheim, Germany) (Hickel & Kunzelmann, 1997; Manhart & others, 1999c; Mörmann & Brandestini, 1989; Mörmann & Krejci, 1992; Sjögren & others, 1992).

Failure of dental restorations is a major problem in dental practice, especially in the treatment of permanent teeth. Placement and replacement of restorations still constitutes the major workload in general dental practice, although preventive programs and an increased awareness of oral health have had positive effects on the DMFT-index in many countries (Mjör, Jokstad & Qvist, 1990; Rykke, 1992). About 60% of all operative work done is attributed to the replacement of restorations (Mjör, 1989). The clinical assessment of restoration failure differs according to the diagnostic criteria applied and will reflect the interpretative variability of different operators. The examination of patients for treatment needs frequently reveals restorations that do not meet precise criteria for excellent or good restorations but are capable of further clinical service and do not necessarily require replacement. In view of the high cost of delivering restorative dentistry to the community, it is of particular interest to know how long dental restorations may be expected to survive (Crabb, 1981; Paterson, 1984).

A direct comparison of the longevity of different types of restorations among different studies as reported by different authors is problematic for various reasons (el-Mowafy & others, 1994). The variables in study design are often poorly described or omitted, or differences in clinical procedures, materials used and variations in study characteristics make direct comparisons impossible. Irrespective of these limitations, however, certain trends are apparent from comparisons of the results of different clinical studies. Retrospective cross-sectional surveys differ from controlled longitudinal studies in which the clinicians operate under ideal conditions. However, results from controlled clinical studies do not reflect the situation in general dental practice (Mjör, 1997b). Results from longitudinal clinical studies may not be generalized and are difficult to compare with retrospective cross-sectional investigations since the outcome is highly dependent on the individual skills of the dentist and the care taken in placing the restoration (Roulet, 1997a). However, performance differences in the tested materials can be detected earlier in highly standardized longitudinal studies.

This review analyzed the literature, predominantly since 1990, for the clinical longevity of stress-bearing dental restorations in permanent posterior teeth (Black Class I and II) and identified factors contributing to the survival and failure of these restorations. Only clinical studies with an observation time of at least two years and at least 10 restorations at risk at the last recall were considered for calculations in this survey.

LONGEVITY OF DIRECT POSTERIOR RESTORATIONS

Amalgam Restorations

Table 1 summarizes the results of clinical studies on the longevity of amalgam restorations (Allan, 1969, 1977; Bjertness & Sonju, 1990; Burke & others, 1999; Cichon & Kerschbaum, 1999; Crabb, 1981; Hawthorne & Smales, 1997; Jokstad, Mjör & Qvist, 1994b; Jokstad & Mjör, 1991; Kamann & Gängler, 1999; Kiremitci & Bolay, 2003; Kreulen & others, 1998; Lavelle, 1976; Letzel & others, 1989, 1997; Mahmood & Smales, 1994; Mair, 1998; Martin & Bader, 1997; Mjör, 1997b; Mjör & Jokstad, 1993; Mjör & Toffenetti, 1992; Moffa, 1989; Osborne, Normann & Gale, 1991; Paterson, 1984; Pieper & others, 1991; Plasmans, Creugers & Mulder, 1998; Qvist, Qvist & Mjör, 1990; Robinson, 1971; Roulet, 1997b; Setcos, Staninec & Wilson, 1999; Smales, 1991; Smales, Gerke & White, 1990; Smales, Webster & Leppard, 1991a, 1991b; Smales & Hawthorne, 1996, 1997; Summitt & others, 2001; Van Nieuwenhuysen & others, 2003; Welbury & others, 1990; Wilson, Wastell & Norman, 1996). Annual failure rates range between 0 and 7.4% for non-gamma-2 and gamma-2 containing alloys with observation periods of up to 20 years.

Secondary caries, a high incidence of bulk and tooth fracture, cervical overhang and marginal ditching have been reported as the main problems limiting the survival of amalgam restorations (Burke & others, 1999; Dahl & Oilo, 1994; Mjör, 1989, 1997b; Moffa, 1989). Several authors found a higher survival time and a

lower annual failure rate for amalgam restorations in Class I defects compared with Class II cavities (Allan, 1969; Crabb, 1981; Jokstad & others, 1994a, 1994b; Kamann & Gängler, 1999). Cavity size influenced the longevity of amalgam restorations (Maryniuk & Kaplan, 1986; Mjör, 1992a). Large amalgam restorations exhibit more deterioration than moderate- and small-sized restorations (Wilson & others, 1996). In contrast to the adhesive capabilities with modern composite systems, the lack of adhesive stabilization of hard tooth tissues, in combination with amalgam, frequently results in infraction or fracture of restored teeth. The zinc and copper content of the alloy has been found to have a strong impact on the survival rates of amalgam restorations, as it influences the corrosion resistance of the amalgam. High-copper amalgams have higher survival rates than conventional amalgams (Letzel & others, 1997).

Direct Posterior Composite Restorations

Meanwhile, there is widespread use of resin composites for the direct restoration of posterior teeth, even in stress-bearing areas. Table 2 summarizes the results of clinical studies on the longevity of direct posterior composite restorations (Baratieri & Ritter, 2001; Barnes & others, 1991; Burke & others, 1999; Busato & others, 2001; el-Mowafy & others, 1994; Ernst & others, 2001, 2003; Freilich & others, 1992; Gaengler, Hoyer & Montag, 2001; Geurtsen & Schoeler, 1997; Gordan & others, 2002; Helbig & others, 1998; Hugo & others, 2001b; Jokstad & others, 1994b; Köhler, Rasmusson & Ödham, 2000; Letzel, 1989; Lindberg, van Dijken & Lindberg, 2003; Lopes & others, 2003; Lundin & Koch, 1989, 1999; Mair, 1998; Manhart & others, 2000b, 2002b; Mertz-Fairhurst & others, 1998; Mjör & others, 1990; Mjör, 1997b; Mjör & Jokstad, 1993; Moffa, 1989; Collins, Bryant & Hodge, 1998; Nordbo, Leirskar & von der Fehr, 1998; Pallesen & Qvist, 2003; Qvist & others, 1990; Raskin & others, 1999, 2000; Scheibenbogen-Fuchsbrunner & others, 1999; Smales & others, 1990; Türkün, Aktener & Ates, 2003a; Türkün, Türkün & Ozata, 2003b; Türkün & Aktener, 2001; van Dijken, 2000, 2002, 2003a; Van Nieuwenhuysen & others, 2003; Vilkinis, Hörsted-Bindslev & Baelum, 2000; Wassell, Walls & McCabe, 1995, 2000; Welbury & others, 1990; Wilder & others, 1999; Wilson & others, 1988, 2000; Wucher, Grobler & Senekal, 2002). Annual failure rates range between 0 and 9%.

Insufficient wear resistance resulting in loss of anatomic form and interproximal contacts with general degradation were the main problems of direct composite restorations in the ’70s and early ’80s (Leinfelder & others, 1980). Improvements in filler technology and the formulation of composite materials have resulted in changes in the reasons for restoration replacement, as well as the increasing trend to insert composite restorations in stress-bearing areas of posterior teeth. Marginal opening with secondary caries, fracture of the restorations, marginal deterioration, discoloration and wear are now the principle modes of failure and reasons for limitations in the longevity of resin-based composites (Barnes & others, 1991; Geurtsen & Schoeler, 1997; Jokstad & others, 1994b; Mjör, 1996, 1997b; Pallesen & Qvist, 2003; Scheibenbogen-Fuchsbrunner & others, 1999; Smales & others, 1991b). Microfilled composites exhibited more fracture-related failures, especially in high-stress Class II cavities, compared with hybrid composites, because of their inferior mechanical properties (Hickel, 1997). The relatively high incidence of secondary caries may be explained with the low efficacy of older generation bonding agents, in particular, when cavity finish lines were lying within dentin. Despite improvements in the formulation of new bonding agents with enhanced marginal adaptation and bond strengths, a perfect marginal seal is still not achievable. Premolars generally offer more favorable conditions for composite restorations compared with molars (Geurtsen & Schoeler, 1997; Rykke, 1992; Scheibenbogen-Fuchsbrunner & others, 1999). Cavities tend to become smaller and, as a consequence, the effect of the chewing forces is less intense. A 17-year study of ultraviolet-cured posterior composites by Wilder and others (1999) demonstrated an excellent success rate of 76%. Color matching (94% Alpha), marginal discoloration (100% Alpha), marginal integrity (100% Alpha), secondary caries (92% Alpha), surface texture (72% Alpha), anatomic form (22% Alpha) and a mean occlusal wear of 264 µm was recorded after 17 years. Most wear (75%) occurred in the first five years, confirming the findings of Raskin and others (1999).

Ormocers

The negative effects of polymerization shrinkage, besides wear, were often cited as common causes of failure of direct posterior composite restorations (Leinfelder & others, 1980). An inherent weakness of resin composite restorations has been attributed to the organic matrix component. Ormocers (ormocer = acronym for organically modified ceramics) were introduced in the European market in 1998 and are characterized by a novel inorganic-organic copolymer matrix (Hickel & others, 1998; Manhart & others, 1999a, 1999b, 2000a; Wolter, Storch & Ott, 1994). For this new class of composite restorative materials based on ormocer technology, only the preliminary data of few clinical trials are available, indicating a wide range of annual failure rates between 0% and 12.7% (Lopes & others, 2002, 2003; Manhart & others, 2002b; Oberländer & others, 2001; Rosin & others, 2003). However, in all of these studies, the same restorative material (Definite, Degussa, Hanau) was used, mostly in combination with the self-etching adhesive Etch&Prime 3.0 (Degussa, Hanau). In laboratory tests, this adhesive has shown

significantly lower bond strength compared to other tested systems (Gernhardt, Salhab & Schaller, 2001).

Direct Posterior Composite Restorations with Inserts

Only a very limited number of clinical studies (Table 3) that report the performance of direct composite restorations in combination with prefabricated glass-ceramic inserts are available (Hugo & others, 2001a; Kiremitci, Bolay & Gurgan, 1998; Lösche, 1996; Ödman, 2002; Sjögren & others, 2000). Annual failure rates range between 0 and 10.3%.

Lösche (1996) restored 24 Class II cavities in premolars with composite and glass-ceramic inserts and observed a 100%-success rate after two years. Kiremitci (Kiremitci & others, 1998) observed a survival rate of 95.5% for Class II composite restorations reinforced with Beta-quartz glass-ceramic inserts after two years. However, after three years of clinical service, Sjögren and others (2000) rated only 69% of 39 Heliomolar Beta-quartz insert restorations in Class I and II cavities as satisfactory. Four restorations lost their inserts, seven fractured or exhibited flaking surfaces and one failed because of recurrent caries.

Compomer Restorations

Several clinical trials are ongoing to determine the clinical performance of compomers in stress-bearing Class I and II cavities in the permanent dentition. The annual failure rates of compomer restorations range between 0 and 3.3%. The results of clinical studies are summarized in Table 4 (Demirci & Ücok, 2002; Huth & others, 2003; Jedynakiewicz, Martin & Fletcher, 2002; Luo & others, 2002; Manhart & others, 2002c, 2002e; Wucher & others, 2002).

These results reflect up to three years with a limited validity but with excellent results. It will be worth monitoring the performance of these restorations over time. The relative ease of handling of compomer materials is claimed to be the main reason for their wide acceptance by practitioners, especially in European countries (Hickel, 1997; Höhnk & Hannig, 1998; Manhart & Hickel, 1999a), although several compomer materials have exhibited significantly inferior wear rates compared with hybrid composites (Hickel & others, 1998; Powers & others, 1998). However, compomers comprise a rather inhomogeneous class of restorative materials concerning their physical and mechanical data, with some materials being stronger than microfilled composites and packable and hybrid composites (Flessa & others, 2001; Manhart & others, 2001b, 2001c). Main failure reasons are similar to resin composites, including wear and secondary caries (Huth & others, 1999; Luo & others, 2000, 2002). In some clinical studies, enamel and dentin are not separately acid etched prior to adhesive application, but self-conditioning adhesive systems are used. However, more recent laboratory investigations have shown that acid-etching enamel margins yielded superior results when compared with the results of self-conditioning adhesives that were available when compomer restorative materials were introduced into the market (Buchalla, Attin & Hellwig, 1996; Cortes, García Godoy & Boj 1993; Manhart & others, 1998; Triolo, Barkmeier & Los, 1995).

Glass Ionomer Cement Restorations

The annual failure rates of posterior glass ionomer restorations range within 0 and 14.3%. Table 5 summarizes the results of clinical studies (Frencken, Makoni & Sithole, 1998; Gao & others, 2003; Hasselrot, 1993, 1998; Hickel & others, 1988; Ho, Smales & Fang, 1999; Krämer & others, 1994; Mallow, Durward & Klaipo, 1998; Mjör, 1997b; Mjör & Jokstad, 1993; Nicolaisen & others, 2000; Phantumvanit & others, 1996; Pilebro & van Dijken, 2001; Smales & others, 1990, 1991b; Strand & others, 1996, 2000; Svanberg, 1992; Ziraps & Honkala, 2002).

Glass ionomer cements are not considered as having adequate mechanical strength for general use as definitive restorations in stress-bearing posterior teeth (Hickel & others, 1998; Mjör & others, 1990, 1997b; Mjör & Jokstad, 1993). Many glass ionomer restorations have failed because of bulk fractures due to their low mechanical strength (Krämer & others, 1994; Mjör & Jokstad, 1993). Silver particles sintered into the glass ionomer powder particles were claimed to increase the strength and radiopacity; however, metal-reinforced glass ionomer cements (cermet) are not suitable as a long-term restorative material for use in Class II cavities (Hickel & others, 1988; Krämer & others, 1994; Mjör & Jokstad, 1993). In contrast to expectations and despite the release of fluoride ions, in some studies, secondary caries has been found to be the main reason for the clinical failure of glass ionomer restorations (Burke & others, 1999; Hasselrot, 1998; Mjör, 1997b). The release of fluoride ions has been anticipated as reducing the incidence of secondary caries. The longevity of glass ionomer restorations is further dependent on the use of appropriate clinical techniques, as these materials tend to be rather technique sensitive, especially with respect to water adsorption and dehydration (Smales & others, 1990; Svanberg, 1992).

*Tunnel Restoration*s

Tunnel restorations (Table 5) comprise a special subgroup within all glass ionomer restorations. Partial tunnel preparations (Class I) with an unbroken approximal enamel wall need to be distinguished from total tunnel configurations (Class II) with perforated interproximal enamel (Hasselrot, 1998). Failures are mainly caused by fractures of the marginal ridge, secondary caries and progressive enamel cavitation and/or degra-

dation of the glass ionomer restorations (Hasselrot, 1998; Strand & others, 1996; Svanberg, 1992).

ART Restorations

ART restorations (atraumatic restorative treatment) (Table 5, section 3) are based on the removal of tooth decay with hand instruments only after which the cleaned cavity is filled with a glass ionomer cement (Frencken, Makoni & Sithole, 1996). This technique has been established to provide a minimum of dental health care to rural areas in developing countries, where no electricity-driven dental equipment can be used. Most studies are limited to Class I cavity configurations. Annual failure rates of this type of restoration are rather high and are listed with 33% in maximum for Class II cavities (Frencken & others, 1994). This results from the limited conditions present during placement of the restorations. Furthermore, these ART restorations are often placed by dental nursing students, rather than clinically experienced dentists (Mallow, Durward & Klaipo, 1995, 1998). Wear, fracture and recurrent caries are reported as the main reasons for failure (Ho & others, 1999; Yip & others, 2002).

LONGEVITY OF INDIRECT POSTERIOR RESTORATIONS

Composite Inlays and Onlays

Composite inlays are indicated for the restoration of large defects. The major advantage is that most of the composite used is displaced by an inert body of the composite inlay, which is inserted in the cavity using a minimum of resin cement to be polymerized in the mouth and a better control of anatomic form and proximal contacts (Bessing & Lundqvist, 1991; Burke & Qualtrough, 1994; Donly & others, 1999; O'Neal, Miracle & Leinfelder, 1993; Roulet, 1997a; Scheibenbogen-Fuchsbrunner & others, 1999). Table 6 summarizes the results of clinical studies (Donly & others, 1999; Frederickson & Setcos, 1994; Haas & others, 1992, 1996; Hannig, 1996; Klimm, Wolff & Natusch, 1999; Krämer & others, 1996; Leirskar & others, 2003; Manhart & others, 2002a,b,d; Pallesen & Qvist, 2003; Scheibenbogen-Fuchsbrunner & others, 1999; Thordrup, Isidor & Hörsted-Bindslev, 2001; van Dijken, 1994, 2000; Wassell & others, 1995, 2000; Wendt & Leinfelder, 1992; Wiedmer, Krejci & Lutz, 1997). Annual failure rates of posterior composite inlays and onlays range between 0 and 10%.

Many of the problems associated with the direct placement of large posterior composite restorations can be overcome with the use of an indirect composite inlay technique. Composite inlays have been concluded to be a good, longer lasting alternative to direct plastic composite restorations in large Class II situations (van Dijken, 1994). Indications for esthetic inlays include teeth in which strengthening of the remaining structure is indicated, the cavity is free from marked undercuts and the patients are requesting tooth-colored restorations in posterior teeth (Burke & Qualtrough, 1994; Hickel & Kunzelmann, 1997). Strict patient and case selection, that is, frequent attenders with a good standard of oral hygiene and cavities that allow adequate moisture control, will increase the longevity of adhesive inlays.

The indirect technique allows the production of restorations in the laboratory with appropriate proximal contours and contacts and control of anatomic form. Polymerization shrinkage is limited to the width of the luting space. Post-curing the inlay with heat, pressure and/or light increases the degree of conversion through an annealing process, improving the mechanical properties of the composite and resulting in better wear resistance (Burke & Qualtrough, 1994; Wassell & others, 1995; Wendt & Leinfelder, 1992). Several authors have indicated that premolars offer more favorable conditions for indirect composite restorations than molars (Donly & others, 1999; Rykke, 1992; Scheibenbogen-Fuchsbrunner & others, 1999). A premolar restoration is subjected to much less occlusal stress than a molar restoration, the access for dental treatment is easier and oral hygiene measures are more easily controlled by the patient. The main reasons for failure of composite inlays include fracture, marginal opening, secondary caries and postoperative sensitivity (Donly & others, 1999; Krämer & others, 1996; Pallesen & Qvist, 2003; van Dijken, 2000; Wassell & others, 1995).

Ceramic Inlays and Onlays

Ceramic inlays are traditionally made of feldspathic ceramic materials by sintering or by way of a more recent technique of milling from prefabricated blocks. Glass ceramic inlays can be cast or pressed, using the lost wax technique, or they can be produced by milling (Hickel & Kunzelmann, 1997; Roulet, 1997a; Studer & others, 1996). Table 7 summarizes the results of clinical studies that investigate laboratory-fabricated ceramic inlays and onlays made by dental technicians (Barghi & Berry, 2002; Felden & others, 1998; Felden, Schmalz & Hiller, 2000; Fradeani, Aquilano & Bassein, 1997; Frankenberger, Petschelt & Krämer, 2000; Friedl & others, 1996, 1997; Fuzzi & Rapelli, 1998, 1999; Haas & others, 1992, 1996; Hayashi & others, 1998, 2000; Höglund, van Dijken & Olofsson, 1992, 1994; Isidor & Brondum, 1995; Jensen, 1988; Krämer & others, 1999; Krejci, Krejci & Lutz, 1992; Lehner & others, 1998; Malament & Socransky, 1999; Manhart & others, 2002a; Molin & Karlsson, 1996, 2000; Qualtrough & Wilson, 1996; Roulet, 1997b; Schulz, Johansson & Arvidson, 2003; Stenberg & Matsson, 1993; Studer & others, 1996; Thonemann & others, 1997; Thordrup & others, 2001; Tidehag & Gunne, 1995; van Dijken,

2003b; van Dijken, Hoglund-Aberg & Olofsson, 1998; van Dijken, Örmin & Olofsson, 1999; van Dijken & others, 2001). The annual failure rates of ceramic inlay restorations range between 0 and 7.5%.

Bulk fracture is the most frequent cause of failure of ceramic inlays (Roulet, 1997b; Schulz & others, 2003; Studer & others, 1996; van Dijken & others, 2001; Walther, Reiss & Toutenburg, 1994). The risk of a ceramic inlay fracturing depends, among other factors, on the strength of the material. Ceramic materials are brittle and susceptible to failure in tensile mode, while their resistance to compression is high. Flaws at internal or external surfaces are, in many cases, the origin of cracks that can propagate and lead to catastrophic failure (Chen & others, 1999; Martin & Jedynakiewicz, 1999). Other important factors, such as the design of the cavity preparation, the shape of the restoration (minimum restoration thickness: 1.5 mm) and the internal fit, influence the strength of the ceramic restoration. Strict case selection, avoiding placement of the ceramic inlays in patients suffering from heavy parafunctional activities or bruxing/clenching and situations that require crowns, increases the probability for success of the ceramic inlay (Roulet, 1997b). In general, preparation dimensions have an important influence on the fracture resistance of all-ceramic restorations (Martin & Jedynakiewicz, 1999; Walther & others, 1994). Failure to achieve necessary cavity dimensions may contribute more to failure by fracture than the nature of the ceramic system. Wear of the resin cement in the luting gap results in marginal deterioration of ceramic restorations, especially in the first years after placement of the restoration (Friedl & others, 1997; Hayashi & others, 1998; Krämer & others, 1999; Krejci & others, 1992; Manhart & others, 2001a). It is of crucial importance to remove flashes of the luting agent after adhesive cementation and to avoid gingival inflammation and periodontal problems.

Different modes of ceramic inlay cementation, adhesive vs conventional, or the use of chemically-cured vs dual-cured composites are studied in the literature (Höglund & others, 1992, 1994; Stenberg & Matsson, 1993; van Dijken, 2003b; van Dijken & others, 1998, 1999, 2001). Höglund and others (1992) studied the effect of the luting agent on the clinical performance of 118 fired Mirage inlays in Class II cavities. Half of the restorations were cemented with either a dual-cured composite cement or a glass ionomer cement. After two years, the assessment exhibited a 2% failure rate for resin cemented inlays, while 15% of the restorations cemented with the glass ionomer luting agent failed. The three-year follow-up examination confirmed these observations (Höglund & others, 1994). At the six-year follow-up examination, a significantly higher failure rate could be revealed for the ceramic inlays placed with a glass ionomer cement (74% survival) compared with the resin cement bonded restorations (88% survival) (van Dijken & others, 1998). Similar failure rates were observed for cast Dicor glass ceramic inlays inserted with a glass ionomer luting cement, showing 8% failure after two years (Stenberg & Matsson, 1993). Van Dijken (van Dijken, 2003b; van Dijken & others, 1999) reported after two and five years no significant difference for pressed Empress inlays cemented with a resin-modified glass ionomer or a chemically-cured composite. No significant differences were found when comparing a dual-cured vs a chemically-cured luting resin for the cementation of Empress restorations (van Dijken & others, 2001).

CAD/CAM Ceramic Inlays and Onlays

CAD/CAM-technology is implemented in the Cerec system which mills ceramic inlays from industrial blocks of ceramic material that are prefabricated under optimum and controlled conditions (Martin & Jedynakiewicz, 1999). It is possible to obtain a high, uniform quality ceramic without the inevitable material variations seen in manually produced restorations (Mörmann & Brandestini, 1989; Sjögren & others, 1992). Table 8 presents the longevity data of computer generated ceramic restorations (Berg & Derand, 1997; Bindl & Mörmann, 2003; Gladys & others, 1995; Haas & others, 1992; Heymann & others, 1996; Molin & Karlsson, 2000; Mörmann & Krejci, 1992; Otto, 1995; Otto & de Nisco, 2002; Pallesen & van Dijken, 2000; Posselt & Kerschbaum, 2003; Reiss & Walther, 1991, 1998, 2000; Sjögren & others, 1992; Sjögren, Molin & van Dijken, 1998; Thordrup, Isidor & Hörsted-Bindslev, 1999; Thordrup & others, 2001; Walther & others, 1994; Zuellig-Singer & Bryant, 1998). The annual failure rates of CAD/CAM ceramic restorations range between 0 and 5.6%.

The main reasons for failure of machined ceramic inlays, onlays and partial crowns are restoration fracture, tooth fracture, postoperative symptoms and recurrent caries (Otto & de Nisco, 2002; Pallesen & van Dijken, 2000; Reiss & Walther, 1991; Sjögren & others, 1992; Thordrup & others, 1999; Walther & others, 1994). Cerec inlays that were cemented either with a dual-cured or a chemically-cured luting resin exhibited no significant difference in clinical performance after five years of clinical service (Sjögren & others, 1998). Zuellig-Singer and Bryant (1998) compared Cerec-Vita Mark II restorations in Class II cavities luted with three different resin cements or a glass ionomer cement. After three years of service, 100% (but only nine restorations in this subgroup) of the latter were still intact, while one inlay (3.6%) inserted with a composite cement fractured.

Cast Gold Inlays and Onlays

Usually, cast gold restorations tend to be used in patients with excellent oral hygiene, which influences

the results of clinical studies significantly (Mjör, 1992a). Table 9 summarizes the results of clinical studies that examine the longevity of cast gold inlays (Bentley & Drake, 1986; Crabb, 1981; Donly & others, 1999; Erpenstein, Kerschbaum & Halfin, 2001; Fritz, Fischbach & Harke, 1992; Haas & others, 1992, 1996; Hawthorne & Smales, 1997; Jokstad & others, 1994b; Leempoel & others, 1985; Mahmood & Smales, 1994; Mjör & Medina, 1993; Molin & Karlsson, 2000; Pelka, Schmidt & Petschelt, 1996; Schlösser & others, 1993; Smales & Hawthorne, 1996; Stoll & others, 1999; Studer & others, 2000; Wagner, Hiller & Schmalz, 2003). Annual failure rates of cast gold restorations range between 0 and 5.9%.

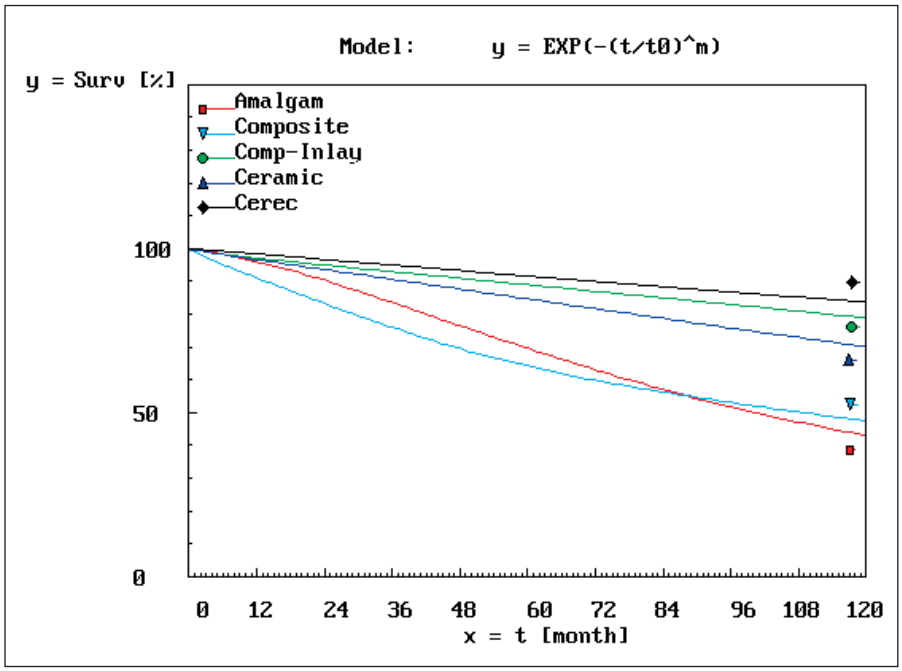


Figure 1. *Plots of a 2-parameter fit of the published survival curves (Tables 1, 2 and 6 through 8).*

Compared to other restorations for posterior teeth, cast gold restorations are considered costly, but long lasting (Stoll & others, 1999). The relative cost factor of gold restorations has been calculated to be 3.8 to 6.3 times that of amalgam restorations (Mjör, 1992a; Smales & Hawthorne, 1996). Gold restorations are, however, considered the most durable for posterior teeth. Tooth fracture, marginal defects, insufficient retention and secondary caries are main reasons for the failure of cast gold inlays (Donly & others, 1999; Fritz & others, 1992; Mjör & Medina, 1993; Stoll & others, 1999; Studer & others, 2000). If the size of a lesion requires replacement of one or more cusps, gold onlays or partial crowns are still an excellent method for achieving tooth restoration despite the possibilities offered by adhesively bonded all-ceramic restorations. Smales and Hawthorne (1996) found that posterior cast gold restorations had significantly greater longevity than cuspal replacement amalgam restorations.

Statistical Evaluation

The values recorded for annual failure rates in Tables 1 through 9 were subsequently processed by calculating mean, standard deviation and median for each class of restoration (Tables 10 and 11). Only annual failure values with an observation time of restorations at risk for at least two years were processed. One study detailed several failure rates for different materials, each annual failure rate was processed as a single case in the statistical procedure. With regard to clinical studies published with different observation periods, only the most recent published failure rates were processed. Where appropriate, subgroups were built for longitudinal vs cross-sectional studies, studies published before 1990 vs those published in 1990 and later, Black Class I vs Class II cavities and for studies using survival curve analysis (*Kaplan-Meier*) vs others. Statistical analysis was performed using ANOVA and post-hoc LSD-test ($p<0.05$) (Table 10).

Statistical comparison of amalgam restorations, direct composites and all different types of indirect restorations exhibited a significant difference among these types of dental restorations ($p=0.0107$). For amalgam restorations, the post hoc LSD-test revealed a significantly higher annual failure rate for gold inlays than laboratory-made ceramic inlays and CAD-CAM-manufactured ceramic restorations. Also, gold and CAD-CAM ceramic inlays exhibited significantly lower failure rates than composite inlays. When only data from longitudinal studies covering Class II restorations published in 1990 and later were investigated, slightly different results were presented (Table 10). Figure 1 depicts the plots of a 2-parameter fit of the published survival curves listed in Tables 1 and 2 and Tables 6 through 8 (Hamm, Manhart & Hickel, 2003). Besides amalgam, for most groups, only few restoration data with an observation period of more than eight years have been published. There were no differences in annual failure rates for all groups of materials when studies with an observation period of at least two years with studies of at least four-years duration were compared. Calculations showed a rather constant annual failure rate over time when publications of less than two years of observation were excluded.

Statistical analysis of the subgroups (Table 10) created for study design (longitudinal vs cross-sectional), cavity design (Black Class I vs Class II) and statistical analysis of the individual clinical studies (*survival curves*:

yes vs no) detailed no significant influence of subclassification, but there were some recognizable tendencies. Only studies investigating the longevity of dental restorations published before 1990 showed significantly higher annual failure rates for amalgam and direct composite restorations compared to the results of clinical investigations printed in 1990 or later. However, publication dates had no significant influence on the performance of gold restorations.

When the data for direct placement (amalgam, composite) and indirect restorations (composite inlays, ceramic, CAD/CAM, gold) were pooled, the statistical analysis showed a significantly lower (p=0.0031) mean annual failure rate for indirect restorations (mean 2.0% ± 2.0; median 1.3%) compared to direct techniques (mean 3.0% ± 2.9; median 2.3%). When only publications with survival curves were included for comparison of direct vs indirect restorations, 50% of the pooled direct restorations (amalgam and composite) failed after about nine years; whereas, after 10 years, 75% of all adhesive inlays remained *in situ* (Figure 2). Gold inlays were not included in this 2-parameter fit calculation, as not enough published survival curve data were available.

For direct composite restorations with inserts, compomers and all subclasses of glass ionomer restorations, no statistical evaluation was performed due to the limited data material available, only descriptive values (mean, standard deviation, median) were calculated (Table 11).

All studies showing median survival times in their results (only cross-sectional studies) were compared as another set of data. Medium survival times were calculated for four groups (Figure 3). Again, gold restorations performed best. But, in contrast to the first—and larger—set of data comprised by annual failure rates (with many controlled longitudinal studies), the ranking of amalgam and composite changed. In this data set, gold restorations now showed significantly better performance than direct composite restorations. Amalgam restorations (medium 8.5 years) were not significantly different from direct composites (medium 5.5 years) and gold inlays (medium 12.2 years).

DISCUSSION

The handling of dental restorative material under ideal circumstances produces a restoration that can last for many years. However, the longevity of the restoration is dependent upon many factors, which are patient-, dentist- and material-related (Table 12) (Hickel, 1996).

Studies that did not provide survival or failure rates, but median survival times, were all cross-sectionally designed. They mainly reflect the situation in daily practice. In contrast to the other set of studies, where mainly controlled longitudinal clinical investigations were included, failure rates for all groups of restorations were drastically higher. In both data sets, gold restorations performed best. However, in contrast to the set of cross-sectional studies where amalgam restorations showed a trend of being superior to direct composites, the situation changed in the other set of data (mainly longitudinal studies).

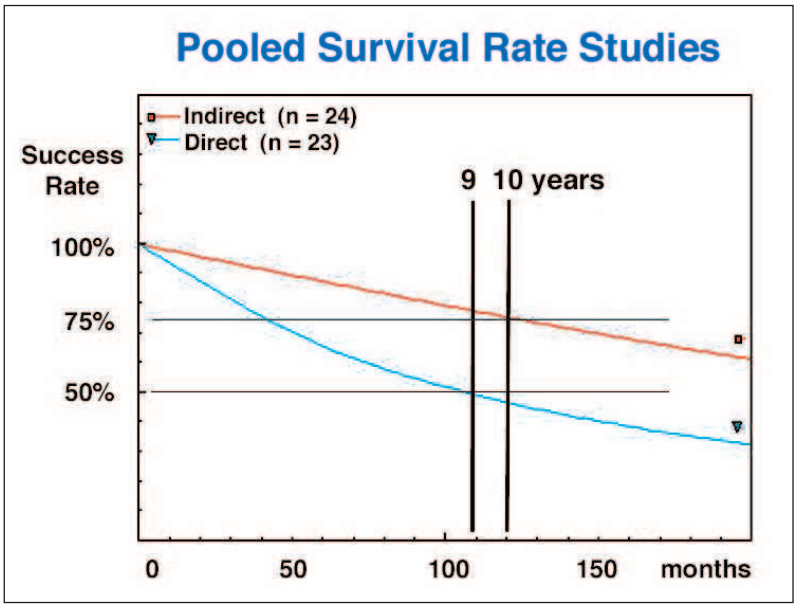


Figure 2. *Comparison of the published survival curves of direct restorations (amalgam and composite) vs indirect adhesive inlays with the 2-parameter fit.*

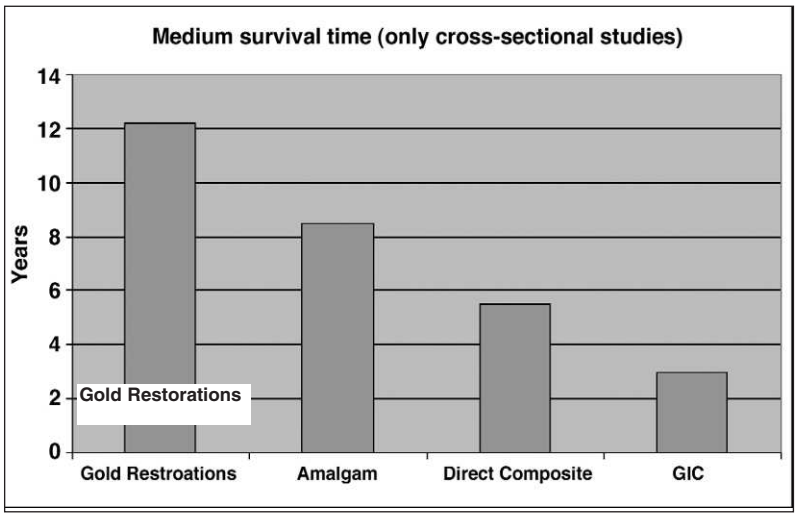


Figure 3. *Medium survival times of studies indicating median survival times in their results.*

In a survival analysis of posterior restorations using an insurance claim database (Bogacki & others, 2002) with a huge number of restorations (207,558 amalgam and 93,195 direct composite), it was stated that the survival probability for amalgam (94% after five years) was slightly higher than for direct composite restorations (93% after five years). However, seven-year survival probability decreased significantly from 92% to 60% when the patients changed to different dentists for recall examinations.

The findings of several studies supported the view that single-surface restorations show greater longevity than multi-surface restorations (Crabb, 1981; Krämer & others, 1994; Maryniuk & Kaplan, 1986; Mjör, 1992b). However, restorations limited to the occlusal surface may incur significantly more failures due to new caries formation compared to Class II restorations (Fritz & others, 1992; Robinson, 1971; Stoll & others, 1999). The development of new lesions of caries on unprotected approximal surfaces during the lifetime of Class I restorations was considered to account for this phenomenon. Furthermore, premolars were found to offer significantly more favorable conditions for the survival of adhesive inlays than molars (Fuzzi & Rappelli, 1998; Geurtsen & Schoeler, 1997; Rykke, 1992; Scheibenbogen-Fuchsbrunner & others, 1999). The age of the patient had an important influence on treatment outcome (Bentley & Drake, 1986; Plasmans & others, 1998; Smales & others, 1991a). Smales and others (1991a) mentioned an age effect but found no statistical evidence; whereas, Plasmans and others (1998) and Bentley and Drake (1986) found a superior survival rate for restorations placed in younger adults compared with older patients. Differences in the quality of oral hygiene measures, fluoride availability, dietary habits and periodontal problems may be associated with these findings.

Postoperative hypersensitivity is a complication frequently encountered after the luting of an adhesive inlay (Hickel, 1990; Otto, 1995; Roulet & Herder, 1989; Sjögren & others, 1992; Thordrup, Isidor & Hörsted-Bindslev, 1994). The risk of post-placement hypersensitivity has been attributed to the method of luting and could be significantly reduced by improved dentin bonding agents and resin cements, in addition to the meticulous use of recommended techniques and avoiding tooth desiccation. While in 1990, up to 16% hypersensitivity could be observed after placement of adhesive restorations (Hickel, 1990); these figures have decreased significantly, with an incidence of 0 to 3% today (Manhart & Hickel, 1999b).

In general, early failures of dental restorations encountered after weeks or months need to be distinguished from late failures after several years of clinical service. The early failures are a result of severe treatment faults (for example, incorrect manipulation of the materials, insufficient polymerization that results in weak material properties), selecting an incorrect indication for the restorative material or postoperative symptoms. Late failures are predominantly caused by fractures (tooth and/or restoration), the occurrence of secondary caries and wear or deterioration of the respective materials.

Cross-sectional clinical studies differ in many aspects from controlled, prospective, longitudinal clinical investigations on the performance of dental restorations (Mjör & others, 1990; Mjör, 1997b). In the design of a controlled longitudinal study, a very limited number of excellent dentists, specially trained and standardized for the specific procedure, are placing restorations under almost ideal conditions. The patient population is often selected from reliable, easily available individuals, such as dental students and dental school staff and faculty who have good compliance and are highly motivated for oral health and hygiene (Roulet, 1997a). The lesions treated are usually strictly selected and restricted to the indications of the investigated materials. The recall criteria are defined and, ideally, the calibrated recall assessments are performed by dentists different from those inserting the restorations (Mjör, 1997b). Replicas and color photographs may be recorded on a regular basis to assist in the evaluation of the restorations over time (Scheibenbogen-Fuchsbrunner & others, 1998). The early detection of performance differences among tested materials (failure rate and failure mode) is the major advantage of these highly standardized longitudinal studies with strict clinical protocols. Cross-sectional clinical investigations of dental restorations are less strictly defined than longitudinal studies. Important factors such as restorative materials can usually only be differentiated as types of materials. The technical procedure used, the conditions of the cavity preparation and the use of a base material are mostly unknown factors (Mjör, 1997b). In most cases, it is at least difficult, if not impossible, to determine the exact age of the restorations (Rykke, 1992). One major advantages of cross-sectional clinical studies is that a large number of restorations can be assessed in a relatively short time and reflects the type of dental care the patients receive, rather than the performance of restorations in almost ideal conditions of longitudinal studies. Practice-based research is a source from which clinically relevant problems can be identified (Mjör, 2004).

CONCLUSIONS

The longevity of dental restorations is dependent upon many different factors including materials-, dentist- and patient-related factors. The main reasons for failure were predominated by the formation of secondary caries, fracture of the bulk of the restoration or the tooth, marginal deficiencies and wear. The importance of direct-placement esthetic restorative materials is

still increasing. Amalgam restorations are being replaced more frequently, because of alleged adverse health effects and inferior esthetic appearance. However, all alternative restorative materials and procedures have certain limitations. Direct composite restorations require a time-consuming and more costly treatment procedure. Currently, glass ionomers can only be considered as long-term provisional restorations in stress-bearing cavities. Future treatment regimens that are only made possible by the development of sophisticated preparation techniques, improved dentin bonding agents and resin-based restorative materials, will result in the therapy of more small-sized lesions rather than large restorations. The importance of indirect inlay techniques will shift more and more towards direct restoratives. As if the cavities become smaller, it is to be expected that the use of improved direct restorative materials will provide excellent longevity even in stress-bearing situations.

(Presented 19 February 2004)

Table 1: *Longevity of Amalgam Restorations in Posterior Teeth (SD = Study Design; CS = cross-sectional; L = longtudinal; MA = meta analysis)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)	Median Survival Time (years)
1969	Allan	10	I II	Amalgam (alloys not specified, gamma-2 alloys)	78 92		Defined criteria for clinical failure	CS	54 39	4.6 6.1	
1971	Robinson	20	I and II	Amalgam (alloys not specified, gamma-2 alloys)	145		Defined criteria for clinical failure	CS	22.8	3.9	10
1976	Lavelle	20	I and II	Amalgam (alloys not specified, gamma-2 alloys)	6000		Defined criteria for clinical failure	CS		4.8	
1976	Lavelle	20	I and II	Amalgam (alloys not specified, gamma-2 alloys)	400		Defined criteria for clinical failure	L		7	<10
1977	Allan	20	I and II	Amalgam (alloys not specified, gamma-2 alloys)	148		Defined criteria for clinical failure	CS	14	4.3	8
1981	Crabb	10	I II	Amalgam (alloys not pecified, gamma-2 alloys)	269 530		Defined criteria for clinical failure	CS	59.5 37.2	4.1 6.3	>10 8
1984	Paterson	15	I II	Solila	854 1490		Defined criteria for clinical failure	CS			8 7
1989	Letzel	5-7	I II	Conventional and high-copper alloys	2341		Defined criteria for clinical failure	L	88-91		
1989	Moffa	5	I II	Amalgam (alloys not specified)	314		Modified USPHS criteria		90 75	2 5	
1990	Bjertness	17	I and II	Amalgam (alloys not specified)	782		Defined criteria for clinical failure	CS	78	1.3	
1990	Qvist		I II	Amalgam (alloys not specified)			Defined criteria for clinical failure	CS			9.5 8
1990	Smales	3	I	Dispersalloy	13		Defined criteria for clinical failure	L	100	0	
1990	Welbury	5	I	Amalcap	150	103	Modified USPHS criteria	L	92.7	1.5	
1991	Jokstad	7-10	II	4 non-gamma-2 alloys 1 conventional alloy	256	141	USPHS criteria		73.5	2.7-3.8	
1991	Osborne	14	I and II	5 gamma-2 alloys and 7 non-gamma-2 alloys	367	40	Defined criteria for clinical failure and photographs	L	87.2	0.9	
1991	Pieper	9-11	I II	Amalgam (alloys not specified)	129 413		Modified USPHS criteria	CS	85.3	1.3-1.6	
1991	Smales	11-18	I and II	New True Dentalloy, Dispersalloy, Indiloy, Shofu Spherical	1680			CS		1.0-1.7 and 6.3	
1991	Smales	18	I and II	New True Dentalloy, Shofu Spherical, Dispersalloy, Tytin, Indiloy	1801		Defined criteria for clinical failure	CS	70	1.7	
1991	Smales	15	II		768				72	1.9	
1992	Mjör			Amalgam (alloys not specified)	360		Defined criteria for clinical failure	CS			4.7
1993	Mjör	5	II	Dispersalloy	88		Modified USPHS criteria	L	95	1	

Table 1: *Longevity of Amalgam Restorations in Posterior Teeth (continued)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%) (%)	Annual Failure Rate	Median Survival Time (years)
1994	Jokstad	> 10	I II	Amalgam (alloys not specified)	803 > 3000			CS			14 7-11
1994	Mahmood	> 14	I and II	Amalgam (alloys not specified)	245 (P) 455 (A)		Defined criteria for clinical failure	CS			7.9 9
1996	Smales	15	II	Amalgam (alloys not specified)	160			CS	47.8	3.5	
1996	Wilson	5	I and II	High-copper amalgams (Sybralloy, Dispersalloy, Tytin)	172		Defined criteria for clinical failure	L	94.8	1	
1997	Hawthorne		I and II	Amalgam (alloys not specified)	1371		Defined criteria for clinical failure	CS			22.5
1997	Letzel	13	I and II	Conventional zinc-free a. Convent. zinc-contain. a. High-copper zinc-free a. High-copper zinc-cont. alloys	3119 (all)			MA	25 70 70 85	5.8 2.3 2.3 1.2	
1997	Martin	5	II: 4 surface II: 5-surface	Valiant PhD	2038 1626		Defined criteria for clinical failure	CS	72 65	5.6 7	
1997	Mjör	>25	I and II	Amalgam (alloys not specified)	282		Defined criteria for clinical failure	CS			9
1997	Roulet	6	I and II	5 high-copper amalgams (Amalcap plus, Contour, Permite C, Dispersalloy, Si-Am-Kap)	163	43	Modified USPHS criteria	CS	87.5	2.1	
1997	Smales	5 10 15	II	Amalgam (alloys not specified)	160		Defined criteria for clinical failure	CS	77.6 66.7 47.8	4.5 3.3 3.5	14.6
1998	Collins	8	I and II	Amalgam (Dispersalloy) Composite: - Heliomolar radiopaque - Herculite XR - P30 APC	52 55 52 54	46	Modified USPHS criteria	L	94.2 83.6 84.6 90.7	0.7 2.1 1.9 1.2	
1998	Kreulen	15	II	New True Dentalloy, Tytin, Cavex	1117	183	Defined criteria for clinical failure	L	83	1.1	
1998	Mair	10	II	New True Dentalloy, Solila Nova	35		Modified USPHS criteria	L	94.3	0.6	
1998	Plasmans	8	II	Cavex (non-gamma-2)	266	130	Defined criteria for clinical failure	L	88	1.5	
1999	Burke		I II	Amalgam (alloys not specified)	268 1142			CS			7.4 6.6
1999	Cichon	8	1-surface 2-surfaces 3-surfaces	Amalgam (alloys not specified)	820		Defined criteria for clinical failure	CS	80 73.2 71.1	2.5 3.4 3.6	
1999	Kamann	6	I II	Luxalloy	62 21			L	83.9 66.7	2.7 5.6	
1999	Setcos	2	II	Dispersalloy (adhesively bonded) Dispersalloy (not bonded)	55 50	31 (all)	Modified USPHS criteria	L	100 94	0 3	
2001	Summitt	5	II	Tytin (bonded) Tytin (pin-retained)	21 19	28 (all)	Modified USPHS criteria	L	90.5 63.2	1.9 7.4	
2003	Kiremitci	3	I	Galloy (Gallium restorative alloy) Permite-C (high-copper amalgam)	32 32	14	Modified USPHS criteria	L	84.4 100	5.2 0	
2003	Van Nieuwen-huysen		I and II	Amalgam Composite	722 115		Defined criteria for clinical failure	L			12.8 7.8

Table 2: *Longevity of Direct Composite Restorations in Posterior Teeth (SD = study design; CS = cross-sectional; L = longitudinal)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)	Median Survival Time (years)
1988	Wilson	5	I and II	Occlusin	67		Modified USPHS criteria	L	86	2.8	
1989	Letzel	4	I and II	Occlusin	711		Defined criteria for clinical failure	L	94	1.5	
1989	Lundin	4	I and II	Occlusin and PC4502	137	65	Modified USPHS criteria	L	84	4	
1989	Moffa	5	I II	Composite resins (not specified)	356		Modified USPHS criteria		80 55	4 9	
1990	Mjör		II	Composite resins) (not specified							4
1990	Qvist		I II	Composite resins (not specified)			Defined criteria for clinical failure	CS			3 3
1990	Smales	3	I	Visio-Molar P-30	42 251		Defined criteria for clinical failure	L	93.9 100	2 0	
1990	Welbury	5	I	Prisma-Fil and Prisma-Shield	150	103	Modified USPHS criteria	L	94.7	1.1	
1991	Barnes	5 8	I and II	Ful-Fil	33	12	Modified USPHS criteria	L	90 77	2 2.9	
1992	Freilich	3	I and II	Heliomolar, Marathon, P-30, Experimental composite	105	46	Modified USPHS criteria	L	99	0.3	
1993	Mjör	5	II	P-10	91		Modified USPHS criteria	L	85	3	
1994	el Mowafi	5	I and II	Composite resins (not specified)	191		Meta-analysis		89.5	2.1	
1994	Jokstad	> 10	I II	Composite resins (not specified)	22 79			CS			4 4-7
1995	Wassell	3	I and II	Brilliant	71	54	Modified USPHS criteria	L	96	1.3	
1997	Geurtsen	4	I II	Herculite XR	109 1100	412 (all)	Modified USPHS criteria	CS	87	3.3	9
1997	Mjör	> 25		Composite resins specified)	537		Defined criteria for clinical failure	CS			6
1998	Helbig	5	I and II	P-50	27	22	Modified USPHS criteria	L	88.9	2.2	
1998	Mair	10	II	P-30, Occlusin, Clearfil Posterior	56		Modified USPHS criteria	L	92.9	0.7	
1998	Collins	8	I and II	Amalgam (Dispersalloy) Composite: - Heliomolar radiopaque - Herculite XR - P30 APC	52 55 52 54	46	Modified USPHS criteria	L	94.2 83.6 84.6 90.7	0.7 2.1 1.9 1.2	
1998	Mertz-Fairhurst	10	I	Miradapt + Delton sealant	85		Modified USPHS criteria	L	80	2	
1998	Nordbo	7	II	Occlusin Ful-Fil	34 17	37 (all)	Modified USPHS criteria	L	88 59	1.7 5.9	
1999	Burke		I II	Composite resins (not specified)	27 71			CS			3.3 4.6
1999	Lundin	5 10	I and II	PC4575 (= Occlusin) PC4502 PC4575 (= Occlusin) PC4502	61 66 55 62	65 (all)	Modified USPHS criteria	L	88.5 90.9 67.3 88.7	2.3 1.8 3.3 1.1	
1999	Raskin	10	I and II	Occlusin	100	36	Modified USPHS criteria	L	50-60	4-5	
1999	Scheiben-bogen-Fuchsbrunner	2	I and II	Tetric, Pertac-Hybrid Unifil, blend-a-lux	43		Modified USPHS criteria	L	90	5	
1999	Wilder	17	I and II	Estilux, Nuva-Fil, Nuva-Fil PA, Uvio-Fil	85	33	Modified USPHS criteria	L	76	1.4	
2000	Köhler	5	II	Superlux Molar, P-50	51	45	Modified USPHS criteria	L	68.6	6.3	
2000	Manhart	3	I and II	Tetric, Pertac-Hybrid Unifil, blend-a-lux	30		Modified USPHS criteria	L	87	4.3	
2000	Vilkinis	2	II	Z100 Z100 + Vitremer (open sandwich rest)	67 74	118 (all)	Modified USPHS criteria	L	92.5 86.5	3.8 6.8	
2000	Wassell	5	I and II	Brilliant Dentin	62		Modified USPHS criteria	L	92.5	1.5	

Table 2: *Longevity of Direct Composite Restorations in Posterior Teeth (continued)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)	Median Survival Time (years)
2000	Raskin	10	I and II	Occlusin	60		Modified USPHS criteria	L	46.7	5.3	
2000	Van Dijken	11	II	Direct composite inlays and onlays (Brilliant DI) Direct composite	96 33	40 (all)	Modified USPHS criteria	L L	82.3 72.7	1.6 2.5	
2000	Wilson	3-4	II	Tetric	31	22	Modified USPHS	L	100	0	
2001	Baratieri	4	I II	Z100	260 466	204 (all)	Modified USPHS criteria	L	100 100	0 0	
2001	Busato	6	I and II	Z100 Tetric Charisma	29 29 32	13 (all)	Defined criteria for clinical failure	L	100 79.3 78.1	0 3.5 3.7	
2001	Ernst	1 2 3	I and II	Solitaire I	165	120 (all)	Modified USPHS criteria	L	93.6 84 79	6.4 8 7	
2001	Gaengler	10	I and II	Visio-Molar radiopaque	62		CPM Index	L	74.2	2.6	
2001	Hugo	1-3	II	Tetric Ceram, Tetric Flow + Tetric Ceram	109	50	Modified USPHS criteria	L	96.3	1.2-3.7	
2001	Türkün	2	I and II	Prisma TPH Z100 Clearfil Ray-Posterior	36 35 36	38	Modified USPHS criteria	L	100 100 100	0 0 0	
2002	Gordan	2	I and II	Beautifil	60	30	Modified USPHS criteria	L	96.7	1.7	
2002	Manhart	2	I and II	Definite (ormocer) Pertac 2	67 44		Modified USPHS criteria	L	74.6 95.4	12.7 2.3	
2002	Van Dijken	3	I and II	Ariston pHc (ion-releasing composite)	65	34	Modified USPHS criteria	L	74	8.7	
2002	Wucher	3	II	Spectrum TPH (composite) Dyract (compomer)	20 20	20 20	Modified USPHS criteria	L	100 100	0 0	
2003	Ernst	2	II	Prodigy condensable Prodigy condensable + Revolution (flowable composite as base)	116 (all)	50	Modified USPHS criteria	L	94.6 92.8	2.7 3.6	
2003	Lindberg	3	II	Compomer/Composite sandwich restorations Dyract/Prisma TPH) Direct composite restorations (Prisma TPH)	73 73	56	Modified USPHS criteria	L	97.3 95.9	0.9 1.4	
2003	Lopes	2	I and II	Prodigy condensable Definite (ormocer)	36 38	34 (all)	Modified USPHS criteria	L	100 94.7	0 2.7	
2003	Pallesen	11	II	Brilliant Dentin Estilux	27 27	27 (all)	Modified USPHS criteria	L	89 81	1 1.7	
2003	Türkün	2	I and II	SureFil	50 (all)	36	Modified USPHS criteria	L	96	2	
2003	Türkün	7	I and II	Z100 Clearfil Ray posterior Prisma TPH	23 26 21	38 (all)	Modified USPHS criteria	L	95.7 96.2 90.5	0.6 0.5 1.4	
2003	Van Dijken	6	I	Compomer/Composite laminate restorations (Dyract/Prisma TPH) Direct composite restorations (Prisma TPH)	41 41	29	Modified USPHS criteria	L	97.6 97.6	0.4 0.4	
2003	Van Nieuwen-huysen		I and II	Composite Amalgam	115 722		Defined criteria for clinical failure	L			7.8 12.8

Table 3: *Longevity of Direct Composite Restorations with Inserts in Posterior Teeth (SD = study design; CS = cross-sectional; L = longitudinal)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)
1996	Lösche	2	II	Herculite and P-50 + glass ceramic inserts	24		Modified USPHS criteria and SEM	L	100	0
1998	Kiremitci	2	I and II	Charisma + Beta-quartz inserts	22		Modified USPHS criteria and SEM	L	95.5	2.3
2000	Sjögren	3	I II	Heliomolar + Beta-quartz inserts	9 30	16 (all)	CDA criteria	L	69	10.3
2001	Hugo	1.5	II	Tetric Flow + Tetric Ceram + Sonicsys approx inserts	213	79	Modified USPHS criteria	CS	99.5	0.3
2002	Ödman	3	I and II	Cerana inserts	59		CDA criteria	L	84.3	5.2

Table 4: *Longevity of Compomer Restorations in Posterior Teeth (SD = study design; CS = cross-sectional; L = longitudinal)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)
2002	Demirci	1 2	I	Dyract	87	36	Modified USPHS criteria	L	100 98.9	0 0.6
2002	Jedynakiewicz	3	I and II	Dyract AP	21		Modified USPHS criteria	L	100	0
2002	Luo	2	I II	Dyract AP	33 43	33	Modified USPHS criteria	L	93.4	3.3
2002	Manhart	2	I	Hytac	34	22	Modified USPHS criteria	L	97.1	1.5
2002	Manhart	3	I	Hytac	40	25	Modified USPHS criteria	L	97.5	0.8
2002	Wucher	3	II	Dyract (compomer) Spectrum TPH (composite)	20 20	20 20	Modified USPHS criteria	L	100 100	0 0
2003	Huth	3	I and II	Hytac	39	14	Modified USPHS criteria	L	92.3	2.6

Table 5: *Longevity of Glass Ionomer Restorations in Posterior Teeth (SD = study design; CS = cross-sectional; L = longitudinal)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)	Median Survival Time (years)
1988	Hickel	3.5	I II II (modif.)	Ketac-Silver (cermet)	87 104 52		Defined criteria for clinical failure	L	88.5 50 80.8	3.3 14.3 5.5	
1990	Smales	3	I	Ketac-Silver (cermet)	132		Defined criteria for clinical failure	L	56.8	14.4	
1991	Smales	9	All classes	Aaps, Fuji II, Ketac	465		Defined criteria for clinical failure	CS			2.2
1993	Mjör	5	II	Ketac-Silver (cermet)	95		Modified USPHS criteria	L	55	9	
1994	Krämer	4	I II	Ketac-Silver (cermet)	49 39	50 (all)	Modified USPHS criteria	L	89.8 71.8	2.6 7.1	
1997	Mjör	> 25	All classes	Glass ionomers (not specified)	155		Defined criteria for clinical failure	CS			3
TUNNEL RESTORATIONS											
1992	Svanberg	3	II	Ketac-Silver	18	18	Defined criteria for clinical failure	L	94.4	1.9	
1993	Hasselrot	3.5	I and II	Base Line and Ketac-Silver	282		Defined criteria for clinical failure	L	73.5	7.6	
1996	Strand	3	I and II	Ketac-Silver (cermet)	161	85	Defined criteria for clinical failure	L	70	10	
1998	Hasselrot	7	I II	Base Line Ketac-Silver (cermet)	232 35	193 (all)	Defined criteria for clinical failure	L		7 7	6 6
2000	Nicolaisen	3-6	I and II	Glass ionomers (not specified)	182	94	Defined criteria for clinical failure	CS	65		
2000	Strand	2-4.5	I and II	Ketac Silcer (cermet)	302	179	Modified USPHS criteria	L	57		
2001	Pilebro	3	I and II	Ketac Silcer (cermet) Ketac Silver + FulFil (composite)	305	272	Defined criteria for clinical failure	L	72.7	9.1	
ART RESTORATIONS											
1996	Phantum-vanit	1 2 3	I	Glass ionomer cement	241	144	Defined criteria for clinical failure	L	93 83 71	7 8.5 9.7	
1998	Frencken	1 2 3	I	Fuji IX	297	142	Defined criteria for clinical failure	L	98.6 93.8 88.3	1.4 3.1 3.9	
1998	Mallow	1 3	I	Fuji II	89	53	Defined criteria for clinical failure	L	76.3 57.9	 14	
1999	Ho	2	I	Fuji IX ChemFil Superior	55 45	21	Defined criteria for clinical failure	L	93	3.5	
2002	Ziraps	2	I	Chem-Flex (GIC)	40		Defined criteria for clinical failure	L	92.5	3.8	
2003	Gao	2.5	I	ART hand instrument preparation of cavity • Fuji IX GP • Ketac Molar Conventional rotary cavity preparation • Fuji IX GP • Ketac Molar • Non-Gamma-2-Amalgam (GK Amalgam)	 8 9 6 4 8	17	Modified USPHS criteria	L	 87.5 100 100 100 100	 5 0 0 0 0	

Table 6: *Longevity of Composite Inlays and Onlays (SD = study design; CS = cross-sectional, L = longitudinal)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)
1992	Haas	2	Inlays	Coltene-composite Kulzer-composite	30 30		Defined criteria for clinical failure	L	80 80	10 10
1992	Wendt	3	I and II	Occlusin	60		Modified USPHS criteria		96.7	1.1
1994	Frederickson	3	Inlays and Onlays	Concept	31	23	Modified USPHS criteria		100	0
1994	van Dijken	5	II	Brilliant	100		Modified USPHS criteria	L	88	2.4
1995	Wassell	3	I and II	Brilliant	71	54	Modified USPHS criteria	L	92	2.7
1996	Haas	10	Inlays and Partial crowns	Gold Composite Ceramic (Dicor)	344 80 340		Modified USPHS criteria	L	91 78 81	0.9 2.2 1.9
1996	Hannig	5	I II	Isosit	20 20		Defined criteria for clinical failure and SEM	L	92.5	1.5
1996	Krämer	6	I and II	Visio-Gem	118	28	Modified USPHS criteria	L	41	9.8
1997	Wiedmer	5	I and II	Brilliant, APH	24		Modified USPHS criteria and SEM	L	100	0
1999	Donly	7	Inlays Onlays	Concept	32 4	18 (all)	Modified USPHS criteria	L	75 75	3.6 3.6
1999	Klimm	2	II	Herculite XRV	13		Modified USPHS criteria	L	100	0
1999	Scheiben-bogen Fuchsbrunner	2	I and II	Composite inlays (Tetric, Pertac-Hybrid Unifil, blend-a-lux) Direct composite restrorations	45 43		Modified USPHS criteria	L	93 90	3.5 5
2000	Van Dijken	11	II	Direct composite inlays and onlays (Brilliant DI) Direct composite restorations (FulFil)	96 33	40 (all)	Modified USPHS criteria	L	82.3 72.7	1.6 2.5
2000	Wassell	5	I and II	Brilliant Dentin (direct composite inlay)	57		Modified USPHS criteria	L	82.6	3.5
2001	Thordrup	5	I and II	Brilliant Direct Inlay (direct composite inlay) Estilux (indirect composite inlay)	10 9		CDA criteria	L	82.1 91.7	3.6 1.7
2002	Manhart	4	I and II	Tetric, Pertac-Hybrid Unifil, blend-a-lux Ceramic (Empress)	36 21		Modified USPHS criteria	L	86 100	3.5 0
2002	Manhart	3	I and II	Artglass Charisma	49 63		Modified USPHS criteria	L	89.8 84.1	3.4 5.3
2002	Manhart	2	I and II	Definite (ormocer)	55		Modified USPHS criteria	L	89.1	5.5
2003	Leirskar	5	Inlays and Onlays	Tetric Z100 Maxxim	35 16 13	25 (all)	Modified USPHS criteria	L	95.3	0.9
2003	Pallesen	11	II	Composite inlays: - Brilliant Dentin - Estilux - Isosit Direct composite restorations: - Brilliant Dentin - Estilux	 27 27 27 27 27	27 (all)	Modified USPHS criteria	L	 85 89 78 89 81	 1.4 1 2 1 1.7

Table 7: *Longevity of Laboratory Fabricated Ceramic Inlays and Onlays (SD = study design; CS = cross-sectional; L = longitudinal)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)
1988	Jensen	2	I and II	Mirage	310	59	Modified USPHS criteria		95.8	2.1
1992	Haas	3	Inlays	Dicor Optec Hi-Ceram Du-Ceram	30 30 30 30		Defined criteria for clinical failure	L	93.3 80 80 90	2.2 6.7 6.7 3.3
1992	Höglund	2	II	Mirage (resin cement) Mirage (GI cement)	59 59	50	Modified USPHS criteria		98 85	1 7.5
1992	Krejci	1.5	II	Empress	10	10	Modified USPHS criteria and SEM	L	100	0
1993	Stenberg	2	II	Dicor (GI cement)	25	20	Modified USPHS criteria		92	4
1994	Höglund	3	II	Mirage (resin cement) Mirage (GI cement)	59 59	50	Modified USPHS criteria		96.6 84.7	1.3 5.1
1995	Isidor	2-4.5	II	Mirage	25		Defined criteria for clinical failure	L	52	
1995	Tidehag	2	II	Empress	62	18	CDA criteria	L	98.4	0.8
1996	Friedl	2	II	Mirage II	50	20	Modified USPHS criteria and SEM	L	100	0
1996	Molin	3	I II	Optec	12 133 (81 m, 64 pm)	47	CDA criteria	L	85.5	4.8
1996	Qualtrough	3	I and II	Mirage	50	27	Modified USPHS criteria and SEM	L	82	6
1996	Studer	2	I and II	Empress	130	36	Modified USPHS criteria	L	97.5	1.3
1996	Haas	10	Inlays and Partial crowns	Gold Composite Ceramic (Dicor)	344 80 340		Modified USPHS criteria	L	91 78 81	0.9 2.2 1.9
1997	Fradeani	4.5	Inlays and Onlays	Empress	125	29	Modified USPHS criteria	L	95.6	1
1997	Friedl	4	II	Mirage II	50	20	Modified USPHS criteria and SEM	L	100	0
1997	Roulet	6	I and II	Dicor	123	29	Modified USPHS criteria	CS	76	4
1997	Thonemann	2	I II	Empress	14 37	11	Modified USPHS criteria and SEM		100	0
1998	Felden	7	Inlays and Onlays	Dicor, Empress, Mirage II, Cerec Vita Mark I, Duceram LFC	287	106	Modified USPHS criteria	CS	94.2	0.8
1998	Fuzzi	10	I and II	Microbond Natural Ceramic and FortuneCeramic	183	67	Defined criteria for clinical failure	L	97	0.3
1998	Hayashi	6	I and II	G-Cera Cosmotech II	49	29	Modified USPHS criteria and SEM	L	92	1.3
1998	Lehner	6	Inlays Onlays	Empress	138 17	43	Modified USPHS criteria	L	94.9	0.9
1998	van Dijken	6	II	Mirage (resin cement) Mirage (GI cement)	58 57	50 (all)	Modified USPHS criteria	L	88 74	2 4.3
1999	Malament	11.3	Inlays Onlays	Dicor	74 25		Modified USPHS criteria	L	90 96	0.9 0.4
1999	Krämer	4	Inlays and Onlays	Empress	96	34	Modified USPHS criteria and SEM	L	93	1.8
1999	van Dijken	2	II	Empress luted with RMGI Empress luted with Panavia 21	73	27 (all)	Modified USPHS criteria	L	100 100	0 0
1999	Fuzzi	11.5	I and II	Microbond Natural Ceramic and Fortune Ceramic	182	66	Defined criteria for clinical failure	L	95	0.4
2000	Felden	7	Partial crowns	Empress I	42	22	Modified USPHS criteria	CS	81	2.7

Table 7: *Longevity of Laboratory fabricated Ceramic Inlays and Onlays (continued)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)
2000	Frankenberger	6	Inlays Onlays	Empress	67 (all)	34 (all)	Modified USPHS criteria	L	93	1.2
2000	Hayashi	8	I II	G-Cera G-Cerca Cosmotech II	12 33	25	Modified USPHS criteria and SEM	L	80	2.5
2000	Molin	5	II	Empress Mirage (fired ceramic)	20 20	20 20	CDA criteria	L	80 95	4 1
2001	Thordrup	5	I and II	Vita Dur N (fired ceramic)	11		CDA criteria	L	85.1	3
2001	van Dijken	5	Partial crowns	Empress (dual-cured resin cement) Empress (chemically cured resin cement)	58 124	110 (all)	Modified USPHS criteria	L	94.8 91.9	1 1.6
2002	Barghi	4	Onlays and Overlays	High leucite-content ceramic	21	12	Modified USPHS criteria	L	100	0
2002	Manhart	4	I and II	Empress	21		Modified USPHS criteria	L	100	0
2003	Schulz	6.3	II	Mirage	107	51	Modified USPHS criteria	CS	84.1	2.5
2003	van Dijken	5	II	Empress (chemically-cured RMGI, Fuji Plus) Empress (chemically cured resin cement, Panavia 21)	71 (all)	26 (all)	Modified USPHS criteria	L	93	1.4

Table 8: *Longevity of Computer Generated Ceramic Inlays and Onlays (CAD/CAM) (SD = study design; CS = cross-sectional; L = longitudinal)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)
1991	Reiss	3	I and II	Cerec	426	142	Modified USPHS criteria	L	95	1.7
1992	Haas	3	Inlays	Cerec	30		Defined criteria for clinical failure	L	86.7	4.4
1992	Mörmann	5	II	Cerec; Vita Cerec Mark I	8	8	Modified USPHS criteria and SEM		100	0
1992	Sjögren	1-2	I and II	Cerec; Vita Cerec Mark I and II	205	72	CDA criteria		98	
1994	Walther	5	I and II	Cerec	1011	299	Defined criteria for clinical failure	L	95	1
1995	Gladys	3	II	Cerec 1; Dicor MGC Cerec 1; Vita Mark I P50 (composite inlay)	32 (all)	20 (all)	Defined criteria for clinical failure	L	100	0
1995	Otto	5	II	Cerec; Vita-Blocks	100	62	Defined criteria for clinical failure	L	98	0.4
1996	Heymann	4	I II	Cerec; Dicor MGC	19 31	28 (all)	Modified USPHS criteria and SEM	L	100	0
1997	Berg	5	II	Cerec 1	51	46	Defined criteria for clinical failure and SEM	L	94.1	1.2
1998	Reiss	7.5	I and II	Cerec	1011	299	Defined criteria for clinical failure	L	91.6	1.1
1998	Sjögren	5	II	Cerec; dual-cured resin cement Cerec; chemically-cured resin cement	33 33	27 (all)	CDA criteria	L	84.8 93.9	3 1.2
1998	Zuellig-Singer	3	II	Cerec; Vita Mark II (RC) Cerec; Vita Mark II (GIC)	28 9	18 (all)	SEM	L	96.4 100	1.2 0
1999	Thordrup	3	II	Cerec; Cerec Vita Blocs Celay; Vita Celay Blanks	15 15	17	CDA criteria	L	86.7 86.7	4.4 4.4
2000	Molin	5	II	Cerec	20	20	CDA criteria	L	90	2
2000	Pallesen	8	II	Cerec; Vita Mark II Cerec; Dicor MGC	16 16	16 (all)	Modified USPHS criteria	L	87.5 93.8	1.6 0.8
2000	Reiss	10 12	I and II	Cerec	1010	299	Defined criteria for clinical failure	L	90 84.9	1 1.3
2001	Thordrup	5	I and II	Cerec	14		CDA criteria	L	92.9	1.4
2002	Otto	10	I and II	Cerec 1; Vita Mark I	187	108	Modified USPHS criteria	L	90.4	0.9
2003	Bindl	4.8 3.2 1.4	II	Cerec 1 Cerec 2 + standard wall software Cerec 2 + wall-spacing software	18 16 17	18 16 17	Modified USPHS criteria	CS	94.4 100 100	5.6 0 0
2003	Posselt	9	I and II	Cerec 1 and 2	2328	794	Modified USPHS criteria	CS	95.5	0.5

Table 9: *Longevity of Cast Gold Inlays and Onlays (SD = study design; CS = cross-sectional; L = longitudinal)*

Year of Publication	First Author	Observation Period (years)	Black Class	Restorative Materials	# of Restorations (n)	# of Patients (n)	Method(s)	SD	Survival Rate (%)	Annual Failure Rate (%)	Median Survival Time (years)
1981	Crabb	10	II	Gold	146		Defined criteria for clinical failure	CS	41.1	5.9	7
1985	Leempoel	5 11 5 11	Partial crown Partial crown Crown Crown	Gold	895 785 				96 91 99 97	0.8 0.8 0.2 0.3	
1986	Bentley	10	I and II Crown	Gold	173 295			CS	95 89	0.5 1.1	
1992	Fritz	10	I II (2-surfaces) II (3-surfaces) Partial crown	Gold	2717 (all)	548 (all)		CS	65 60 68 70	3.5 4 3.2 3	
1992	Haas	5	Inlays	Gold (RC) Gold (CC)	30 30		Defined criteria for clinical failure	L	100 100	0 0	
1993	Mjör		Cast gold restorations	Gold	1689			CS			15-16
1993	Schlösser	9	Partial crown Crown	Gold	725 390			CS	87.3 92.1	1.3 0.9	
1994	Jokstad	> 10	II	Gold	> 250			CS			15-17
1994	Mahmood	> 12		Gold	120 (P) 80 (A)		Defined criteria for clinical failure	CS			5 10.6
1996	Pelka	> 10	Inlays Onlays	Gold	520	56	Modified USPHS criteria	CS	94.6		
1996	Haas	10	Inlays and Partial crowns	Gold Composite Ceramic (Dicor)	344 80 340		Modified USPHS criteria	L	91 78 81	0.9 2.2 1.9	
1997	Hawthorne		I and II	Gold	49		Defined criteria for clinical failure	CS			13.8
1996	Smales	15	I and II	Gold	96			CS	78	1.5	
1999	Donly	7	Inlays Onlays	Gold	11 7	18 (all)	Modified USPHS criteria	L	81.8 85.7	2.6 2	
1999	Stoll	10	I II (MO) II (OD) II (MOD) Partial crown	Gold	171 294 427 862 1679	890 (all)	Defined criteria for clinical failure	CS	76.1 88.3 83.4 87.5 86.1	2.4 1.2 1.7 1.3 1.4	
2000	Molin	5	II	Gold	20	20	CDA criteria	L	95	1	
2000	Studer	10 15 20 30	Inlays and onlays	Gold	303	50	Modified USPHS criteria	CS	96.1 92.2 87 73.5	0.4 0.5 0.7 0.9	
2001	Erpenstein	25	I II (2 surfaces) II (3 surfaces) II (>3 surfaces)	Gold	139 846 434 619	531 (all)	Defined criteria for clinical failure	CS	52 64.3 75.8 84.8	1.9 1.4 1 0.6	
2003	Wagner	7	Partial crowns	Gold Ceramic	42 42	42 22	Modified USPHS criteria	CS	96 81	0.6 2.7	

Table 10: *Range, mean (SD) and median values of annual failure rates (%) of clinical investigations analyzing the longevity of dental restorations in stress-bearing cavities of permanent teeth, subdivided for longitudinal and cross-sectional studies, Black Class I and II cavities, publication date, and survival curve analysis, where appropriate. Although it is problematic to directly compare different studies of different authors, and keeping all the limitations in mind, a trend can be observed. In the section "all studies," statistical analysis with ANOVA and LSD-test was performed (p<0.05). Missing values: not enough studies available for comparison.*

	Annual Failure Rate (%)											
Restoration Type	**All Studies**				**Longitudinal Studies**		**Cross-sectional Studies**		**Black Class I**		**Black Class II**	
	Range	**Mean (SD)**	**LSD-test**	**Median**	**Mean (SD)**	**Median**	**Mean (SD)**	**Median**	**Mean (SD)**	**Median**	**Mean (SD)**	**Median**
Amalgam	0-7.4	3.0 (1.9)	C	2.6	2.3 (2.4)	1.5	3.7 (1.5)	3.7	2.1 (1.6)	2.2	3.3 (2.0)	2.9
Direct composite	0-9.0	2.2 (2.0)	ABC	1.8					1.8 (1.4)	2.0	2.3 (2.1)	1.8
Composite inlays/onlays	0-10.0	2.9 (2.6)	BC	2.3								
Ceramic inlays/onlays	0-7.5	1.9 (1.8)	AB	1.3	1.9 (1.9)	1.3	2.5 (1.3)	2.6				
CAD/CAM inlays/onlays	0-5.6	1.7 (1.6)	A	1.2								
Gold inlays/onlays	0-5.9	1.4 (1.4)	A	1.0	0.9 (1.0)	0.9	1.6 (1.4)	1.3	2.2 (1.1)	1.9	1.4 (1.6)	0.9
	Longitudinal Studies, Class II, 1990 - 2003				**Publication Date < 1990**		**Publication Date ≥ 1990**		**Survival Curve Analysis: No**		**Survival Curve Analysis: Yes**	
	Range	**Mean (SD)**	**LSD-test**	**Median**	**Mean SD**	**Median**	**Mean (SD)**	**Median**	**Mean (SD)**	**Median**	**Mean (SD)**	**Median**
Amalgam	0-7.4	2.6 (2.0)	B	2.0	4.8 (1.4)	4.7	2.4 (1.7)	2.0				
Direct composite	0-7.0	2.2 (1.9)	AB	1.7	4.2 (2.8)	4.0	2.0 (1.8)	1.7				
Composite inlays/onlays	0-10.0	2.9 (2.6)	B	2.3								
Ceramic inlays/onlays	0-7.5	1.9 (1.9)	AB	1.3					2.1 (2.0)	1.3	1.5 (1.1)	1.0
CAD/CAM inlays/onlays	0-5.6	1.7 (1.4)	AB	1.2					1.9 (1.8)	1.3	1.0 (0.4)	1.0
Gold inlays/onlays	0-4.0	0.9 (1.1)	A	0.9	2.4 (3.0)	0.8	1.3 (1.1)	1.0	2.1 (1.9)	1.5	1.1 (0.8)	0.9

Table 11: *Range, Mean (SD) and Median Values of Annual Failure rates (%) of Insert Restorations, Compomers and Glass Ionomers. For Inserts, Compomers and ART Restorations, Only Results Up to Three Years Observation Time Are Published*

Restoration Type	All Studies		
	Range	Mean (SD)	Median
Direct composite + insert	0-10.3	3.6 (4.2)	2.3
Compomer	0-3.3	1.1 (1.2)	0.7
Glass ionomer (regular)	0-14.3	7.2 (5.6)	7.1
Glass ionomer (tunnel)	1.9-10.0	7.1 (2.8)	7.3
Glass ionomer (ART)	0-14.0	6.0 (4.6)	3.6

Table 12: *Factors Influencing the Longevity of Dental Restorations*

Patient	Dentist	Material
Oral Hygiene, Dietary Habits	Correct Indication	Strength (fractures)
Preventive measures, fluoride availability	Cavity preparation (size, type, finishing)	Fatigue/degradation
Compliance in recall	Handling and application (for example, incremental vs bulk placement)	Wear resistance (occlusal contact areas, contact-free areas)
Oral environment (Quality of tooth structure, saliva, etc) and systemic diseases	Curing mode (device, time, light intensity)	Bond strength, polymerization shrinkage, post-operative sensitivity
Size, shape, location of the lesion and tooth (number of surfaces, vital vs non-vital tooth, premolar vs molar)	Mode of finishing and polishing of the restoration	Chemical compatibility of restorative systems (DBA, composite)
Cooperation during treatment	Correct occlusion	Technique sensitivity
Bruxism/parafunctions/habits	Experience (with material and restorative technique)	Caries inhibiting effects (release of substances?)

References

Allan DN (1969) The durability of conservative restorations *British Dental Journal* **126** 172-177.

Allan DN (1977) A longitudinal study of dental restorations *British Dental Journal* **143** 87-89.

Baratieri LN & Ritter AV (2001) Four-year clinical evaluation of posterior resin-based composite restorations placed using the total-etch technique *Journal of Esthetic Dentistry* **13** 50-57.

Barghi N & Berry TG (2002) Clinical evaluation of etched porcelain onlays: A 4-year report *Compendium of Continuing Education in Dentistry* **23** 657-60, 662, 664.

Barnes DM, Blank LW, Thompson VP, Holston AM & Gingell JC (1991) A 5- and 8-year clinical evaluation of a posterior composite resin *Quintessence International* **22** 143-151.

Bentley C & Drake CW (1986) Longevity of restorations in a dental school clinic *Journal of Dental Education* **50** 594-600.

Berg NG & Derand T (1997) A 5-year evaluation of ceramic inlays (Cerec) *Swedish Dentistry Journal* **21** 121-127.

Bessing C & Lundqvist P (1991) A 1-year clinical examination of indirect composite resin inlays: A preliminary report *Quintessence International* **22** 153-157.

Bindl A & Mörmann WH (2003) Clinical and SEM evaluation of all-ceramic chair-side CAD/ CAM-generated partial crowns *European Journal of Oral Sciences* **111** 163-169.

Bjertness E & Sonju T (1990) Survival analysis of amalgam restorations in long-term recall patients *Acta Odontologica Scandinavica* **48** 93-97.

Bogacki RE, Hunt RJ, del Aguila M & Smith WR (2002) Survival analysis of posterior restorations using an insurance claims database *Operative Dentistry* **27** 488-492.

Buchalla W, Attin T & Hellwig E (1996) Influence of dentin conditioning on bond strength of light-cured ionomer restorative materials and polyacid-modified composite resins *Journal of Clinical Dentistry* **7** 81-84.

Burke FJT, Cheung SW, Mjör IA & Wilson NHF (1999) Restoration longevity and analysis of reasons for the placement and replacement of restorations provided by vocational dental practitioners and their trainers in the United Kingdom *Quintessence International* **30** 234-242.

Burke FJT & Qualtrough AJE (1994) Aesthetic inlays: Composite or ceramic *British Dental Journal* **176** 53-60.

Busato ALS, Loguercio AD, Reis A & De Oliveira Carrilho MR (2001) Clinical evaluation of posterior composite restorations: 6-year results *American Journal of Dentistry* **14** 304-308.

Chen HY, Hickel R, Setcos JC & Kunzelmann KH (1999) Effects of surface finish and fatigue testing on the fracture strength of CAD/CAM and pressed ceramic crowns *Journal of Prosthetic Dentistry* **82** 468-475.

Cichon P & Kerschbaum T (1999) [Verweildauer zahnärztlicher Restaurationen bei Behinderten] *Deutsche Zahnärztliche Zeitschrift* **54** 96-102.

Collins CJ, Bryant RW & Hodge KLV (1998) A clinical evaluation of posterior composite resin restorations. 8-year findings *Journal of Dentistry* **26** 311-317.

Cortes O, García-Godoy F & Boj JR (1993) Bond strength of resin-reinforced glass ionomer cements after enamel etching *American Journal of Dentistry* **6** 299-301.

Crabb HSM (1981) The survival of dental restorations in a teaching hospital *British Dental Journal* **150** 315-318.

Dahl BL & Oilo G (1994) *In vivo* wear ranking of some restorative materials *Quintessence International* **25** 561-565.

Demirci M & Ücok M (2002) Two-year clinical evaluation of Dyract in small Class I cavities *American Journal of Dentistry* **15** 312-316.

Donly KJ, Jensen ME, Triolo P & Chan D (1999) A clinical comparison of resin composite inlay and onlay posterior restorations and cast-gold restorations at 7 years *Quintessence International* **30** 163-168.

el-Mowafy OM, Lewis DW, Benmergui C & Levinton C (1994) Meta-analysis on long-term clinical performance of posterior composite restorations *Journal of Dentistry* **22** 33-43.

Ernst CP, Canbek K, Aksogan K & Willershausen B (2003) Two-year clinical performance of a packable posterior composite with and without a flowable composite liner *Clinical Oral Investigations* **7** 129-134.

Ernst CP, Martin M, Stuff S & Willershausen B (2001) Clinical performance of a packable resin composite for posterior teeth after 3 years *Clinical Oral Investigations* **5** 148-155.

Erpenstein H, Kerschbaum T & Halfin T (2001) Long-term survival of cast-gold inlays in a specialized dental practice *Clinical Oral Investigations* **5** 162-166.

Felden A, Schmalz G, Federlin M & Hiller KA (1998) Retrospective clinical investigation and survival analysis on ceramic inlays and partial ceramic crowns: Results up to 7 years *Clinical Oral Investigations* **2** 161-167.

Felden A, Schmalz G & Hiller K-A (2000) Retrospective clinical study and survival analysis on partial ceramic crowns: Results up to 7 years *Clinical Oral Investigations* **4** 199-205.

Flessa HP, Manhart J, Ilie N, Chen HY, Kunzelmann KH & Hickel R (2001) Mechanical properties of microfilled composite restorative materials *Proceedings of the Continental European Division of the IADR* 05.-08.09.2001 Rome, Italy.

Fradeani M, Aquilano A & Bassein L (1997) Longitudinal study of pressed glass-ceramic inlays for four and a half years *Journal of Prosthetic Dentistry* **78** 346-353.

Frankenberger R, Petschelt A & Krämer N (2000) Leucite-reinforced glass ceramic inlays and onlays after six years: Clinical behavior *Operative Dentistry* **25** 459-465.

Frederickson D & Setcos J (1994) Clinical evaluation of indirect posterior composite restorations over three years *Journal of Dental Research* **73** 381.

Freilich MA, Goldberg AJ, Gilpatrick RO & Simonsen RJ (1992) Direct and indirect evaluation of posterior composite restorations at three years *Dental Materials* **8** 60-64.

Frencken JE, Makoni F & Sithole WD (1996) Atraumatic restorative treatment and glass ionomer sealants in a school oral health programme in Zimbabwe: Evaluation after 1 year *Caries Research* **30** 428-433.

Frencken JE, Makoni F & Sithole WD (1998) ART restorations and glass ionomer sealants in Zimbabwe: Survival after 3 years *Community Dental Oral Epidemiology* **26** 372-381.

Frencken JE, Songpaisan Y, Phantumvanit P & Pilot T (1994) An atraumatic restorative treatment (ART) technique: Evaluation after one year *International Dental Journal* **44** 460-464.

Friedl KH, Hiller KA, Schmalz G & Bey B (1997) Clinical and quantitative marginal analysis of feldspathic ceramic inlays at 4 years *Clinical Oral Investigations* **1** 163-168.

Friedl KH, Schmalz G, Hiller KA & Saller A (1996) *In-vivo* evaluation of a feldspathic ceramic system: 2-year results *Journal of Dentistry* **24** 25-31.

Fritz U, Fischbach H & Harke I (1992) [Langzeitverweildauer von Goldgußfüllungen] *Deutsche Zahnärztliche Zeitschrift* **47** 714-716.

Fuzzi M & Rapelli G (1999) Ceramic inlays: Clinical assessment and survival rate *Journal of Adhesive Dentistry* **1** 71-79.

Fuzzi M & Rappelli G (1998) Survival rate of ceramic inlays *Journal of Dentistry* **26** 623-626.

Gaengler P, Hoyer I & Montag R (2001) Clinical evaluation of posterior composite restorations: The 10-year report *Journal of Adhesive Dentistry* **3** 185-194.

Gao W, Peng D, Smales RJ & Yip KH (2003) Comparison of atraumatic restorative treatment and conventional restorative procedures in a hospital clinic: Evaluation after 30 months *Quintessence International* **34** 31-37.

Gernhardt CR, Salhab J & Schaller HG (2001) [Die Zugfestigkeit verschiedener Dentinhaftvermittler auf trockenem und perfundiertem Dentin] *Deutsche Zahnärztliche Zeitschrift* **56** 467-471.

Geurtsen W & Schoeler U (1997) A 4-year retrospective clinical study of Class I and Class II composite restorations *Journal of Dentistry* **25** 229-232.

Gladys S, Van Meerbeek B, Inokoshi S, Willems G, Braem M, Lambrechts P & Vanherle G (1995) Clinical and semiquantitative marginal analysis of four tooth-coloured inlay systems at 3 years *Journal of Dentistry* **23** 329-338.

Gordan VV, Mjör IA, Vazquez O, Watson RE & Wilson N (2002) Self-etching primer and resin-based restorative material: Two-year clinical evaluation *Journal of Esthethetic Restorative Dentistry* **14** 296-302.

Haas M, Arnetzl G, Pertl C, Polansky R & Smetan M (1996) Cerec vs laboratory inlays in Mörmann WH (ed) *CAD/CIM in Aesthetic Dentistry. Cerec 10-year Anniversary Symposium* Quintessence Publishing Chicago 1996 299-312.

Haas M, Arnetzl G, Wegscheider WA, Konig K & Bratschko RO (1992) [Klinische und werkstoffkundliche Erfahrungen mit Komposit-, Keramik- und Goldinlays] *Deutsche Zahnärztliche Zeitschrift* **47** 18-22.

Hamm G, Manhart J & Hickel R (2003) A simple 2 parameter fit to Kaplan-Meier-survival-curves characterizing the longevity of dental restorations *Proceedings Triannual Meeting of the European Federation of Conservative Dentistry (EFCD)* ConsEuro 2003 Munich Germany 05.-07.06.2003.

Hannig M (1996) Five-year clinical evaluation of a heat- and pressure-cured composite resin inlay system *Journal of Dental Research* **75** 260.

Hasselrot L (1993) Tunnel restorations. A 3 1/2-year follow up study of Class I and II tunnel restorations in permanent and primary teeth *Swedish Dental Journal* **17** 173-182.

Hasselrot L (1998) Tunnel restorations in permanent teeth. A 7-year follow up study *Swedish Dental Journal* **22** 1-7.

Hawthorne WS & Smales RJ (1997) Factors influencing long-term restoration survival in three private dental practices in Adelaide *Australian Dental Journal* **42** 59-63.

Hayashi M, Tsuchitani Y, Kawamura Y, Miura M, Takeshige F & Ebisu S (2000) Eight-year clinical evaluation of fired ceramic inlays *Operative Dentistry* **25** 473-481.

Hayashi M, Tsuchitani Y, Miura M, Takeshige F & Ebisu S (1998) 6-year clinical evaluation of fired ceramic inlays *Operative Dentistry* **23** 318-326.

Helbig EB, Klimm W, Haufe E & Richter G (1998) [Klinische Fünfjahresstudie zum Feinpartikelhybrid P-50 in Kombination mit Scotchbond 2] (Acta Medicinae Dentinum Helvetica) *Schweizer Monatsschrift für Zahnmedizin* **3** 171-177.

Heymann HO, Bayne SC, Sturdevant JR, Wilder-AD J & Roberson TM (1996) The clinical performance of CAD-CAM-generated ceramic inlays: A four-year study *Journal of the American Dental Association* **127** 1171-1181.

Hickel R (1990) [Zur Problematik hypersensibler Zähne nach Eingliederung von Adhäsivinlays] *Deutsche Zahnärztliche Zeitschrift* **45** 740-742.

Hickel R (1996) Glass ionomers, cermets, hybrid ionomers and compomers - (long-term) clinical evaluation *Trans Academy Dental Materials* **9** 105-129.

Hickel R (1997) [Moderne Füllungswerkstoffe] *Deutsche Zahnärztliche Zeitschrift* **52** 572-585.

Hickel R, Dasch W, Janda R, Tyas M & Anusavice K (1998) New direct restorative materials *International Dental Journal* **48** 3-16.

Hickel R & Kunzelmann KH (1997) [Keramikinlays und Veneers] Hanser Publishing, Munich.

Hickel R, Petschelt A, Maier J, Voß A & Sauter M (1988) [Nachuntersuchung von Füllungen mit Cermet-Zement (Ketac-Silver)] *Deutsche Zahnärztliche Zeitschrift* **43** 851-853.

Ho TFT, Smales RJ & Fang DTS (1999) A 2-year clinical study of two glass ionomer cements used in the atraumatic restorative treatment (ART) technique *Community Dentistry and Oral Epidemiology* **27** 195-201.

Höglund CH, van Dijken JW & Olofsson AL (1992) A clinical evaluation of adhesively luted ceramic inlays. A two-year follow-up study *Swedish Dental Journal* **16** 169-171.

Höglund CH, van Dijken JW & Olofsson AL (1994) Three-year comparison of fired ceramic inlays cemented with composite resin or glass ionomer cement *Acta Odontologica Scandinavica* **52** 140-149.

Höhnk HD & Hannig M (1998) Dentalmaterialien mit Potential: Compomere & Co Phillip J **15** 264-269.

Hugo B, Otto A, Stassinakis A, Hofmann N & Klaiber B (2001a) [Retrospektive *In-vivo*-Untersuchung von Sonicsys-approx-Füllungen] *Schweizer Monatsschrift für Zahnmedizin* **111** 152-158.

Hugo B, Stassinakis A, Hofmann N, Hausmann P & Klaiber B (2001b) [*In-vivo*-Untersuchung von kleinen Klasse-II-Kompositfüllungen] *Schweizer Monatsschrift für Zahnmedizin* **111** 11-18.

Huth K, Manhart J, Hickel R & Kunzelmann K-H (2003) Three-year clinical performance of a compomer in stress-bearing restorations in permanent posterior teeth *American Journal of Dentistry* **16** 255-259.

Huth K, Selbertinger S, Kunzelmann KH & Hickel R (1999) Compomer for Class I/II restorations—results after 6 months *Journal of Dental Research* **78** 285.

Isidor F & Brondum K (1995) A clinical evaluation of porcelain inlays *Journal of Prosthetic Dentistry* **74** 140-144.

Jedynakiewicz NM, Martin N & Fletcher JM (2002) A clinical evaluation of a posterior compomer restorative at 3 years *Journal of Dental Research* **81** 52.

Jensen ME (1988) A two-year clinical study of posterior etched-porcelain resin-bonded restorations *American Journal of Dentistry* **1** 27-33.

Jokstad A & Mjör IA (1991) Analyses of long-term clinical behavior of Class-II amalgam restorations *Acta Odontologica Scandinavica* **49** 47-63.

Jokstad A, Mjör IA, Nilner K & Kaping S (1994a) Clinical performance of three anterior restorative materials over 10 years *Quintessence International* **25** 101-108.

Jokstad A, Mjör IA & Qvist V (1994b) The age of restorations *in situ Acta Odontologica Scandinavica* **52** 234-248.

Kamann WK & Gängler P (1999) [Zur Funktionszeit von Amalgam-, Komposite- und Goldhämmerfüllungen] *ZWR* **108** 270-273.

Kiremitci A & Bolay S (2003) A 3-year clinical evaluation of a gallium restorative alloy *Journal of Oral Rehabilitation* **30** 664-667.

Kiremitci A, Bolay S & Gurgan S (1998) Two-year performance of glass-ceramic insert-resin composite restorations: Clinical and scanning electron microscopic evaluation *Quintessence International* **29** 417-421.

Klimm W, Wolff U & Natusch I (1999) Evaluation of Class II composite and ceramic restorations *in vivo* and *in vitro* *Journal of Dental Research* **78** 308.

Köhler B, Rasmusson C-G & Ödman P (2000) A five-year clinical evaluation of Class II composite resin restorations *Journal of Dentistry* **28** 111-116.

Krämer N, Frankenberger R, Pelka M & Petschelt A (1999) IPS Empress inlays and onlays after four years—a clinical study *Journal of Dentistry* **27** 325-331.

Krämer N, Kunzelmann KH, Mumesohn M, Pelka M & Hickel R (1996) [Langzeiterfahrungen mit einem mikrogefüllten Komposit als Inlaysystem] *Deutsche Zahnärztliche Zeitschrift* **51** 342-344.

Krämer N, Kunzelmann KH, Pollety T, Pelka M & Hickel R (1994) [Langzeiterfahrungen mit Cermet-Zementfüllungen in Klasse-I/II-Kavitäten] *Deutsche Zahnärztliche Zeitschrift* **49** 905-909.

Krejci I, Krejci D & Lutz F (1992) Clinical evaluation of a new pressed glass ceramic inlay material over 1.5 years *Quintessence International* **23** 181-186.

Krejci I & Lutz F (1991) Marginal adaptation of Class V restorations using different restorative techniques *Journal of Dentistry* **19** 24-32.

Kreulen CM, Tobi H, Gruythuysen RJM, van Amerongen WE & Borgmeijer PJ (1998) Replacement risk of amalgam treatment modalities: 15-year results *Journal of Dentistry* **26** 627-632.

Lavelle CLB (1976) A cross-sectional survey into the durability of amalgam restorations *Journal of Dentistry* **4** 139-143.

Leempoel PJB, Eschen S, De Haan AF & van-'t-Hof MA (1985) An evaluation of crowns and bridges in general dental practice *Journal of Oral Rehabilitation* **12** 515-528.

Lehner C, Studer S, Brodbeck U & Schärer P (1998) Six-year clinical results of leucite-reinforced glass ceramic inlays and onlays (Acta Medicinae Dentinum Helvetica) *Schweizer Monatsschrift für Zahnmedizin* **3** 137-146.

Leinfelder KF, Sluder TB, Santos JFF & Wall JT (1980) Five-year clinical evaluation of anterior and posterior restorations of composite resins *Operative Dentistry* **5** 57-65.

Leirskar J, Nordbo H, Thoresen NR, Henaug T & der Fehr FR (2003) A four to six years follow-up of indirect resin composite inlays/onlays *Acta Odontologica Scandinavica* **61** 247-251.

Letzel H (1989) Survival rates and reasons for failure of posterior composite restorations in multicentre clinical trial *Journal of Dentistry* **17** 10-17.

Letzel H, van-'t-Hof MA, Marshall GW & Marshall SJ (1997) The influence of the amalgam alloy on the survival of amalgam restorations: A secondary analysis of multiple controlled clinical trials *Journal of Dental Research* **76** 1787-1798.

Letzel H, van-'t-Hof MA, Vrijhoef MM, Marshall-GW J & Marshall SJ (1989) A controlled clinical study of amalgam restorations: Survival, failures, and causes of failure *Dental Materials* **5** 115-121.

Lindberg A, van Dijken JW & Lindberg M (2003) 3-year evaluation of a new open sandwich technique in Class II cavities *American Journal of Dentistry* **16** 33-36.

Lopes LG, Cefaly DF, Franco EB, Mondelli RF, Lauris JR & Navarro MF (2003) Clinical evaluation of two "packable" posterior composite resins: Two-year results *Clinical Oral Investigations* **7** 123-128.

Lopes LG, Cefaly DFG, Franco EB, Mondelli RFL, Lauris JRP & Navarro MFL (2002) Clinical evaluation of two "packable" posterior composite resins *Clinical Oral Investigations* **6** 83.

Lösche GM (1996) [Klasse-II-Kompositfüllungen mit und ohne konfektionierte Glaskeramik-Inserts. Eine *in-vivo*-Studie] *Deutsche Zahnärztliche Zeitschrift* **51** 389-394.

Lundin SA & Koch G (1989) Class I and II composite resin restorations: 4-year clinical follow-up *Swedish Dental Journal* **13** 217-227.

Lundin SA & Koch G (1999) Class I and II posterior composite resin restorations after 5 and 10 years *Swedish Dental Journal* **23** 165-171.

Luo Y, Lo ECM, Fang DTS & Wei SHY (2000) Clinical evaluation of polyacid-modified resin composite posterior restorations: One-year results *Quintessence International* **31** 630-636.

Luo Y, Lo ECM, Fang DTS, Smales RJ & Wei SH (2002) Clinical evaluation of Dyract AP restorative in permanent molars: 2-year results *American Journal of Dentistry* **15** 403-406.

Mahmood S & Smales RJ (1994) Longevity of dental restorations in selected patients from different practice environments *Australian Dental Journal* **39** 15-17.

Mair LH (1998) Ten-year clinical assessment of three posterior resin composites and two amalgams *Quintessence International* **29** 483-490.

Malament KA & Socransky SS (1999) Survival of Dicor glass-ceramic dental restorations over 14 years. Part II: Effect of thickness of Dicor material and design of tooth preparation *Journal of Prosthetic Dentistry* **81** 662-667.

Mallow P, Durward C & Klaipo M (1995) Comparison of two glass ionomer cements using the ART technique *Journal of Dental Research* **74** 405.

Mallow PK, Durward CS & Klaipo M (1998) Restoration of permanent teeth in young rural children in Cambodia using the atraumatic restorative treatment (ART) technique and Fuji II glass ionomer cement *International Journal of Paediatric Dentistry* **8** 35-40.

Manhart J, Chen HY, Albrecht A, Neuerer P & Hickel R (2002a) Four-year clinical evaluation of composite and ceramic inlays *Journal of Dental Research* **81** 332.

Manhart J, Chen HY, Kunzelmann KH & Hickel R (1999a) [Werkstoffkundliche Charakterisierung eines Füllungsmateriales auf Ormocer-Basis im Vergleich zu einem Komposit und einem Kompomer] *ZMK* **15** 807-812.

Manhart J, Chen HY, Neuerer P, Scheibenbogen-Fuchsbrunner A & Hickel R (2001a) Three-year clinical evaluation of composite and ceramic inlays *American Journal of Dentistry* **14** 95-99.

Manhart J, Chen HY, Spoerlein F & Hickel R (2002b) 2-year clinical evaluation of Definite ormocer-restorations in posterior teeth *Journal of Dental Research* **81** 80.

Manhart J, Glomb C, Stueckgen D, Neuerer P, Flessa H-P & Hickel R (2002c) Clinical evaluation of a self-etching adhesive at 2 years *Journal of Dental Research* **81** 233.

Manhart J & Hickel R (1999a) Esthetic compomer restorations in posterior teeth using a new all-in-one adhesive: Case presentation *Journal of Esthethic Dentistry* **11** 250-258.

Manhart J & Hickel R (1999b) [Klinische Studie zum Einsatz eines All-in-one-Adhäsivs. Erste Ergebnisse nach 6 Monaten] *Quintessenz der zahnärztlichen Literatur* **50** 1277-1288.

Manhart J, Hollwich B, Mehl A, Kunzelmann KH & Hickel R (1999b) [Randqualität von Ormocer- und Kompositfüllungen in Klasse-II-Kavitäten nach künstlicher Alterung] *Deutsche Zahnärztliche Zeitschrift* **54** 89-95.

Manhart J, Huetzen D, Chen HY, Neuerer P & Hickel R (2002d) Three-year clinical evaluation of Artglass- and Charisma-inlays *Journal of Dental Research* **81** 333.

Manhart J, Huth K, Glomb C, Stueckgen D, Neuerer P, Flessa H-P & Hickel R (2002e) Clinical evaluation of a self-etching adhesive at 3 years *Program and Abstracts 2002 of the European Festival of Oral Science*.

Manhart J, Ilie N, Dullin P, Draegert U, Chen HY, Kunzelmann KH & Hickel R (2001b) Flexure mechanical properties of compomer materials *Journal of Dental Research* **80** 204.

Manhart J, Ilie N, Neuerer P, Chen HY, Kunzelmann KH & Hickel R (2001c) Flexure mechanical properties of packable and hybrid composites *Journal of Dental Research* **80** 203.

Manhart J, Kunzelmann KH, Chen HY & Hickel R (2000a) Mechanical properties and wear behavior of light-cured packable composite resins *Dental Materials* **16** 33-40.

Manhart J, Li D, Powers JM & Hickel R (1998) Bonding of compomers to deep dentin under various surface conditions *Journal of Dental Research* **77** 787.

Manhart J, Mehl A, Schroeter R, Obster B & Hickel R (1999c) Bond strength of composite to dentin treated by air abrasion *Operative Dentistry* **24** 223-232.

Manhart J, Neuerer P, Scheibenbogen-Fuchsbrunner A & Hickel R (2000b) Three-year clinical evaluation of direct and indirect composite restorations in posterior teeth *Journal of Prosthetic Dentistry* **84** 289-296.

Martin JA & Bader JD (1997) Five-year treatment outcomes for teeth with large amalgams and crowns *Operative Dentistry* **22** 72-78.

Martin N & Jedynakiewicz NM (1999) Clinical performance of Cerec ceramic inlays: A systematic review *Dental Materials* **15** 54-61.

Maryniuk GA & Kaplan SH (1986) Longevity of restorations: Survey results of dentists' estimates and attitudes *Journal of the American Dental Association* **112** 39-45.

Mertz-Fairhurst EJ, Curtis JW, Ergle JW, Rueggeberg FA & Adair SM (1998) Ultraconservative and cariostatic sealed restorations: Results at year 10 *Journal of the American Dental Association* **129** 55-66.

Mjör IA (1989) Amalgam and composite resin resin restorations: Longevity and reasons for replacement in Anusavice K (ed) *Quality Evaluation of Dental Restorations* Quintessence Publishing, Chicago, 1989, 61-80.

Mjör IA (1992a) Long-term cost of restorative therapy using different materials *Scandinavian Journal of Dental Research* **100** 60-65.

Mjör IA (1992b) Problems and benefits associated with restorative materials: Side-effects and long-term cost *Advanced Dental Research* **6** 7-16.

Mjör IA (1996) Glass ionomer restorations and secondary caries. A preliminary report *Quintessence International* **27** 171-174.

Mjör IA (1997a) Selection of restorative materials in general dental practice in Sweden *Acta Odontologica Scandinavica* **55** 53-57.

Mjör IA (1997b) The reasons for replacement and the age of failed restorations in general dental practice *Acta Odontologica Scandinavica* **55** 58-63.

Mjör IA & Jokstad A (1993) Five-year study of Class II restorations in permanent teeth using amalgam, glass polyalkenoate (ionomer) cerment and resin-based composite materials *Journal of Dentistry* **21** 338-343.

Mjör IA, Jokstad A & Qvist V (1990) Longevity of posterior restorations *International Dentistry Journal* **40** 11-17.

Mjör IA & Medina JE (1993) Reasons for placement, replacement, and age of gold restorations in selected practices *Operative Dentistry* **18** 82-87.

Mjör IA & Toffenetti F (1992) Placement and replacement of amalgam restorations in Italy *Operative Dentistry* **17** 70-73.

Mjör IA (2004) A recurring problem: Research in restorative dentistry *Journal of Dental Research* **83** 92.

Moffa JP (1989) Comparative performance of amalgam and composite resin restorations and criteria for their use in Anusavice K (ed) *Quality Evaluation of Dental Restorations* Quintessence Publishing, Chicago 125-133.

Molin M & Karlsson S (1996) A 3-year clinical follow-up study of a ceramic (Optec) inlay system *Acta Odontologica Scandinavica* **54** 145-149.

Molin MK & Karlsson SL (2000) A randomized 5-year clinical evaluation of 3 ceramic inlay systems *International Journal of Prosthodontics* **13** 194-200.

Mörmann W & Brandestini M (1989) [Die Cerec Computer Rekonstruktion] *Inlays, Onlays und Veneers* Quintessence Publishing Berlin.

Mörmann W & Krejci I (1992) Computer-designed inlays after 5 years *in situ*: Clinical performance and scanning electron microscopic evaluation *Quintessence International* **23** 109-115.

Nicolaisen S, der Fehr FR, Lunder N & Thomsen I (2000) Performance of tunnel restorations at 3-6 years *Journal of Dentistry* **28** 383-387.

Nordbo H, Leirskar J & von der Fehr FR (1998) [Schüsselförmige Kavitätenpräparation für approximale Kompositrestaurationen im Seitenzahnbereich - Beobachtungen bis zu 10 Jahren] *Quintessenz der zahnärztlichen Literatur* **49** 773-779.

O'Neal SJ, Miracle RL & Leinfelder KF (1993) Evaluating interfacial gaps for esthetic inlays *Journal of the American Dental Association* **124** 48-54.

Oberländer H, Hiller KA, Thonemann B & Schmalz G (2001) Clinical evaluation of packable composite resins in Class-II restorations *Clinical Oral Investigations* **5** 102-107.

Ödman P (2002) A 3-year clinical evaluation of Cerana prefabricated ceramic inserts *International Journal of Prosthodontics* **15** 79-82.

Osborne JW, Normann RD & Gale EN (1991) A 14-year clinical assessment of 12 amalgam alloys *Quintessence International* **22** 857-864.

Otto T (1995) [Cerec-Restaurationen] *Schweizer Monatsschrift für Zahnmedizin* **105** 1039-1044.

Otto T & de Nisco S (2002) Computer-aided direct ceramic restorations: A 10-year prospective clinical study of Cerec CAD/CAM inlays and onlays *International Journal of Prosthodontics* **15** 122-128.

Pallesen U & Qvist V (2003) Composite resin fillings and inlays. An 11-year evaluation *Clinical Oral Investigations* **7** 71-79.

Pallesen U & van Dijken JWV (2000) An 8-year evaluation of sintered ceramic and glass ceramic inlays processed by the Cerec CAD/CAM system *European Journal of Oral Science* **108** 239-246.

Paterson N (1984) The longevity of restorations. A study of 200 regular attenders in a general dental practice *British Dental Journal* **157** 23-25.

Pelka M, Schmidt G & Petschelt A (1996) [Klinische Qualitätsbeurteilung von gegossenen Metallinlays und –onlays] *Deutsche Zahnärztliche Zeitschrift* **51** 268-272.

Peters MCRB, Roeters JJM & Frankenmolen FWA (1996) Clinical evaluation of Dyract in primary molars: 1-year results *American Journal of Dentistry* **9** 83-87.

Phantumvanit P, Songpaisan Y, Pilot T & Frencken JE (1996) Atraumatic restorative treatment (ART): A three-year community field trial in Thailand—survival of one-surface restorations in the permanent dentition *Journal of Public Health Dentistry* **56** 141-145.

Pieper K, Meyer G, Marienhagen B & Motsch A (1991) [Eine Langzeitstudie an Amalgam- und Kunststoff-Füllungen] *Deutsche Zahnärztliche Zeitschrift* **46** 222-225.

Pilebro CE & van Dijken JWV (2001) Analysis of factors affecting failure of glass cermet tunnel restorations in a multi-center study *Clinical Oral Investigations* **5** 96-101.

Plasmans PJJM, Creugers NHJ & Mulder J (1998) Long-term survival of extensive amalgam restorations *Journal of Dental Research* **77** 453-460.

Posselt A & Kerschbaum T (2003) Longevity of 2328 chairside Cerec inlays and onlays *International Journal of Compendium Dentistry* **6** 231-248.

Powers JM & Farah J (eds) (1998) Compomers *The Dental Advisor* **15** 1-5.

Qualtrough AJE & Wilson NHF (1996) A 3-year clinical evaluation of a porcelain inlay system *Journal of Dentistry* **24** 317-323.

Qvist V, Qvist J & Mjör IA (1990) Placement and longevity of tooth-colored restorations in Denmark *Acta Odontologica Scandinavica* **48** 305-311.

Raskin A, Michotte-Theall B, Vreven J & Wilson NHF (1999) Clinical evaluation of a posterior composite 10-year report *Journal of Dentistry* **27** 13-19.

Raskin A, Setcos JC, Vreven J & Wilson NHF (2000) Influence of the isolation method on the 10-year clinical behaviour of posterior resin composite restorations *Clinical Oral Investigations* **4** 148-152.

Reiss B & Walther W (1991) [Überlebensanalyse und klinische Nachuntersuchungen von zahnfarbenen Einlagefüllungen nach dem CEREC-Verfahren] *Zahnärztliche Welt - Zahnärztliche Rundschau - Zahnärztliche Reform* **100** 329-332.

Reiss B & Walther W (1998) [Ereignisanalyse und klinische Langzeitergebnisse mit Cerec-Keramikinlays] *Deutsche Zahnärztliche Zeitschrift* **53** 65-68.

Reiss B & Walther W (2000) Clinical long-term results and 10-year Kaplan-Meier analysis of Cerec restorations *International Journal of Computerized Dentistry* **3** 9-23.

Robinson AD (1971) The life of a filling *British Dental Journal* **130** 206-208.

Rosin M, Steffen H, Konschake C, Greese U, Teichmann D, Hartmann A & Meyer G (2003) One-year evaluation of an Ormocer restorative—a multipractice clinical trial *Clinical Oral Investigations* **7** 20-26.

Roulet JF (1997a) Benefits and disadvantages of tooth-coloured alternatives to amalgam *Journal of Dentistry* **25** 459-473.

Roulet JF (1997b) Longevity of glass ceramic inlays and amalgam—results up to 6 years *Clinical Oral Investigations* **1** 40-46.

Roulet JF & Herder S (1989) [Keramik als Füllungsmaterial für Seitenzahnkavitäten] *Zahnärztl.Mitteilungen* **79** 908-913.

Rykke M (1992) Dental materials for posterior restorations *Endodontology Dental Traumatology* **8** 139-148.

Scheibenbogen-Fuchsbrunner A, Manhart J, Kunzelmann KH & Hickel R (1998) One-year clinical evaluation of composite and ceramic inlays in posterior teeth *Journal of Prosthetic Dentistry* **80** 410-416.

Scheibenbogen-Fuchsbrunner A, Manhart J, Kremers L, Kunzelmann KH & Hickel R (1999) Two-year clinical evaluation of direct and indirect composite restorations in posterior teeth *Journal of Prosthetic Dentistry* **82** 391-397.

Schlösser R, Kerschbaum T, Ahrens FJ & Cramer M (1993) [Überlebensrate von Teil- und Vollgußkronen] *Deutsche Zahnärztliche Zeitschrift* **48** 696-698.

Schulz P, Johansson A & Arvidson K (2003) A retrospective study of Mirage ceramic inlays over up to 9 years *The International Journal of Prosthodontics* **16** 510-514.

Setcos JC, Staninec M & Wilson NH (1999) A two-year randomized, controlled clinical evaluation of bonded amalgam restorations *Journal of Adhesive Dentistry* **1** 323-331.

Sjögren G, Bergman M, Molin M & Bessing C (1992) A clinical examination of ceramic (Cerec) inlays *Acta Odontologica Scandinavica* **50** 171-178.

Sjögren G, Hedlund SO, Jonsson C & Sandström A (2000) A 3-year follow-up study of preformed beta-quartz glass-ceramic insert restorations *Quintessence International* **31** 25-31.

Sjögren G, Molin M & van Dijken JW (1998) A 5-year clinical evaluation of ceramic inlays (Cerec) cemented with a dual-cured or chemically cured resin composite luting agent *Acta Odontologica Scandinavica* **56** 263-267.

Smales RJ (1991) Longevity of cusp-covered amalgams: Survivals after 15 years *Operative Dentistry* **16** 17-20.

Smales RJ, Gerke DC & White IL (1990) Clinical evaluation of occlusal glass ionomer, resin, and amalgam restorations *Journal of Dentistry* **18** 243-249.

Smales RJ & Hawthorne WS (1996) Long-term survival and cost-effectiveness of five dental restorative materials used in various classes of cavity preparations *International Dental Journal* **46** 126-130.

Smales RJ & Hawthorne WS (1997) Long-term survival of extensive amalgams and posterior crowns *Journal of Dentistry* **25** 225-227.

Smales RJ, Webster DA & Leppard PI (1991a) Survival predictions of amalgam restorations *Journal of Dentistry* **19** 272-277.

Smales RJ, Webster DA & Leppard PI (1991b) Survival predictions of four types of dental restorative materials *Journal of Dentistry* **19** 278-282.

Stenberg R & Matsson L (1993) Clinical evaluation of glass ceramic inlays (Dicor) *Acta Odontologica Scandinavica* **51** 91-97.

Stoll R, Sieweke M, Pieper K, Stachniss V & Schulte A (1999) Longevity of cast gold inlays and partial crowns—a retrospective study at a dental school clinic *Clinical Oral Investigations* **3** 100-104.

Strand GV, Nordbo H, Leirskar J, von der Fehr FR & Eide GE (2000) Tunnel restorations placed in routine practice and observed for 24 to 54 months *Quintessence International* **31** 453-460.

Strand GV, Nordbo H, Tveit AB, Espelid I, Wikstrand K & Eide GE (1996) A 3-year clinical study of tunnel restorations *European Journal of Oral Science*s **104** 384-389.

Studer S, Lehner C, Brodbeck U & Scharer P (1996) Short-term results of IPS-Empress inlays and onlays *Journal of Prosthodontology* **5** 277-287.

Studer SP, Wettstein F, Lehner C, Zullo TG & Schärer P (2000) Long-term survival estimates of cast gold inlays and onlays with their analysis of failures *Journal of Oral Rehabilitation* **27** 461-472.

Summitt JB, Burgess JO, Berry TG, Robbins JW, Osborne JW & Haveman CW (2001) The performance of bonded vs pin-retained complex amalgam restorations. A five-year clinical evaluation *Journal of the American Dental Association* **132** 923-931.

Svanberg M (1992) Class II amalgam restorations, glass-ionomer tunnel restorations, and caries development on adja-

cent tooth surfaces: A 3-year clinical study *Caries Research* **26** 315-318.

Thonemann B, Federlin M, Schmalz G & Schams A (1997) Clinical evaluation of heat-pressed glass-ceramic inlays *in vivo:* 2-year results *Clinical Oral Investigations* **1** 27-34.

Thordrup M, Isidor F & Hörsted-Bindslev P (1994) A one-year clinical study of indirect and direct composite and ceramic inlays *Scandinavian Journal of Dental Research* **102** 186-192.

Thordrup M, Isidor F & Hörsted-Bindslev P (1999) A 3-year study of inlays milled from machinable ceramic blocks representing 2 different inlay systems *Quintessence International* **30** 829-836.

Thordrup M, Isidor F & Hörsted-Bindslev P (2001) A 5-year clinical study of indirect and direct resin composite and ceramic inlays *Quintessence International* **32** 199-205.

Tidehag P & Gunne J (1995) A 2-year clinical follow-up study of IPS Empress ceramic inlays *International Journal of Prosthodontics* **8** 456-460.

Triolo PT, Barkmeier WW & Los SA (1995) Bonding efficacy of a compomer using different conditioning procedures *Journal of Dental Research* **74** 107.

Türkün LS & Aktener BO (2001) Twenty-four month clinical evaluation of different posterior composite resin materials *Journal of the American Dental Association* **132** 196-203.

Türkün LS, Aktener BO & Ates M (2003a) Clinical evaluation of different posterior resin composite materials: A 7-year report *Quintessence International* **34** 418-426.

Türkün LS, Turkun M & Ozata F (2003b) Two-year clinical evaluation of a packable resin-based composite *Journal of the American Dental Association* **134** 1205-1212.

van Dijken JW (2003a) A 6-year clinical evaluation of Class I poly-acid modified resin composite/resin composite laminate restorations cured with a two-step curing technique *Dental Materials* **19** 423-428.

van Dijken JW (2003b) Resin-modified glass ionomer cement and self-cured resin composite luted ceramic inlays. A 5-year clinical evaluation *Dental Materials* **19** 670-674.

van Dijken JWV (1994) 5-6 year evaluation of direct composite inlays *Journal of Dental Research* **73** 327.

van Dijken JWV (2000) Direct resin composite inlays/onlays: An 11 year follow-up *Journal of Dentistry* **28** 299-306.

van Dijken JWV (2002) Three-year performance of a calcium-, fluoride-, and hydroxyl-ions-releasing composite *Acta Odontologica Scandinavica* **60** 155-159.

van Dijken JWV, Hasselrot L, Örmin A & Olofsson AL (2001) Restorations with extensive dentin/enamel-bonded ceramic coverage: A 5-year follow-up *European Journal of Oral Science*s **109** 222-229.

van Dijken JWV, Höglund-Aberg C & Olofsson AL (1998) Fired ceramic inlays: A 6-year follow up *Journal of Dentistry* **26** 219-225.

van Dijken JWV, Örmin A & Olofsson AL (1999) Clinical performance of pressed ceramic inlays luted with resin-modified glass ionomer and autopolymerizing resin composite cements *Journal of Prosthetic Dentistry* **82** 529-535.

Van Nieuwenhuysen JP, D'Hoore W, Carvalho J & Qvist V (2003) Long-term evaluation of extensive restorations in permanent teeth *Journal of Dentistry* **31** 395-405.

Vilkinis V, Hörsted-Bindslev P & Baelum V (2000) Two-year evaluation of Class II resin-modified glass-ionomer cement/composite open sandwich and composite restorations *Clinical Oral Investigations* **4** 133-139.

Wagner J, Hiller KA & Schmalz G (2003) Long-term clinical performance and longevity of gold alloy vs ceramic partial crowns *Clinical Oral Investigations* **7** 80-85.

Walther W, Reiss B & Toutenburg H (1994) [Longitudinale Ereignisanalyse von Cerec-Einlagefüllungen] *Deutsche Zahnärztliche Zeitschrift* **49** 914-917.

Wassell RW, Walls AWG & McCabe JF (1995) Direct composite inlays versus conventional composite restorations: Three-year clinical results *British Dental Journal* **179** 343-349.

Wassell RW, Walls AWG & McCabe JF (2000) Direct composite inlays versus conventional composite restorations: 5-year follow-up *Journal of Dentistry* **28** 375-382.

Welbury RR, Walls AW, Murray JJ & McCabe JF (1990) The management of occlusal caries in permanent molars. A 5-year clinical trial comparing a minimal composite with an amalgam restoration *British Dental Journal* **169** 361-366.

Wendt SL & Leinfelder KF (1992) Clinical evaluation of a heat-treated resin composite inlay: 3-year results *American Journal of Dentistry* **5** 258-262.

Wiedmer CS, Krejci I & Lutz F (1997) [Klinische, röntgenologische und rasterelektronenoptische Untersuchung von Kompositinlays nach fünfjähriger Funktionszeit] (Acta Medicinae Dentinum Helvetica) *Schweizer Monatsschrift für Zahnmedizin* **2** 301-307.

Wilder AD, May KN, Bayne SC, Taylor DF & Leinfelder KF (1999) Seventeen-year clinical study of ultraviolet-cured posterior composite Class I and II restorations *Journal of Esthethetic Dentistry* **11** 135-142.

Wilson NHF, Cowan AJ, Unterbrink G, Wilson MA & Crisp RJ (2000) A clinical evaluation of Class II composites placed using a decoupling technique *Journal of Adhesive Dentistry* **2** 319-329.

Wilson NHF, Wastell DG & Norman RD (1996) Five-year performance of high-copper content amalgam restorations in a multiclinical trial of a posterior composite *Journal of Dentistry* **24** 203-210.

Wilson NHF, Wilson MA, Wastell DG & Smith GA (1988) A clinical trial of a visible light cured posterior composite resin restorative material: Five-year results *Quintessence International* **19** 675-681.

Wolter H, Storch W & Ott H (1994) New inorganic/organic copolymers (ORMOCERs) for dental applications *Materials Research Society Symposia Proceedings* **346** 143-149.

Wucher M, Grobler SR & Senekal PJC (2002) A 3-year clinical evaluation of a compomer, a composite and a compomer/composite (sandwich) in Class II restorations *American Journal of Dentistry* **15** 274-278.

Yip KHK, Smales RJ, Gao W & Peng D (2002) The effect of two cavity preparation methods on the longevity of glass ionomer cement restorations. An evaluation after 12 months *Journal of the American Dental Association* **133** 744-751.

Ziraps A & Honkala E (2002) Clinical trial of a new glass ionomer for an atraumatic restorative treatment technique in Class I restorations placed in Latvian school children *Medical Principles of Practice* **11**(**Supplement 1)** 44-47.

Zuellig-Singer R & Bryant RW (1998) Three-year evaluation of computer-machined ceramic inlays: Influence of luting agent *Quintessence International* **29** 573-582.

©Operative Dentistry, 2004, **29-5,** 509-514

Clinical Research

Effect of Luting Cement on Dental Biofilm Composition and Secondary Caries Around Metallic Restorations *In Situ*

JS Moura • EMCX Lima • AF Paes Leme
AA Del Bel Cury • CPM Tabchoury • JA Cury

Clinical Relevance

When fluoride toothpaste is used, the anticariogenic properties of luting cements appear not to be relevant on secondary caries reduction.

SUMMARY

Since the importance of luting cement on secondary caries in enamel and dentin is unknown, an *in situ* crossover study was conducted in three phases over 21 days using a fluoride-containing toothpaste. One hundred and twenty-six metallic restorations were cemented into the dentinoenamel junction of slabs of human teeth with zinc phosphate (ZP), resin-modified glass ionomer (GI) or resinous cement (RC). The slabs were inserted onto flanges of the removable partial acrylic dentures of 14 volunteers and covered with gauze to enhance dental plaque accumulation. The volunteers used fluoride toothpaste (1.100 µg F/g, w/w). After 21 days, the biofilm that formed on the slabs was collected for biochemical and microbiological analyses, and the demineralization in enamel-dentin around the restorations was evaluated. The fluoride concentration of biofilm in the GI group was higher ($p<0.05$) than the ZP and RC groups. Also, the concentration of Zinc in biofilm formed on the slabs cemented with ZP was higher ($p<0.05$) than the other groups. However, the effect of the luting material on enamel or dentin demineralization was not statistically significant ($p>0.05$). The data suggest that when fluoride toothpaste is used, the anticariogenic property of the luting cement may not be relevant to the reduction of secondary caries.

JS Moura, Faculty of Dentistry of Piracicaba, University of Campinas, Piracicaba, SP, Brazil

EMCX Lima, Faculty of Dentistry of Piracicaba, University of Campinas, Piracicaba, SP, Brazil

AF Paes Leme, Faculty of Dentistry of Piracicaba, University of Campinas, Piracicaba, SP, Brazil

*Altair A Del Bel Cury, Faculty of Dentistry of Piracicaba, University of Campinas, Piracicaba, SP, Brazil

CPM Tabchoury, Faculty of Dentistry of Piracicaba, University of Campinas, Piracicaba, SP, Brazil

JA Cury, Faculty of Dentistry of Piracicaba, University of Campinas, Piracicaba, SP, Brazil

*Reprint request: Av Limeira, 901, Piracicaba, SP 13414 – 903, Brazil; e-mail: altcury@fop.unicamp.br

INTRODUCTION

Improvements in life expectancy and oral health have resulted in an increase in individuals who keep their natural teeth. Therefore, procedures that strengthen and restore tooth structure, such as indirect restorations, are necessary (Ettinger & others, 1998). Jokstad and Mijor (1996) observed that the longevity of this kind of procedure is 80% to 85% in five years and 71% to 81% in 10 years, with secondary caries being the

most common cause of failure (Musa, Pearson & Gelbier, 1996).

The development of secondary caries may be related to the marginal integrity of a restoration and, in indirect restorations, is related to microfracture, infiltration and plastic deformation of the luting cement. Thus, selection of the luting cement is decisive for restoration longevity (Christensen, 1997).

A desired property of luting cements is anticariogenicity provided by fluoride release, which could reduce the severity of cariogenic challenges when available during an acid attack (Forsten, 1998). In spite of its effectiveness as a restorative material (Kotsanos, 2001) in preventing caries lesions, the cariostatic effect of resin-modified glass ionomer, when used as a luting cement, is questionable since Ettinger and others (1998) and Shinkai, Del Bel Cury and Cury (2001) did not verify any influence of the luting material on the extension of artificial caries on enamel and root dentin. Besides, there is no evidence that the luting cement could modify the biochemical and microbiological composition of dental biofilm. However, most studies on luting cements did not use fluoride-containing toothpaste, which prevents demineralization on dental enamel even in high cariogenic challenges (Duggal & others, 2001).

There is also evidence that zinc, when presented in dental biofilm, interacts with fluoride and enhances its antimetabolic activity on mutans streptococci (Izaquirre-Fernandez, Eisenberg & Curzon, 1989). Thus, this work evaluated the inhibitory effect of resin-modified glass ionomer on secondary caries around indirect restorations and its effect on fluoride concentration and mutans streptococci counts in dental biofilm

METHODS AND MATERIALS

Experimental Design

A crossover *in situ* study was carried out for 21 days each in three phases, where 14 healthy adult volunteers (mean age 59 ± 11.2), whose treatment involved an inferior Class I RPD in association with a superior complete denture, were selected. The volunteers signed an informed written consent approved by the Ethics Committee of Faculty of Dentistry of Piracicaba (FOP-UNICAMP process #18/2001). None of the subjects were smokers or used antimicrobials or chemical products for mouth rinse; they were instructed to maintain their normal dietary habits.

The volunteers wore removable partial dentures (RPD) containing four enamel-dentin slabs with cemented indirect restorations at each phase (Figure 1). Each sample was previously restored with an indirect metallic restoration and covered with gauze (Polyester Knit Fabric, Bard, Billerica, MA, USA) (Figures 2 and 3). The volunteers were submitted to three treatment groups according to the luting cement: zinc phosphate - ZP (Cimento De Zinco, SS White Ltda, Rio de Janeiro, Brazil), resin-modified glass ionomer - GI (Rely X Luting Vitremer, 3M ESPE, St Paul, MN, USA) and resinous cement - RC (Rely X, 3M ESPE). The treatment effect on caries in enamel and root dentin around the restoration was evaluated by cross-sectional microhardness test since there is a good correlation (0.91) between enamel microhardness and the percentage of mineral in caries lesions (Featherstone & others, 1983). Biochemical and microbiological analyses of the biofilm formed on the slabs were made, and two of four gauzes were used to determine the fluoride (F) and zinc (Zn) concentrations; total cultivable flora (TF), mutans streptococci (MS) counting and the percentage of mutans streptococci (%MS) were quantified in the other two gauzes. The volunteers were randomly assigned to treatment at the beginning of the study and after three phases; all were submitted to all treatments. Between treatments, seven-day washout periods were conducted to prevent any carry over effect. During the experiment, the volunteers used a silica-based fluoride-containing toothpaste (1,100 µg F/g, w/w, as NaF) (Sorriso Fresh, Kolynos Do, Brasil, São Bernardo do Campo, Brazil).

Preparation and Restoration of the Samples

One hundred and twenty-six slabs were made of third human molars extracted for clinical purposes and stored in thymol 0.1% for at least one month. The dental surfaces were manually scaled and polished with slurry of pumice at low speed. The inclusion criteria for use of the third molars included more than two-thirds of formed root, sufficient volume and sound cervical region. The middle-third of crowns and roots were longitudinally sectioned into three blocks. Cavities 1.6 mm in diameter and 0.8 mm in depth were prepared by a patronizing machine at the crown-root junctions, where half of the cavity margin was located in enamel and half in root dentin (Shinkai & others, 2001; Figure 3).

Stainless steel precision ball bearings (Rolamentos Fag, Ltda, São Paulo, Brazil), with a diameter of 1.5 mm, were used to simulate a cast metallic restoration. The metallic restorations were obtained from grinding the ball bearings using an electric polishing machine and 320-grit silicon carbide paper disc until approximately half of their diameter was achieved. The restoration surfaces to be luted were then blasted with 50 µm aluminum oxide. For each sample, one cavity was randomly assigned for restoration luted with one of the three cements (Shinkai & others, 2001).

Intraoral Phase

For each volunteer, one superior, complete denture and two inferior RPDs were made. In one of the RPDs, four insertions 4x4x4 mm were made on the acrylic buccal flange to set the dental slabs.

At the beginning of each phase, the luting cements were manipulated according to manufacturers' instructions and the metallic restorations cemented in the tooth samples. The excess material was removed with a #15 surgical blade to avoid damage to tooth structures. For each phase, four samples of the same group were randomly inserted into the RPDs' flanges.

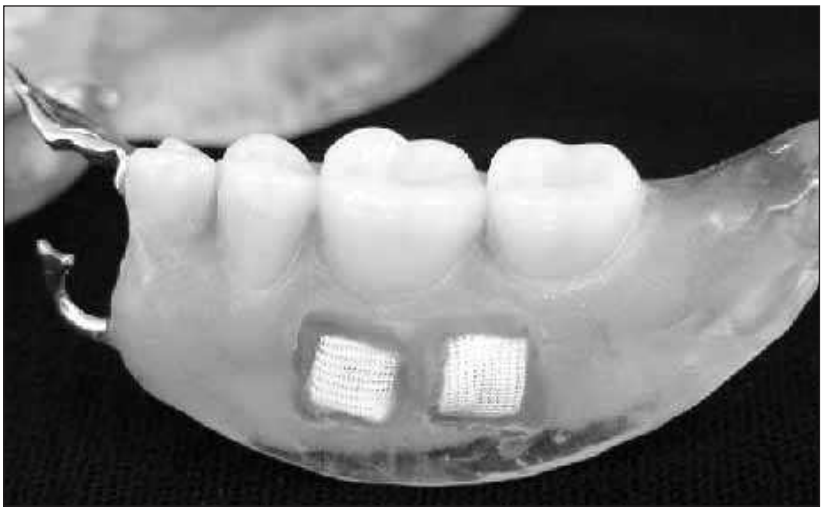


Figure 1. *Removable prosthesis fabricated for this study.*

Dental Biofilm Analyses

On day 21 of each intraoral phase and 12 hours after the last meal and oral hygiene procedure, two of the four gauzes were removed with #15 surgical blades in order to analyze the total cultivable flora (TF), mutans streptococci (MS) counting and to calculate the percentage of mutans streptococci (% MS). Gauzes containing biofilm were weighed in pre-weighed test tubes containing 1 mL of sterilized 0.9% NaCl solution. The test tubes were homogenized in a vortex mixer and sonicated (Vibra Cell 400W, Sonics and Materials, Inc, Danbury, CT, USA) six times in 9.9-second bursts. The extract was then serially diluted in 0.9% NaCl solution (1:1 to $1:10^5$) and automatically plated in duplicate (Spiral Plater, DW Scientific Ltd, West Yorkshire, England), in mitis salivarius agar (MSB) containing sucrose and bacitracin (Gold, Jordan & Van Houte, 1973) to determine MS and blood agar base (5% sheep's blood) to estimate TF. The plates inoculated in MSB were kept at 37°C for 48 hours at 10% CO_2 and the plates inoculated in blood agar base were kept at 37°C for 48 hours at 10% CO_2 and 37°C for an additional 24 hours in an aerobic environment (IG 150, Jouan, Saint Herblain, France). The gauze containing the material not extracted by the saline solution was cleaned with NaOH M solution, dried and weighed to obtain the difference in the wet weight of biofilm formed.

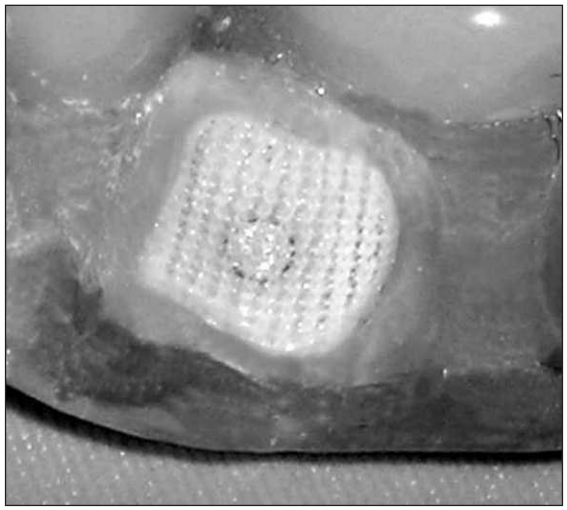


Figure 2. *Close up view of the testing specimens covered in a gauze and positioned in a removable partial prosthesis.*

For F and Zn analyses, the remaining gauze was placed in pre-weighed microcentrifuge tubes and dried in a vacuum for 24 hours over P_2O_5. To each tube, 0.5 M HCl was added in the proportion of 0.1 mL/1.0 mg dry weight, and after three hours at room temperature under constant agitation, the same volume of TISAB II (20 g NaOH/L) was added to the tube as a buffer (Cury & others, 2000). The suspension was centrifuged (11,000 x g) for one minute and the supernatant retained for determination of acid-soluble fluoride and zinc. Fluoride was analyzed with ion selective electrode Orion 96-09 using an ion analyzer (Orion EA-940, Boston, MA, USA) (Cury & others, 2000). The Zn concentration was determined by atomic absorption with an espectrophotometer (Varian, Mulgrave Victoria, Australia), using nitrous oxide, acetylene flame and a hollow cathode lamp at 213.9 nm. The spectrophotometer was calibrated with five standard solutions ranging

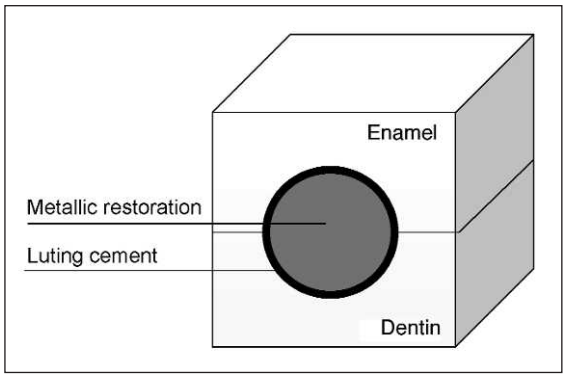


Figure 3. *Representation of a sample with an indirect metallic restoration.*

from 0.1 to 1.6 µg Zn/mL and all samples were analyzed straight, with no pre-treatment procedure. The results were expressed in µg/g of dry weight of biofilm and the net weight of biofilm was obtained as described before for the microbiological analysis.

Enamel-dentin Demineralization Assessment

Each slab of restored enamel-dentin was sectioned in two segments with a longitudinal cut through one of the extremes of the restoration. Then, the section containing the restoration was embedded in acrylic resin and polished sequentially using 320, 400, 600 and 1,200 µm grit silicon carbide paper discs, followed by 1 µm diamond suspension with the respective polishing cloth.

A Shimadzu HMV-2000 tester (Shimadzu Corp, Kyoto, Japan) with a Knoop diamond under a 25-g or 5-g load, respectively, for enamel or dentin, was used for five seconds for the microhardness analysis. Indentations were made considering the distance from the edge of the metallic restoration and the depth from the anatomical surface of the enamel and root dentin as illustrated in Figure 4.

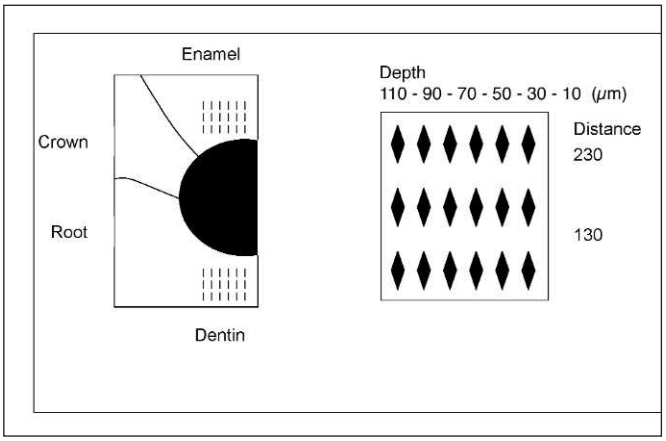


Figure 4. *Representation of the indentations made on enamel and dentin around the restoration. Indentations were made at six depths from the outer anatomical surface of enamel and dentin (10, 30, 50, 70, 90 and 110 µm), and at three distances (30, 130 and 230 µm) from the edge of the metallic restoration.*

Statistical Analysis

The assumptions of equality of variances and normal distribution of errors were checked with Bartlett, Shapiro Wilks and Kolmogorov-Smirnov tests for the response variables data. The assumptions were satisfied for F and microhardness, and two-way analysis of variance (ANOVA) was made, followed by Tukey test, when applied. Since the assumptions were not satisfied for the bacterial counts and Zn, the two-way Friedman non-parametric test was used for these data. The analyses were performed with SAS System 6.11 software (SAS Institute, Inc) and the significance limit was set at 5%.

RESULTS

Table 1 shows the results of the statistical analysis. The effect of the luting cements on total cultivable flora, mutans streptococci counts and percentage of mutans streptococci on the biofilm formed was not statistically significant, but with regard to the concentration of F and Zn in the biofilm, the effect was statistically significant (Table 1). Table 2 shows that the concentration of F in the biofilm formed on the Gl group was highest and statistically different ($p<0.05$) compared to that formed in the ZP group. Table 2 also shows that the Zn concentration in the biofilm formed on metallic restorations cemented with zinc phosphate was statistically higher than that found in the other conditions evaluated.

With regard to enamel and dentin demineralization (Table 1), the effect of materials was not statistically significant ($p>0.05$). A higher average of microhardness was observed either in enamel or dentin around the slabs cemented with glass ionomer compared to those with the other materials, as showed in Table 3, but differences among treatments were not statistically significant ($p>0.05$).

Table 1: *Statistical Results (p-values)*

	Response Variables						
Factors and Interactions	TF[a]	MS[b]	%MS[c]	F	Zn	Enamel MH[d]	Dentin MH
Materials	0.73	0.92	0.92	<0.001	<0.001	0.757	0.178
Distance	[e]-	-	-	-	-	0.011	0.218
Depth	-	-	-	-	-	<0.001	<0.001
Materials* Distance	-	-	-	-	-	<0.001	0.962
Materials* Depth	-	-	-	-	-	0.8434	0.786
Mat* Dist* Depth	-	-	-	-	-	0.984	0.997

[a] *TF = Total cultivable flora in biofilm*

[b] *MS = Mutans streptococci in biofilm*

[c] *%MS = MS/TF*

[d] *MH = Microhardness*

[e] *- not applicable*

DISCUSSION

Caries risk due to chronic diseases and pharmacotherapies grows with the increase in the life expectancy. However, the elderly population wishes for more conservative dental treatments (Ettinger & others, 1998), such as restorative procedures with fixed prosthesis, which depends on the longevity of the material used as luting cement (Jokstad & Mijor, 1996).

The three major groups of luting materials used in dental practice are: zinc phosphate, resin-modified glass ionomer and resinous cement, which show different properties and advantages. Zinc phosphate is widely used due to its longevity, resin-modified glass ionomer, because it contains fluoride and chemically bonds to tooth structure, while resinous cement is used for its strength and chemical attachment to tooth structure through a bonding agent (Christensen, 1997).

In this study, the fluoride-releasing property of the ionomeric material was shown, since the highest concentration of F was found in the biofilm formed on the GI group (Table 2). The fluoride concentration in biofilm formed on the other conditions studied was also high and can be explained by the fact that the volunteers used fluoride toothpaste. Today, the use of fluoride toothpaste by the population is routine, and the patient can have caries protection independent of the anticariogenic property of the dental material used. In fact, the counts of mutans streptococci in the biofilm formed on the ionomeric material was not statistically different from that formed on the resinous material, which does not release fluoride. These data do not agree with publications where ionomeric materials reduce mutans in dental biofilm (Sëppa, Salmenkivi & Forss, 1992; Benelli & others, 1993), but this can be explained by the small area of luting material exposed or by the fact that, in this study, the volunteers regularly used fluoride dentifrice.

Zinc is a recognized antibacterial substance (Izaguirre-Fernández & others, 1989), and a higher concentration was found in the biofilm formed on the dental slabs of the ZP group (Table 2). It seems that this is the first publication which shows that Zn is released from the cement, but the concentration was not enough to change the level of mutans in the dental biofilm. One possible reason for these results is that the pellicle of this luting cement had too small an area, which was not enough to release an antibacterial concentration of Zn.

In this study, the enamel demineralization evaluated by microhardness did not indicate higher property of any material in preventing demineralization on enamel and dentin. These results agree with White, Furuichi and Kyomen (1995) and Shinkai and others (2001), and there was no evidence relative to the influence of the luting cement on the longevity of indirect restorations *in vivo* when fluoride-containing toothpaste was used (Jokstad & Mjör, 1996). However, such results are not in agreement with some studies which demonstrated that microinfiltration values could be influenced by the luting cements (White & others, 1994; Ettinger & others, 1998). Such differences could be explained by the influence of embedded resin and tooth intrinsic factors, while it is possible that the method of testing the demineralization (microhardness) of this study may not have been sensitive enough to measure differences in demineralization. Also, Ettinger and others (1998) studied conventional glass ionomers, while White and others (1994) studied an adhesive composite resin-glass ionomer hybrid luting agent, which has different mechanical properties. Furthermore, samples containing the indirect restorations of these studies were not exposed to fluoride-containing toothpaste.

Different results were also found in *in situ* and *in vivo* studies of secondary caries around direct restorations of conventional glass ionomer (Serra & Cury, 1992; Benelli & others, 1993; ten Cate, Buijs & Damen, 1995) and resin-modified glass ionomers (Donly & others, 1999; Kotsanos, 2001). These different findings could be explained by the fact that in such studies, direct restorations were evaluated, where a larger area of the materials was exposed and no fluoride-containing toothpaste was used.

In spite of fluoride release, resin-modified glass ionomer was not effective in influencing Knoop hardness. Similar results were found by Chung and others (1998), where fluoride released by the same material, when used as an

Table 2: *Concentration of Fluoride and Zinc in Dental Biofilm (Mean ± SD; n=14)*

Group/Treatments	F, µg/g	Zn, µg/mg
Zinc Phosphate	350.4 ± 350.2[A]	3.9 ± 6.6 [B]
Glass Ionomer	7,451.0 ± 12587.0[B]	0.6 ± 0.7 [A]
Resinous Cement	3,830.4 ± 8351.3[A]	1.2 ± 3.5 [A]

Treatments whose means are followed by distinct superscript letters differ statistically (5%).

Table 3: *Knoop Microhardness (KHN) of Enamel and Dentin Around the Restorations According to the Luting Materials (mean ± SD, n=14)*

	KHN, kg/mm²	
	Enamel	**Dentin**
Group/Treatments		
Zinc Phosphate	289.4 ± 44.2	33.1 ± 13.1
Glass Ionomer	297.8 ± 50.5	38.5 ± 13.6
Resinous Cement	296.0 ± 48.5	36.0 ± 14.6

The difference among treatments were not statistically significant (5%).

orthodontic cement, did not present any cariostatic effect when compared to resinous cement, probably due to the effect of fluoride toothpaste. Donly and others (1999) studied the influence of fluoride toothpaste on the efficacy of direct restorations *in situ*, and it was verified that resin-modified glass ionomer was more effective in preventing tooth demineralization than other materials only when a non-fluoride toothpaste was used. Furthermore, a recent *in situ* study, where volunteers wore oral lower appliances, showed that sugar can be consumed up to seven times a day if fluoride dentifrice is used, but enamel demineralization occurs after only three sugar exposures if a non-fluoridated toothpaste is used (Duggal & others, 2001).

Another explanation for the data in this research, showing no difference in the luting cement on caries reduction, could result from the fact that the volunteers' diet was of low cariogenicity. Moreover, the intraoral *in situ* model used, which placed the specimens in removable lower prosthesis, favors the saliva effect controlling caries development due to a permanent action.

CONCLUSIONS

The data showed that although the restorations cemented with resin-modified glass ionomer released F, and those cemented with zinc phosphate released Zn, they were not more effective in caries reduction than the resinous material. Considering the volunteers' regular use of fluoride dentifrice, this factor can be more relevant than the cementing material property on caries reduction.

(Received 14 October 2003)

References

Benelli EM, Serra MC, Rodrigues AL Jr & Cury JA (1993) *In situ* anticariogenic potential of glass ionomer cement *Caries Research* **27(4)** 280-284.

Christensen GJ (1997) Cements used for full crown restorations: A survey of the American Academy of Esthetic Dentistry *Journal of Esthetic Dentistry* **9(1)** 1 20-26.

Chung CK, Millett DT, Creanor SL, Gilmour WH & Foye RH (1998) Fluoride release and cariostatic ability of a compomer and a resin-modified glass ionomer cement used for orthodontic bonding *Journal of Dentistry* **26(5-6)** 533-538.

Cury JA, Rebelo MA, Del Bel Cury AA, Derbyshire MT & Tabchoury CP (2000) Biochemical composition and cariogenicity of dental plaque formed in the presence of sucrose or glucose and fructose *Caries Research* **34(6)** 491-497.

Donly KJ, Segura A, Wefel JS & Hogan MM (1999) Evaluating the effects of fluoride-releasing dental materials on adjacent interproximal caries *Journal of the American Dental Association* **130(6)** 817-825.

Duggal MS, Toumba KJ, Amaechi BT, Kowash MB & Higham SM (2001) Enamel demineralization *in situ* with various frequencies of carbohydrate consumption with and without fluoride toothpaste *Journal of Dental Research* **80(8)** 1721-1724.

Ettinger RL, Kambhu PP, Asmussen CM & Damiano PC (1998) An *in vitro* evaluation of the integrity of stainless steel crown margins cemented with different luting agents *Special Care Dentist* **18(2)** 78-83.

Featherstone JD, ten Cate JM, Shariati M & Arends J (1983) Comparison of artificial caries-like lesions by quantitative microradiography and microhardness profiles *Caries Research* **17(5)** 385-391.

Forsten L (1998) Fluoride release and uptake by glass-ionomers and related materials and its clinical effect *Biomaterials* **19(6)** 503-508.

Gold OD, Jordan HV & Van Houte J (1973) A selective medium for streptococcus mutans *Archives of Oral Biology* **18(11)** 1357-1364.

Izaguirre-Fernández EJ, Eisenberg AD & Curzon ME (1989) Interactions of zinc with fluoride on growth, glycolysis and survival of streptococcus mutans GS-5 *Caries Research* **23(1)** 18-25.

Jokstad A & Mjör JA (1996) Ten years' clinical evaluation of three luting cements *Journal of Dentistry* **24(5)** 309-315.

Kotsanos N (2001) An intraoral study of caries induced on enamel in contact with fluoride-releasing restorative materials *Caries Research* **35(3)** 200-204.

Musa A, Pearson GJ & Gelbier M (1996) *In vitro* investigation of fluoride ion release from four resin-modified glass polyalkenoate cements *Biomaterials* **17(10)** 1019-23.

Sëppa L, Salmenkivi S & Forss H (1992) Enamel and plaque fluoride following glass ionomer application *in vivo Caries Research* **26(5)** 340-344.

Serra MC & Cury JA (1992) The *in vitro* effect of glass-ionomer cement restoration on enamel subjected to a demineralization and remineralization model *Quintessence International* **23(2)** 143-147.

Shinkai RS, Cury AA & Cury JA (2001) *In vitro* evaluation of secondary caries development in enamel and root dentin around luted metallic restoration *Operative Dentistry* **26(1)** 52-59.

ten Cate JM Buijs MJ & Damen JJ (1995) The effects of GIG restorations on enamel and dentin demineralization and remineralization *Advanced Dental Research* **9(4)** 384-388.

White SN, Yu Z, Tom JF & Sangsurasak S (1994) *In vivo* microleakage of luting cements for cast crowns *Journal of Prosthetic Dentistry* **4(71)** 333-338.

White SN, Furuichi R & Kyomen SM (1995) Microleakage through dentin after crown cementation *Journal of Endodontics* **21(1)** 9-12.

©Operative Dentistry, 2004, **29-5,** 515-523

Clinical Evaluation of a Polyacid-modified Resin Composite Under Different Conditioning Methods in Primary Teeth

MD Turgut • M Tekçiçek • S Ölmez

Clinical Relevance

The clinical performance of Dyract AP was not affected by the conditioning method used in primary teeth.

SUMMARY

This study evaluated the clinical performance of a polyacid-modified resin composite, Dyract AP (De Trey/Dentsply), under different conditioning methods in primary teeth. Eighty-one box-shaped cavity preparations (three restorations per patient) on the mesial or distal surfaces of primary first or second molars were prepared in 27 patients. The cavity preparations were either non pre-treated or pre-treated with 36% phosphoric acid or NRC (De Trey/Dentsply). The teeth were restored with Prime & Bond NT (De Trey/Dentsply) and Dyract AP. The restorations were evaluated at baseline and 6, 12 and 18-month recalls according to the modified Ryge criteria by two calibrated operators. The data obtained from the clinical assessment of all restorations were subjected to statistical analysis by chi-square-tests at a 0.05 level of significance. The ratings of each criteria were compared among each evaluation period between treatment groups. Statistical analysis revealed no significant difference among each evaluation period between groups in regard to color match, marginal discoloration, marginal adaptation, secondary caries, surface texture, anatomic form and retention.

*Melek D Turgut, DDS, PhD, research assistant, Department of Pediatric Dentistry, Faculty of Dentistry, Hacettepe University, Ankara, Turkey

Meryem Tekçiçek, DDS, PhD, associate professor, Department of Pediatric Dentistry, Faculty of Dentistry, Hacettepe University, Ankara, Turkey

Seval Ölmez, DDS, PhD, associate professor, Department of Pediatric Dentistry, Faculty of Dentistry, Hacettepe University, Ankara, Turkey

*Reprint request: Kibrıs Caddesi, Deniz Apartmani, B Blok No: 9/5, 06600, Kurtulus, Ankara, Turkey; e-mail: melekturgut@hotmail.com

INTRODUCTION

Polyacid-modified resin composites, commonly called compomers, have been the material of choice for the restoration of primary anterior and posterior teeth. These materials can be used to restore early childhood caries and Class I, II, III and V lesions (García-Godoy, 2000).

Polyacid-modified resin composites were developed to combine the mechanical properties of resin composites with the fluoride characteristic of glass ionomers. They are essentially resin-based materials and contain acid-modified monomers and basic glass fillers. The initial setting of the material is achieved by light curing and only a limited degree of acid/base reaction occurs. Ion-

exchange adhesion never occurs, as in conventional glass ionomers (Mount, 1999; Lutz, 1996).

Esthetic stability and wear resistance of polyacid-modified resin composites are inferior to resin composites (Gross, Griffen & Casamassimo, 2001). However, these materials are suitable to primary teeth compared to resin composites, since primary teeth have a limited life span that reduces the demands on wear resistance and are less wear resistant than permanent teeth (Gross & others, 2001).

Many new restorative materials have been introduced to the dental market as new versions are developed. Short-term laboratory studies provide some information about the physical properties of new restorative materials. Long-term clinical studies, however, complement these studies and provide further information regarding the performance of these materials over an acceptable time period and their cost-effectiveness (Knibbs, 1997).

In all resin-based materials, acid etching has been a standard procedure prior to the application of enamel and dentin bonding agents. However, the manufacturers of polyacid-modified resin composites, including Dyract AP (De Trey/Dentsply), suggest that these materials can be used without a phosphoric acid pre-treatment.

A number of clinical studies have been conducted using Dyract in primary molars without acid pre-treatment (Gross & others, 2001; Papagiannoulis & others, 1999; Marks & others, 1999; Mass, Gordon & Fuks, 1999; Welbury & others, 2000; Andersson-Wenckert, Folkesson & van Dijken, 1997). To date, no clinical study has been reported regarding the clinical performance of polyacid-modified resin composites with and without acid pre-treatment in primary teeth.

This study investigated the clinical performance of a polyacid-modified resin composite, Dyract AP, under different conditioning methods in primary teeth.

METHODS AND MATERIALS

Twenty-seven children, 5 to 8 years of age, participated in the study. Selection criteria included three primary first and/or second molars with clinically and radiographically detected caries extending into dentin. All selected teeth had occlusal and proximal contacts and a predicted survival of at least two years until exfoliation. The purpose and clinical procedure of the study were explained and a written, signed informed consent obtained from the children's parents.

Prior to the onset of the study, each treatment group was randomly assigned to teeth requiring treatment. No more than three restorations were placed per patient to distribute each treatment group equally in each child. A total of 81 lesions, 44 in first and 37 in second primary molars, were treated. Two operators performed all restorations. Prior to the start of the study, a consensus evaluation was obtained between the operators for inter-operator calibration regarding the cavity preparations and restorative procedures.

Table 1: *Direct Clinical Evaluation Criteria*

Criteria	Modified Ryge Criteria
Retention	A. No loss of the restoration B. Partial loss of the restoration C. Total loss of the restoration
Color-match	A. The restoration matches the adjacent tooth structure in color, shade and translucency B. There is a light mismatch in color, shade and/or translucency but within the normal range of adjacent tooth structure C. There is a mismatch in color, shade and/or translucency outside the normal range of adjacent tooth structure
Marginal Discoloration	A. There is no discoloration anywhere along the margin between the restoration and the tooth structure B. There is slight discoloration along the margin between the restoration and the tooth structure but the discoloration has not penetrated along the margin in a pulpal direction C. The discoloration has penetrated along the margin in a pulpal direction
Secondary Caries	A. No evidence of caries B. Evidence of caries along the margin
Marginal Adaptation	A. There is no visible evidence of a crevice along the margin B. There is a visible evidence of crevice along the margin into which the explorer will penetrate or catch C. There is a visible evidence of crevice along the margin into which the explorer will penetrate. The dentin is exposed D. The restoration is fractured mobile or missing, either in part or total
Surface Texture	A. The restoration surface is as smooth as the adjacent enamel B. The restoration surface is rougher than the adjacent enamel C. There is a crevice and fracture on the surface of the restoration
Anatomic Form	A. The restoration is continuous with existing anatomic form B. The restoration is discontinuous with existing anatomic form, but the material is not sufficient to expose dentin C. Sufficient material is lost to expose dentin

The teeth were prepared using a high-speed handpiece with air and water spray and a #835 fissure bur (Diatech Dental AG, Heerbrugg, Switzerland). Cavity preparations were limited to caries removal, and box-shaped cavity preparations were prepared on the distal or mesial surfaces of primary teeth. The teeth with caries extending down the gingiva were not included in the study, and the gingival wall of all cavity preparations was above the gingiva. No lining cement was placed. Cotton rolls and saliva ejectors were used to isolate the working field. In Group I, the preparations were acid etched with 36% phosphoric acid for 30 seconds starting at the enamel margins (García-Godoy, 2000), rinsed with water for 20 seconds and gently dried with cotton pellets to leave the surfaces wet (Ferrari & others, 1998). Prime & Bond NT (De Trey/Dentsply, Konstanz, Germany) was applied to the wet surfaces with a brush, left undisturbed for 20 seconds and the excess solvent was removed with a gentle stream of air and light cured with a visible light unit (Translux EC, Kulzer, Germany) emitting 500mW/cm^2 light intensity for 20 seconds. The curing light intensity of the light unit was verified throughout the study before each use. Dyract AP (De Trey/Dentsply) was placed in 2-mm increments, with each increment light cured for 40 seconds. In Group II, instead of 36% phosphoric acid etching, a non-rinse conditioner (NRC, a self-priming conditioning liquid with organic acidic components that co-polymerize with the subsequently applied bonding agent) was applied to the preparations, left undisturbed for 20 seconds and gently air-dried. The teeth were then restored with Prime & Bond NT and Dyract AP as in the first group. In Group III, the preparations were restored with Prime & Bond NT and Dyract AP without any pre-treatment.

Following removal of the matrix band and wedge, each restoration was light cured for an additional 40 seconds. All restorations were finished with 12-fluted finishing burs (Diatech Dental AG, Heerbrugg) under water spray and polished with Sof-Lex disks (3M Dental Products, St Paul, MN, USA).

Each restoration was assessed for retention, color-match, cavosurface margin discoloration, secondary caries, marginal adaptation, surface texture and anatomical form using modified Ryge criteria (Ryge, 1980) as detailed in Table 1. A (alpha) indicates the clinically ideal restoration. B (bravo) is a clinically acceptable situation except for secondary caries. C (charlie) and D (delta) indicate clinically unacceptable restorations that must be replaced. All evaluations were carried out under normal clinical conditions with a dental operating light, a mouth mirror and a dental explorer. The evaluation of all restorations was carried out at baseline (one week after placement) and after 6, 12 and 18 months for inter-examiner reliability testing by two authors as a blind test without knowledge about the restorative group under examination. The intra-examiner reliability was analyzed with an interval of seven days in 10 children selected at different recall appointments. The intra- and inter-examiner reliabilities were analyzed with Kappa test.

After six-month recall, one patient failed to keep appointments. Therefore, 26 patients were available for 12-month recall. A total of 24 patients were evaluated at 18-month recall. Two patients withdrew from the study because one lost three restorations and the other patient failed to keep the 18-month recall appointment. The data obtained from the clinical assessment of all restorations were subjected to statistical analysis by chi-square-tests at a 0.05 level of significance. If differences in scores between the two evaluators occurred, the worse score was registered. The ratings of each criteria were compared among each evaluation period between treatment groups.

RESULTS

The Kappa values for inter-examiner and intra-examiner agreements were 0.80 and 0.84, respectively. Statistical analysis revealed no significant differences among groups at all recalls in regard to retention, color match, marginal discoloration, secondary caries, marginal adaptation, surface texture and anatomic form (Tables 2 and 3).

Retention

In Group I, 7%, 11.5% and 12.5% of the restorations were lost after 6, 12 and 18 months, respectively. In Group II, 30% of the restorations were lost after six months. Two additional teeth needed to be extracted due to acute apical abscess formation after 12 months. At 18-month recall, the total loss rate was 42% (10 restorations) and the partial loss rate was 4% (one restoration). In Group III, 18.5% of the restorations after six months and 23% of the restorations after 12 months were lost. At 18-month recall, the total loss rate was 25% (six restorations) and the partial loss rate was 4% (one restoration). As seen in Figure 1, higher amounts of restorations were lost in Groups II and III; however, no statistically significant differences were found among groups at all recalls (p=0.11, p=0.076, p= 0.148 for 6, 12 and 18 months, respectively).

Color Match

During the first year, all restorations in Groups II and III had ideal color match (alpha score). On the other hand, the rates for clinically acceptable color match (bravo score) for Group I were 8%, 13% and 14% after 6, 12 and 18 months, respectively. An increase in the rate of bravo score with respect to 12-month recall was observed in all groups at the 18-month recall. In detail, 14% for Group I, 21% for Group II and 6% for Group III (Figure 2). No statistically significant differences were found at all evaluation periods among groups

Table 2: *Percentages for the Categories Retention, Color Match, Marginal Discoloration and Secondary Caries at Baseline, 6,12 and 18-month-recall Evaluations for Groups I, II and III*

Group 1											
		Retention				**Color Match**		**Marginal Disc**		**Secondary Caries**	
	n*	A	B	C	n	A	B	A	B	A	B
Baseline	27	100	-	-	27	100	-	100	-	100	-
6 mos	27	93	-	7	25	92	8	92	8	100	-
12 mos	26	88.5	-	11.5	23	87	13	65	35	100	-
18 mos	24	87.5	-	12.5	21	86	14	57	43	90	10
Group 2											
		Retention				**Color Match**		**Marginal Disc**		**Secondary Caries**	
	n	A	B	C	n	A	B	A	B	A	B
Baseline	27	100	-	-	27	100	-	96	4	100	-
6 mos	27	70	-	30	19	100	-	89.5	10.5	100	-
12 mos	26	61.5	-	38.5	16	100	-	69	31	100	-
18 mos	24	54	4	42	14	79	21	29	71	100	-
Group 3											
		Retention				**Color Match**		**Marginal Disc**		**Secondary Caries**	
	n	A	B	C	n	A	B	A	B	A	B
Baseline	27	100	-	-	27	100	-	100	-	100	-
6 mos	27	81.5	-	18.5	22	100	-	73	27	100	-
12 mos	26	77	-	23	20	100	-	60	40	100	-
18 mos	24	71	4	25	18	94	6	50	50	83	17

p=*0.11* p=*0.184* p=*0.148*
p=*0.076* p=*0.84* p=*0.857*
p=*0.148* p=*0.414* p=*0.242* p=*0.278*

**sample size larger for retention than for other criteria because lost restorations were unavailable for evaluation.*
***p values are for 6, 12 and 18-month recalls, respectively.*

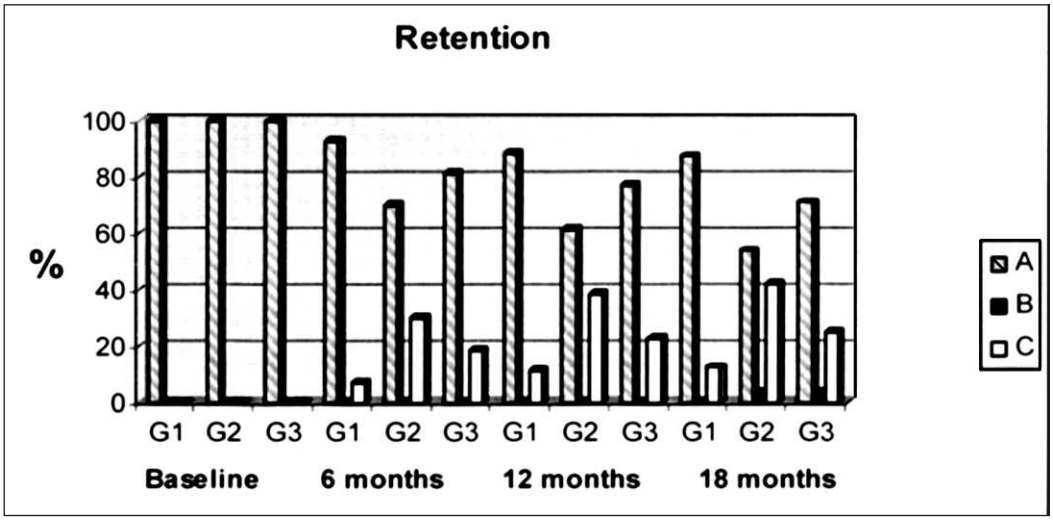


Figure 1. *Retention of Group I, II and III at baseline, 6, 12 and 18- month-recall evaluations.*

(*p*=0.184, *p*=0.84, *p*=0.414 for 6, 12 and 18 months, respectively).

Marginal Discoloration

All groups showed moderate marginal discoloration (bravo score) with a tendency to increase with time during the study. In Group I, 8%, 35% and 43%; in Group II, 10.5%, 31% and 71% and in Group III, 27%, 40% and 50% of the restorations demonstrated a bravo score after 6, 12 and 18 months, respectively (Figure 3). Statistical analysis revealed no significant differences for all evaluation periods among groups (*p*=0.148, *p*=0.857, *p*=0.242 for 6, 12 and 18 months, respectively).

Table 3: *Percentages for the Categories Marginal Adaptation, Surface Texture and Anatomic Form at Baseline, 6, 12 and 18-month-recall Evaluations for Groups I, II and III*

Group 1										
		Marginal Adaptation			**Surface Texture**			**Anatomic Form**		
	n	A	B	D	A	B	C	A	B	C
Baseline	27	100	-	-	100	-	-	100	-	-
6 mos	25	63	30	7	100	-	-	100	-	-
12 mos	23	46	42	12	96	4	-	100	-	-
18 mos	21	42	46	12	95	5	-	95	5	-
Group 2										
		Marginal Adaptation			**Surface Texture**			**Anatomic Form**		
	n	A	B	D	A	B	C	A	B	C
Baseline	27	96	4	-	100	-	-	100	-	-
6 mos	19	52	18	30	95	5	-	100	-	-
12 mos	16	31	31	38	87.5	12.5	-	94	6	-
18 mos	14	17	37	46	86	7	7	86	7	7
Group 3										
		Marginal Adaptation			**Surface Texture**			**Anatomic Form**		
	n	A	B	D	A	B	C	A	B	C
Baseline	27	100	-	-	100	-	-	100	-	-
6 mos	22	56	26	18	100	-	-	95.5	4.5	-
12 mos	20	42	35	23	90	10	-	90	10	-
18 mos	18	33	38	29	83	11	6	78	17	6
p=*0.33*		p=*0.285*		p=*0.362*						
p=*0.264*		p=*0.698*		p=*0.32*						
p=*0.117*		p=*0.725*		p=*0.514*						

***p values are for 6, 12 and 18-month recalls, respectively.*

Secondary Caries

No secondary caries was detected in all groups during the 12-month period. Of the first group, 10% and 17% of the third group of the restorations demonstrated secondary caries after 18 months (Figure 4). However, no statistically significant difference was found between groups (p=0.278).

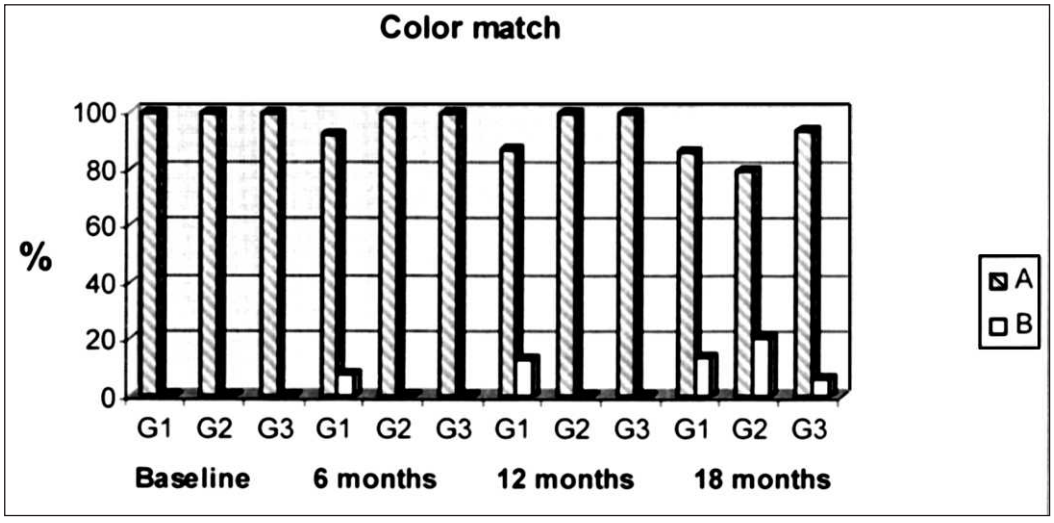


Figure 2. *Color match of Group I, II and III at baseline, 6, 12 and 18-month-recall evaluations.*

Marginal Adaptation

All groups demonstrated a gradually increasing bravo score during the study period (Figure 5). In Group I, 30%, 42% and 46%; in Group II, 18%, 31% and 37%; in Group III, 26%, 35% and 38% of the restorations were evaluated with bravo ratings after 6, 12 and 18 months, respectively. No statistically significant differences were found at all evaluation periods between groups (p=0.33, p=0.264, p=0.117 for 6, 12 and 18 months, respectively).

Surface Texture

All of the restorations in Groups I and III were as smooth as the surrounding enamel (alpha score) after

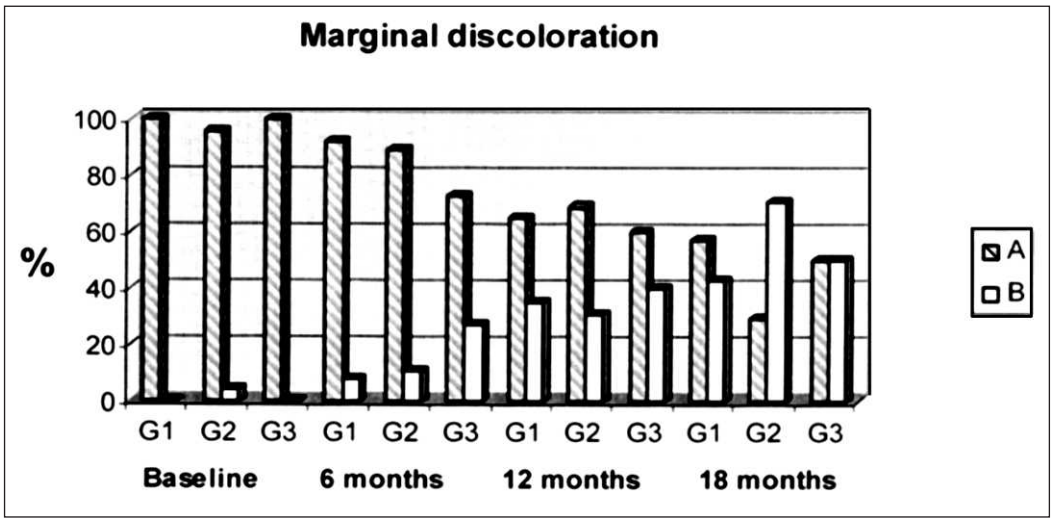


Figure 3. *Marginal discoloration of Group I, II and III at baseline, 6, 12 and 18- month-recall evaluations.*

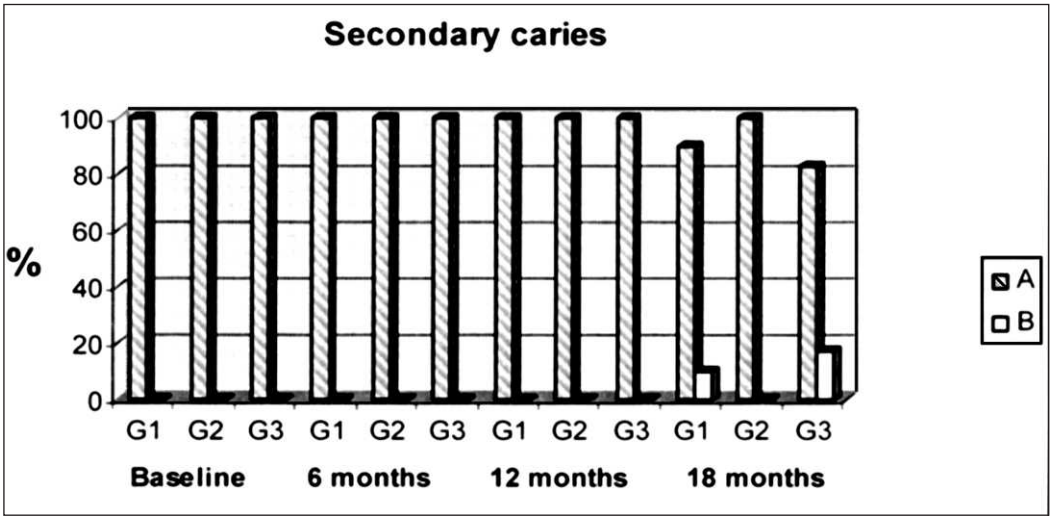


Figure 4. *Secondary caries of Group I, II and III at baseline, 6, 12 and 18-month-recall evaluations.*

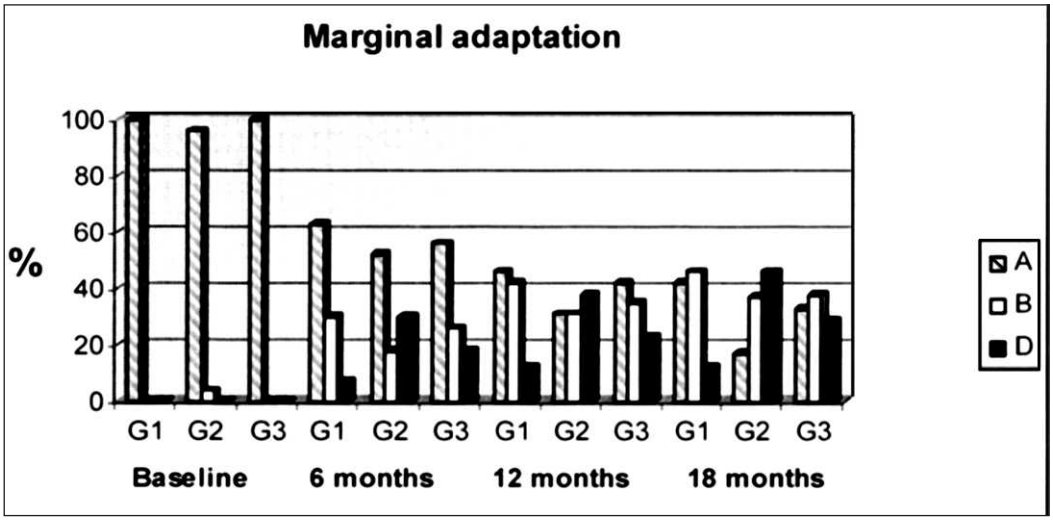


Figure 5. *Marginal adaptation of Group I, II and III at baseline, 6, 12 and 18- month-recall evaluations.*

six months. Only one restoration (5%) in Group II was rougher than the surrounding enamel (bravo score) (Figure 6). After 12 months, one restoration in Group I (4%), two restorations in Group II (12.5%) and two restorations in Group III (10%) were classified with a bravo rating. A partial fracture of one restoration in both Groups II and III were detected after 18 months, and these restorations were evaluated with a charlie rating (7% and 6% for Groups II and III, respectively). One restoration in Groups I and II (5% and 7%, respectively) and two restorations (11%) in Group III received a bravo score after 18 months. Statistical analysis revealed no significant differences between groups (p=0.285, p=0.698, p=0.725 for 6, 12 and 18 months, respectively).

Anatomic Form

All of the restorations in Group I had an excellent anatomical form after six and 12 months. Only one restoration (5%) was discontinuous with existing anatomical form after 18 months (bravo score) (Figure 7). In Group II, one restoration (7%) received a bravo score after 18 months. The fractured restorations in Groups II (7%) and III (6%) were evaluated with a charlie rating at 18-month recall. In Group III, one restoration (4.5%) after six months, two restorations (10%) after 12 months and three restorations (17%) after 18 months were classified with a bravo rating. No

statistically significant differences were found at all evaluation periods between groups (p=0.362, p=0.32, p=0.514 for 6, 12 and 18 months, respectively).

DISCUSSION

The use of an additional pre-treatment agent before application of a bonding agent is a time consuming procedure that increases the risk of saliva contamination in children. Polyacid-modified resin composites provide a better alternative than resin composites, since they claim to be used without a separate acid etching step (Attin & others, 2000). Therefore, this study aimed to investigate the clinical performance of Dyract AP restorations with and without pre-treatment agents, phosphoric acid and NRC in primary teeth. The results revealed no statistically significant differences among all treatment groups.

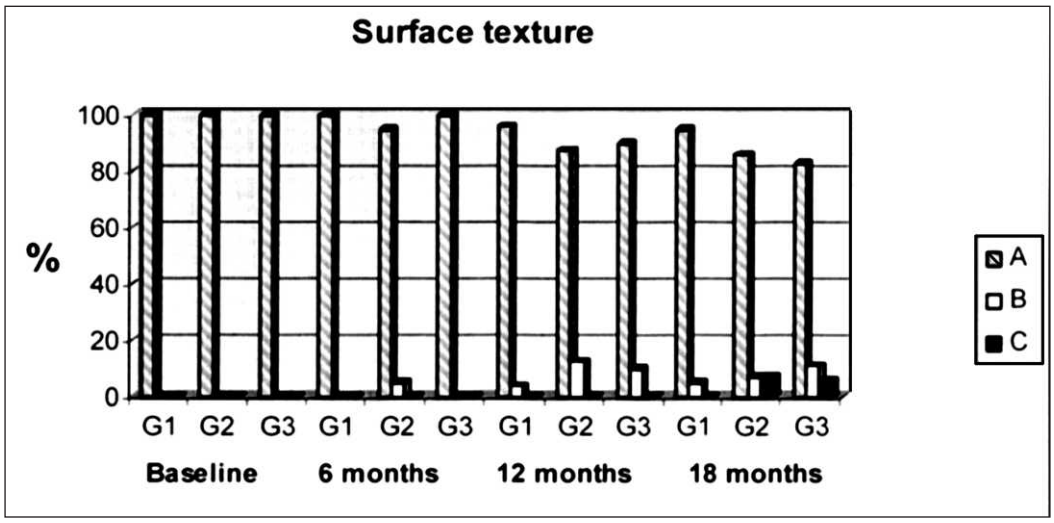


Figure 6. *Surface texture of Group I, II and III at baseline, 6, 12 and 18-month-recall evaluations.*

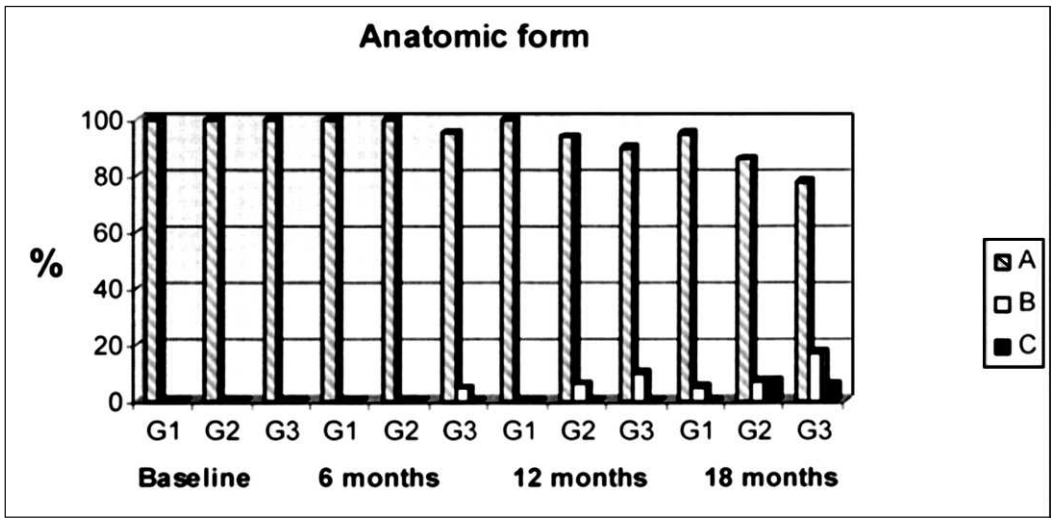


Figure 7. *Anatomic form of Group I, II and III at baseline, 6, 12 and 18-month-recall evaluations.*

The highest bond strengths of polyacid-modified resin composites to both permanent enamel and dentin were attained by using phosphoric acid in combination with bonding agents (Tate, You & Powers, 1999, 2000). However, different results were obtained from studies in which the pre-treatments, phosphoric acid and NRC, were compared with Prime & Bond NT in permanent teeth. Of these, Ferrari and others (1999) reported the unsatisfactory etching ability of Prime & Bond NT on both enamel and dentin. Luo and others (2000b) found no statistically significant effect of two pre-treatments on enamel marginal adaptation but observed positive effects on dentinal marginal adaptation of Dyract AP restorations. On the contrary, Rosa and Perdigão (2000) obtained higher enamel bond strengths with acid and NRC pre-treatments but reported higher dentinal bond strengths even with Prime & Bond NT, alone.

Prime & Bond NT contains PENTA (dipentaerythritolpentacrylate phosphoric acid), a molecule of mild acidity that behaves as a conditioning agent (Perdigão & others, 1994). Thus, it claimed to penetrate the smear layer and infiltrate dentin instead of requiring removal of the smear layer by separate acid etching (Luo & others, 2000a). Moreover, it claimed that since the hydrophilic phosphate group on the PENTA in Prime & Bond NT bonds ionically to dentinal calcium, etching the dentin could deplete the surface calcium and compromise the bond (Tyas, 1998).

Primary teeth are shown to be less mineralized and more reactive to acidic conditioners than permanent teeth. It was shown that the same treatment for dentin bonding produces a relatively thicker hybrid layer than permanent teeth (Nör & others, 1997). Therefore, it might be conflicting to extrapolate the results of the aforementioned *in vitro* studies to primary teeth.

Prime & Bond NT was found to be capable of reducing enamel microleakage as effective as phosphoric acid and NRC in primary teeth. Moreover, Prime & Bond NT alone was found to be as effective as phosphoric acid and better than NRC in reducing the dentinal microleakage of Dyract AP restorations (Turgut & others, 2001).

NRC is a self-priming conditioning liquid with two organic acids. Maleic acid acts as a conditioning agent, while itaconic acid behaves as a priming agent that copolymerizes with the subsequently applied bonding agent. The pH of NRC was reported to be higher than phosphoric acid but lower than Prime & Bond NT (1.3, 0.1 and 2.4, respectively) (Clinical Research Associates, 2000). The manufacturer suggests that the debris dissolved by NRC and left on the tooth surface copolymerizes with Prime & Bond NT (De Trey/Dentsply, 1998).

Polyacid-modified resin composites are advocated for use in Class II cavity preparations in primary teeth (García-Godoy 2000; Duke, 1999). The manufacturer suggests the use of Dyract/Dyract AP without a pre-treatment in non-stress bearing areas. Another suggestion by the manufacturer is the usage of NRC instead of phosphoric acid even in stress bearing restorations. However, there have been a number of clinical studies with these materials placed into box-shaped or conventional Class II cavity preparations where the stress cannot be overlooked. Failure rates of these studies range from 2% to 20% (Gross & others, 2001; Papagiannoulis & others, 1999; Mass & others, 1999; Marks & others, 1999; Andersson-Wenckert & others, 1997). In this study, therefore, all the above treatment modalities were included. The main cause of failure rate was found to result from the loss of restorations. The rates of restoration loss were 12.5% for Group I, 42% for Group II (partial loss 4%) and 25% for Group III (partial loss 4%). While a relatively lesser restoration failed in the phosphoric acid etched group (Group I) for all evaluation periods, no statistically significant differences were found among all groups.

The retention of the restorations depends on the bond between the restorative material and tooth structure. Cavity design and the use of a rubber dam influence retention, as well. Lower failure rates were reported in clinical studies when compomer restorations were placed to conventional or mechanically-retentive cavity preparations with the use of rubber dam in primary teeth (Gross & others, 2001; Papagiannoulis & others, 1999; Mass & others, 1999). Andersson-Wenckert and others (1997) reported a higher failure rate of Dyract restorations with minimal mechanical retention in primary teeth without the use of rubber dam. In contrast, Marks and others (1999) obtained low failure rates of compomer restorations with rubber dam even with non-retentive cavities in primary teeth. The increased risk of saliva contamination without the use of a rubber dam deteriorates the bond between the tooth and the restoration and may lead to restoration loss (Andersson-Wenckert & others, 1997; Feigal & others, 2000). Thus, the high failure rates obtained in this study may be attributed to the non-retentive cavity preparation associated with the absence of a rubber dam.

In addition, the marginal discoloration of restorations may be a signal of failure of the bond between the tooth and restoration interface (Tyas, 1998). In primary teeth, low percentages of marginal discoloration with rubber dam isolation were reported (Welbury & others, 2000; Papagiannoulis & others, 1999). Attin and others (2000) reported a 33% marginal discoloration rate of Compoglass restorations placed without rubber dam. The marginal discoloration in this study was found to be higher than these studies (50%, 71% and 50% for Groups I, II and III, respectively). Whether the NRC group showed a greater marginal discoloration rate than the other groups, no statistically significant difference was found among all groups.

Low rates of secondary caries were reported with Dyract restorations in primary teeth (Marks & others, 1999; Welbury & others, 2000; Mass & others, 1999; Papagiannoulis & others 1999). In this study, no secondary caries was detected throughout the first 12-month period. Only two restorations in Group I and three restorations in Group III exhibited secondary caries at 18-month recalls. Therefore, no relationship was found between tooth loss and secondary caries. However, radiographs were not included in this study with respect to x-ray load in children; thus, the status of the gingival margin could not be evaluated perfectly.

Dyract AP suggested exhibiting better surface performance, greater wear resistance, hardness and strength than its predecessor, Dyract, due to its modified organic matrix by adding cross-linking monomer and finer filler particles (Luo & others, 2000a). Excellent anatomic form, surface texture and color-match were reported in clinical studies both with Dyract or Dyract AP in both permanent teeth (Çehreli & Altay, 2000; Luo & others, 2000a; Ermis, 2002; Folwaczny & others, 2000; Demirci, Ersev & Üçok, 2002; Tyas, 1998; 2000) and primary teeth (Marks & others, 1999; Welbury & others, 2000; Mass & others, 1999; Papagiannoulis & others 1999).

The results of this study demonstrated no significant effect of phosphoric acid or NRC pre-treatment on the clinical success of Dyract AP restorations in primary teeth. Di Lenarda, Cadenaro and De Stefano Dorigo (2000) found no difference in the retention rates of Dyract applied with and without acid pre-treatment after 48 months in permanent teeth. They reported significant differences in both marginal adaptation loss and marginal discoloration between two groups during this period and concluded that marginal adaptation of Dyract is enhanced by acid pre-treatment. However, no

clinical study in primary teeth with Dyract or Dyract AP included a separate acid etching step.

CONCLUSIONS

Within the limits of this study, it can be concluded that NRC or phosphoric acid pre-treatments had no effect on the clinical success of Dyract restorations in primary teeth. However, long-term clinical follow-up is mandatory to evaluate the cumulative failure or success rates.

(Received 21 October 2003)

References

Andersson-Wenckert IE, Folkesson UH & van Dijken JW (1997) Durability of a polyacid-modified composite resin (compomer) in primary molars. A multicenter study *Acta Odontologica Scandinavica* **55(4)** 255-260.

Attin T, Opatowski A, Meyer C, Zingg-Meyer B & Monting JS (2000) Class II restorations with a polyacid-modified composite resin in primary molars placed in a dental practice: Results of a two-year clinical evaluation *Operative Dentistry* **25(4)** 259-264.

Clinical Research Associates (2000) Enamel-dentin adhesives, self-etching primers *CRA Newsletter* **24(11)** 1-3.

Çehreli ZC & Altay N (2000) Three-year clinical evaluation of a polyacid-modified resin composite in minimally invasive occlusal cavities *Journal of Dentistry* **28(2)** 117-122.

Demirci M, Ersev H & Üçok M (2002) Clinical evaluation of a polyacid-modified resin composite (Dyract) in Class III cavities: Three year results *Operative Dentistry* **27(3)** 223-230.

De Trey/Dentsply (1998) NRC Non-rinse conditioner *Technical Manual*.

Di Lenarda R, Cadenaro M & De Stefano Dorigo E (2000) Cervical compomer restorations. The role of cavity etching in a 48-month clinical evaluation *Operative Dentistry* **25(5)** 382-387.

Duke ES (1999) From composite resins to compomers: What have we gained? *Compendium* **20(1)** 34-37.

Ermis B (2002) Two-year clinical evaluation of four polyacid-modified resin composites and a resin-modified glass-ionomer cement in Class V lesions *Quintessence International* **33(7)** 542-548.

Feigal RJ, Musherure P, Gillespie B, Levy-Polack M, Quelhas I & Hebling J (2000) Improved sealant retention with bonding agents: A clinical study of two-bottle and single bottle systems *Journal of Dental Research* **79(11)** 1850-1856.

Ferrari M, Vichi A, Mannocci F & Davidson CL (1998) Sealing ability of two "compomers" applied with and without phosphoric acid treatment for Class V restorations *in vivo Journal of Prosthetic Dentistry* **79(2)** 131-135.

Ferrari M, Mannocci F, Kugel G & García-Godoy F (1999) Standardized microscopic evaluation of the bonding mechanism of NRC/Prime & Bond NT *American Journal of Dentistry* **12(2)** 77-83.

Folwaczny M, Loher C, Mehl A, Kunzelmann KH & Hinkel R (2000) Tooth-colored filling materials for the restoration of cervical lesions: A 24-month follow-up study *Operative Dentistry* **25(4)** 251-258.

García-Godoy F (2000) Resin-based composites and compomers in primary molars *Dental Clinics of North America* **44(3)** 541-570.

Gross LC, Griffen AL & Casamassimo PS (2001) Compomers as Class II restorations in primary molars *Pediatric Dentistry* **23(1)** 24-27.

Knibbs PJ (1997) Methods of clinical evaluation of dental restorative materials *Journal of Oral Rehabilitation* **24(2)** 109-123.

Luo Y, Lo EC, Fang DT & Wei SH (2000a) Clinical evaluation of polyacid-modified resin composite posterior restorations: One-year results *Quintessence International* **31(9)** 630-636.

Luo Y, Tay FR, Lo EC & Wei SH (2000b) Marginal adaptation of a new compomer under different conditioning methods *Journal of Dentistry* **28(7)** 495-500.

Lutz F (1996) State of the art of tooth-colored restoratives *Operative Dentistry* **21(6)** 237-248.

Marks LA, Weerheijm KL, van Amerongen WE, Groen HJ & Martens LC (1999) Dyract versus Tytin Class II restorations in primary molars: 36 months' evaluation *Caries Research* **33(5)** 387-392.

Mass E, Gordon M & Fuks AB (1999) Assessment of compomer proximal restorations in primary molars: A retrospective study in children *Journal of Dentistry for Children* **66(2)** 93-97.

Mount GJ (1999) Glass ionomers: A review of their current status *Operative Dentistry* **24(2)** 115-124.

Nör JE, Feigal RJ, Dennison JB & Edwards CA (1997) Dentin bonding: SEM comparison of the dentin surface in primary and permanent teeth *Pediatric Dentistry* **19(4)** 246-252.

Papagiannoulis L, Kakaboura A, Pantaleon F & Kavvadia K (1999) Clinical evaluation of a polyacid-modified resin composite (compomer) in Class II restorations of primary teeth: A two year follow-up study *Pediatric Dentistry* **21(4)** 231-234.

Perdigão J, Swift EJ Jr, Denehy GE, Wefel JS & Donly KJ (1994) *In vitro* bond strengths and SEM evaluation of dentin bonding systems to different substrates *Journal of Dental Research* **73(1)** 44-55.

Rosa BT & Perdigão J (2000) Bond strengths of non-rinsing adhesives *Quintessence International* **31(5)** 353-358.

Ryge G (1980) Clinical criteria *International Dental Journal* **30(4)** 347-358.

Tate WH, You C & Powers JM (1999) Bond strength of compomers to dentin using acidic primers *American Journal of Dentistry* **12(5)** 235-242.

Tate WH, You C & Powers JM (2000) Bond strength of compomers to human enamel *Operative Dentistry* **25(4)** 283-291.

Turgut MD, Uzami S, Tekçiçek M, Attar N & Sargon MF (2001) Microleakage of two polyacid-modified resin composites under different conditioning methods in primary teeth *Journal of Dentistry for Children* **68(5-6)** 300, 311-315.

Tyas MJ (1998) Clinical evaluation of a polyacid-modified resin composite (Compomer) *Operative Dentistry* **23(2)** 77-80.

Tyas MJ (2000) Three-year clinical evaluation of a polyacid-modified resin composite (Dyract) *Operative Dentistry* **25(3)** 152-154.

Welbury RR, Shaw AJ, Murray JJ, Gordon PH & McCabe JF (2000) Clinical evaluation of paired compomer and glass ionomer restorations in primary molars: Final results after 42 months *British Dental Journal* **189(2)** 93-97.

©Operative Dentistry, 2004, **29-5**, 524-531

Laboratory Research

Fluoride Releasing Materials: Their Anti-cariogenic Properties Tested in *In Vitro* Caries Models

LT Sá • C González-Cabezas • MA Cochran
M Fontana • BA Matis • BK Moore

Clinical Relevance

The results of this study indicate that fluoride-releasing materials alone may not prevent secondary caries in human dentin.

SUMMARY

The anti-cariogenic properties of three fluoride-releasing materials on root surfaces were evaluated using two different caries models. Standardized cavities were prepared in dentin specimens and restored with either glass-ionomer, resin-modified glass-ionomer, polyacid-modified resin composite or resin composite material. Two groups of 56 specimens were demineralized using a microbial caries model for three days, while another 56 specimens were demineralized using a chemical demineralization model for three days. Lesions around the restorations were measured with a confocal laser scanning microscope. Glass ionomers demonstrated significant anti-cariogenic properties when exposed to the chemical model. However, no significant anti-cariogenic properties were observed with the microbial caries model. In conclusion, the fluoride releasing materials showed different anti-cariogenic properties in root surfaces under the different caries models, suggesting that caution should be exercised when trying to extrapolate the results of *in vitro* studies to the clinical situation.

*Lorena Taddei Sá, DDS, MSD, Graduate Operative Department, Indiana University School of Dentistry, Indianapolis, IN, USA

Carlos González-Cabezas, DDS, MSD, PhD, assistant professor, Oral Biology, Oral Health Research Institute, Indianapolis, IN, USA

Michael A Cochran, DDS, MSD, professor, director of Graduate Operative Dentistry, Indiana University School of Dentistry, Indianapolis, IN, USA

Margherita Fontana, DDS, PhD, assistant professor, director, Microbial Caries Facility, Indiana University School of Dentistry, Indianapolis, IN, USA

Bruce A Matis, DDS, MSD, associate professor, Department of Operative Dentistry, Indiana University School of Dentistry, Indianapolis, IN, USA

B Keith Moore, PhD, professor, director of Graduate Dental Materials, Indiana University School of Dentistry, Indianapolis, IN, USA

*Reprint request: 1121 W Michigan Street, Indianapolis, Indiana 46202, USA; e-mail: lsa@iupui.edu

INTRODUCTION

Frequent restoration replacement remains an unresolved problem in the practice of dentistry (Fontana & Gonzalez-Cabezas, 2000). The cycle of re-restoration leads to larger restorations, weaker teeth and increases the potential for more complex treatment needs (Elderton & Osman, 1991). Studies on the frequency of

restoration replacement in dental offices agree that secondary caries is the primary reason given for replacement (Mjör, Moorhead & Dahl, 2000).

Restorative materials capable of releasing fluoride are used with the intention of preventing secondary caries (Swartz & others, 1976; Arends, Ruben & Dijkman, 1990; Dijkman & others, 1993; Hicks & Flaitz, 2000; Tabchoury & others, 2002). The amount of fluoride released by these products has been shown to vary among different materials (Forsten, 1995; Verbeeck & others, 1998; Williams, Billington & Pearson, 1999; Vermeersch, Leloup & Vreven, 2001). However, definitive long-term clinical evidence of the anti-cariogenic potential from these materials has been difficult to demonstrate.

Protection of enamel (Souto & Donly, 1994; Wandera, 1998) and dentin (Gilmour & others, 1993; Pereira & others, 1996; Hsu & others, 1998) around freshly placed glass ionomer restorations has been shown *in vitro* (Nagamine & others, 1997) and *in situ* (Tabchoury & others, 2002). In spite of the significant amount of *in vitro/in situ* evidence demonstrating the anticariogenic potential of fluoride releasing materials, clinical studies have shown controversial results, and most cross-sectional epidemiological studies seem to contradict the *in vitro* data (Mjör, 1996, 1997; Wilson, Burke & Mjör, 1997).

This study evaluated the *in vitro* anti-cariogenicity of different fluoride-releasing restorative materials (conventional glass ionomer, resin-modified glass ionomer and polyacid-modified resin composite) in human dentin, using a chemical caries model commonly used for *in vitro* testing of anti-cariogenic products and using a microbial caries model that more closely reproduces the oral environment.

METHODS AND MATERIALS

Experiment 1: Microbial Caries Model

One hundred and fifty recently extracted (less than three months old) human maxillary canines (stored in 0.1% thymol) were selected and had their anatomic crown removed with a water-cooled trimmer (Lapcraft's L'il Trimmer, model 4R5, Lapcraft Inc, West Olentangy, Powell, OH, USA). These teeth had been collected from private practices and stored at the Oral Health Research Institute, Indianapolis, IN, USA. All teeth selected were mature (complete apical closure) and their root surface areas showed no visible damage or signs of demineralization. The roots were mounted on acrylic plates and cut to 4 mm x 4 mm using a low speed saw (Isomet, Buehler, Lake Bluff, IL, USA). The specimens were ground and polished on their facial surfaces using a silicone polishing pad (Procter & Gamble, Cincinnati, OH, USA) and a polishing compound (Gamma micropolish alumina 3B, 0.05 µm, Buehler). Specimens were then screened and discarded if they had a thickness below 2 mm or they presented any surface irregularity.

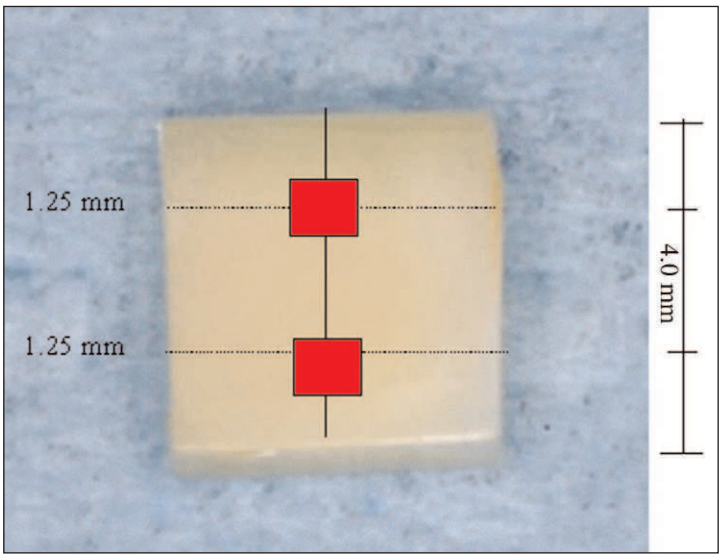


Figure 1. *Photograph of a representative specimen. Red squares illustrate the two areas scanned and used for the confocal analysis.*

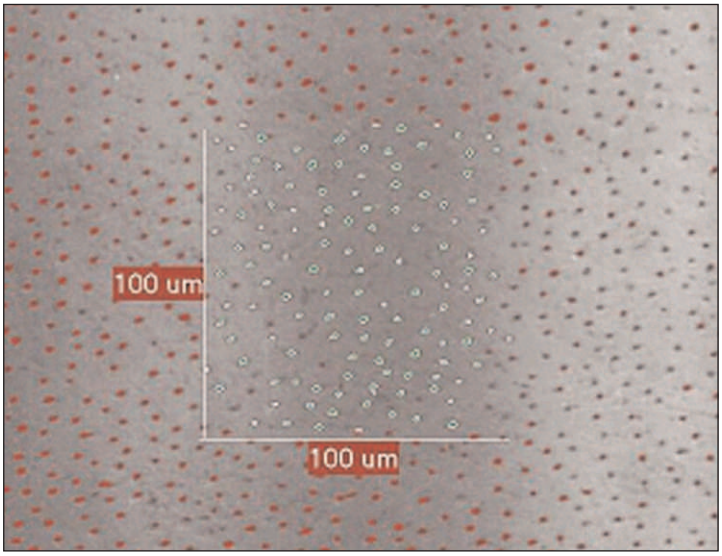
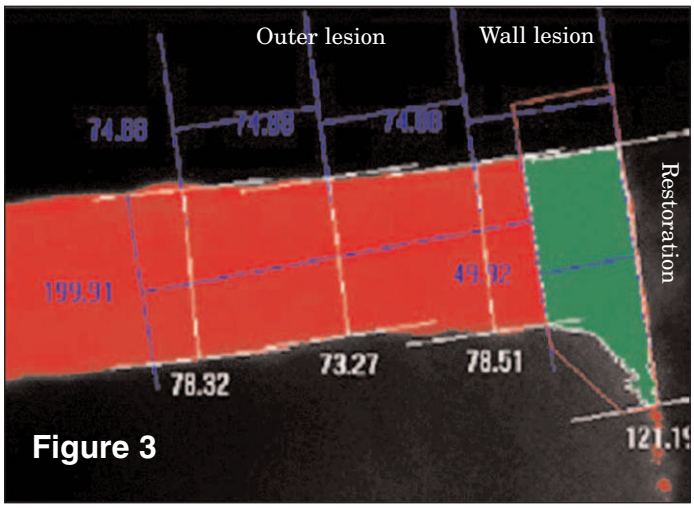
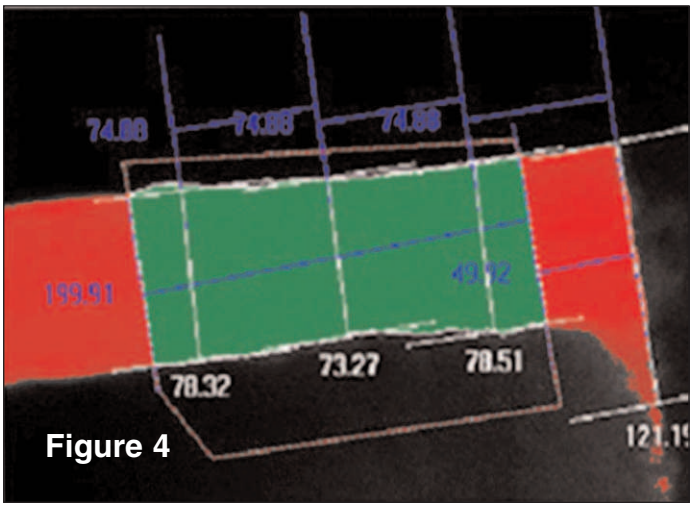


Figure 2. *Confocal micrograph of an area used for screening after imaging analysis. A gray scale threshold was determined and used to distinguish the tubules from the background. Two standard deviations higher and two lower from the average tubule size and number of tubules were taken as the range for acceptance for the study (510x, reflective signal).*

To reduce dentin tubule variability, the specimens were screened using a confocal laser scanning microscope (Odyssey Confocal Laser Microscope, Noran Instruments, Inc, W Beltline Highway Middleton, WI, USA) equipped with Image Processing and Analysis software (MetaMorph, Universal Images Corp, West Chester, PA, USA). Images from two areas (100 x 100 mm) within the specimen were acquired (Figure 1)





Figures 3 and 4. *Confocal micrographs of a lesion composed by a wall and an outer area being analyzed. The green area represents the lesion being measured (wall lesion on Figure 3, outer lesion on Figure 4). The wall lesion depth was obtained by measuring the depth at the material-dentin interface. The outer lesion depth measurements were obtained by averaging the depth measurement at 75, 150 and 225 µm from the material-dentin interface. The total area, average fluorescence and total fluorescence were obtained from the first 50 µm from the material-dentin interface (wall lesion) and from the next 200 µm (outer lesion).*

using the reflective channel. The average diameter and total number of dentinal tubules per area were obtained (Figure 2). Acceptable specimens included those with values within two standard deviations of the average tubule diameter and those with an average tubule count within each batch of specimens.

The lingual surfaces of the specimens were attached to acrylic rods, and an oblong-shaped cavity (2 mm long x 1.5 mm wide x 1.5 mm deep) was prepared using a 1159 carbide bur (Brasseler, Savannah, GA, USA) in a high-speed drill using air-water cooling on a mechanical lathe-cut machine. The specimens were observed by stereomicroscopy; those cavities with any irregularity at the margins were discarded. Four groups of 14 specimens were extracted from the remaining specimens:

Group 1: Glass ionomer cement (Ketac-Fil Plus Aplicap, 3M ESPE America, Inc, Norristown, PA, USA). The cavities were conditioned for 10 seconds (10% polyacrylic acid), rinsed, blot-dried and filled with glass ionomer. A glaze was applied (Ketac-Glaze, 3M ESPE America, Inc) and the finishing/polishing procedure was conducted in a standardized method using different grits of paper discs (Sof-Lex discs, 3M ESPE America, Inc).

Group 2: Resin-modified glass ionomer cement (Photac-Fil Plus Aplicap, 3M ESPE America Inc). The same restorative procedures as described above were conducted, after which the material was light-cured for 40 seconds. The same glaze material was applied and the restorations were finished and polished in the same way as Group 1.

Group 3: Polyacid-modified resin composite (Dyract AP, Dentsply International Inc, Milford, DE, USA). The cavities were etched for 15 seconds (37% phosphoric acid), rinsed and blot-dried. A one-bottle adhesive (Prime & Bond Universal Dental Adhesive, Dentsply International Inc) was applied and light cured for 20 seconds. The cavities were restored and the material light cured for 40 seconds. Finishing/polishing was the same as Group 1.

Group 4 (control): Resin composite (Point 4, Kerr Corporation, Orange, CA, USA). The cavities were etched for 15 seconds (37% phosphoric acid), rinsed and blot-dried. A one-bottle adhesive (OptiBond Solo Plus, Kerr Corporation) was applied and light cured for 20 seconds. The cavities were restored and the material was light cured for 40 seconds. Finishing/polishing was the same as Group 1.

An acid-resistant varnish was applied around all restoration margins leaving a 1-mm window. The specimens were gas sterilized under humid conditions and placed in the microbial caries model as described by Fontana and others (1996). Briefly, the groups were distributed equally among four vessels, inoculated with a mixed culture of Streptococcus mutans + Lactobacillus casei and exposed at 37°C to circulating trypticase soy broth with 5% sucrose three times a day and to an artificial saliva (fluoride-free mineral washing solution) for 22.5 hours per day. Total incubation time was three days. Bacteria viability, pH and lack of contamination were determined.

The specimen surfaces were then covered with resin and sectioned perpendicular to the restoration long-axis. The specimens were then stained overnight with a solution of 0.1 mM Rhodamine B (Aldrich Chem Co, Milwaukee, WI, USA) and analyzed without drying

using the confocal laser scanning microscope. Images from both sides of the restoration were obtained and analyzed with Metamorph software (MetaMorph, Universal Images Corp). Wall lesion depth was obtained by measuring the depth at the material-dentin interface (Figure 3). Outer lesion depth represented the average of three measurements (75 mm, 150 mm and 225 mm) from the material-dentin interface (Figure 4). Area, average fluorescence and total fluorescence were obtained from the first 50 mm from the material-dentin interface (wall lesion—Figure 3) and from the next 200 mm (outer lesion—Figure 4). Representative images taken from one specimen of each group in Experiment 1 are shown in Figure 5, left column.

Experiment 2: Testing Validity of Experiment 1

One hundred and thirty-five maxillary canines were selected and prepared as described in Experiment 1. To simplify the specimen preparation process, some changes were made in the procedure. Round, 4 mm-diameter specimens were prepared utilizing a customized machine. They were then ground and polished on their facial surfaces with a custom-made grinding machine using 600-grit sandpaper (Carbinet, Buehler), a custom-made rotator and a Silicone Polishing Pad (Procter & Gamble) with a polishing paste (Gamma Micropolish Alumina 3, Buehler).

The specimens had the same screening procedures, cavity preparation, restoration, demineralization in the microbial model and confocal laser scanning analysis as described in Experiment 1.

Experiment 3: Chemical Caries Model

The specimens were prepared as described in Experiment 2, gas sterilized in humid conditions and exposed to a demineralizing solution (0.1 mol/L lactic acid and 0.2 % carbopol, 50% saturated with hydroxyapatite) for 72 hours at 37°C.

Sectioning and confocal laser scanning microscope analyses were done in the same way as Experiments 1 and 2. Representative images taken from one specimen of each group in Experiment 3 are shown in Figure 5, right column.

Statistical Analysis: One-Way Analysis of Variance (ANOVA) was used to detect the existence of significant differences ($p<0.001$) among different materials in each of the parameters analyzed and Tukey test was used to find significant differences ($p<0.05$) between each pair of groups.

RESULTS

Experiment 1: Microbial Caries Model

No statistically significant differences were found among the different materials for any of the parameters measured ($p>0.001$) (Figures 6 through 8).

Experiment 2: Testing Validity of Experiment 1

The three fluoride releasing materials used did not exhibit statistically significant smaller lesions than the resin control in all the parameters measured ($p>0.001$) (Figures 6 through 8). The polyacid-modified resin composite also performed worse than the glass ionomer cement for total lesion depth ($p=0.025$).

Experiment 3: Chemical Caries Model

Glass-ionomer cements had significantly shallower total lesions and wall lesions compared to the resin control material ($p<0.05$). No significant differences were found between conventional and resin-modified glass ionomers for any of the measured parameters. Both glass ionomer materials significantly inhibited secondary caries at wall and total lesion when compared with the control material. The conventional glass ionomer showed no significant difference in the outer lesion when compared with the control ($p>0.05$).

Figure 5 shows illustrations of representative lesions from the different caries models.

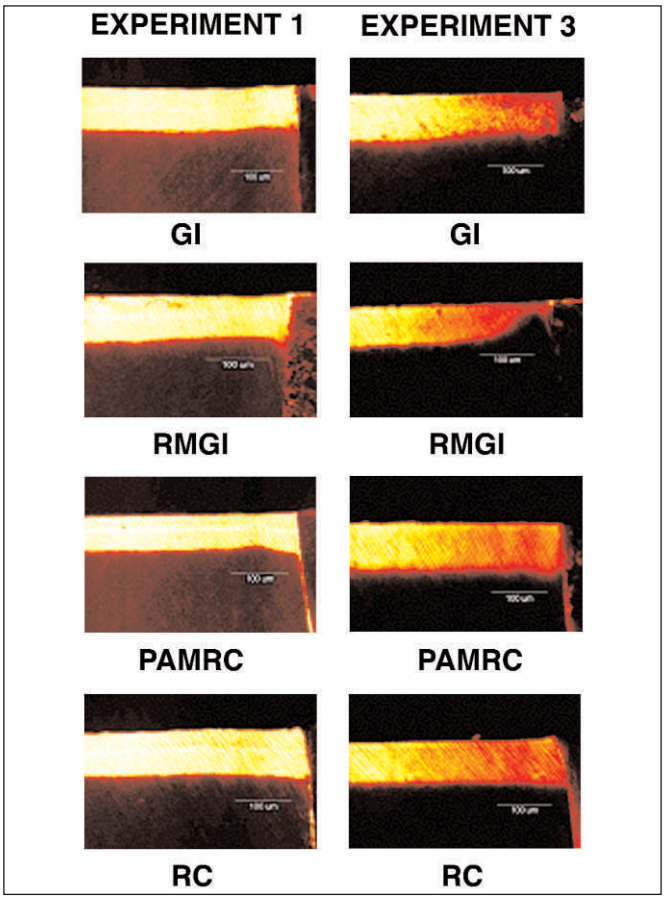


Figure 5. *Confocal micrographs of representative lesions from each of the four different materials on Experiments 1, left column and 3, right column. Inhibition zones of demineralization were observed around the glass ionomers in Experiment 3, while in Experiment 1, both glass ionomers cements presented similar lesions to the resin composite control.*

DISCUSSION

The demineralization of dentinal tissue occurs faster and in a different way from demineralization of enamel *in vitro* (Arends & others, 1992). Several studies have suggested that fluoride-releasing materials could prevent the formation of secondary caries. A study has shown that high fluoride levels are needed to inhibit dentin demineralization in a high caries challenge. In this study, the preventive properties of three fluoride-releasing materials under different *in vitro* conditions were investigated. Under a microbial caries model, fluoride-releasing materials showed no benefit when compared to the control material and showed preventive properties under a chemical caries model.

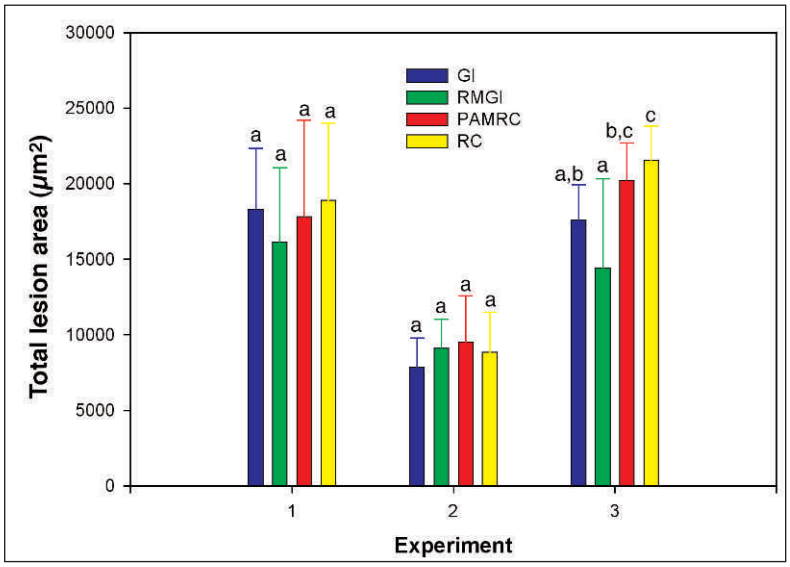


Figure 6. *Total lesion area. Significant differences are represented by different letters, and a<b<c.*

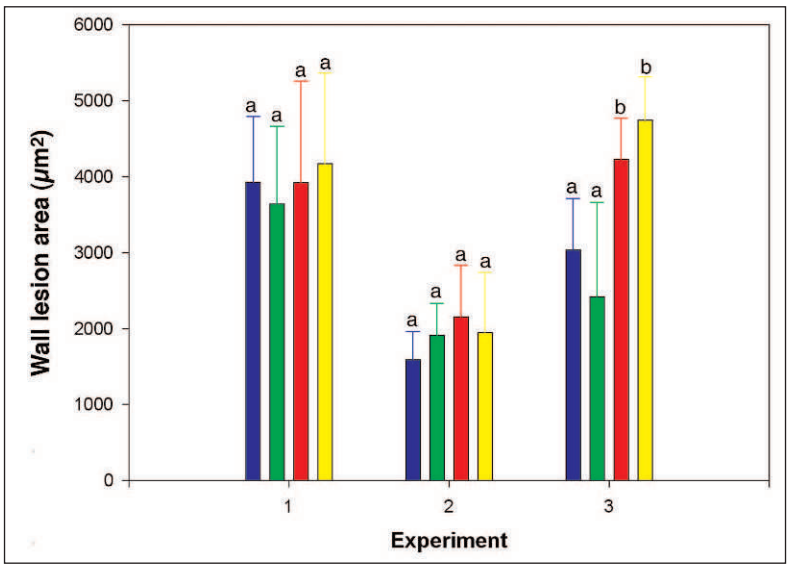


Figure 7. *Wall lesion area. Significant differences are represented by different letters, and a<b.*

Figures 6 through 8 show the results from the three experiments. Although this study measured lesion area and depth, the authors chose to display lesion area, because area is more representative of the whole lesion. The depth of the lesions is not always uniform and can have significant variability at different locations.

In the first and second experiments (microbial caries model), the authors found no statistically significant differences between fluoride releasing materials and the resin composite control. There was a trend towards the resin-modified glass ionomer group preventing more demineralization than the other three materials, but this protection was not statistically significant. It is possible that the finishing procedure that was applied and later removed from the surface of the conventional glass ionomer had a strong impact on the fluoride releasing properties of this material. A study demonstrated that glazing the surface of conventional glass ionomer cements negatively affected their short-term fluoride release, even if the glaze was removed later (McKnight-Hanes & Whitford, 1992). Finishing and glazing were accomplished according to the manufacturer's recommendations, and a second finishing procedure was done eight hours later in an attempt to remove the glaze and reproduce the wear that occurs *in vivo* within a few days. In this study, it is possible that by finishing the varnished surface eight hours after mixing, the authors removed the leachable glass particles that are responsible for short-term fluoride release (Momoi & McCabe, 1993). Despite this, the authors think that the value of the materials tested falls within the capability of preventing secondary caries from forming in the long-term. During the initial two weeks after restoration placement, the action of the fluoride released may not be significant in the restoration's life, considering that caries is a slow forming multi-factorial disease. Also, all the specimens in this study were prepared in the same way for each experiment, so if an effect also occurred in the third experiment, the authors found no differences in prevention between conventional or resin-modified glass ionomer materials. A glaze was not used on the resin-modified glass ionomer material, as its manufacturer leaves it to the operator whether or not to use the glaze material.

The significant differences in results between Experiments 1 and 2 can be attributed to the variable behavior of each experiment under a microbial model dependent on microbiologic variables (bacteria viability, reproduction and acidic production). For this reason, an independent com-

parison of material behavior between the different experiments is inappropriate. A comparison of the lesions can only be made within the same experiment, which is why the four different materials were exposed to the same environment at the same time.

The results of these studies contradict data from earlier studies that show protection from the demineralization of fluoride-releasing materials under different *in vitro* cariogenic bacterial models. Those models differed from the model used in this study in at least two important factors: clearance flow and inoculation. Nagamine and others (1997) and Torii and others (2001) inoculated the specimens with S mutans and incubated them in a sucrose-rich medium, which was replaced every three to four days. Hsu and others (1998) changed the media every 48 hours. It has been suggested that, because of the salivary clearance, the anti-caries activity of fluoride-releasing materials might be limited in the clinical situation, even when high fluoride-releasing materials are used (Torii & others, 2001). The model used in this study had a constant flow of artificial saliva with a corresponding constant clearance. Additionally, the model used a more complex inoculum composed of L casei and S mutans. Because of these factors, the authors consider this model to more closely replicate clinical conditions than the previously mentioned microbial models.

These results also contradict some available *in situ* data. ten Cate and Lagerweij (1996) found significantly shallower dentinal lesions and significantly less mineral loss around freshly restored glass ionomers than around resin composites placed on bovine specimens. Remineralization of a previously formed lesion in dentin by fluoride-releasing materials has also been shown *in situ* (Jordan & others, 1999). Some studies used bovine material (ten Cate & Lagerweij, 1996), which is known to be different and have another demineralization pattern than human dental tissue (Featherstone & Mellberg, 1981). This might partially explain the discrepancy with the results of this study.

Some of the problems resulting from using resin composite as a restorative material are related to dimensional changes that can result in the formation of gaps in the tooth-material interface. In this study, a thermocycling procedure was not done in an attempt to prevent shrinkage and avoid detrimental effects on the control material. The authors also used a bonding technique that tries to minimize difficulties in obtaining a good seal in dentinal tissue, thus avoiding dentin dehydration during the procedure. Because of this, it is possible that the control group had "ideal" restorations with less likelihood of developing secondary caries around them.

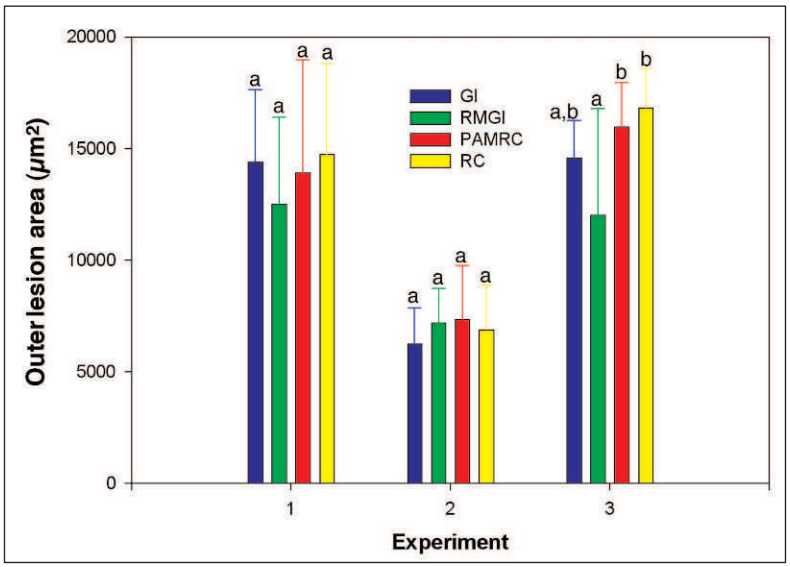


Figure 8. *Outer lesion area. Significant differences are represented by different letters, and a<b.*

In Experiment 3, the glass ionomers more effectively prevented demineralization than the resin composite control. These results are in agreement with those of previous chemical model studies in enamel (Jensen & others, 1990; Seppä, 1994; Hicks & Flaitz, 2000) and dentin (Pereira & others, 1996; Pereira, Inokoshi & Tagami, 1998). Also, the authors found no differences between conventional and resin-modified glass ionomers that were in agreement with some previous findings (Souto & Donly, 1994) but contradicted a previous study (Pereira & others, 1998). This chemical model was concluded to be more sensitive to the amount of fluoride released from the materials than the microbial model.

It is important to consider that *in vitro* studies have multiple limitations and that *in situ* and *in vivo* studies more closely replicate reality. Despite this, definitive evidence of the anti-cariogenic properties of fluoride-releasing materials has been difficult to demonstrate *in vivo*. Data from a clinical trial showed some evidence of the protection from fluoride-releasing materials on enamel demineralization (Tyas, 1991). In another study, protection from fluoride-releasing materials was found when lesions produced *in vivo* were analyzed *in vitro* (Donly & others, 1999). On the other hand, cross-sectional epidemiological studies have found that fluoride-releasing materials do not prevent secondary decay from occurring (Mjör, 1997; Wilson & others, 1997; Mjör & others, 2000).

Because of the considerable difficulties in conducting *in vivo* studies on secondary caries in dentin, there is a need for *in vitro* methods to be developed that closely reproduce the oral environment. Microbial models are considered more reliable in imitating the oral environment for the presence of bacteria and their products.

CONCLUSIONS

In this study, fluoride-releasing materials had different anti-cariogenic behaviors in dentinal tissue when subjected to different caries models. In the chemical model, a preventive effect was seen with glass ionomer cements. In the microbial model, glass ionomers did not perform significantly better than the resin composite control; therefore, no relation could be shown between the fluoride-releasing capacity of a material and its capacity to prevent recurrent decay in this model. These findings suggest that caution should be taken when trying to extrapolate the results of *in vitro* studies to the clinical situation.

(Received 2 May 2003)

References

Arends J, Ruben J & Dijkman AG (1990) Effect of fluoride release from a fluoride-containing composite resin on secondary caries: An *in vitro* study *Quintessence International* **21(8)** 671-674.

Arends J, Christoffersen J, Buskes JA & Ruben J (1992) Effects of fluoride and methanehydroxydiphosphate on enamel and on dentine demineralization *Caries Research* **26(6)** 409-417.

Dijkman GE, de Vries J, Lodding A & Arends J (1993) Long-term fluoride release of visible light-activated composites *in vitro*: A correlation with *in situ* demineralization data *Caries Research* **27(2)** 117-123.

Donly KJ, Segura A, Kanellis M & Erickson RL (1999) Clinical performance and caries inhibition of resin-modified glass ionomer cement and amalgam restorations *Journal of the American Dental Association* **130(10)** 1459-1466.

Elderton RJ & Osman YI (1991) Preventive versus restorative management of dental caries *Journal of the Dental Association of South Africa* **46(4)** 217-221.

Featherstone JD & Mellberg JR (1981) Relative rates of progress of artificial carious lesions in bovine, ovine and human enamel *Caries Research* **15(1)** 109-114.

Fontana M, Dunipace AJ, Gregory RL, Noblitt TW, Li Y, Park KK & Stookey GK (1996) An *in vitro* microbial model for studying secondary caries formation *Caries Research* **30(2)** 112-118.

Fontana M & Gonzalez-Cabezas C (2000) Secondary caries and restoration replacement: An unresolved problem *Compendium of Continuing Education in Dentistry* **21(1)** 15-18, 21-24, 26.

Forsten L (1995) Resin-modified glass ionomer cements: Fluoride release and uptake *Acta Odontologica Scandinavica* **53(4)** 222-225.

Gilmour AS, Edmunds DH, Newcombe RG & Clark MF (1993) An *in vitro* study into the effect of a bacterial artificial caries system on the enamel adjacent to composite and amalgam restorations *Caries Research* **27(3)** 169-175.

Hicks MJ & Flaitz CM (2000) Resin-modified glass-ionomer restorations and *in vitr*o secondary caries formation in coronal enamel *Quintessence International* **31(8)** 570-578.

Hsu CY, Donly KJ, Drake DR & Wefel JS (1998) Effects of aged fluoride-containing restorative materials on recurrent root caries *Journal of Dental Research* **77(2)** 418-425.

Jensen ME, Wefel JS, Triolo PT & Hammesfahr PD (1990) Effects of a fluoride-releasing fissure sealant on artificial enamel caries *American Journal of Dentistry* **3(2)** 75-78.

Jordan TH, Bradshaw R & Frahm S (1999) Effect of F Releasing restorative material on De/Remin of roots *Journal of Dental Research* **78** 124 IADR Abstract #151.

McKnight-Hanes C & Whitford GM (1992) Fluoride release from three glass ionomer materials and the effects of varnishing with or without finishing *Caries Research* **26(5)** 345-350.

Mjör IA (1996) Glass-ionomer cement restorations and secondary caries: A preliminary report *Quintessence International* **27(3)** 171-174.

Mjör IA (1997) The reasons for replacement and the age of failed restorations in general dental practice *Acta Odontologica Scandinavica* **55(1)** 58-63.

Mjör IA, Moorhead JE & Dahl JE (2000) Reasons for replacement of restorations in permanent teeth in general dental practice *International Dental Journal* **50(6)** 361-366.

Momoi Y & McCabe JF (1993) Fluoride release from light-activated glass ionomer restorative cements *Dental Materials* **9(3)** 151-154.

Nagamine M, Itota T, Torii Y, Irie M, Staninec M & Inoue K (1997) Effect of resin-modified glass ionomer cements on secondary caries *American Journal of Dentistry* **10(4)** 173-178.

Pereira PNR, Inokoshi S, Yamada T & Tagami J (1996) Artificial root caries lesions around fluoride releasing restorative materials *Journal of Dental Research* **75** IADR Abstract #1222 p 170.

Pereira PN, Inokoshi S & Tagami J (1998) *In vitro* secondary caries inhibition around fluoride releasing materials *Journal of Dentistry* **26(5-6)** 505-510.

Seppä L (1994) Fluoride release and effect on enamel softening by fluoride-treated and fluoride-untreated glass ionomer specimens *Caries Research* **28(6)** 406-408.

Souto M & Donly KJ (1994) Caries inhibition of glass ionomers *American Journal of Dentistry* **7(2)** 122-124.

Swartz ML, Phillips RW, Norman RD, Elliason S, Rhodes BF & Clark HE (1976) Addition of fluoride to pit and fissure sealants—a feasibility study *Journal of Dental Research* **55(5)** 757-771.

Tabchoury CPM, Dalcico R, Del Bel Cury AA, Rosalen P & Cury JA (2002) *In situ* anticariogenic potential of conventional and resin-modified glass ionomer cement *Journal of Dental Research* **81(32)** IADR Abstract #0026.

ten Cate JM & Lagerweij MD (1996) An *in situ* study of (Inhibition of) caries around restorations *Journal of Dental Research* **75** 233 IADR Abstract #1722.

Torii Y, Itota T, Okamoto M, Nakabo S, Nagamine M & Inoue K (2001) Inhibition of artificial secondary caries in root by fluoride-releasing restorative materials *Operative Dentistry* **26(1)** 36-43.

Tyas MJ (1991) Cariostatic effect of glass ionomer cement: A five-year clinical study *Australian Dental Journal* **36(3)** 236-239.

Verbeeck RM, De Maeyer EA, Marks LA, De Moor RJ, De Witte AM & Trimpeneers LM (1998) Fluoride release process of (resin-modified) glass-ionomer cements versus (polyacid-modified) composite resins *Biomaterials* **19(6)** 509-519.

Vermeersch G, Leloup G & Vreven J (2001) Fluoride release from glass-ionomer cements, compomers and resin composites *Journal of Oral Rehabilitation* **28(1)** 26-32.

Wandera A (1998) *In vitro* enamel effects of a resin-modified glass ionomer: Fluoride uptake and resistance to demineralization *Pediatric Dentistry* **20(7)** 411-417.

Williams JA, Billington RW & Pearson GJ (1999) The influence of sample dimensions on fluoride ion release from a glass ionomer restorative cement *Biomaterials* **20(14)** 1327-1337.

Wilson NH, Burke FJ & Mjör IA (1997) Reasons for placement and replacement of restorations of direct restorative materials by a selected group of practitioners in the United Kingdom *Quintessence International* **28(4)** 245-248.

©Operative Dentistry, 2004, **29-5,** 532-537

Influence of Interchanging Adhesive Resins and Self-etching Primers on the Mechanical Properties of Adhesive Resins

M Yamada • M Miyazaki • BK Moore

Clinical Relevance

Data suggests that the curing characteristics for adhesive resins of self-etching primer systems can be influenced when mixed with primer. In addition to the etching effect and priming ability of self-etching primers, the effect on the mechanical properties of adhesive resins should be considered.

SUMMARY

This study determined the influence of interchanging adhesive resins and self-etching primers on the mechanical properties of adhesive resins. Four commercially available two-step self-etching primer systems were used. To measure microtensile strength, 0.5-mm thick dumbbell-shaped slabs of each combination of adhesive resin and self-etching primer were prepared. After 24 hours storage in 37°C distilled water, these specimens were subjected to microtensile testing at a crosshead speed of 1.0 mm/minute. Fourier transformation infrared spectroscopy was used to determine the degree of conversion. The percentage of residual double bonds, including pendant and monomeric double bonds, was calculated by comparing the obtained ratio against uncured adhesive resin. The degree of conversion of the adhesive resins was obtained by subtracting the %C=C from 100%. Two-way ANOVAs, followed by Tukey tests, were done. The microtensile strengths and degree of conversion varied with different combinations of self-etching primer and adhesive resin. Numerically, the highest microtensile strengths were obtained when the primer/adhesive resin combinations from the same manufacturer were used. When the different combinations of self-etching primers and adhesive resins were mixed, the microtensile strength and degree of conversion of the adhesive resins tended to decrease for some combinations. Within the limitations of this study, which was far removed from clinical situations, the role of the self-etching primers on the

Mitsunori Yamada, DDS, graduate student, Nihon University School of Dentistry, Department of Operative Dentistry, Tokyo, Japan

*Masashi Miyazaki, DDS, PhD, assistant professor, Nihon University School of Dentistry, Department of Operative Dentistry and Dental Research Center, Division of Biomaterial Science, Tokyo, Japan

B Keith Moore, PhD, professor, Indiana University School of Dentistry, Department of Restorative Dentistry, Division of Dental Materials, Indianapolis IN, USA

*Reprint request: 1-8-13, Kanda-Surugadai, Chiyoda-ku, Tokyo 101-8310, Japan; e-mail: miyazaki-m@dent.nihon-u.ac.jp

mechanical properties of adhesive resins should be considered to create an authentic resin-dentin interface.

INTRODUCTION

The two-step self-etching primer system is one of the bonding systems developed to simplify and shorten bonding procedures by combining dentin conditioning and priming steps (Van Meerbeek & others, 1998; Perdigão & Lopes, 1999). A self-etching primer forms a continuous layer between the resin composite and tooth surface. This layer is simultaneously demineralized by the acidic monomers followed by adhesive resin penetration into the tooth substrate (Barkmeier, Los & Triolo, 1995; Ikemura, Kuoro & Endo, 1996; Gordan & others, 1997). The harmony of the degree of demineralization with the self-etching primer and the integrity of the resin monomer infiltration is an important key to creating high quality dentin bonding (Miyazaki, Onose & Moore, 2002). It has been reported that poor infiltration of adhesive resin into demineralized dentin leaves nano-spaces in the hybrid layer (Sano & others, 1995a; Spencer & Swafford, 1999). The existence of porosity at the base of the hybrid layer and within the adhesive resin one year after placement has been shown in an *in vivo* study (Sano & others, 1999). After infiltration of the resin monomers into the partially demineralized dentin, subsequent polymerization of monomers is required to create a stable bond. If polymerization of these monomers is not complete, hydrophilic monomers or small oligomers might be extracted or hydrolyzed by nanoleakage (Sano & others, 1999). Proper demineralization of dentin substrate, uniform resin impregnation and sufficient mechanical strength of cured adhesive resin are important factors to creating a high quality hybrid layer for good dentin bonding (Miyazaki & others, 2003).

Self-etching primers are applied to the tooth surface prior to applying the adhesive resin to ensure maximum adhesion by improving monomer penetration into the hydrophilic dentin substrate and improving wettability of the tooth surface by the adhesive resin. The self-etching primer is not rinsed off, in contrast to a conventional etchant, such as phosphoric acid. After application of the self-etching primer, the primed dentin surface is air dried, because primer contains solvents, such as water, ethanol and acetone (Miyazaki & others, 1999). These solvents may have an adverse effect on the polymerization of the adhesive resin applied after the priming step (Hotta, Kondoh & Kamemizu, 1998). The question has been raised whether the mechanical properties of adhesive resin might be adversely affected by the presence of self-etching primers, since the mechanical properties of the cured adhesive resin are some of the most important factors that determine bond strength (Takahashi & others, 2002). A combination of self-etching primer and adhesive resin, as recommended by the manufacturers, is thought to be an important factor in good clinical results. However, little is known about the influence of interchanging adhesive resins and self-etching primers on their mechanical properties.

This study examined the effect of self-etching primers on microtensile strength and the conversion ratio of adhesive resins. The null hypotheses tested was that the interchanging of self-etching primers does not influence the mechanical properties of adhesive resins or their curing ability as measured by the degree of conversion.

METHODS AND MATERIALS

Four commercial self-etching primer systems, Clearfil SE Bond (CB, Kuraray Medical, Tokyo, Japan), Imperva Fluoro Bond (FB Shofu Inc, Kyoto, Japan), Mac Bond II (MB Tokuyama Dental, Tokyo, Japan) and Unifil Bond (UB GC Corp, Tokyo, Japan) were used, as shown in Table 1. An Optilux 501 curing unit (Demetron/Kerr, Danbury, CT, USA), with light intensity above 600 mW/cm^2 as measured with a dental radiometer (Model 100, Demetron/Kerr), was used.

For measuring microtensile strength (μ-TS), the adhesive resins were put into a mold made of additional silicone impression material (Examix Fine, GC Corp, Tokyo, Japan) to make a dumbbell-shaped specimen as described by Sano and others (1995b). Glass cover slips were placed on top of the mold's surface to achieve a flat specimen surface. The adhesive resin or the mixture with the self-etching primer was irradiated for 60 seconds, removed from the mold and trimmed to eliminate flash. The thickness of the specimen was 0.5±0.05 mm and the minimum width was 0.5 mm. Five specimens of each combination of adhesive resin and primer were made at a weight ratio of 10:1.

After storage in 37°C distilled water for 24 hours, the specimens were attached to the grips of a testing apparatus using a small amount of cyanoacrylate adhesive (Aron Alpha, Toagosei Co, Tokyo, Japan) in a universal testing machine (Type 4204 Instron Corp, Canton, MA, USA) at a crosshead speed of 1.0 mm/minute. The tensile strength in MPa was calculated from the peak load at failure divided by the original cross-sectional area at the smallest section.

The degree of conversion (DC) of the adhesive resins mixed with each self-etching primer was determined by use of Fourier transformation infrared spectroscopy (FTIR, 480 Plus, JASCO Corp, Tokyo, Japan). Solutions of each combination of the primer and adhesive resin were inserted into a Teflon mold (4 mm in diameter, 1 mm in height), and the surface was covered with glass followed by 60 seconds with light exposure. The cured specimen was pulverized into a fine powder, mixed with potassium bromide powder and compacted to form a

Table 1: *Self-etching Primer Systems Tested*

Bonding System (Code)	Self-etching Primer (Lot #) Main Component	Adhesive Resin (Lot #) Main Component	Manufacturer
Clearfil Mega Bond (CB)	Primer (00135A) Water, ethanol, MDP, HEMA, CQ, N,N-diethanol p-toluidin	Bond (00088A) MDP, bis-GMA, HEMA, N,N-diethanol p-toluidin, micro filler, CQ	Kuraray Medical (Tokyo, Japan)
Fluoro Bond (FB)	FB Primer (A:060060, B:060076) A: Water, catalyst B: 4-AET, 4-AETA, HEMA acetone, catalyst	FB Bond (060070) 4-AET, HEMA, UDMA, TEGDMA, PRG-filler CQ, accelerator	Shofu Inc (Kyoto, Japan)
Mac-Bond II (MB)	Primer (A:0251, B:013) A: MAC-10, HEMA, acetone, isopropyl alcohol, phosphate monomer B: Ethanol, water	Adhesive resin (0181) MAC-10, HEMA , bis-GMA, TEGDMA, CQ	Tokuyama Dental (Tokyo, Japan)
UniFil Bond (UB)	Self-etching primer (0008281) Water, ethanol, 4-MET, HEMA	Adhesive resin (008301) TEGDMA, HEMA, UDMA, CQ	GC Corp (Tokyo, Japan)

Abbreviations: MDP: 10-methacryloxydecyl di-hydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, bis-GMA: 2, 2bis[4-(2-hydroxy-3-methacryloyloxypropoxy)]phenyl propane, CQ: dl-camphorquinone, 4-AET: 4-acryloyloxyethyl trimellitic acid, 4-AETA: 4-acryloyloxyethyl trimellitate anhydride, UDMA: urethane dimethacrylate, TEGDMA: tri-ethylene glycol di-methacrylate, MAC-10: 11-methacryloxy-1,1-undecandicarboxylic acid, 4-MET: 4-methacryloyloxyethyl trimellitate.

Table 2: *Influence of Interchanging Adhesive Resins and Primers on the Microtensile Strength of the Adhesive Resins*

	Primer				
Adhesive Resin	Control	CBP	FBP	MBP	UBP
CB	88.0 (5.7)[a]	79.4 (8.4)[a]	35.4 (8.5)[b]	39.8 (5.5)[b]	40.1 (6.3)[b]
FB	76.9 (9.8)[d]	56.7 (7.2)[d,e]	66.8 (9.1)[c,d]	49.9 (5.4)[e]	55.4 (6.0)[e]
MB	54.9 (8.0)[f]	45.8 (8.8)[f]	49.1 (9.6)[f]	50.2 (8.4)[f]	43.2 (6.5)[f]
UB	57.5 (7.4)[g]	49.0 (7.3)[g]	48.2 (9.8)[g]	46.9 (6.9)[g]	49.9 (7.3)[g]

Unit: MPa, ( ): SD, n=5
Groups in the same row with the same superscript letters are not significantly different (p>0.05).

Table 3: *Influence of Interchanging Adhesive Resins and Primers on Degree of Conversion of the Adhesive Resins*

	Primer				
Adhesive Resin	Control	CBP	FBP	MBP	UBP
CB	84.9 (1.2)[a]	82.8 (1.4)[a]	53.6 (2.2)[b]	51.1 (2.3)[b]	51.0 (2.3)[b]
FB	86.7 (1.9)[c]	58.1 (3.9)[d]	74.5 (2.3)[e]	46.2 (2.8)[f]	59.9 (3.0)[d]
MB	88.3 (1.5)[g]	72.6 (2.2)[h]	73.8 (2.8)[h]	80.1 (2.7)[i]	70.1 (2.3)[h]
UB	87.4 (1.5)[j]	81.1 (2.4)[k]	81.6 (2.4)[k]	80.4 (2.6)[k]	82.7 (2.6)[k]

Unit: %, ( ): SD, n=5
Groups in the same row with the same superscript letters are not significantly different (p>0.05).

specimen for IR spectroscopy. The IR spectra were obtained from 30 scans over the 400-4000 cm^{-1} ranges at a resolution of 2 cm^{-1}.

Measurement of the residual double bond was made on a relative basis by comparing the C=C unpolymerized methacrylate stretching vibration (1637 cm^{-1}) to that of the C=O stretching vibration of the aromatic ring (1608 cm^{-1}) for adhesive resins containing Bis-GMA or to the C-H stretching vibration of the aliphatic group (2960 cm^{-1}) for adhesive resins containing UDMA. The C=O stretching mode at 1608 cm^{-1} and C-H stretching vibration at 2960 cm^{-1} were used as internal standards for Bis-GMA-based and UDMA-based adhesive resins, respectively.

The intensity of the individual bands was obtained from the peak areas using curve fitting software (Spectra manager, JASCO Corp) designed to calculate a Lorentzian curve. In this way, the ratios of the two band areas were calculated at each measuring point and the percentage of DC was calculated by subtracting the percentage of remaining C=C from 100%. It should be noted that the percentages obtained were based on the assumption that the linearity of the ratios between uncured and cured states existed and the unreacted pendant methacrylate groups bound to the polymer network were not available to be leached as residual monomer. Five specimens for each combination of adhesive resin and primer were measured.

The results were analyzed by calculating the mean and standard deviation for each group. The data for each specimen were subjected to one-way ANOVA followed by Duncan's multiple range test at a *p*-value of 0.05. Statistical analysis was carried out with the Sigma Stat software system (SPSS Inc, Chicago, IL, USA).

Table 4: *Comparison of μ-tensile Strength and DC, Arranged Highest to Lowest*

CB	DC	FB	DC	MB	DC	UB	DC
μ tensile		**μ tensile**		**μ tensile**		**μ tensile**	
Control	Control	Control	Control	Control	Control	Control	Control
CBP	CBP	FBP	FBP	MBP	MBP	UBP	UBP
UBP	FBP	CBP	UBP	FBP	FBP	CBP	FBP
MBP	MBP	UBP	CBP	CBP	CBP	FBP	CBP
FBP	UBP	MBP	MBP	UBP	UBP	MBP	MBP

Groups in columns connected by vertical lines are not statistically, significantly different $p<0.05$.

RESULTS

Table 2 shows the results of μ-TS with various combinations of self-etching primers and adhesive resins. After 24 hours storage in water, the μ-TS of the adhesive resins were 88.0 MPa for CB, 76.9 MPa for FB, 54.9 MPa for MB and 57.5 MPa for UB. When the self-etching primers were mixed with adhesive resins, the μ-TS of the adhesive resins tended to decrease, but no significant differences were found for MB and UB compared to the controls. The μ-TS varied with the self-etching primers used and ranged from 35.4 to 79.4 MPa for CB, from 49.9 to 66.8 MPa for FB, from 43.2 to 50.2 MPa for MB and from 46.9 to 49.9 MPa for UB. Although numerically the highest μ-TS were obtained when the primer/adhesive resin combinations from the same manufacturer were used, there were no statistically significant differences found between the control and its primer for any of the adhesives.

Table 3 shows the results of DC with the various combinations of self-etching primers and adhesive resins. After 24 hours storage in water, the DC of the adhesive resins were 84.9% for CB, 86.7 for FB, 88.3% for MB and 87.4% for UB. When the self-etching primers were mixed with adhesive resins, the %DC of the adhesive resins decreased significantly except for CB mixed with the same manufacturer's self-etching primer. The DC varied with the dentin primers used and ranged from 51.0 to 82.8% for CB, from 46.2 to 74.5% for FB, from 70.1 to 80.1 % for MB and from 80.4 to 82.7% for UB. Numerically, mixing primer and adhesive from the same manufacturer gave the highest DC for all combinations, which was statistically significant with the exception of UB. Table 4 shows the statistical analyses for both strength and DC.

DISCUSSION

One of the most important functions of self-etching primer is etching the dentin substrate to modify the smear layer, thus allowing the subsequently applied resin monomers to penetrate. As acidic primers infiltrate into intertubular dentin, the liquid phase of the primer replaces hydroxyapatite crystallites that occupied spaces between the collagen fibrils. Adhesive resin monomers must be supplied to infiltrate into the exposed collagen fibril network in the demineralized dentin to establish an authentic hybrid layer. After infiltration into the etched/primed dentin, the adhesive resins must polymerize. Hypothetically, stronger resins might lead to stronger bonding to dentin (Pashley & others, 1995). The strength of cured adhesive resin is dependent on the composition, degree of conversion and length of polymer chain. Unreacted resin monomer remaining in adhesive resins may alter their mechanical properties. Thus, evaluation of the mechanical properties of the adhesive resin is important to the durability of bonding to dentin.

Most general practitioners use dental materials according to each material's instruction, since they know this might lead to the best clinical results. Due to the fact that self-etching primers act as etching agents as well as dentin primer (Van Meerbeek & others, 1998; Perdigão & Lopes, 1999), a self-etching primer would be effective for any kind of adhesive resin of two-step self-etching primer systems. The objective of this *in vitro* study was to clarify the influence of the interchanging of self-etching primers and adhesive resins on mechanical properties. From the results of this study, similar trends are seen in the data for tensile strength and DC. Numerically, the order from high to low for both is very similar (Table 4) and significant relationships were found (r^2=0.904~0.980) from the linear regression analysis. For CB, in particular, the results from the statistical analysis are the same for both. DC and tensile strength are not significantly influenced by the addition of their own primer to CB but is significantly lower for other combinations. For resin UB on the other hand, the addition of different primers has little influence on either DC or strength. With the addition of self-etching primers into adhesive resins, μ-TS of CB and FB were affected by some combinations of the primer and adhesive resin. Notably, the strength of both of these adhesives was not statistically influenced by the addition of their own primers. μ-TS of the adhesive resin MB and UB were not significantly lowered regardless of the primer employed. Similar trends were seen for the DC data.

One reason for the decreasing μ-TS observed for CB and FB could be that incorporating the solvent from the primers into the adhesive resin might alter polymerization reaction of the resin monomers (Hotta & others, 1998). Another explanation for the decreasing μ-TS could be related to the acidic functional monomers in the self-etching primers. The adhesive resins used in this study are all cured by a free radical polymerization reaction utilizing camphorquinone (CQ) as a visible-light activated photoinitiator (Taira & others, 1988; Jakubiak & Rabek, 1999). CQ requires a co-initiator for an effective polymerization process to occur, and a tertiary amine photo-reductant is employed. The tertiary amine interacts with an activated CQ to produce reactive radicals for polymerization (Cook, 1992). There is a possibility that the tertiary amines in adhesive resins could be neutralized by acidic functional monomers in the self-etching primers, resulting in poor polymerization and reduced mechanical properties (Sanares & others, 2001). Incompatibility between acidic functional monomers of the self-etching primers and resin monomers does not occur under the primer/adhesive resin combination recommended by each manufacturer as reported by Imazato and others (2003). However, improper polymerization occurs when the different combinations of primer/adhesive resin were mixed, which is thought to be caused by adverse interaction between the nucleophilic tertiary amine and acidic functional monomers (Tay & others, 2001). On the other hand, the dysfunction of resin polymerization for the adhesive resins MB and UB was not observed. One reason for such differences was the different compositions of monomers, polymerization initiators and inhibitors (Sideridou, Tserki & Papanastasiou, 2002).

Several approaches are employed for measuring conversion in resin composites; the majority of analytical studies have been done with the use of FTIR (Asmussen, 1982; Ferracane & Greener, 1984; Ruyter & Øysæd, 1987; Rueggeberg, Hashinger & Fairhurst, 1990). The DC in methacrylate groups depends on the resin monomer composition (Shimomura, 1987) and the concentration of polymerization initiators (Yoshida & Greener, 1994). From the results of this study, the DC of adhesive resin appears to depend upon the kind of self-etching primers mixed with them. Incorporation of the self-etching primers interfered with polymerization reaction of the adhesive resins and significant reductions of DC were found for all combinations except for CB mixed with the primers recommended by each manufacturer. The data of DC had a tendency to correspond to that of μ-TS of the adhesive resins, and the reduction of DC was caused by mixing with the primers. For CB mixed with primer, as recommended by the same manufacturer as adhesive resin, a slight but not significant decrease in DC was found. It has been suggested that including reducing agents other than the tertiary amines in adhesive resin resulted in increased bond strength (Munksgaard, Irie & Asmussen, 1985). The adhesive system CB contains a sulphinic acid salt, and this ingredient might act as a scavenger for oxygen to maintain DC even after mixing with self-etching primer.

Other factors that affect residual double bonds in adhesive resin would be an elution of monomers into water during the storage period of the specimen (Ferracane, 1994). The rate of elution of components from resin composites has been reported to be rapid during the initial period of immersion in solvent, leading to substantial solution within hours. There might be a possibility that the uncured monomers leach during storage in water, thus reducing the amount of uncured remaining double bonds. The presence of residual monomer can make the polymer matrix more susceptible to oxidation degradable reactions and lead to lower mechanical strength.

The experimental null hypothesis was not confirmed. The μ-TS of the adhesive resins was influenced by the primers mixed with, and the DC of the adhesive resins were affected by the incorporation of primers.

CONCLUSIONS

Within the limitation of this *in vitro* study, the mechanical properties of adhesive resin had a tendency to deteriorate by mixing the self-etching primers. This tendency was more pronounced for the different combinations of adhesive resins and self-etching primers that were recommended by each manufacturer. Further research is needed to determine the effects on the clinical performance of self-etching primer restorative systems.

Acknowledgements

This work was supported, in part, by a Grant-in-Aid for Scientific Research (C) (2) 14571826 from the Japan Society for the Promotion of Science and by a Grant from the Dental Research Center, Nihon University School of Dentistry, 2003.

(Received 27 May 2003)

References

Asmussen E (1982) Factors affecting the quantity of remaining double bonds in restorative resin polymers *Scandinavian Journal of Dental Research* **90(6)** 490-496.

Barkmeier WW, Los SA & Triolo PT Jr (1995) Bond strengths and SEM evaluation of Clearfil Liner Bond 2 *American Journal of Dentistry* **8(6)** 289-293.

Cook WD (1992) Photopolymerization kinetics of dimethacrylates using camphorquinone amine initiator system *Polymer* **33(3)** 600-609.

Ferracane JL & Greener EH (1984) Fourier transform infrared analysis of degree of polymerization in unfilled resins–methods comparison *Journal of Dental Research* **63(8)** 1093-1095.

Ferracane JL (1994) Elution of leachable components from composites *Journal of Oral Rehabilitation* **21(4)** 441-452.

Gordan VV, Vargas MA, Cobb DS & Denehey GE (1997) Evaluation of adhesive systems using acidic primers *American Journal of Dentistry* **10(5)** 219-223.

Hotta M, Kondoh K & Kamemizu H (1998) Effect of primers on bonding agent polymerization *Journal of Oral Rehabilitation* **25(10)** 792-799.

Ikemura K, Kouro Y & Endo T (1996) Effect of 4-acryloxyethyltrimellitic acid in a self-etching primer on bonding to ground dentin *Dental Materials Journal* **15(2)** 132-143.

Imazato S, Kinomoto Y, Tarumi H, Ebisu S & Tay FR (2003) Antibacterial activity and bonding characteristics of an adhesive resin containing antibacterial monomer MDPB *Dental Materials* **19(4)** 313-319.

Jakubiak J & Rabek JE (1999) Photoinitiators for visible light polymerization *Polimery* **44(7-8)** 447-461.

Miyazaki M, Hirohata N, Takagaki K, Onose H & Moore BK (1999) Influence of self-etching primer drying time on enamel bond strength of resin composites *Journal of Dentistry* **27(3)** 203-207.

Miyazaki M, Onose H & Moore BK (2002) Analysis of the dentin-resin interface by use of laser Raman spectroscopy *Dental Materials* **18(8)** 576-580.

Miyazaki M, Onose H, Iida N & Kazama H (2003) Determination of residual double bonds in resin-dentin interface by Raman spectroscopy *Dental Materials* **19(3)** 246-251.

Munksgaard EC, Irie M & Asmussen E (1985) Dentin-polymer bond promoted by Gluma and various resins *Journal of Dental Research* **64(12)** 1409-1411.

Pashley DH, Ciucchi B, Sano H, Carvalho RM & Russell CM (1995) Bond strength versus dentine structure: A modeling approach *Archives of Oral Biology* **40(12)** 1109-1118.

Perdigão J & Lopes M (1999) Dentin bonding— Questions for the new millennium *Journal of Adhesive Dentistry* **1(3)** 191-209.

Rueggeberg FA, Hashinger DT & Fairhurst CW (1990) Calibration of FTIR conversion analysis of contemporary dental resin composites *Dental Materials* **6(4)** 241-249.

Ruyter IE & Øysæd H (1987) Composites for use in posterior teeth: Composition and conversion *Journal of Biomedical Materials and Research* **21(1)** 11-23.

Sano H, Takatsu T, Ciucci B, Horner JA, Matthews WG & Pashley DH (1995a) Nanoleakage: Leakage within the hybrid layer *Operative Dentistry* **20(1)** 18-25.

Sano H, Takatsu T, Ciucchi B, Russell CM & Pashley DH (1995b) Tensile properties of resin-infiltrated demineralized human dentin *Journal of Dental Research* **74(4)** 1093-1102.

Sano H, Yoshikawa T, Pereira PN, Kanemura N, Morigami M, Tagami J & Pashley DH (1999) Long-term durability of dentin bonds made with a self-etching primer, *in vivo Journal of Dental Research* **78(4)** 906-911.

Shimomura H (1987) Photochemical studies on composite resins cured by visible light *Dental Materials Journal* **6(1)** 9-27.

Sideridou I, Tserki V & Papanastasiou G (2002) Effect of chemical structure on degree of conversion in light-cured dimethacrylate-based dental resins *Biomaterials* **23(8)** 1819-1829.

Spencer P & Swafford JR (1999) Unprotected protein at the dentin-adhesive interface *Quintessence International* **30(7)** 501-507.

Sanares AM, Itthagarun A, King NM, Tay FR & Pashley DH (2001) Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites *Dental Materials* **17(6)** 542-556.

Taira M, Urabe H, Hirose T, Wakasa K & Yamaki M (1988) Analysis of photo-initiators in visible-light-cured dental composite resins *Journal of Dental Research* **67(1)** 24-28.

Takahashi A, Sato Y, Uno S, Pereira PNR & Sano H (2002) Effects of mechanical properties of adhesive resins on bond strength to dentin *Dental Materials* **18(3)** 263-268.

Tay FR, King NM, Suh BI & Pashley DH (2001) Effect of delayed activation of light-cured resin composites on bonding of all-in-one adhesives *Journal of Adhesive Dentistry* **3(3)** 207-225.

Yoshida K & Greener EH (1994) Effect of photoinitiator on degree of conversion of unfilled light-cured resin *Journal of Dentistry* **22(5)** 296-299.

Van Meerbeek B, Perdigão J, Lambrechts P & Vanherle G (1998) The clinical performance of adhesives *Journal of Dentistry* **26(1)** 1-20.

©Operative Dentistry, 2004, **29-5,** 538-546

Immunohistochemical Analysis of Collagen Fibrils within the Hybrid Layer: A FEISEM Study

L Breschi • C Prati • P Gobbi
D Pashley • G Mazzotti • G Teti • J Perdigão

Clinical Relevance

The immunohistochemical technique utilized in this study allows for a very fine distinction between the adhesive agent and exposed collagen fibrils in the hybrid layer. The identification of exposed collagen fibrils indicates incomplete hybridization of the matrix by resin. The presence of naked collagen fibrils may compromise the longevity of resin-dentin bonds in aesthetic restorations.

SUMMARY

This study evaluated the immunohistochemical labeling pattern of dentin collagen fibrils within hybrid layers created by different bonding systems using high resolution SEM.

Four different adhesive materials, including self-etching and total-etching systems, were examined: Prime & Bond NT, OptiBond SOLO *Plus*, Single Bond and Clearfil Protect Bond. All materials were applied to the dentin of extracted human third molars. After cutting the bonded specimens transversely, an anti-collagen type I antibody was incubated on the surface of the dentin-adhesive interface and gold-conjugated secondary antibody was applied to reveal collagen labeling under high resolution SEM.

The hybrid layers showed a significant number of collagen fibrils embedded in the resin matrix. The presence of exposed dentin collagen fibrils, as determined by positive labeling with an anti-type I collagen monoclonal antibody, is considered an indication of the presence of incompletely embedded fibrils and, thus, the quality of dentin matrix hybridization. The hybrid layers produced by total-etching systems showed higher labeling compared to those produced by the self-etching system. Positive collagen labeling was also found along the resin tags produced by total-etching adhesive systems.

*Lorenzo Breschi, DDS, PhD, associate professor, Dipartimento di Scienze Chirurgiche Specialistiche, University of Trieste, Trieste, Italy

Carlo Prati, MD, PhD, professor, Department of Dental Science, University of Bologna, Bologna, Italy

Pietro Gobbi, MD, PhD, associate professor, Dipartimento di Scienze Morfologiche, University of Urbino, Urbino, Italy

David Pashley, DMD, PhD, regents professor, Department of Oral Biology, School of Dentistry Medical College of Georgia, Augusta GA, USA

Giovanni Mazzotti, MD, PhD, professor, Department of SAU & FAL, University of Bologna, Bologna, Italy

Gabriella Teti, PhD, Department of SAU & FAL, University of Bologna, Bologna, Italy

Jorge Perdigão, DMD, MD, PhD, associate professor, Department of Restorative Sciences, University of Minnesota, Minneapolis, MN, USA

*Reprint request: Via Stuparich, 1, I-34129 Trieste, Italy; e-mail: lbreschi@units.it

INTRODUCTION

Bonding to dentin requires the infiltration of adhesive co-monomers into the matrix of decalcified dentin, thus forming the so-called hybrid layer (HL) (Nakabayashi, Kojima & Masuhara, 1982; Van Meerbeek & others, 1993). Dentin demineralization is achieved by preliminary acid etching (total-etch technique) or simultaneously acid etching with adhesive infiltration using acidic adhesive monomers (self-etching adhesive systems) (Perdigão & Lopes, 1999). The different etching techniques result in distinct morphological interphases of the adhesives to dentin, as demonstrated by different thicknesses of the hybrid layers, resin tag morphology and collagen fibrils (CF) distribution (Van Meerbeek & others, 1992, 1993; Tay & Pashley, 2001; Prati & others, 1999). In the total-etching adhesive systems, a preliminary etching step of the dentin surface with phosphoric acid is followed by water rinsing to extract solubilized minerals. The matrix is left moist to facilitate the penetration of adhesive co-monomers into the exposed superficial dentin collagen matrix and into opened tubules, thus, forming resin tags. The final goal of the procedure is full infiltration of the exposed collagen fibrils by hydrophilic resin to fill all porosities created by etching procedures (Nakabayashi & others, 1982; Van Meerbeek & others, 1993). On the other hand, the self-etching materials produce partial demineralization of the smear layer, smear plugs and the most superficial collagen fibrils on top of sound dentin (Tay & Pashley, 2001). The solubilized mineral phase remains in the resin as a suspension. The distinct approaches of the two adhesive procedures may lead to different types of hybrid layers.

Previous studies have investigated the infiltration pattern of the collagen fibril network by adhesive agents by determining the distribution of silver nitrate within the HL (Sano & others, 1995a). This technique of identifying submicron porosities within the HL has been termed nanoleakage and is thought to result from incomplete penetration of adhesives into the demineralized substrate (Sano & others, 1995b). Further studies revealed that the presence of uninfiltrated collagen fibrils exposed by etching is susceptible to proteolytic enzymes and thus may represent a possible pathway to secondary bacteria colonization (Vargas, Cobb & Armstrong, 1997). Moreover, recent studies on the micropermeability of HL revealed that it can absorb and release water, acting as a semi-permeable membrane (Tay & others, 2002). These data suggest that part of the organic phase of the dentin matrix type I collagen, proteoglycans and/or other non-collagenous protein) (Marshall & others, 1997) may be responsible for water sorption and thus is not embedded by the adhesive resin.

This study evaluated, with an immunocytochemical technique and high resolution SEM, the morphological labeling pattern of collagen fibrils within HL using both self-etching and total-etching systems. The presence of positive labeling by a monoclonal anti-type I collagen antibody was considered evidence of naked collagen fibrils embedded within the HL and gives an indication of the quality of hybridization.

METHODS AND MATERIALS

Specimen Preparation

The occlusal enamel of four impacted third molars was removed immediately after extraction. Middle dentin was exposed using a slow speed diamond saw (Remet, Casalecchio di Reno, Bologna, Italy) under irrigation with 0.2 M phosphate buffered (pH 7.2) solution. The dentin surface was divided in four quarters using the diamond saw and a metallic matrix was placed to separate the quarters. Four different dentin bonding materials were applied on each quarter of the same dentin specimen:

1. Prime & Bond NT (P&B NT, Dentsply DeTrey, Konstanz, Germany): Dentin was etched with 35% phosphoric acid gel (Scotchbond Etchant, 3M ESPE Dental Products, St Paul, MN, USA) for 15 seconds and rinsed for 30 seconds with water. Dentin was blotted with a cotton pellet and P&B NT was applied with a small sponge. Excess solvent was removed by gently drying with air for at least five seconds. The adhesive was light-cured for 10 seconds.
2. OptiBond SOLO *Plus* (OB, Kerr Co, Orange, CA, USA): Dentin was etched with 35% phosphoric acid gel (Scotchbond Etchant) for 15 seconds and rinsed for 30 seconds with water. The excess water was removed with a cotton pellet. One layer of adhesive was applied to dentin for 15 seconds using a light brushing motion, air thinned for three seconds and light cured for 20 seconds.
3. Single Bond (SB, 3M ESPE Dental Products, St Paul, MN, USA): Dentin was etched with 35% phosphoric acid gel (Scotchbond Etchant) for 15 seconds and rinsed with water for 30 seconds. The excess water was removed with a cotton pellet. Two coats of SB were applied on the etched dentin surface. After gently air-drying to evaporate the solvent, the adhesive was light-cured for 10 seconds.
4. Clearfil Protect Bond (CPB, Kuraray Medical Co, Ltd, Osaka, Japan): This antibacterial, fluoride containing self-etching primer system was dispersed, applied and left undisturbed on the dentin surface for 20 seconds. Primer was then gently air dried and "BOND" was applied. The adhesive was light cured for 10 seconds.

A flowable resin composite (Filtek Flow, 3M ESPE) was applied to all bonded specimens and light cured. All adhesives and resin composites were light cured using a previously tested unit (Curing Light XL3000, 3M ESPE working at 600 mW/cm^2).

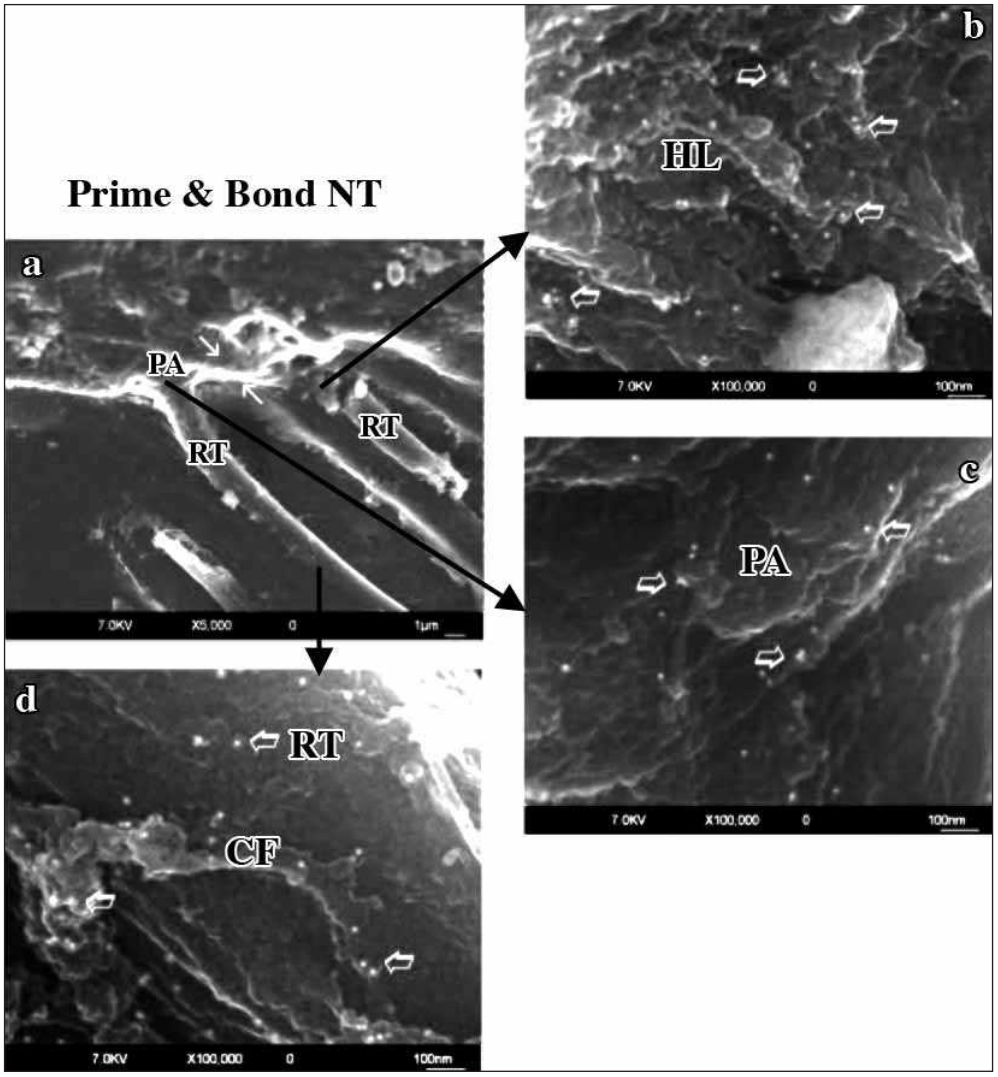


Figure 1. *FEISEM micrographs of the dentin-P&B NT interface after the immunohistochemical labeling with a monoclonal antibody:*
Figure. 1a*: FEISEM secondary electron image after the application of phosphoric acid and P&B NT. A 3-5 mm thick hybrid layer (HL) (white arrows) with long resin tags (RT) are clearly seen. No visible gaps or porosities could be detected at this level of magnification, confirming a good adaptation of the adhesive agent on the dentin surface. Infiltration of the adhesive agent is evident also within the peritubular area (PA).*
Figure 1b*: FEISEM mixed image of secondary and back-scattered electrons. The hybrid layer (HL) reveals intense collagen fibrils labeling (arrows) with localization of the gold nano-particles throughout the HL thickness. No visible gaps or porosities are evident.*
Figure 1c*: FEISEM mixed image of secondary and back-scattered electrons. Peritubular area (PA) reveals resin infiltration of the funneled dentin tubule. Gold labeling (arrows) is present with homogenous distribution. Labeling appears to be lower than in HL.*
Figure 1d*: FEISEM mixed image of secondary and back-scattered electrons. The resin tag (RT) reveals gold labeling (arrows) on its surface where a collagen fibril appears emanating from the resin surface. Positive labeling can also be found on the surrounding dentin surface that was acid-etched for three seconds during specimen preparation. Such dentin surfaces serve as internal control, in that they show the maximum amount of antibody binding in the absence of exposure to bonding resins.*

Immunocytochemical Technique

Specimens were immediately transversally cut in small sticks 1 x 1 mm and 4-mm high (approximately 2 mm of dentin and 2 mm of restorative materials) to expose the bonded interface. An aqueous solution of 35% phosphoric acid was applied on the surface for three seconds to remove cutting debris and challenge the integrity of the resin infiltration. The specimens were rinsed in saline solution for 15 minutes, followed by Tris buffer solution (TBS) 0.05 M at pH 7.6. Nonspecific antigen saturation was obtained with normal goat serum in TBS 0.05 M at pH 7.6 for 30 minutes at room temperature. Overnight incubation was performed at 4°C using a monoclonal anti-type I collagen antibody (Sigma Chemical Co, St Louis, MO, USA) (Breschi & others, 2003a). Gold labeling was performed with a goat IgG anti-mouse IgG (British BioCell International, Cardiff; United Kingdom) conjugated with 15 nm colloidal gold diluted in 0.02 M TBS at pH 8.2 (Breschi & others, 2002). Specimens were then rinsed in distilled water, fixed in 2.5% glutaraldehyde in 0.1 M cacodylate buffer pH 7.2 for four hours and further rinsed in 0.1M cacodylate buffer. After dehydration in ascending ethanol series, the specimens were HMDS-dried (Perdigão & others, 1995) in preparation for field emission in-lens scanning electron microscopy (FEISEM). Two operators performed observations on blind specimens, and images were taken from areas most representative of the observed specimens.

Control specimens were processed as described, then incubated in TBS 0.05 M at pH 7.6 without the primary antibody.

RESULTS

The FEISEM examination of the specimens confirmed HL formation for all four adhesive systems. Gold particle labeling patterns were evaluated in three zones: within the HL thickness (Figures 1b, 2b, 3b, 4b), in the peritubular zone (Figures 1c, 2c, 3c, 4c) and along the resin tags within the tubules (Figures 1d, 2d, 3c, 3d). Most of the collagen fibrils were embedded in polymerized adhesive, in all of the adhesives that were tested.

The HL formed by the three total-etch adhesive agents (Figures 1-3) was characterized by a compact and homogenous structure, revealing no visible porosities or gaps. Intense gold labeling identifying residual exposed collagen fibrils within the HL was visible, especially in the deepest part of the HL (Figures 1b, 2b and 3b). In particular, OB and SB (Figures 2b and 3b) revealed a gradient in the gold labeling, showing higher concentrations of gold particles in the deepest part of the HL, while P&B NT revealed a homogenous labeling throughout the HL thickness (Figure 1b). Very intense labeling was also found in the peritubular zone for specimens bonded with P&B NT (Figure 1c), where the adhesive agent contributed to the formation of resin tags infiltrating the peritubular region of intertubular dentin. Specimens bonded with P&B NT also revealed intense labeling along the wall of the resin tags, showing several naked collagen fibrils within the resin matrix (Figure 1d). Specimens bonded with OB and SB revealed only minor labeling in the peritubular zone (Figures 2c and 3c) and almost no labeling along the resin tags (Figures 2d and 3d)

Much less collagen labeling was observed in HL formed by the self-etching adhesive system tested in this study (CPB, Figure 4b) compared to total-etch adhesive systems. Positive gold labeling was also found in the peritubular zone, while almost no labeling was found along the short resin tags (Figure 4c and 4d).

Sound dentin surfaces located some distance from the bonded interfaces showed intense labeling due to three second acid etching with phosphoric acid, while no labeling was observed in the resin composite, on resin surfaces or on control specimens, thus confirming no cross reaction between the secondary antibody and the substrate (data not shown).

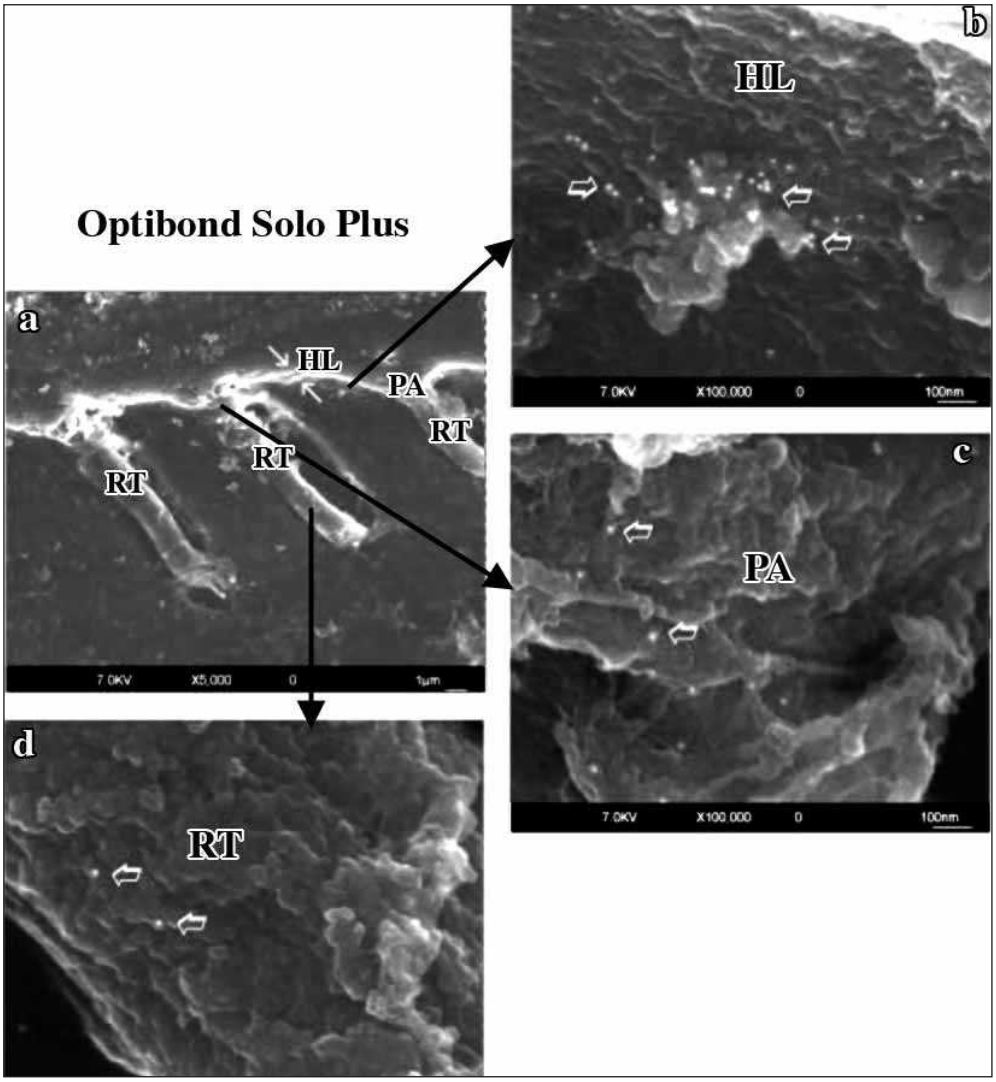


Figure 2. *FEISEM images of the dentin-OB interface after the immunohistochemical labeling of collagen fibrils with a monoclonal antibody:*

Figure 2a*: FEISEM secondary electron image after applying phosphoric acid and OB. Compact and homogenous hybrid layer (HL, between white arrows) and with resin tags (RT) are clearly detectable. Peritubular area is indicated as PA.*

Figure 2b*: FEISEM mixed image of secondary and back-scattered electrons. Scarce labeling is present on the superficial part of the HL, while intense collagen fibril labeling can be seen in the deeper areas of the HL (arrows), close to the sound dentin structure. No visible gaps or porosities are evident.*

Figure 2c*: FEISEM mixed image of secondary and back-scattered electrons. The peritubular area (PA) infiltrated by the resin agent reveals weak and homogenous labeling (arrows).*

Figure 2d: *FEISEM mixed image of secondary and back-scattered electrons. The resin tag (RT) reveals almost no gold labeling on its surface (arrows indicate few gold particles).*

DISCUSSION

Today, the use of transmission electron microscope (TEM), field emission SEM, atomic force microscopy (AFM) and micro-Raman analysis allows for investigation of the quality and composition of HL and micromorphologically describes the interaction between the adhesive agents and the demineralized dentin surface (Van Meerbeek & others, 2000; Spencer & others, 2000; Eliades, Vougiouklakis & Palaghias, 2001; Wang & Spencer, 2002; Spencer & Wang, 2002). Previous studies on HL formation revealed that incomplete infiltration of demineralized dentin may occur after application of an adhesive agent (Sano & others, 1995a,b). Most previous microscopic reports based the morphological analysis of dentin collagen fibrils within the HL only on morphological parameters such as fibrils dimensions and collagen banding (Van Meerbeek & others, 2000). Several micro-Raman spectroscopy studies have shown a gradient of resin distribution within the amount of resin at the bottom of the hybrid layers produced by total-etch adhesive systems (Spencer & others, 2000; Wang & Spencer, 2002; Spencer & Wang, 2002).

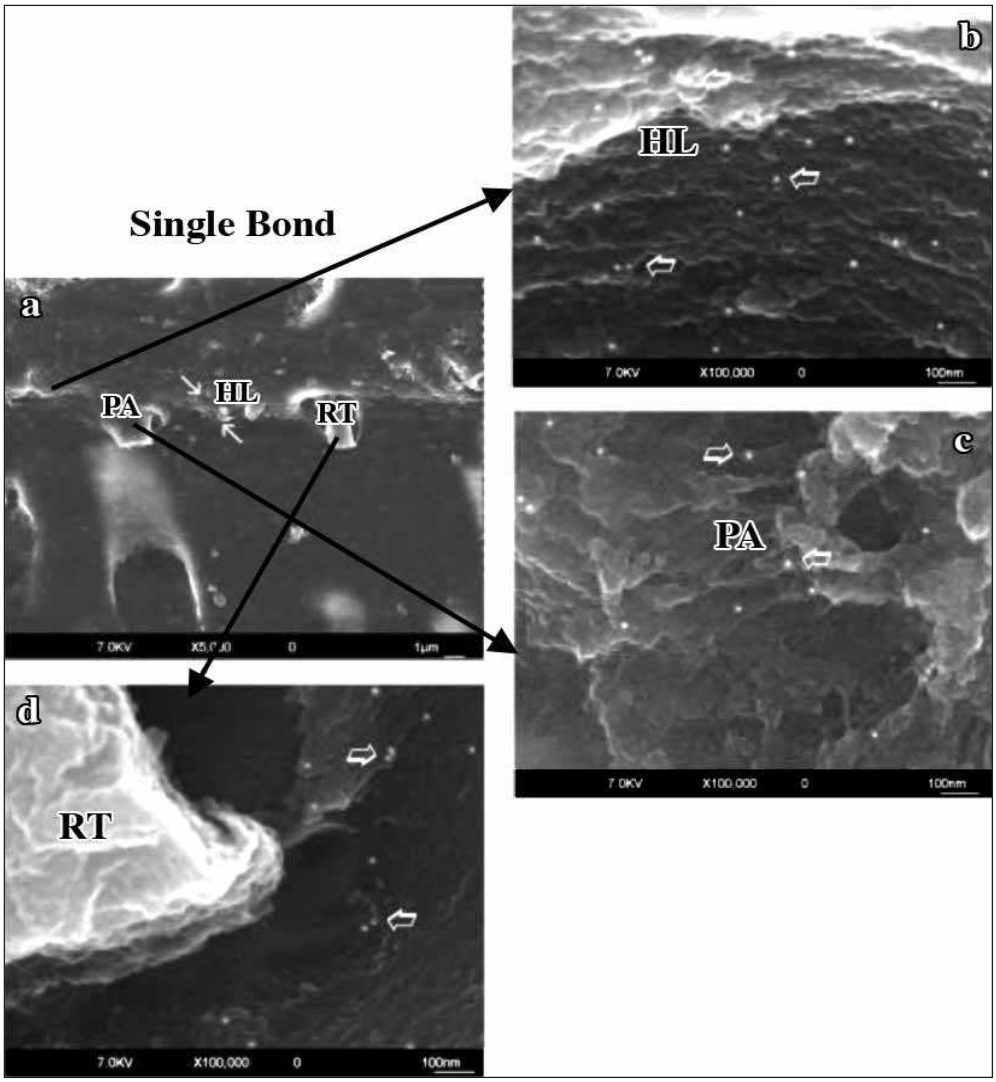


Figure 3. *FEISEM images of the dentin-SB interface after the immunohistochemical labeling of collagen with a monoclonal antibody:*
Figure 3a*: FEISEM secondary electron image after applying phosphoric acid and SB. The typical hybrid layer (HL, between white arrows), resin tags (RT) and areas of peritubular infiltration (PA) formed by total etching systems are clearly visible.*
Figure 3b*: FEISEM mixed image of secondary and back-scattered electrons. Intense labeling is detectable on the HL. The deeper areas of the HL appear to be particularly labeled (arrows) compared to the superficial HL.*
Figure 3c*: FEISEM mixed image of secondary and back-scattered electrons. The peritubular area (PA) infiltrated by the resin agent reveals some labeling (arrows) at the base of the resin tag, then very weak labeling present entering the lumen of the tubule.*
Figure 3d: *FEISEM mixed image of secondary and back-scattered electrons. The resin tag (RT) reveals almost no gold labeling on the surface.*

The hypothesis tested in this study is that some collagen fibrils within HL may not be fully infiltrated by resin adhesive and can react with anti-type I collagen antibodies (Breschi & others, 2003a). Previous findings on the labeling pattern of monoclonal anti-collagen Type I antibody to etched dentin matrix allowed for the demonstration of a significant number of immunolabeled collagen fibrils after dentin treatment with phosphoric, citric and maleic acid and EDTA (Breschi & others, 2003b). The results of this study support the hypothesis that HL may not be fully infiltrated with resin.

In this study, positive labeling patterns were found in all bonded specimens. The dentin surface revealed the presence of scattered labeling of a few exposed collagen fibrils, but no labeling was evident on the resin composite surface. Considering that antibody binding may occur only when the antigen epitope is exposed, the authors identified three different areas of dentin-resin interactions: HL, the peritubular hybridization zone and resin tags.

The HL created by total-etching systems (particularly OB and SB) revealed minor labeling on the top of HL (superficial

HL) but an intense labeling of collagen fibrils in the deepest part of HL. This supports the hypothesis that with total-etching systems, different degrees of resin-collagen fibril interactions may occur depending on the degree of penetration of the adhesive into the demineralized dentin matrix. The collagen fibrils in superficial HL seem to be fully impregnated (reduced labeling), while the deepest area of HL shows a great number of exposed collagen fibrils that remain partially available for binding the antibodies. This work supports the recent observation that some adhesive monomers can mask antibody binding to collagen (Vaidyanathan, Chinnaswamy & Vaidyanathan, 2003). In particular, small but frequent areas of poorly impregnated fibrils are present inside the deeper area of the hybrid layer network, close to the mineralized dentin. This area seems to have a large number of exposed, uncovered collagen fibrils that react with anti-collagen antibodies. P&B NT revealed intense labeling throughout the entire thickness of HL, showing many exposed collagen fibrils.

In contrast, the HL created by the self-etching system (CPB) did not reveal a gradient in the labeling pattern, but showed only weak, uniform gold labeling. This weak acidic self-etching system infiltrates though the smear layer but only 0.5 µm into the underlying sound dentin surface (Tay & Pashley, 2001). Collagen fibrils within the smear layer may be partially denaturated by thermal and shear stresses created by cutting procedures and are no longer available for binding the antibody. The anti-collagen type I antibody tested in this study reacts only with native collagen (Breschi & others, 2003a). Another possible explanation for the lower degree of gold labeling may relate to the fact that self-etching systems produce a more homogenous resin infiltration of demineralized collagen fibrils compared to total-etching systems (Nakabayashi & Saimi, 1996). Since the self-etching systems do not include a rinsing step, there is no loss of inorganic matrix (Tay & Pashley, 2001). Since the depth of HL is only 0.5-1.0 µm, the diffusion distance for resin infiltration is shorter than in total-etch systems. The final conse-

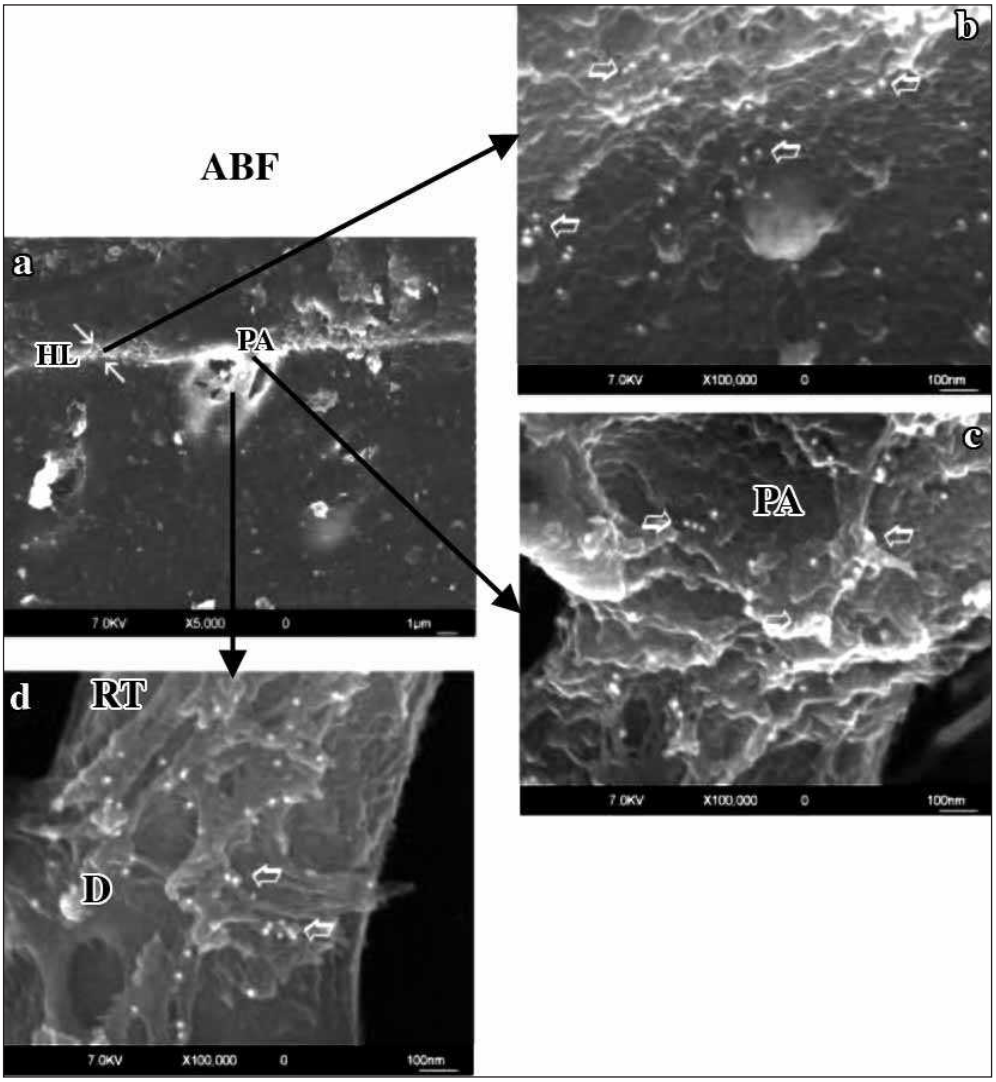


Figure 4. *FEISEM images of the dentin-CPB interface after the immunohistochemical labeling of collagen fibrils with a monoclonal antibody:*

Figure 4a*: FEISEM secondary electron image after applying CPB as per manufacturers' instructions. The hybrid layer (HL, between white arrows) and peritubular area (PA) are visible.*

Figure 4b*: FEISEM mixed image of secondary and back-scattered electrons. Scarce labeling (arrows) is present throughout the thickness of the HL. No labeling difference was found between the deeper and most superficial areas of the HL.*

Figure 4c*: FEISEM mixed image of secondary and back-scattered electrons. A weak and homogenous labeling (arrows) can be observed at the peritubular area (PA). The brief phosphoric acid etch used to clean the surface of cutting debris removed the mineralized peritubular dentin matrix.*

Figure 4d*: FEISEM mixed image of secondary and back-scattered electrons. The resin tag (RT) reveals almost no gold labeling on the surface, while intense labeling (arrows) is visible on the dentin organic matrix (D), which is in close relation to the resin tag. This collagen is represented by several fibrils overlapping each other and constituting a complex network that was exposed by the three seconds etch used to clean the surface of grinding debris during specimen preparation.*

quences for these factors are represented by the lower number of labeling sites.

Analysis of the peritubular dentin hybridization of total-etching systems revealed few labeled collagen fibrils on the resin surface. The relatively low labeling density present in this area may be related to the scarcity of collagen fibrils (Perdigão & others, 1996) and to a relatively complete infiltration of the peritubular dentin due to funneling created by etching (Perdigão & others, 1996). CPB revealed a moderate labeling at this level, probably related to the collagen fibrils in the smear plugs and the fact that less acidic self-etching monomers fail to remove mineralized peritubular dentin (Tay & Pashley, 2001).

Positive gold labeling was also found on the resin tags of P&B NT bonded specimens. Considering that most proteins constituting the *lamina limitans* contained inside the dentin tubules are proteoglycans (Thomas & Carella, 1984), the gold label on the surface of resin tags (and visible on Figure 1d) may have come from the overlying lateral tubular walls (Marshall & others, 1997). These collagen fibrils appear to be embedded in the resin matrix of the resin tag. However, such labeling was not seen in resin tags created by other adhesive systems.

CPB-bonded specimens revealed no labeling along the resin tag, confirming mild acidic activity of the self-etching system relative to the buffering potential of mineralized peritubular dentin. The authors speculate that low gold labeling in the peritubular zone and along the resin tags produced by CPB is due to, in part, to the inability of the acidic primer to remove all the mineral coating of the collagen fibrils (Tay & Pashley, 2001) that is necessary for antibody binding or the superior ability of the resin to completely infiltrate the entire surface of exposed collagen fibrils.

Considering that the self-etching bonding system showed a reduced labeling index (defined as number of gold-particles per area unit), the authors believe that this system created a less permeable HL than total-etching systems. In fact, the morphological disposition of labeling was quite different, suggesting a reduced number of porosities and a better protection of collagen fibrils. Moreover, self-etching agents do not require exposure (by preliminary etching) of the collagen fibrils. Etching procedures may represent a critical step in bonding procedures, in which a great number of collagen fibrils remain exposed, partially collapsed and poorly infiltrated by resin systems. Such exposed collagen fibrils may be rapidly hydrolyzed by oral enzymes (Vargas & others, 1997). Removal of the inorganic phase of dentin by etching procedures leaves a sizeable volume in the dentin matrix that must be filled by hydrophilic adhesive monomer. Exposure of the collagen fibrils and their labeling by specific antibodies suggests that it is not possible, at least with the four bonding systems tested, to obtain complete hybridization of the matrix. The total-etching procedures are probably too strong, and the moist bonding technique leaves too many porosities that remain unfilled by hydrophilic resin. The obvious consequence is the high number of exposed, naked collagen fibrils. This phenomenon forms a porous zone within the HL that permits uptake of silver nitrate tracer (nanoleakage) (Sano & others, 1995a,b). Several studies have investigated nanoleakage and have tried to define possible pathways for penetration of the tracer. A recent TEM study on nanoleakage revealed that there is no association between the presence of nanoleakage in HL and the presence of hiati in the dentin demineralized matrix (Agee & others, 2003). Nanoleakage is also related to other factors, such as water rich domains in heterogeneous resins, water sorption by collagen/proteoglycans molecules (Breschi & others, 2002; Goldberg & Takagi, 1993) and incomplete polymerization of the resin matrix (Sano & others, 1999). Moreover, it is possible that dentin matrix proteins exposed by etching (in particular, collagen fibrils and proteoglycans) may adsorb and release water (Breschi & others, 2003b). This is supported by recent studies on the permeability of HL (Tay & Pashley, 2003; Burrow, Inokoshi & Tagami, 1999; Pashley & Tay, 2002; Itthagarun & others, 2003). In particular, water-tree formation (Tay & Pashley, 2003) recently described within HL suggests that HL acts as a semi-permeable membrane (Tay & others, 2002).

The immunohistochemical approach used in this study not only revealed the presence of naked collagen fibrils within the thickness of HL, but also indicated that these fibrils retained their native structure due to the selective binding capabilities of the monoclonal antibody anti-collagen type-1 used in this investigation. In fact, the antibody used in this study is specific for intact, native collagen (Breschi & others, 2003a). Denatured collagen or collagen fibrils that are completely enveloped by resin do not bind this antibody.

CONCLUSIONS

1. This technique represents a very selective approach to the morphological study of HL, allowing for discrimination between the adhesive agent and exposed collagen fibrils versus embedded and, thus, unlabeled collagen fibrils. High numbers of gold-labeled collagen fibrils represent incomplete hybridization. The quality and durability of the composite-dentin interface may be affected by the quality of this interface.
2. Different anti-collagen antibody labeling patterns were seen between total-etching and self-etching techniques, revealing differences in the adhesive/matrix interactions. Major gold labeling

was found within the HL of total-etching techniques, while the self-etching system revealed weaker labeling. This data may indicate reduced exposure of collagen fibrils by CPB compared to the other total-etching systems.

3. A high level of gold labeling was also found along the surface of resin tags for all tested total-etching systems. These data suggest that many collagen fibrils remain uncovered and not enveloped by resin. The contribution of this incomplete hybridization phenomenon to the bond strength and durability of bonding should be further investigated.

Acknowledgement

This investigation was supported by ex 40% and 60% MIUR grants (Italy) and by RO1DE014911 and DEO 15306 from the national Institute of Dental and Craniofacial Research (PI, D Pashley). The authors thank Mr Massimo Gamberini for laboratory technical assistance and Mr Aurelio Valmori for the extensive photographic work.

(Received 30 May 2003)

References

Agee KL, Pashley EL, Itthagarun A, Sano H, Tay FR & Pashley DH (2003) Submicron hiati in acid etched dentin are artifacts of desiccation *Dental Materials* **19(1)** 60-68.

Breschi L, Lopes M, Gobbi P, Mazzotti G, Falconi M & Perdigão J (2002) Dentin proteoglycans: An immunocytochemical FEISEM study *Journal of Biomedical Materials Research* **61(1)** 40-46.

Breschi L, Perdigão J, Gobbi P, Mazzotti G, Falconi M & Lopes M (2003a) Immunocytochemical identification of type I collagen in acid-etched dentin *Journal of Biomedical Materials Research* **66A(4)** 764-769.

Breschi L, Gobbi P, Lopes M, Prati C, Falconi M, Teti G & Mazzotti G (2003b) Immunocytochemical analysis of dentin: A double labeling technique *Journal of Biomedical Materials Research* **67A(1)** 11-17.

Burrow MF, Inokoshi S & Tagami J (1999) Water sorption of several bonding resins *American Journal of Dentistry* **12(6)** 295-298.

Eliades G, Vougiouklakis G & Palaghias G (2001) Heterogeneous distribution of single-bottle adhesive monomers in the resin-dentin interdiffusion zone *Dental Materials* **17(4)** 277-283.

Goldberg M & Takagi M (1993) Dentine proteoglycans: Composition, ultrastructure and functions *Histochemical Journal* **25(11)** 781-806.

Itthagarun A, Tay FR, Pashley DH, Wefel JS, García-Godoy F & Wei SHY (2003) Single-step, self-etch adhesives behave as permeable membranes after polymerization Part III Evidence from fluid conductance and artificial caries inhibition *American Journal of Dentistry* in press.

Marshall GW Jr, Marshall SJ, Kinney JH & Balooch M (1997) The dentine substrate: Structure and properties related to bonding *Journal of Dentistry* **25(6)** 441-458.

Nakabayashi N & Saimi Y (1996) Bonding to intact dentin *Journal of Dental Research* **75(9)** 1706-1715.

Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials Research* **16(3)** 265-273.

Pashley DH & Tay FR (2002) Single-step adhesives are semipermeable membranes I Nanoleakage and fluid conductance evidence *Journal of Dental Research* **81** Abstract #3828 p 468.

Perdigão J & Lopes M (1999) Dentin bonding—Questions for the new millennium *Journal of Adhesive Dentistry* **1(3)** 191-209.

Perdigão J, Lambrechts P, Van Meerbeek B, Tomé AR, Vanherle G & Lopes AB (1996) Morphological field emission-SEM study of the effect of six phosphoric acid etching agents on human dentin *Dental Materials* **12(4)** 262-271.

Perdigão J, Lambrechts P, Van Meerbeek B, Vanherle G & Lopes AL (1995) Field Emission SEM comparison of four post-fixation drying techniques for human dentin *Journal of Biomedical Materials Research* **29(9)** 1111-1120.

Prati C, Chersoni S, Mongiorgi R, Montanari G & Pashley DH (1999) Thickness and morphology of resin-infiltrated dentin layer in young, old and sclerotic dentin *Operative Dentistry* **24(2)** 66-72.

Sano H, Takatsu T, Ciucchi B, Horner JA, Matthews WG & Pashley DH (1995a) Nanoleakage: Leakage within the hybrid layer *Operative Dentistry* **20(1)** 18-25.

Sano H, Yoshiyama M, Ebisu S, Burrow MF, Takatsu T, Ciucchi B, Carvalho R & Pashley DH (1995b) Comparative SEM and TEM observations of nanoleakage within the hybrid layer *Operative Dentistry* **20(4)** 160-167.

Sano H, Yoshikawa T, Pereira PN, Kanemura N, Morigami M, Tagami J & Pashley DH (1999) Long-term durability of dentin bonds made with self-etching primer, *in vivo* *Journal of Dental Research* **78(4)** 906-911.

Spencer P, Wang Y, Walker MP, Wieliczka DM & Swafford JR (2000) Interfacial chemistry of the dentin/adhesive bond *Journal of Dental Research* **79(7)** 1458-1463.

Spencer P & Wang Y (2002) Adhesive phase separation at the dentin interface under wet bonding conditions *Journal of Biomedical Materials Research* **62(3)** 447-456.

Tay FR & Pashley DH (2001) Aggressiveness of contemporary self-etching systems. I: Depth of penetration beyond dentin smear layers *Dental Materials* **17(4)** 296-308.

Tay FR, Pashley DH, Suh BI, Carvalho RM & Itthagarun A (2002) Single-step adhesives are permeable membranes *Journal of Dentistry* **30(7-8)** 371-382.

Tay FR & Pashley DH (2003) Water treeing—a potential mechanism for degradation of dentin adhesives *American Journal of Dentistry* **16(1)** 6-12.

Thomas HF & Carella P (1984) Correlation of scanning and transmission electron microscopy of human dentinal tubules *Archives of Oral Biology* **29(8)** 641-646.

Vaidyanathan J, Chinnaswamy K & Vaidyanathan TK (2003) Biomimetic recognition and immunochemical assay of ligand binding to collagen *Journal of Adhesive Dentistry* **5(1)** 7-17.

Van Meerbeek B, Dhem A, Goret-Nicaise M, Braem M, Lambrechts P & Vanherle G (1993) Comparative SEM and TEM examination of the ultrastructure of the resin-dentin interdiffusion zone *Journal of Dental Research* **72(2)** 495-501.

Van Meerbeek B, Inokoshi S, Braem M, Lambrechts P & Vanherle G (1992) Morphological aspects of the resin-dentin interdiffusion zone with different dentin adhesive systems *Journal of Dental Research* **71(8)** 1530-1540.

Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Perdigão J, Lambrechts P & Vanherle G (2000) Microscopy investigations. Techniques, results, limitations *American Journal of Dentistry* **13(Spec No)** 3D-18D.

Vargas MA, Cobb DS & Armstrong SR (1997) Resin-dentin shear bond strength and interfacial ultrastructure with and without a hybrid layer *Operative Dentistry* **22(4)** 159-166.

Wang Y & Spencer P (2002) Quantifying adhesive penetration in adhesive/dentin interface using confocal Raman microspectroscopy *Journal of Biomedical Materials Research* **59(1)** 46-55.

©Operative Dentistry, 2004, **29-5,** 547-553

Effects of Aging on Mechanical Properties of Composite Restoratives: A Depth-sensing Microindentation Approach

AUJ Yap • SM Chung
Y Rong • KT Tsai

Clinical Relevance

Changes in hardness and modulus after aging in water were material-dependent. While no significant change in mechanical properties was detected for composites and the posterior compomer, a significant decrease in hardness and modulus was observed for the conventional compomer over time.

SUMMARY

This study investigated the effects of aging on the hardness and modulus of two composites (Tetric Ceram [TC], Vivadent; Esthet X [EX], Dentsply), a conventional (Compoglass [CG], Vivadent) and a posterior compomer (Dyract Posterior [DP], Dentsply) using a depth-sensing microindentation approach. Seven specimens (3-mm wide x 3-mm long x 2-mm deep) of each material were made and conditioned in distilled water at 37°C. Hardness and modulus of the materials were determined at seven and 30 days using depth-sensing microindentation testing with the Instron MicroTester. Hardness was determined by dividing the peak load over the maximum projected contact area while effective modulus was calculated by analysis of the loading/unloading P-h curves and the analytical model according to Oliver and Pharr (1992). Results were analyzed using one-way ANOVA/Scheffe's post-hoc test and Independent Samples *t*-test at significance level 0.05. Mean Vickers Hardness (HV) ranged from 46.60 to 58.67 and 44.44 to 59.41 at seven and 30 days, respectively. Mean indentation modulus ranged from 9.57 to 9.95 and 9.19 to 10.03 for the same time periods. At both time periods, EX was significantly harder than all the other materials and HV values for TC were significantly greater than CG. No significant difference in hardness and modulus was observed between seven and 30 days for all materials with the exception of CG. For the latter, a significant decrease in mechanical properties was detected over time.

*Adrian UJ Yap, BDS, MSc, PhD, FAMS, FADM, FRSH, associate professor, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

SM Chung, BEng (Hons), Meng, research fellow, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

Y Rong, BEng (Hons), student, Graduate Programme in Bioengineering, National University of Singapore, Republic of Singapore

KT Tsai, BSc, PhD, CEng, MIM, applications manager, Instron Singapore Pte Ltd, Republic of Singapore

*Reprint request: 5 Lower Kent Ridge Road, Singapore 119074, Republic of Singapore; e-mail: rsdyapuj@nus.edu.sg

INTRODUCTION

Resin composites have become the most commonly used materials for direct aesthetic restorations. They are, by definition, three-dimensional combinations of at least two chemically different materials with a distinct interface (Phillips, 1981). Dental composites consist of a resin matrix (organic phase), inorganic filler particles (dispersed phase), filler-matrix coupling agent (interface) and minor additions, including coloring pigments, stabilizers and polymerization initiators. Compomers or polyacid-modified composites are resin composites that contain either or both of the essential components of glass ionomer cements but at levels insufficient to promote acid-base cure reaction in the dark (McLean, Nicholson & Wilson, 1994). They were developed to combine the ease of use, aesthetics and physical properties of composites with the fluoride release of glass ionomer cements. While composites can be used to restore all cavity classes in anterior and posterior teeth, compomers are usually indicated for restoration of non-stress bearing areas like Class III and V cavities. The mechanical properties of conventional compomers are generally inferior to their composite counterparts (Peutzfeldt, García-Godoy & Asmussen, 1997; Yap & others, 2000a). In addition, these materials are designed to absorb water, which facilitates the acid-base reaction of the glass components to occur (Yap & others, 2000b; Huang & others, 2002). The latter may result in deterioration of mechanical properties over time. Compomer materials have been recently re-formulated for use in stress-bearing areas (Class I, II and IV cavities). Little is known about the effects of aging on the mechanical properties of these new compomers (Munack & others, 2001; Musanje, Shu & Darvell, 2001; Munksgaard, 2002).

Hardness may be defined as the resistance of a material to indentation or penetration (O'Brien, 1997). Among the properties related to hardness of a material are strength, proportional limit and ductility. Hardness has also been used to predict the wear resistance of a material and its ability to abrade or be abraded by opposing dental structures and materials (Anusavice, 1996). In addition to hardness, elastic modulus is another mechanical property important in determining the resistance to occlusal forces. Elastic modulus describes the relative stiffness of a material. Together with adhesive properties, elastic modulus plays an important role in preventing microleakage, secondary caries and/or filling dislodgement (Sabbagh, Vreven & Leloup, 2002). In stress-bearing areas, materials with low elastic modulus will deform more under masticatory stresses, resulting in catastrophic failures (Lambrechts, Braem & Vanherle, 1987). A high elastic modulus is required to withstand deformation and cusp fracture. For cervical cavities (Class V), materials must have a low modulus to allow the material to flex during tooth flexure.

Indentation tests provide important information on material deformation, while flexural and tensile tests yield results on both deformation and fracture of bulk specimens (Xu & others, 2000). Indentation offers information that may be more relevant to applications that involve localized, non-uniform deformation or point-contact, such as occlusal contacts with opposing teeth and surface asperities/third bodies during chewing and wear. It is especially useful when specimen dimensions are limited, such as in the case of composite restoration in a tooth preparation. In addition to hardness and fracture toughness (Ferracane, 1989; Xie & others, 2000; Yap, Low & Ong, 2000), instrumented indentation techniques can be used to continuously monitor loading-unloading during an indentation. This process provides information on the energy absorbed by the materials during indentation (Xu, Smith & Jahanmir, 1996) and the elastic modulus (Oliver & Pharr, 1992; Xu & others, 1998; Giannakopoulos & Suresh, 1999).

This study investigated the effects of aging on the hardness and modulus of composite and compomer restorative materials using depth-sensing microindentation testing. The mechanical properties of the different materials were also compared at various time intervals.

METHODS AND MATERIALS

Two resin composites (Tetric Ceram [TC], Esthet X [EX]), a conventional (Compoglass [CG]) and a posterior compomer (Dyract Posterior [DP]), were selected for this study. Table 1 shows the technical profiles of the materials. All materials were of the A2 shade. The 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 acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide was placed over the acetate strip and pressure was applied to extrude excess materials. The specimens were then light polymerized using the Spectrum light curing unit (Dentsply-Caulk, Milford, DE, USA) according to manufacturers' cure times. The mean intensity of the light source (460 ± 4 mW/cm^2) was determined with a commercial radiometer (Cure Rite, EFOS Inc, Ontario, Canada). Immediately after light polymerization, the acetate strips were removed and the specimens stored in distilled water at 37°C. Seven specimens were made for each material.

The composite and compomer specimens were subjected to depth-sensing microindentation testing after seven and 30 days of aging in water. The specimens were blotted dry, positioned centrally beneath a custom-designed indentation head unit (Figure 1) mounted to an Instron Micro-Test system (Model 5848, Instron Corporation, Canton, MA, USA). A static load cell of 1 kN capacity was built into the indentation head unit for force measurement and penetration depth was

measured directly using a LVDT (linear variable differential transducer) that was in contact with the specimen surface. This surface reference technique was employed to eliminate compliance issues associated with the test system. A digital imaging system was also incorporated into the indentation head unit. This consisted of an optical transmission unit (Moritex Corp, Tokyo, Japan), an illumination light source (MHF-M1002; Moritex Corp), a microscopic digital camera system (DP12; Olympus Optical Co Ltd, Tokyo, Japan) and image analysis software (Micro Image 4.0, Media Cybernatics, Silver Spring, MD, USA). The imaging system was used to inspect the specimen surface before and after indentation testing and to compute indentation size. A pneumatic actuating system was used to toggle between the Vickers indenter (four-sided square indenter with known geometry) and the objective lens (CF Pan 10-50X; Nikon Corp, Tokyo, Japan) which was mounted to the imaging system.

Table 1: *Technical Profiles of the Materials Evaluated*

Material (Lot #)	Cure Time	Resin Matrix	Filler Type	Filler Size (μm)	Filler Content (% vol)
Tetric Ceram (TC) (D54267)	40 seconds	BisGMA, UDMA, TEGDMA	Barium glass, Ytterbium trifluoride, Ba-Al-fluorosilicate, Silicon dioxide	0.04 – 1.0	60
Esthet X (EX) (0108102)	20 seconds	BisGMA, BisEMA, TEGDMA	Ba-Al-fluorosilicate, Silicon dioxide	0.04 – 1.0	60
Compoglass (CG) (D51370)	40 seconds	UDMA, PEGDMA, DCDMA	Ytterbium trifluoride, Ba-Al-fluorosilicate	1.0 (mean)	55
Dyract Posterior (DP) (0107001376)	20 seconds	UDMA, TCB	Strontium-fluoro-silicate, strontium fluoride	0.8 (mean)	47

BisEMA = Ethoxylated bisphenol-A-glycidyl methacrylate
BisGMA = Bisphenol-A-dimethacrylate
DCDMA = Cycloaliphatic dicarbonic acid dimethacrylate
PEGDMA = Polyethylene glycoldimethacrylate
TEDGMA = Triethylene glycol dimethacrylate
TCB = Reaction product butane tetracarboxylic acid and HEMA
UMDA = Urethane dimethacrylate

The specimens were indented at a rate of 0.0005 mm/second until a maximum load of 10 N was attained. The peak load was then held for 10 seconds and unloaded fully at a rate of 0.0002 mm/seconds. Indentation load (P) and the corresponding indenter displacement (h) was recorded continuously during the loading-unloading cycle. Figure 2 shows a typical P-h curve during a loading-unloading cycle. The moduli of the materials were then computed using the following equation, which was derived from previously developed analytical solutions in engineering and materials science (Doerner & Nix, 1986; Sneddon, 1965; Stilwel & Tabor, 1961; Bulychev & others, 1975):

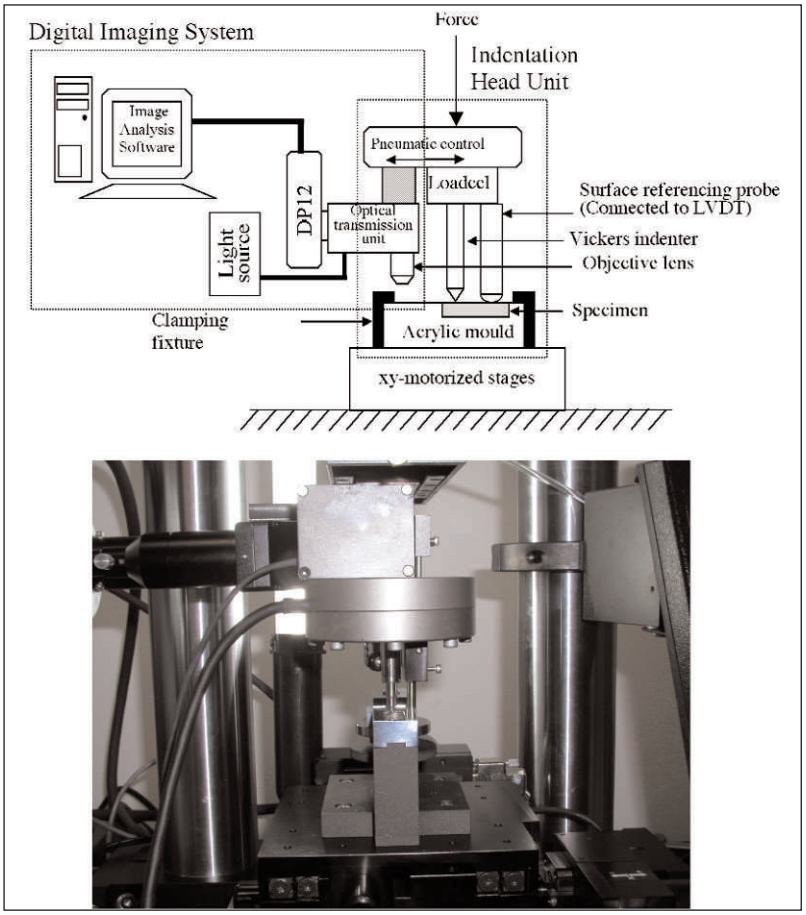


Figure 1. *The depth-sensing microindentation test set-up.*

where

$$E_{in} = \frac{1-v_{in}^2}{\left(\frac{2\delta\sqrt{A_{max}}}{\sqrt{\pi}S} - \frac{(1-v_o^2)}{E_o}\right)}$$

E_{in} = elastic modulus of the test material

E_o = elastic modulus of the indenter

v_o = Poisson ratio of the indenter

v_{in} = Poisson ratio of the test material

δ = numerical factor

A_{max} = maximum projected contact area for the Vickers indenter

S = unloading contact stiffness derived from the first derivative of the fitted power-law equation at the maximum indentation load

For the diamond Vickers indenter, E_o = 1141 GPa, v_o= 0.71 and δ = 1.012 (Simmons & Wang, 1971; King, 1987). The poisson ratio for the test material was modeled at 0.3 based on previous studies and the authors' pilot study (Nakayama & others, 1974; Chabrier, Lloyd & Scrimgeour, 1999). To account for "pile-up" or "sink-in" effects in the indented materials, the model proposed by Oliver and Pharr (1992) was used to determine the projected contact area (A_{max}) at peak load:

$$A_{max} = 24.5h_c^2 \text{ and } h_c = h_{max} - \varepsilon\frac{P_{max}}{S}$$

where

P_{max} is the maximum load,

h_{max} is the indentation depth at maximum load,

ε is the geometric constant (0.75 for Vickers indenter)

Vicker's hardness was calculated using the following equation:

$$H = \frac{P_{max}}{A_{max}}$$

Inter-material comparisons at the two time intervals were analyzed using one-way ANOVA/Scheffe's post-hoc test; and the effects of aging on mechanical properties were determined using Independent Sample's *t*-test at significance level 0.05. Correlation between modulus and hardness was assessed using Pearson's correlation at significance level 0.01.

RESULTS

Table 2 reflects the mean modulus and hardness of the different materials. The results of statistical analysis are shown in Table 3.

Mean modulus ranged from 9.57 to 9.95 GPa and 9.19 to 10.02 GPa for seven and 30 days, respectively. Mean hardness ranged from 46.60 to 58.67 HV at seven days and 44.44 to 59.41 at 30 days. Although no significant difference in modulus was observed between materials at seven and 30 days, significant differences in hardness were detected. Esthet X (EX) was significantly harder than the other materials evaluated at both time intervals. In addition, Tetric Ceram (TC) was significantly harder than Compoglass (CG). At 30 days, the HV of DP (Dyract Posterior) was also significantly greater than CG. With the exception of CG, no significant change in modulus and hardness was observed after aging. For CG, a significant decrease in both modulus and hardness was observed after aging in water. The correlation between modulus and hardness was significant and positive; however, it was weak with a correlation coefficient of r=0.35.

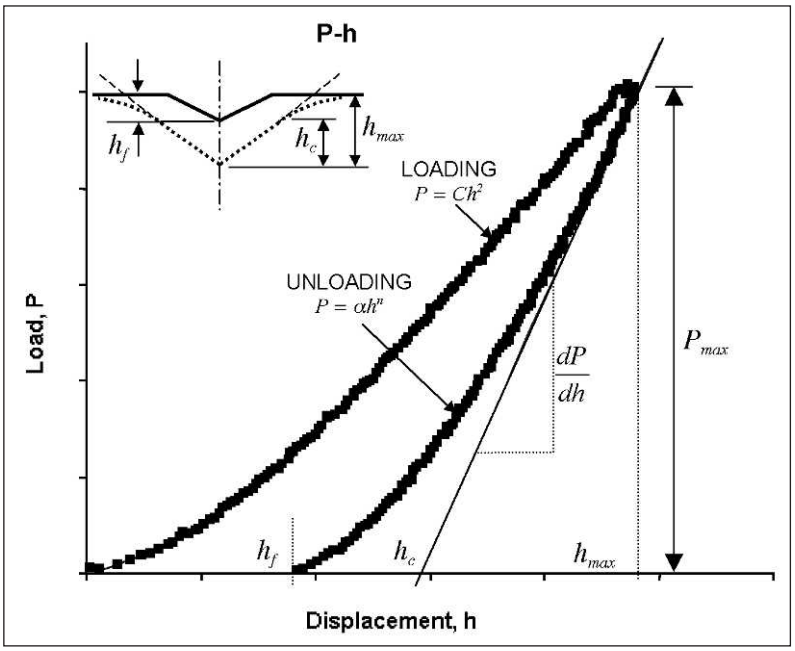


Figure 2. *A typical* P-h *curve during a loading-unloading cycle where* h_{max} *is the maximum indenter displacement at peak indentation load (*P_{max}*),* $\frac{dP}{dh}$ *is the slope of the* P-h *curve during the initial unloading stage,* h_f *and* h_c *are the final (residual) and contact depth of the indent impression, respectively.*

DISCUSSION

The use of nanoindenters for the characterization of mechanical properties of tooth tissues and materials has been widely reported in the literature (Willems & others, 1993; Xu & others, 1998, 2000; Mahoney & others, 2000; Marshall & others, 2001). Elastic properties, hardness and other related properties can be obtained without the visualization of indentations. Although nanoindentation techniques are useful for the characterization of homogenous materials, they cannot be used to characterize the bulk properties of biphasic materials. Most dental materials, including resin composites, are biphasic in nature. Composite restoratives

consist of hard glass fillers within a softer polymer resin. As the glass fillers can be relatively larger than the nanoindenters, data obtained from nanoindentation may only reflect the mechanical properties of the fillers and not the composite material. Microindentation techniques provide an efficient solution to the above mentioned problem, as it represents the bulk behavior of materials (Alcalá, Giannakopoulos & Suresh, 1998). Elastic modulus data obtained from microindentation testing may be more clinically relevant as alternative testing methodologies, such as flexural tests, often involve large specimens that are not clinically realistic and difficult to prepare without flaws (Yap & Teoh, 2003). In addition to wasting material and time, these large specimens are also inhomogeneous due to the need for overlapping irradiation during preparation.

Table 2: *Mean Modulus and Hardness of the Various Materials at the Two Time Intervals*

Materials	Elastic Modulus (GPa)		Vickers Hardness (HV)	
	7 days	30 days	7 days	30 days
Tetric Ceram (TC)	9.95 (0.68)	9.74 (0.59)	52.65 (2.99)	50.67 (2.37)
Esthet X (EX)	9.77 (0.35)	9.68 (0.48)	58.67 (1.27)	59.41 (1.23)
Compoglass (CG)	9.68 (0.44)	9.19 (0.22)	46.60 (2.26)	44.44 (1.11)
Dyract Posterior (DP)	9.57 (0.37)	10.02 (0.71)	49.69 (1.71)	50.31 (1.98)

Standard deviations in parentheses.

Table 3: *Comparison of Modulus and Hardness Between Materials*

Time	Variables	Differences
7 days	**Modulus**	NS
	Hardness	EX > TC, DP, CG, TC > CG
30 days	**Modulus**	NS
	Hardness	EX > TC, DP, CG, TC, DP > CG

Results on one-way ANOVA and Scheffe's test ($p<0.05$); > indicates statistical significance, while NS indicates no statistical significance.

Instrumented indentation methods provide a continuous record of the variation of indentation load as a function of depth of penetration (Zidi & others, 2000). The basic results from this test include the indentation curve giving the normal load as a function of penetration depth (*P-h* curve). Interpretation of the graph is not straightforward, and careful mechanical analysis is required to obtain intrinsic material properties from the global response of a complicated system involving the whole composite structure (fillers, matrix and interface) under a highly heterogeneous stress state. Simplified assumptions are often made in order to obtain tractable results. Complications preventing clear interpretation of indentation results arise from the "pile-up" or "ink-in" of the material around the indenter, which is primarily affected by the plastic properties of the material (Giannakopoulos, Larsson & Vestergaard, 1994). The latter may be determined in part by the volume percent of resin in the composite restorative. As a consequence of "pile-up" or "sink-in," vast differences may arise between the true contact area, which is often difficult to assess *in situ* during indentation and the apparent contact area, which is usually observed after indentation (Giannakopoulos & Suresh, 1999). Knowledge of the relationship between indentation load and the true (projected) area is, however, essential to extract the mechanical properties from instrumented indentation. This difficulty can be overcome if explicit expressions relating the true contact area (A) and the depth of penetration of the indenter (h) into the composite materials are known for the different indenter geometries. Oliver and Pharr (1992) proposed a theoretical framework for instrumented sharp indentation and a methodology which enables the determination of elastic and plastic properties of materials by employing instrumented indentation. Their method was selected for this study, as it provides unique correlations between penetration depth (h) and true contact (A) for commercially available sharp indenters, circumvents the need for contact area measurement through the visual observation and takes into account "pile-up" and "sink-in." Their model was based on more general assumptions when compared to other methods in the literature (Dao & others, 2001; Giannakopoulos & Suresh, 1999) and has been validated on a wide spectrum of materials, ranging from ductile metals to brittle ceramics (Oliver & Pharr, 1992).

The modulus of the materials evaluated generally increased with increasing filler volume fraction (DP < CG < EX < TC). This finding corroborated previous studies that investigated the effect of filler volume on composite stiffness (Braem & others, 1989; Kim & others, 1994). However, the difference in modulus between materials was not significant despite the moderate disparity in filler volumes (47% to 60%). This may be attributed in part to the use of acetate strips and the application of pressure during specimen preparation, which results in the predominance of microfillers and resin on specimen surfaces (Yap, Teoh & Tan, 2001). At both time intervals, EX was significantly harder than the other materials and TC was significantly harder than CG. The findings were consistent with the filler loading of the materials and previous studies (Ferracane, 1995). The only exception to the filler volume–hardness trend—is the significant difference in hardness between DP and CG at 30 days. This may be

attributed to the significant softening of CG with storage in water.

With the exception of CG, no significant difference in hardness and modulus was observed between seven and 30 days of water exposure at 37°C. For GC, significant softening and decreased stiffness was observed after aging. In addition to fluoroalumino silicate glass, CG also employs Ytterbium trifluoride (YbF_3) for fluoride release. Yap, Khor and Foo (1999) found that the fluoride release of CG was significantly greater than another compomer and comparable to a resin-modified glass ionomer. They attributed the significantly higher fluoride release of CG to the additional fluoride source provided by YbF_3. Fluoride release is by means of an exchange mechanism (Arends, Dijkman & Dijkman, 1995). Water diffuses into the compomer, reaches the YbF_3 filler and causes fluoride release according to the reaction $YbF_3 + 3OH^- \rightarrow Yb(OH)_3 + 3F^-$. The fluoride ion diffuses out of the compomer in the direction of the lowest fluoride concentration. Although physical and mechanical properties can theoretically be preserved with the use of fluoride-releasing fillers like YbF_3, these fillers may become completely or partially dislodged as the outer layers dissolve. The decreased hardness and modulus of CG could be accounted for by the latter and by water sorption of the resin component. In view of the aforementioned, CG should not be used for stress-bearing areas where a high modulus is required. Although the modulus of DP was similar to composites, its use in posterior stress-bearing situations should still be approached with caution due to its relatively lower hardness.

The depth-sensing microindentation approach to the characterization of hardness and modulus of resin-based filling materials was found to be highly efficient. In addition to time and material savings, it allows for the time-dependent study of material properties using the same specimen (due to the micro-scale of the indentation, numerous indentations can be made on the same specimen over time). The effect of surface finish on microindentation modulus is not known and warrants more in-depth investigations. Although penetration depths at P_{max} would involve contact area with the subsurface layers, the influence of the microfiller/resin rich surface layer on the *P-h* curve has not been established. Further studies pertaining to the development of depth-sensing microindentation technologies for the characterization of dental biomaterials and tissues are currently being undertaken.

CONCLUSIONS

Under the conditions of this *in vitro* study, the following conclusions can be made:

- The effect of aging on hardness and modulus of resin-based filling materials is material-dependent.
- While no significant change in mechanical properties was detected for composites and the posterior compomer, a significant decrease in hardness and modulus was observed for the conventional compomer over time.
- Conventional compomers should not be used in stress-bearing situations.
- The depth-sensing microindentation approach to characterizing the hardness and modulus of resin-based filling materials is efficient and promising.

(Received 5 June 2003)

References

Alcalá J, Giannakopoulos AE & Suresh S (1998) Continuous measurements of load-penetration curves with spherical micro-indenters and the estimation of mechanical properties *Journal of Material Research* **13(5)** 1390-1400.

Anusavice KL (1996) Mechanical properties of dental materials in *Phillip's Science of Dental Materials* 10th edition Philadelphia WB Saunders Co p 69.

Arends J, Dijkman GEHM & Dijkman AG (1995) Review of fluoride release and secondary caries reduction by fluoridating composites *Advanced Dental Research* **9(4)** 367-376.

Braem M, Finger W, Van Doren VE, Lambrechts P & Vanherle G (1989) Mechanical properties and filler fraction of dental composites *Dental Materials* **5(5)** 346-348.

Bulychev SI, Alekhin VP, Shorshorov MK, Ternovskii AP & Shnyrev GD (1975) Determining Young's modulus from the indentation penetration diagram *Industrial Laboratory* **41(9)** 1137-1140.

Chabrier F, Lloyd CH & Scrimgeour SN (1999) Measurement at low strain rates of the elastic properties of dental polymeric materials *Dental Materials* **15(1)** 33-38.

Dao M, Challacoop N, Van Vliet KJ, Venkatesh TA & Suresh S (2001) Computational modeling of the forward and reverse problems in instrumented sharp indentation *Acta Materialia* **49(19)** 3899-3918.

Doerner MF & Nix WD (1986) A method for interpreting the data from depth-sensing indentation instrumentations *Journal of Materials Research* **1(4)** 601-609.

Ferracane JL (1989) Indentation fracture toughness testing and crack propagation mode in dental composites *Materials Research Society Symposium Proceedings* **110** 619-624.

Ferracane JL (1995) Current trends in dental composites *Critical Review in Oral Biology and Medicine* **6(4)** 302-318.

Giannakopoulos AE, Larsson PL & Vestergaard R (1994) Analysis of Vickers indentation *International Journal of Solids and Structures* **31(19)** 2679-2708.

Giannakopoulos AE & Suresh S (1999) Determination of elastoplastic properties by instrumented sharp indentation *Scripta Materialia* **49(10)** 1191-1198.

Huang C, Tay FR, Cheung GS, Kei LH, Wei SH & Pashley DH (2002) Hygroscopic expansion of a compomer and a composite on artificial gap reduction *Journal of Dentistry* **30(1)** 11-19.

King RB (1987) Elastic analysis of some punch problems for a layered medium *Industrial Laboratory* **23(12)** 1657-1664.

Kim KH, Park JH, Imai Y & Kishi T (1994) Microfracture mechanisms of dental resin composites containing spherically-shaped filler particles *Journal of Dental Research* **73(2)** 499-504.

Lambrechts P, Braem M & Vanherle G (1987) Buonocore Memorial Lecture Evaluation of clinical performance for posterior composite resins and dentin adhesives *Operative Dentistry* **12(2)** 53-78.

Mahoney E, Holt A, Swain M & Kilpatrick N (2000) The hardness and modulus of elasticity of primary teeth: An ultra-micro-indentation study *Journal of Dentistry* **28(8)** 589-594.

Marshall GW, Balooch M, Gallagher RR, Gansky SA & Marshall SJ (2001) Mechanical properties of the dentinoenamel junction: AFM studies of nanohardness, elastic modulus and fracture *Journal of Biomedical Material Research* **54(1)** 87-95.

McLean JW, Nicholson JW & Wilson AD (1994) Proposed nomenclature for glass ionomer dental cements and related materials *Quintessence International* **25(9)** 587-589.

Munack J, Haubert H, Dogan S & Geurtsen W (2001) Effects of various storage media on surface hardness and structure of four polyacid-modified composites resins ("compomers") *Clinical Oral Investigations* **5(2)** 254-259.

Munksgaard EC (2002) Changes in expansion and mechanical strength during water storage of a traditional and three modified resin composites *Acta Odontological Scandinavia* **60(4)** 203-207.

Musanje L, Shu M & Darvell BW (2001) Water sorption and mechanical behaviour of cosmetic direct restorative materials in artificial saliva *Dental Materials* **17(5)** 394-401.

Nakayama WT, Hall DR, Grenoble DE & Katz JL (1974) Elastic properties of dental resin restorative materials *Journal of Dental Research* **53(5)** 1121-1126.

O'Brien WJ (1997) *Physical Properties in Dental Materials and Their Selection* Illinois Quintessence Publishing Company p 18.

Oliver WC & Pharr GM (1992) An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments *Journal of Materials Research* **7(6)** 1564-1568.

Peutzfeldt A, García-Godoy F & Asmussen E (1997) Surface hardness and wear of glass ionomers and compomers *American Journal of Dentistry* **10(1)** 15-17.

Phillips RW (1981) Symposium on composite resins in dentistry *Dental Clinics of North America* **25(2)** 209-218.

Sabbagh J, Vreven J & Leloup G (2002) Dynamic and static moduli of elasticity of resin-based materials *Dental Materials* **18(1)** 64-71.

Simmons G & Wang H (1971) *Single Crystal Elastic Constants and Calculated Aggregate Properties: A Handbook* 2nd edition Cambridge MIT Press.

Sneddon IN (1965) The relation between load and penetration in the axissymmetric Boussinesq problem for a punch of arbitrary profile *International Journal of Engineering Science* **3(1)** 47-57.

Stilwel NA & Tabor D (1961) Elastic recovery of conical indentations *Proceedings of the Physical Society of London* **78** 169-179.

Willems G, Celis JP, Lambrechts P, Braem M & Vanherle G (1993) Hardness and Young's modulus determined by nanoindentation technique of filler particles of dental restorative materials compared with human enamel *Journal of Biomedical Materials Research* **27(6)** 747-755.

Xie D, Brantley WA, Culbertson BM & Wang G (2000) Mechanical properties and microstructures of glass ionomer cements *Dental Materials* **16(2)** 129-138.

Xu HH, Smith DT & Jahanmir S (1996) Influence of microstructure on indentation and machining of dental glass-ceramics *Journal of Materials Research* **11(9)** 2325-2327.

Xu HH, Smith DT, Jahanmir S, Romberg E, Kelly JR, Thompson VP & Rekow ED (1998) Indentation damage and mechanical properties of human enamel and dentin *Journal of Dental Research* **77(3)** 472-480.

Xu HH, Smith DT, Schumacher GE, Eichmiller FC & Antonucci JM (2000) Indentation modulus and hardness of whisker-reinforced heat-cured dental resin composites *Dental Materials* **16(4)** 248-254.

Yap AU, Khor E & Foo SH (1999) Fluoride release and antibacterial properties of new-generation tooth-colored restoratives *Operative Dentistry* **24(5)** 297-305.

Yap AU, Low JS & Ong LF (2000) Effect of food-simulating liquids on surface characteristics of composite and polyacid-modified composite restoratives *Operative Dentistry* **25(3)** 170-176.

Yap AU, Tan DT, Goh BK, Kuah HG & Goh M (2000a) Effect of food-simulating liquids on the flexural strength of composite and polyacid-modified composite restoratives *Operative Dentistry* **25(3)** 202-208.

Yap AU & Teoh SH (2003) Comparison of flexural properties of composite restoratives using the ISO and mini-flexural tests *Journal of Oral Rehabilitation* **30(2)** 171-177.

Yap AU, Teoh SH & Tan KB (2001) Three-body abrasive wear of composite restoratives *Operative Dentistry* **26(2)** 145-151.

Yap AU, Wang HB, Siow KS & Gan LM (2000b) Polymerization shrinkage of visible-light-cured composites *Operative Dentistry* **25(2)** 98-103.

Zidi M, Carpentier L, Chateaminois A & Sidoroff F (2000) Quantitative analysis of the micro-indentation behavior of fiber reinforced composites: Development and validation of an analytical model *Composite Science and Technology* **60(3)** 429-437.

©Operative Dentistry, 2004, **29-5,** 554-559

Influence of Environmental Conditions on Dentin Bond Strengths of One-application Adhesive Systems

Y Chiba • M Miyazaki
A Rikuta • BK Moore

Clinical Relevance

Environmental conditions, such as higher relative humidity, might compromise the bonding ability of one-application adhesive systems.

SUMMARY

Resin composites are considered susceptible to environmental conditions that might affect bond strength. This study investigated the influence of relative humidity and temperature on the dentin bond strength of newly developed one-application adhesive systems. Bonding systems employed in this study were five commercial one-application adhesive systems. Labial surfaces of bovine incisors were ground wet on 600-grit SiC paper. The teeth were transferred to a controlled temperature and humidity chamber and the specimens were prepared in six different environmental conditions, A) 25 ± 0.5°C, 50 ± 5% RH, B) 25 ± 0.5°C, 80 ± 5% RH, C) 25 ± 0.5°C, 95 ± 5% RH, D) 37 ± 0.5°C, 50 ± 5% RH, E) 37 ± 0.5°C, 80 ± 5% RH, F) 37 ± 0.5°C, 95 ± 5% RH. The dentin surfaces were treated according to each manufacturer's instructions. Resin composites of each bonding system were condensed into a mold and light irradiated. After storage in 37°C water for 24 hours, 10 specimens per group were tested in a shear mode in a testing machine at a crosshead speed of 1.0 mm/minute. Statistical analysis was carried out with one-way ANOVA followed by Duncan's multiple range tests at a *p*-value of 0.05. The dentin bond strengths of one-application adhesive systems decreased with increasing RH. These data suggest that the relative high humidity in the oral environment needs consideration in developing clinical procedures for management of these one-application adhesive systems.

Yasushi Chiba, DDS, graduate student, Nihon University School of Dentistry, Department of Operative Dentistry, Tokyo, Japan

*Masashi Miyazaki, DDS, PhD, assistant professor, Nihon University School of Dentistry, Department of Operative Dentistry, Tokyo, Japan

Akitomo Rikuta, DDS, PhD, instructor, Nihon University School of Dentistry, Department of Operative Dentistry, Tokyo, Japan

B Keith Moore, PhD, professor, Indiana University School of Dentistry, Department of Restorative Dentistry, Division of Dental Materials, Indianapolis IN, USA

*Reprint request: 1-8-13, Kanda-Surugadai, Chiyoda-Ku, Tokyo 101-8310, Japan; e-mail: miyazaki-m@dent.nihon-u.ac.jp

INTRODUCTION

Good adhesion of resin composite to tooth substrate has been a goal of dentistry for many years. Reliable adhesion would reduce microleakage and provide a stable bond (Van Meerbeek & others, 1998; Perdigão & Lopes, 1999). Significant efforts have been made to develop restorative materials that bond to dentin in the oral environment without complicated clinical procedures. Several new adhesive systems that employ simultan-

eous etching of enamel and dentin with phosphoric acid or a self-etching primer have been introduced (Eick & others, 1997). Recently, one-application bonding systems that combine the function of self-etching primer and bonding agent have been developed (Van Meerbeek & others, 2003). Theoretically, the acidic one-application adhesive dissolves the smear layer, incorporating it into the mixture and demineralizing the superficial dentin, which then hardens after light irradiation (Tay & others, 2001).

Although the most reliable conclusions about the performance of adhesive systems in the oral environment must be derived from long-term clinical trials, laboratory tests are still useful for comparing newly developing adhesive systems. The most common method for evaluating the bonding properties of restorative materials is bond strength measurements that have been used to develop improved adhesive systems. The dentin bonding ability of these materials is usually tested under standard room temperature conditions of 23°C and 50% relative humidity (RH). However, environmental conditions are known to be important factors in influencing bond strengths to dentin, and the influence of extrinsic dentin wetness has been reported to differ among the adhesive systems tested. Some systems were more sensitive to the wetness than others (Plasmans & others, 1993; Burrow & others, 1995; Nystrom & others, 1998).

Since water is an indispensable component to generating hydrogen ions in one-application adhesive systems, a resin monomer which can polymerize in the presence of water is needed. On the other hand, water sorption and dissolution of polymerized resin containing hydrophilic resin monomer could result in the diffusion of water into the resin matrix (Tay & Pashley, 2003). Differential water movement across the cured bond agent has been speculated to occur, leading to decreased bond strength (Tay & others, 2002). If the cured bonding layer of the one-application system acts as a semi-permeable membrane that allows water diffusion, environmental conditions such as temperature and RH in the oral cavity could be important during placement of the resin composite.

This study investigated the influence of RH and temperature on the dentin bond strengths of one-application adhesive systems. The null hypothesis tested in this study is that the environmental humidity and temperature would not alter the dentin bond strengths of recently developed one-application adhesive systems.

METHODS AND MATERIALS

Adhesive systems with the resin composites employed in this study were five one-application systems, Adper Prompt L-Pop with Filtek Z250 (3M ESPE, St Paul, MN, USA), AQ Bond Plus with Metafil C (Sun Medical, Shiga, Japan), One-Up Bond F with Palfique Estelite (Tokuyama Dental, Tokyo, Japan), Reactmer Bond with Reactmer (Shofu Inc, Kyoto, Japan) and Xeno III with Xeno CF (Dentsply DeTrey GmbH, Konstanz, Germany), all of which are listed in Table 1. An Optilux 501 curing unit (Demetron/Kerr, Danbury, CT, USA), whose light intensity was adjusted to 600 mW/cm^2 as measured with a dental radiometer (Model 100, Demetron/Kerr), was used.

Mandibular incisors extracted from two-to-three year old cattle and stored frozen (-20°C) for up to two weeks were used as a substitute for human teeth. After removing the roots with a low-speed saw, the pulps were removed and the pulp chamber of each tooth was filled with cotton to avoid penetration of the embedding media. The labial surfaces of bovine incisors were ground on wet 240-grit SiC paper to a flat dentin surface. Each tooth was then mounted in cold-curing acrylic resin to expose the flattened area and placed in tap water to reduce the temperature rise from the exothermic polymerization reaction. Final finish was accomplished by grinding on wet 600-grit SiC paper, then ultrasonic cleaning with distilled water for three minutes to remove the debris. Double-sided adhesive tape with a 4-mm diameter hole was firmly attached to the flattened surface to restrict the adhesive area.

The embedded teeth were transferred to a controlled temperature and humidity chamber (Type V-85, Atom Corp, Tokyo, Japan). Because the temperature of the samples was different from the temperature inside the chamber, they were left for 10 minutes to equilibrate to one of six environmental conditions; A) 25 ± 0.5°C, 50 ± 5% RH, B) 25 ± 0.5°C, 80 ± 5% RH, C) 25 ± 0.5°C, 95 ± 5% RH, D) 37 ± 0.5°C, 50 ± 5% RH, E) 37 ± 0.5°C, 80 ± 5% RH, F) 37 ± 0.5°C, 95 ± 5% RH.

The dentin surface was then treated according to each manufacturer's instructions, and a Duracon mold (2-mm height, 4-mm internal diameter) was placed on the tooth surface to form and hold the restorative resin. Resin composite from the same manufacturer as the one-application adhesive was condensed into the mold, then irradiated for 30 seconds. The finished specimens were transferred to 37°C distilled water for 24 hours from the start of light exposure to the material. Ten specimens per group were tested in a shear mode in an Instron testing machine (Type 4204, Instron Corp, Canton, MA, USA) at a crosshead speed of 1.0 mm/minute. The fourth was applied to the dentin flattened surface close to the resin bonding area. Shear bond strengths in MPa were calculated from the peak load at failure divided by the specimen surface area.

After testing, the specimens were examined in an optical microscope at a magnification of 10x to determine the location of the bond failure. The test area on the tooth was divided into eight segments, and the per-

Table 1: *One-application Systems Used*

Code	Adhesive Composition	Lot #	Resin Composite	Lot #	Manufacturer
AP	Adper Prompt L-pop Water, parabenes, fluoride complex methacrylates phosphoric acid ester photoinitiator	141994	Filtek Z250	9J03	3M ESPE
AQ	AQ Bond Plus Water, acetone, 4-META, HEMA UDMA, photonitiator *p*-toluenesulfinic acid sodium salt	FW1 FX1	Metafil C	FL1	Sun Medical
OF	One-Up Bond F Water, MAC-10, HEMA, MMA, multifunctional methacrylic monomer fluoroaluminosilicate glass, photonitiator (aryl borate catalyst)	A: 0712 B: 0709	Palfique Estelite	210	Tokuyama Dental
RB	Reactmer Bond Water, acetone, 4-AET, 4-AETA, UDMA, HEMA, PRG filler, fluoroaluminosilicate glass, trimethyl barbituric acid, photonitiator *p*-toluenesulfinic acid sodium salt	A: 100005 B: 100005	Reactmer	110007	Shofu Inc
XE	Xeno III Water, ethanol, HEMA, butylated hydroxy toluene, photonitiator phosphoric acid polymethacrylate resin 4-dimethylamino-ethylamino-ethyl-benzonate	0210000032	Xeno CF	347	Dentsply DeTrey

Table 2: *Influence of Environmental Conditions on Failure Mode of One-application Systems to Bovine Dentin*

Code	25±0.5°C, 50±5%	25±0.5°C, 80±5%	25±0.5°C, 95±5%	37±0.5°C, 50±5%	37±0.5°C, 80±5%	37±0.5°C, 95±5%
AP	0/ 1/ 0/ 9	0/ 0/ 0/ 10	0/ 0/ 0/ 10	0/ 0/ 0/ 10	0/ 1/ 0/ 9	0/ 0/ 0/ 10
AQ	0/ 1/ 0/ 9	0/ 0/ 0/ 10	0/ 0/ 0/ 10	0/ 0/ 0/ 10	0/ 1/ 0/ 9	0/ 0/ 0/ 10
OF	2/ 5/ 1/ 2	1/ 5/ 0/ 4	0/ 4/ 0/ 6	1/ 4/ 0/ 5	1/ 6/ 0/ 3	0/ 1/ 1/ 8
RB	1/ 5/ 2/ 2	0/ 2/ 2/ 6	2/ 5/ 1/ 2	1/ 3/ 3/ 3	1/ 3/ 1/ 5	0/ 3/ 0/ 7
XE	0/ 7/ 0/ 3	0/ 7/ 0/ 3	0/ 0/ 0/ 10	0/ 8/ 0/ 2	0/ 7/ 0/ 3	0/ 0/ 0/ 10

Number of specimens with mode of failure: cohesive failure in resin/cohesive failure in adhesive/cohesive failure indentin/adhesive failure.

centage that was free of adhesive or restorative material was estimated. The types of failures were determined based on the predominant percentage of substrate free material as adhesive failure, cohesive failure in resin composite, cohesive failure in adhesive and cohesive failure in dentin.

The data obtained for each group were subjected to two-way ANOVA at a p-value of 0.05 using a computer statistics package (Sigma Stat Ver 2.03, SPSS Inc, Chicago, IL, USA).

For ultrastructure observation of the resin/dentin interface, bonded specimens stored in 37°C distilled water for 24 hours were embedded in epoxy resin and longitudinally sectioned with a diamond saw. The sectioned surfaces were polished to a high gloss with abrasive discs followed by diamond pastes down to 0.1-µm particle size. The surfaces were fixed in 2.5% glutaraldehyde in a cacodylate buffer solution, dehydrated in ascending grades of *tert*-butyl alcohol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes and 100% for two hours), then transferred from the final 100% bath to a critical-point dryer (Model ID-3, Elionix, Tokyo, Japan) for 30 minutes. The polished surfaces were then subjected to argon-ion beam etching (EIS-200ER, Elionix, Tokyo, Japan) for 15 seconds with the ion beam (accelerating voltage 1.0 kV, ion current density 0.4 mA/cm^2) directed perpendicular to the polished surface. The surfaces were coated in a vacuum evaporator with a thin film of Au. Observation was conducted under a scanning electron microscope (SEM, JSM-5400, LEOL, Tokyo, Japan) at an operating voltage of 15 kV.

RESULTS

Table 2 and Figure 1 present the mean shear bond strengths and fracture modes with various environmental conditions. The effect of the factor temperature on bond strength was not significant at $p<0.05$ for all adhesive systems used. RH, on the other hand, significantly influenced bond strength. No interaction

between temperature and RH was found. For system AP, there was a significant difference between each pair of RH conditions. For the other resins, there was no significant difference between 50% and 80% RH, but both 50% and 80% were significantly different from 95%. The fracture mode of the debonded specimens for AP and AQ was almost entirely adhesive, while the other systems showed an increased tendency to fail in the adhesive mode with higher RH.

Figures 2 and 3 show representative SEM observations of the resin-dentin interface. After argon ion beam etching, close adaptation between resin and dentin with low resistance to argon ion bombardment was observed as a hybrid layer with specimens made under the lowest RH conditions and the width of this layer was 0.5-2.0 μm. The hybrid layer was not clearly seen and porosities and gaps were observed at the adhesive resin-restorative resin interface in specimens made under higher RH. This was a common finding for systems such as AP, AQ and XE.

DISCUSSION

The adhesive of one-application systems is a hydrophilic solution that is extremely effective in wetting the tooth surface (Kitasako & others, 2000). The etching effect of these systems is related to the acidic monomers that interact with the mineral component of the tooth substrate and enhance monomer penetration (Ikemura, Kouro & Endo,

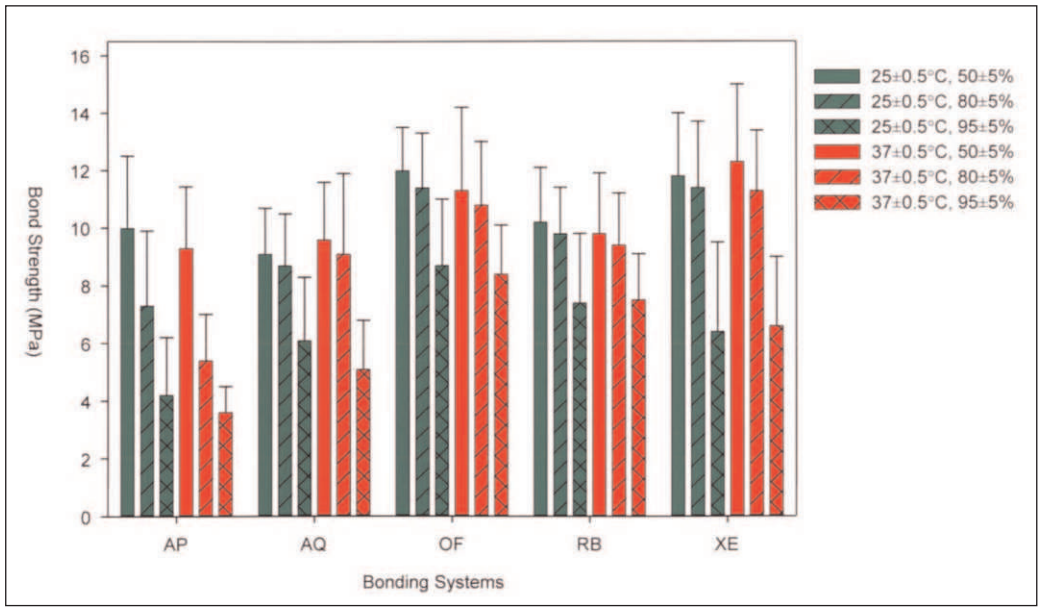


Figure 1: *Bond strengths of bonding systems as a function of temperature and humidity.*

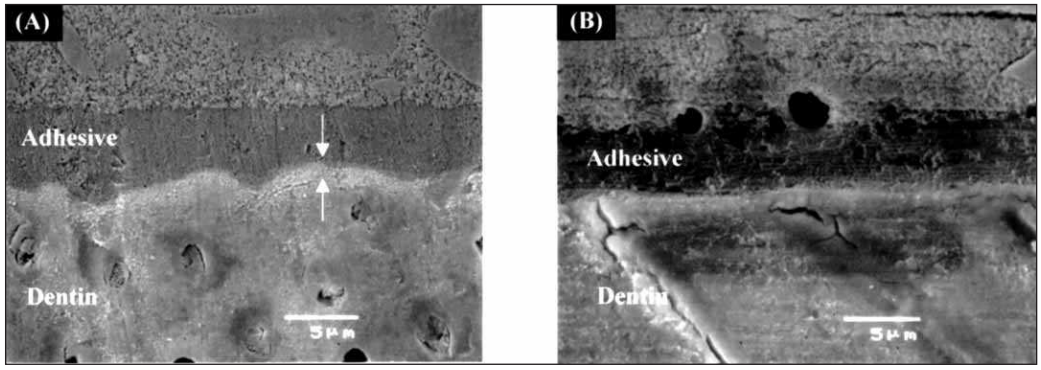


Figure 2. *SEM observation of dentin/resin interface (original magnification 3,500x) of AQ. A thin hybrid layer and close adaptation of the adhesive layer were detected for the specimen made under lower RH condition (A). Small bubbles at the adhesive/resin interface were seen for the specimen made under highest RH condition(B).*

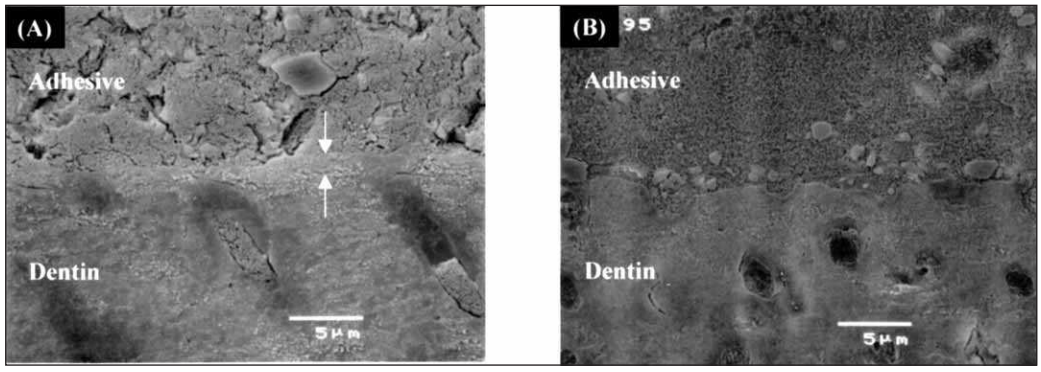


Figure 3. *SEM observation of dentin/resin interface (original magnification 3,500x) of RB. A thin hybrid layer and close adaptation of the adhesive layer were detected for the specimen made under lower RH condition (A). Tough gaps between the adhesive and resin interface were not seen, the hybrid layer was not clearly seen for the specimen made under highest RH condition (B).*

1996). These one-application adhesives may form a continuum between the tooth surface and the adhesive by simultaneous demineralization and resin penetration followed by polymerization to create a hybrid layer (Nakabayashi, Kojima & Masuhara, 1982). To generate the hydrogen ions required for effective dissolution and demineralization, the one-application system has to contain water and water-soluble hydrophilic monomers such as 2-hydroxyethyl methacrylate (HEMA). Water is an essential component in one-application adhesive systems, so that the acidic monomer can dissociate. Protons in solution derived from the acidic monomer then interact with the mineral component of the tooth substrate. After the adhesion was cured, it would be desirable to eliminate any water from the bonding interface. From the results of this study, the dentin bond strengths of the one-application systems studied were influenced by changes in environmental RH. When the specimens were made under 95% RH conditions, the bond strength of all the bonding systems significantly decreased. Evaporation of moisture from the adherent surface may not be sufficient, and the remaining excess moisture on the adherent surface might decrease the concentration of acidic components in the adhesive, resulting in limited demineralization. The presence of HEMA may lower the vapor pressure of water from the adherent surface, resulting in improper polymerization of the resins (Pashley & others, 1998). Excess water may interfere with the dispersion of different monomers and limit the propagation of copolymerization, resulting in lower bond strength (Jacobsen & Söderhold, 1995). Vinyl resin monomers that polymerize via a free radical addition polymerization mechanism are known to be inhibited by oxygen, resulting in the formation of a low polymerization layer (Ruyter, 1981). The thickness of the air inhibition layer depends on the viscosity of the resin monomers that affect the rate of oxygen diffusion (Rueggeberg & Margeson, 1990). Also, an increased concentration of HEMA in the adhesive resin results in an increased thickness of an oxygen inhibition layer (Finger, Lee & Podszun, 1996). Though thickness of the oxygen inhibited layer is related to monomer composition, the presence of extrinsic water might also interfere with the polymerization of adhesive resins (Jacobsen & Söderhold, 1995).

Another explanation for the decreasing dentin bond strength in higher RH condition is the presence of acidic functional monomers on the surface of the adhesives. The adhesive resins used in this study are cured by a free radical polymerization reaction utilizing camphorquinone (CQ) as a visible-light activated photoinitiator (Jakubiak & Rabek, 1999). CQ requires a coinitiator for an effective polymerization process to occur, and a tertiary amine reducing agent is employed to produce reactive radicals for polymerization (Cook, 1992). It is possible that the tertiary amines in adhesive resins can be neutralized by the acidic functional monomers in the unpolymerized adhesive resin layer, resulting in poor bonding (Sanares & others, 2001). To partially overcome problems with acidic monomers, some systems contain *p*-toluenesulfinic acid sodium salt.

The cured adhesive layer in one-application adhesives has been reported to act as semi-permeable membranes that allow water diffusion (Tay & others, 2002). A higher environment RH might cause water movement to the oxygen-inhibited layer of the adhesive, making this inhibition layer thicker. For one-application systems with relatively thinner adhesive layers, the percent of reduction in bond strength at 95% RH might be more pronounced compared with relatively thicker adhesive layers. Manufacturers of unfilled adhesive resins often recommend applying two coats of adhesive to ensure that the adherent surface is adequately covered, since thin adhesive resin can result in decreasing bond strength (Choi, Condon & Ferracane, 2000; Pashley & others, 2002). A thicker adhesive resin layer might contribute to greater reduction in polymerization shrinkage stress and the extent of water diffusion from the oral environment. Such speculation may be further aggravated when these adhesive systems are used on clinically relevant cavities in which an environmental RH is difficult to control.

Although one-application systems utilize water as a component, it is difficult to determine the appropriate extent of wetness in a clinical situation where a more complex environmental condition is present. After applying one-application adhesives, the dentin surface should be air dried, because the adhesive contains solvents such as water, ethanol and acetone. Beyond a critical level, excess water might dilute the one-application adhesive, weaken the etching effect and have an adverse effect on penetration of the resin monomers into dentin. Another reason for decreasing bond strength in higher RH conditions might be the presence of moisture on the surface of hardened adhesives. If the adhesive surface was wetted by oral humidity prior to placing the resin composite, close adaptation between the resin composite and the adhesive might be prevented. Moreover, resin containing hydroxyl functional groups have been known to create internal defects with bound water within the polymer matrices (Tay & Pashley, 2003). The presence of water on the hardened adhesive resin might interfere with polymerization resin composites, so that a poorly polymerized resin layer remains at the adhesive interface, leading to lower bond strength values.

From the results of the study that evaluated the effect of temperature on bond strength to dentin, a significant decrease in bond strength was not observed for higher environmental temperature. The diminished dentin bond strength with higher environmental temperature has been reported, and this phenomenon was described by differences in coefficient of the thermal expansion of

the resin composite compared to tooth structure (Brackett, Covey & Haisch, 2003). The effects of temperature on the flexural behavior of resin composites were investigated, and a significant softening due to temperature under wet immersion test condition was found (Vijayaraghavan & Hsiao, 1994). The plasticizing effect due to higher temperature on adhesive resin might also lead to a decrease in bond strength. Though the authors could not find such an adverse effect on dentin bond strength after 24 hours storage in water, damage accumulation in the bonding interface between dentin and resin composite due to thermal stress during long service in the oral environment might lead to a decrease in bond strength.

CONCLUSIONS

The results of this study suggest that the extrinsic water present from oral humidity above a critical level may adversely affect the dentin bond of the one-application bonding systems studied. Before resin placement, care must be taken when air drying, even though these systems employ hydrophilic components in the adhesive/primer.

Acknowledgement

This work was supported, in part by Grant-in-Aid for Scientific Research 14571826 and 15791111 from the Japan Society for the Promotion of Science, and a Nihon University Research Grant for Assistants and Young Researchers (2003) and a grant from the Ministry of Education, Culture, Sports, Science, and Technology to promote multi-disciplinary research project.

(Received 6 June 2003)

References

Brackett WW, Covey DA & Haisch LD (2003) The effect of elevated temperatures on the dentin adhesion of resin composites *Operative Dentistry* **28(3)** 303-306.

Burrow MF, Taniguchi Y, Nikaido T, Satoh M, Inai N, Tagami J & Takatsu T (1995) Influence of temperature and relative humidity on early bond strengths to dentine *Journal of Dentistry* **23(1)** 41-45.

Choi KK, Condon JR & Ferracane JL (2000) The effects of adhesive thickness on polymerization contraction stress of composite *Journal of Dental Research* **79(3)** 812-817.

Cook WD (1992) Photopolymerization kinetics of dimethacrylates using camphorquinine amine initiator system *Polimery* **33(3)** 600-609.

Eick JD, Gwinnett AJ, Pashley DH & Robinson SJ (1997) Current concepts on adhesion to dentin *Critical Reviews in Oral Biology and Medicine* **8(3)** 306-335.

Finger WJ, Lee KS & Podszun W (1996) Monomers with low oxygen inhibition as enamel/dentin adhesives *Dental Materials* **12(4)** 256-261.

Ikemura K, Kouro Y & Endo T (1996) Effect of 4-acryloxyethyltrimellitic acid in a self-etching primer on bonding to ground dentin *Dental Materials Journal* **15(2)** 132-143.

Jacobsen T & Söderhold KJ (1995) Some effects of water on dentin bonding *Dental Materials* **11(2)** 132-136.

Jakubiak J & Rabek JE (1999) Photoinitiators for visible light polymerization *Polimery* **44(7-8)** 447-461.

Kitasako Y, Nakajima M, Pereira PN, Okuda M, Sonoda H, Otsuki M & Tagami J (2000) Monkey pulpal response and microtensile bond strength beneath a one-application resin bonding system *in vivo Journal of Dentistry* **28(3)** 193-198.

Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials and Research* **16(3)** 265-273.

Nystrom GP, Holtan JR, Phelps RA II, Becker WS & Anderson TB (1998) Temperature and humidity effects on bond strength of a dentinal adhesive *Operative Dentistry* **23(3)** 138-143.

Pashley EL, Zhang Y, Lockwood PE, Rueggeberg FA & Pashley DH (1998) Effects of HEMA on water evaporation from water-HEMA mixtures *Dental Materials* **14(1)** 6-10.

Pashley EL, Agee KA, Pashley DH & Tay FR (2002) Effects of one versus two applications of an unfilled, all-in-one adhesive on dentine bonding *Journal of Dentistry* **30(2-3)** 83-90.

Perdigão J & Lopes M (1999) Dentin bonding–Questions for the new millennium *Journal of Adhesive Dentistry* **1(3)** 191-209.

Plasmans PJ, Reukers EA, Vollenbrock-Kuipers L & Vollenbrock HR (1993) Air humidity: A detrimental factor in dentine adhesion *Journal of Dentistry* **21(4)** 228-233.

Rueggeberg FA & Margeson DH (1990) The effect of oxygen inhibition on an unfilled/filled composite system *Journal of Dental Research* **69(10)** 1652-1658.

Ruyter IE (1981) Unpolymerized surface layers on sealants *Acta Odontologica Scandinavica* **39(1)** 27-32.

Sanares AM, Itthagarun A, King NM, Tay FR & Pashley DH (2001) Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites *Dental Materials* **17(6)** 542-556.

Tay FR, King NM, Suh BI & Pashley DH (2001) Effect of delayed activation of light-cured resin composites on bonding of all-in-one adhesives *Journal of Adhesive Dentistry* **3(3)** 207-225.

Tay FR, Pashley DH, Suh BI, Carvalho RM & Itthagarun A (2002) Single-step adhesives are permeable membranes *Journal of Dentistry* **30(7-8)** 371-382.

Tay FR & Pashley DH (2003) Water treeing-a potential mechanism for degradation of dentin adhesives *American Journal of Dentistry* **16(1)** 6-12.

Van Meerbeek B, Perdigão J, Lambrechts P & Vanherle G (1998) The clinical performance of adhesives *Journal of Dentistry* **26(1)** 1-20.

Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P & Vanherle G (2003) Adhesion to enamel and dentin: Current status and future challenges *Operative Dentistry* **28(3)** 215-235.

Vijayaraghavan TV & Hsiao J (1994) Flexural behavior of visible light-cured composites as a function of temperature under water immersion test conditions *Dental Materials* **10(6)** 347-352.

©Operative Dentistry, 2004, **29-5,** 560-570

Partial Ceramic Crowns: Influence of Preparation Design and Luting Material on Internal Adaptation

M Federlin • S Schmidt • KA Hiller
B Thonemann • G Schmalz

Clinical Relevance

With adhesively bonded partial ceramic crowns (PCC), the choice of luting material is more relevant than preparation design. The resin-modified glass ionomer cement used in this study cannot be recommended as a luting material for feldspathic PCC.

SUMMARY

The influence of three different cavity preparations on the marginal integrity of partial ceramic crowns (PCC) luted with four different luting systems was investigated in this *in vitro* study. PCC preparations were performed in 144 extracted human molars using one of the following preparation designs (n=48/preparation): A–Coverage of functional cusps/butt joint preparation; B–horizontal reduction of functional cusps and C–complete reduction of functional cusps/butt joint preparation. Non-functional cusps were not covered; mesial and distal proximal boxes were extended 1 mm below the cemento-enamel-junction. PCC were fabricated from Vita Mark II ceramic (Vita) with a Cerec 3 Unit (Sirona) and adhesively luted to the cavities using the following luting systems: (VL) Variolink II/Excite (Vivadent), (PA) Panavia F/ED Primer (Kuraray), (DY) Dyract/Prime & Bond NT (DeTrey/Dentsply) and (FU) Fuji Plus/GC Cavity Conditioner (GC). Samples were simultaneously exposed to thermocycling and mechanical loading (TC: 5000x8-55°C, 30 seconds/cycle; ML: 500000x72.5N, 1.6Hz). Marginal adaptation was assessed by evaluating dye penetration on multiple sections by relating the actual penetration distance to the maximal length of the corresponding cavity wall (100%). Ceramic- and tooth-luting material interfaces were evaluated separately. The data were statistically analyzed with the Mann Whitney U-test and Wilcoxon Rank Sum test.

In general, no significant differences could be found between preparations A, B and C. The combination of preparation C and luting material PA showed a tendency for the lowest dye penetration values, especially within dentin (30%). Significant differences could be determined

*Marianne Federlin, Dr med dent, University of Regensburg—Dental School, Department of Operative Dentistry and Periodontology, Regensburg, Germany

Sebastian Schmidt, ZA, University of Regensburg—Dental School, Department of Operative Dentistry and Periodontology, Regensburg, Germany

Karl-Anton Hiller, Dr rer nat, University of Regensburg—Dental School, Department of Operative Dentistry and Periodontology, Regensburg, Germany

Birger Thonemann, Priv Doz Dr med dent, University of Regensburg—Dental School, Department of Operative Dentistry and Periodontology, Regensburg, Germany

Gottfried Schmalz, Prof Dr med dent, University of Regensburg—Dental School, Department of Operative Dentistry and Periodontology, Regensburg, Germany

*Reprint request: Franz Josef Strauß Allee 11, 93042 Regensburg, Germany; e-mail: Marianne.Federlin@klinik.uni-regensburg.de

between luting materials: Composite luting materials PA (0%) and VL (1%) revealed less dye penetration than the compomer DY (6%) and resin-modified glass ionomer cement (RMGIC) FU (26%); use of RMGIC caused fractures of the restorations. The dentin/luting material interface showed the highest penetration values, ranging from 17% to 100%.

In conclusion, with adhesively bonded partial ceramic crowns, the choice of luting material proved to be more relevant than preparation design under the limitations of this study. Margins below the cemento-enamel junction reveal significant loss of adhesion in spite of subsequent application of adhesive luting techniques. RMGIC cannot be recommended as a luting material for feldspathic PCC.

INTRODUCTION

The longevity of adhesively bonded ceramic inlays has been well documented (Frankenberger, Petschelt & Krämer, 2000; Friedl & others, 1997; Sjögren, Molin & van Dijken, 1998; Thonemann & others, 1997; van Dijken, Örmin & Olofsson, 1999), and the technique is recognized throughout the dental profession (Schmalz & Geurtsen, 2001). The survival rate of ceramic inlays has been reported to be in the range of the survival rate of cast gold restorations and amalgam fillings (Felden & others, 1998; Roulet, 1997). Whether *in vitro* (Thonemann & others, 1994) or *in vivo* (Frankenberger & others, 2000), it has been documented that restoration margins within dentin are not a contraindication for the placement of ceramic inlays as long as adhesive luting procedures are subsequently applied. According to Reiss and Walther (2000), the number of more extended ceramic restorations with replacement of up to four cusps (partial ceramic crown; PCC) has increased during the 10-year observation period of their investigation. PCCs are more frequently considered as an alternative to cast gold restorations in order to restore extensively damaged teeth, paying particular attention to tissue conservative procedures and esthetic aspects and reducing the need for full-coverage crowns or post and core placement (van Dijken & others, 2001). Limited clinical data on the longevity of PCC confirm this in the literature. In a retrospective clinical investigation on the survival analysis of Dicor PCC, Felden and others (1998) showed a probability of survival of 55% for seven years. In another retrospective clinical study by Felden, Schmalz and Hiller (2000), PCC fabricated from the Empress I all-ceramic system showed a probability of survival of 81% for seven years. Comparing the longevity of cast gold vs ceramic partial crowns in clinical use for up to seven years, Wagner, Schmalz and Hiller (2002) concluded that the survival rate of cast gold partial crowns as the golden standard for posterior restorations is statistically not superior to partial ceramic crowns. In a five-year follow-up of restorations with extensive dentin/enamel bonded ceramic coverage, van Dijken and others (2001) reported a clinical success rate of 93.4% for PCC in vital teeth.

Factors essential to the longevity of adhesively bonded PCCs are (1) cavity preparation, (2) the nature of the luting material/adhesive system and (3) the ceramic material/manufacturing process. Cavity preparation begins the process of replacing destroyed tooth tissues and the restoration of function, form and esthetics. On one hand, cavity preparation should be tissue conservative, but must also meet the requirements of the restorative material to be used. Despite PCCs being increasingly advocated as alternative restorations for extensively damaged teeth, data concerning preparation designs for PCCs are seldom referred to in the literature and are limited. With respect to the definition of a partial crown, the German Dental Association (DGZMK) recently classified onlay- or overlay-type restorations as partial crowns when one or more cusps are restored (Pröbster, 2001). In the literature, three basic concepts for PCC preparations have been described. To date, the concepts of tooth preparation have evolved from the need for adequate retention forms, because of the limitations of luting agents (zinc phosphate cement) and due to the technical and mechanical requirements determined by the restorative material (cast gold). Therefore, suggested preparation designs for PCCs have been based on traditional preparations utilizing a conventional retention form (Broderson, 1994; Jedynakiewicz & Martin, 1996) (Figure 1a). More recently, the traditional rules and principles of preparation have been stated to be no longer applicable for adhesively luted ceramic restorations (Hansen, 2000; van Dijken & others, 2001). Because of the efficacy of adhesive luting procedures, it has been suggested that the preparation for partial-coverage restorations can be made with less emphasis

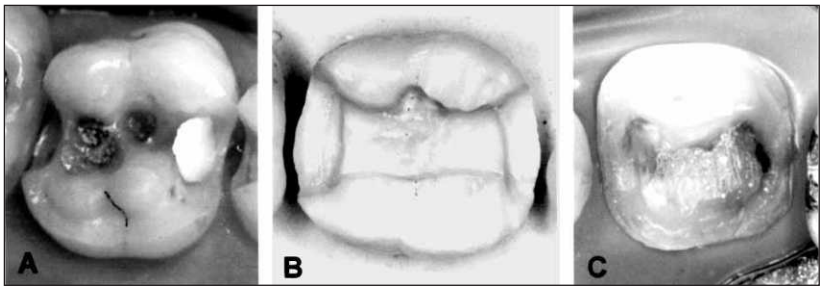


Figure 1: *Different tooth preparations suggested for PCC: (A) retentive type preparation: coverage of functional cusps and preparation of a butt joint. (B) adhesive, onlay- or overlay-type preparation: functional cusp with horizontal bevel and (C) complete reduction of functional cusp and butt joint; buccal cusps included in preparation.*

on retentive form (Kunzelmann, 1999), involving, for example, the mere horizontal reduction of an occluding cusp (Figure 1b). Van Dijken and others (2001) suggest PCC preparations in which the retention of the all-ceramic restoration depends totally on the bond to the underlying dentin and any available enamel, mediated by an adhesive luting system. The preparation is merely defect-oriented (Figure 1c).

With PCC, the durability of the adhesively luted ceramic strongly depends on the strength of the bond between tooth, luting system and ceramic. With ceramic inlays, either light cured, dual cured or chemically cured luting composites have been advocated (Besek, Mörmann & Lutz, 1996; Sorensen & Munksgaard, 1996; Thonemann & others, 1994; van Dijken & others, 2001). Recently, light-curing glass ionomer cements and compomer cements have been suggested as alternative luting materials (Rosenstiel, Land & Crispin, 1998; Thonemann & others, 1995).

Based on reports in the literature, it was hypothesized that both preparation type and luting material have an influence on marginal quality. To test this hypothesis, the internal adaptation of PCC with three different preparation types for the insertion of PCC fabricated from Vita Mark II ceramic blocs (Vita, Bad Säckingen, Germany) with the Cerec 3 system (1.0, Sirona, Bensheim, Germany) and four different luting systems was evaluated after thermocycling and mechanical loading (TCML).

METHODS AND MATERIALS

Sample Preparation

Figure 2 summarizes the procedures followed. Extracted human molars (n=144) stored in chloramine solution from the time of extraction were hand-scaled, cleaned with slurry of pumice, mounted in acrylic resin (Palavit G, Kulzer, Wehrheim, Germany) and stored in physiological saline solution until used. The teeth were randomly assigned to three groups of n=48 specimens. For each group, one of the following preparations was performed using diamond burs (Cerinlay Set, Intensiv, Viganello Lugano, Switzerland) in a high-speed handpiece with sufficient water cooling:

Preparation **A**: Coverage of functional cusps/butt joint preparation;

Preparation **B**: Horizontal reduction of functional cusps and

Preparation **C**: Complete reduction of functional cusps/butt joint preparation (Figure 3).

Non-functional cusps were not covered; mesial and distal proximal margins were placed 1 mm below the cemento-enamel-junction (CEJ) within cementum/dentin. Rounded line angles were prepared. A CAD/CIM method (Cerec, Sirona, Bensheim, Germany) was chosen for the fabrication of partial ceramic crowns (PCC). PCCs were fabricated from Vita Mark II ceramic blocs (Vita Zahnfabrik, Bad Säckingen, Germany) using the Cerec 3 system and corresponding software in function mode (Sirona, software: Cerec 3, 1.0 (600)). PCCs were inserted following adjustment to the cavities using one of four different luting material systems (n=12 specimens per material and preparation): (VL) Variolink II/Excite (Vivadent, Schaan, Liechtenstein)—dual curing luting composite, (PA) Panavia F/ED Primer (Kuraray, Japan)—dual-curing luting composite, (DY) Dyract/Prime & Bond NT (DeTrey/Dentsply, Konstanz, Germany)—compomer and (FU) Fuji Plus/GC Cavity Conditioner (GC Europe, Leuven, Belgium)—chemically curing RMGIC cement. The restorative procedures were performed in a device that simulated proximal contacts to adjacent teeth in order to match the clinical situation as closely as possible. Prior to PCC insertion, clear matrix bands (Hawe Neos, Bioggio, Switzerland) and light reflecting wedges (Hawe Neos) were set in place. Luting material was

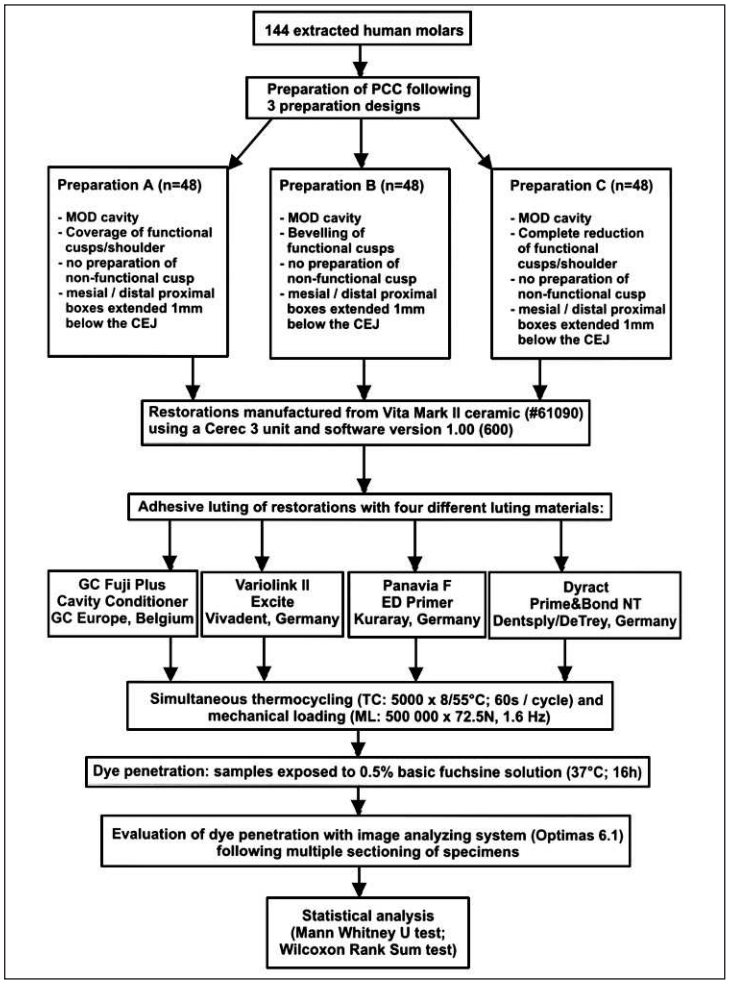


Figure 2. *Flow chart: Methods and materials.*

applied to the cavity surfaces following adhesive conditioning of the cavity and PCC surfaces. Table 1 summarizes the luting procedures. Excess luting material was removed prior to curing. Following insertion procedures, finishing was performed with finishing diamonds (Komet/Brasseler, Lemgo, Germany) and the restorations were polished with flexible discs (Sof-Lex, 3M Dental Products Division, St Paul MN, USA). Prior to TCML, samples were stored in physiological saline solution at 37°C for 24 hours. Impressions (Impregum, ESPE, Seefeld, Germany) for the fabrication of epoxy replicas (Araldit, Ciba-Geigy, Switzerland) were taken immediately before and after TCML. Samples were exposed to thermocycling (TC: 5000x8-55°C, 30-second/cycle) and mechanical loading (ML: 500000x72.5N, 1.6Hz) simultaneously. Mechanical loading was performed by means of a cyclic (1.6 Hz) increase in pressure (72.5N) upon a metal stop representing the opposing cusp. The metal stop was statically placed in the occlusal central fissure of the restoration, the TCML device thus allowed for the simultaneous simulation of static load cycling and temperature changes (Roulet, 1994).

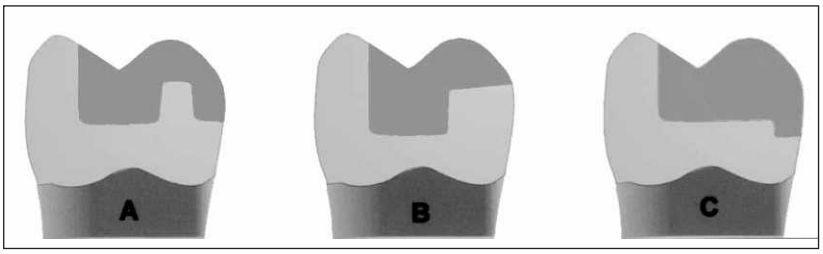


Figure 3: *Schematic drawing of preparations A-C, representing a midline-cut in vestibulo-oral direction: (A) coverage of functional cusps/butt joint preparation; (B) horizontal reduction of functional cusps and (C) complete reduction of functional cusps/butt joint preparation.*

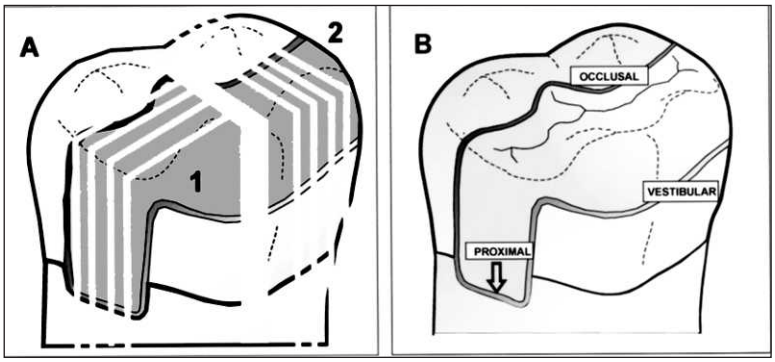


Figure 4: *(A) Schematic drawing indicating multiple sectioning of specimen in (1) mesio-distal and (2) vestibulo-oral direction in order to evaluate dye penetration at occlusal, vestibular and proximal restoration margins (B).*

Dye Penetration

Following TCML, microleakage at the occlusal, vestibular and proximal locations and for tooth- and ceramic-interfaces, separately, was determined by means of dye penetration. Specimens were covered with nail varnish, except for the areas within 1 mm from restoration margins and placed in a 0.5% basic fuchsine solution for 16 hours at 37°C. Specimens were removed from fuchsine solution, cleaned, mounted onto stubs with acrylic resin and sectioned in halves centrally in a bucco-oral direction, rendering two parts/specimen (Figure 4). Consecutively, one part of the specimen was sectioned longitudinally in the mesio-distal direction into as many sections as possible (3 to 4) using a rotating diamond saw (blade thickness 400 µm; Sägemikrotom 1600, Leitz, Germany) with water-cooling for evaluation of dye penetration at the cervical tooth/restoration interface. The second part of each specimen was sectioned in the bucco-oral direction into as many sections as possible (3 to 4) to allow for evaluation of the occlusal and vestibular tooth/restoration interfaces. The sections were approximately 300-µm thick, each section presenting two sites for the evaluation of dye penetration. Digital images of the sections were recorded and microleakage along the cervical (dentin-cementum), vestibular (enamel) and occlusal (enamel) tooth/luting material and luting material/ceramic interfaces on the multiple sections was evaluated with an image analyzing system

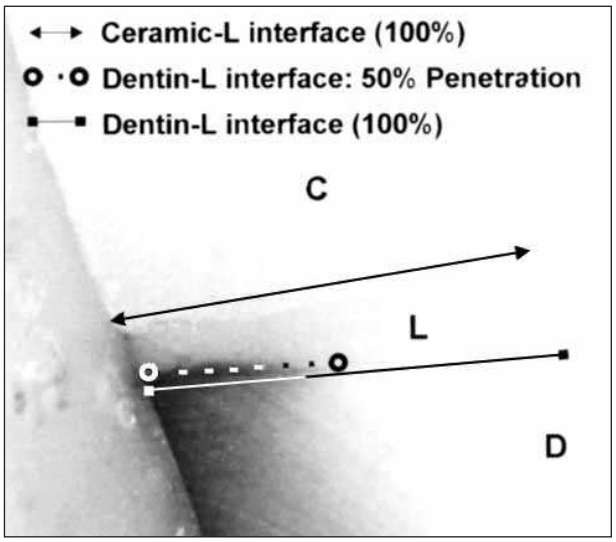


Figure 5: *Determination of dye penetration at proximal restoration margin. (C=ceramic; D=dentin; L=luting agent). Dotted lines indicate pulpoaxial ceramic-luting agent- and luting agent-tooth interface.*

(Optimas 6.1, Stemmer, München, Germany). The extent of dye penetration along the interface was expressed as the percentage of penetration into the depth of the cavity along the cavity margin. The entire length of the margin was used as the 100% reference

Table 1: *Luting Materials, Cavity/Ceramic Conditioning and Luting Procedures*

Luting Material	**Fuji Plus** (resin-modified glass ionomer) (#0006212; GC Europe, Belgium)	**Variolink II** (high viscosity) (composite luting agent) (#B5306; Vivadent, Germany)	**Panavia F** (composite luting agent) (Paste A: #00104A, Paste B: #00005A; Kuraray, Germany)	**Dyract** (compomer material) (#0108000448); Dentsply/DeTrey, Germany)
Conditioning of Ceramic	**Ceramics Etch** gel (#041654; Vita, Germany) (60 seconds, followed by rinsing with water)	**Ceramics Etch** gel (#041654; Vita, Germany) (60 seconds, followed by rinsing with water) **Monobond S** (#C24280; Vivadent, Germany) (applied and dried after 60 seconds)	**Ceramics Etch** gel (#041654; Vita, Germany) (60 seconds, followed by rinsing with water) **Porcelain Bond Activator** (#0007171, Kuraray, Germany) mixed with (#41116; Kuraray, Germany) (applied, blow dried)	**Ceramics Etch** gel (#041654; Vita, Germany) (60 seconds, followed by rinsing with water) **Monobond S** (#C24280; Vivadent, Germany) Silane treatment
Conditioning of Cavity	**GC Cavity Conditioner** (#0010201; GC Europe, Belgium) (20 seconds; followed by rinsing with water/drying of cavity)	**Total Etch** (#D05082; Vivadent, Germany) (Dentin: 20 seconds; Enamel: 40 seconds; followed by water spray and gentle blow drying) **Excite** (#C15056; Vivadent, Germany) (application, after 20 seconds: gentle blow drying and light curing for 20 seconds)	**Total Etch** (#D05082; Vivadent, Germany) (Dentin: 20 seconds; Enamel:40 seconds; followed by water spray and gentle blow drying) **ED Primer Liquid A** (#00103B) **ED Primer Liquid B** (#00110B) (Kuraray, Germany) (mixed; applied for 60 seconds, gentle blow drying, light curing for 20 seconds)	**Conditioner 36** (#0103001067; Dentsply/DeTrey, Germany) (Dentin: 20 seconds; Enamel:40 seconds; followed by water spray and gentle blow drying) **Prime&Bond NT** (#0011000736; Dentsply/DeTrey, Germany) **(**applied for 20 seconds, gentle blow drying and light curing for 20 seconds)
Curing Mode	**Chemically curing; Fuji Coat LC** (#0007171; GC Europe, Belgium) (placed on restoration margins and light-cured for 10 seconds)	**Dual-curing;** (light application 40 seconds from each aspect)	**Dual-curing;** (light application 40 seconds from each aspect) **Oxyguard II** (#00325B, Kuraray, Germany) (placed on restoration margins)	**Light-curing;** (light application 40 seconds from each aspect)

(Figure 5). Two dye penetration measurements were recorded from each section, rendering six to eight measurements per interface. The median of these measurements per interface was selected as the characteristic descriptive value representative of each interface per tooth. The median of n=12 values per tooth and interface was selected as characteristic descriptive value representative of each sample. Additionally, all dye penetration values (medians) were pooled separately for each preparation type, luting system and location, irrespective of all other parameters.

Statistical Analysis

A non-parametric statistical analysis was considered appropriate to analyze the data, because of lack of a normal distribution (Roulet, 1994). Medians and 25% to 75%-percentiles/sample for the dye penetration data (%) were determined separately for all interfaces. Statistical analysis was performed using the Mann Whitney U-test and Wilcoxon Rank Sum test (SPSS/PC+, Vers 6.0, SPSS Inc, Chicago IL, USA) for pairwise comparisons among groups (Roulet, 1994). The level of significance was set to α=0.05.

RESULTS

With *Varionlink*/*Excite* as the luting agent/bonding system combination, the lowest dye penetration values can be found at the ceramic-luting agent (LA)- and enamel-LA-interfaces (Median: 0% to 3%) (Figure 6). Preparation C reveals significantly less dye penetration at the ceramic-LA interface than preparations A ($p \leq 0.0001$) and B ($p \leq 0.001$). The dentin-LA interface

reveals dye penetration values between 55% and 96%. Preparation A (Median: 55%; 25% to 75% percentiles: 32% to 88%) reveals a tendency toward the lowest dye penetration values, followed by preparation B (69%; 41% to 87%) and preparation C (96%; 7% to 100%). No statistically significant differences can be found between preparation types regarding dentin-LA interfaces.

With *Panavia/ED Primer* as luting agent/bonding system combination, the lowest dye penetration values are found at the ceramic-LA and enamel-LA interfaces (0% to 6%) (Figure 7). Preparation A reveals significantly more dye penetration (6%; 0% to 15%) at the enamel-LA interface than preparation B (0%; $p \leq 0.001$)), but no statistically significant differences could be found between preparation types at ceramic-LA and enamel-LA interfaces. The dentin-LA interface reveals dye penetration values between 38% and 51%. Preparation B (51%; 38% to 99%) exhibits highest dye penetration values at the dentin-LA interface followed by preparations A (38%; 30% to 82%) and C (30%; 21% to 87%). Preparation C shows a tendency towards the lowest dye penetration and is significantly better than preparation B ($p \leq 0.01$).

With *Dyract/Prime & Bond NT* as the luting agent/bonding system combination, dye penetration at the ceramic-LA (4% to 8%) and enamel-LA interfaces (0%) is lower than at the dentin-LA interface (70% to 100%) for all preparation types (Figure 8). No statistically significant differences can be found between preparation types A-C at the ceramic-LA and enamel-LA interfaces. At the dentin-LA interface, preparation B shows less dye penetration (70%; 43% to 100%) than preparation A (97%, 62% to 100%) and significantly less dye penetration ($p \leq 0.001$) than preparation C (100%).

With *Fuji Plus/GC Cavity Conditioner* as luting agent, 12 restoration fractures (Figure 9) occurred either prior to TCML after 24-hour storage in saline solution at 37°C (n=5) or following TCML (n=7). With respect to preparation type, preparation A revealed n=6 fractures and preparations B and C n=3 fractures in each group. Dye penetration values were not recorded for fractured specimens. With Fuji Plus/GC Cavity Conditioner, the ceramic-LA interface revealed dye penetration of 24% to 36%, the enamel-LA interface showed dye penetration of 12% to 26% and at the dentin-LA interface, dye penetration values between 17% and 65% were recorded (Figure 10). Preparation C exhibited higher dye penetration values than preparations A and B. At the ceramic–LA and enamel-LA interfaces, preparation C (Ceramic: 35%; 27% to 52%; Enamel: 26%; 10% to 36%) revealed significantly more dye penetration ($p \leq 0.001$) than preparation B (Ceramic: 24%; 19% to 36%; Enamel: 12%; 0 to 21%). At the dentin-LA interface, preparation C (65%; 42% to 99%) showed statistically significant higher dye penetration values than preparations A (17%; 9 to 38%); $p \leq 0.001$) and B (37%; 20% to 45%; $p \leq 0.01$).

Dye penetration data were pooled for preparation type, luting material and location, irrespective of all other criteria. Figure 11 shows summarized overall dye penetration percentages.

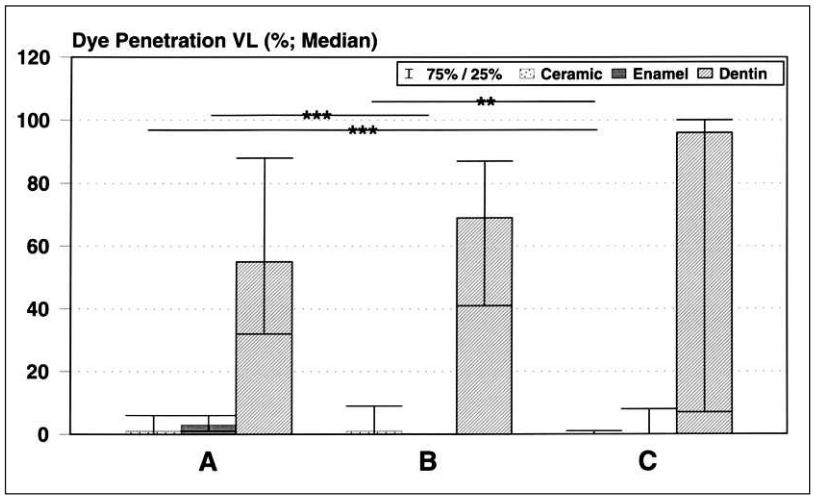


Figure 6: *Results: Dye penetration (%) with luting material Variolink II/Excite at ceramic-, enamel- and dentin-interfaces for preparations A, B and C (n=10 specimens/sample; median and 25% to 75% quartiles). Bars indicate significant differences (*p≤ 0.05; ** p≤ 0.01; ***:p≤ 0.001).*

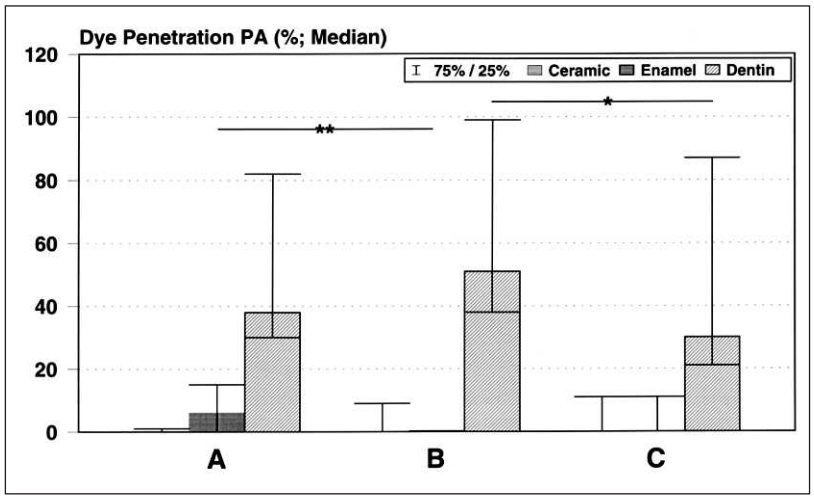


Figure 7: *Results: Dye penetration (%) with luting material Panavia F/ED Primer at ceramic-, enamel- and dentin-interfaces for preparations A, B and C (n= 10 specimens/sample; median and 25% to 75% quartiles). Bars indicate significant differences (*:p≤ 0.05; ** p≤ 0.01; ***:p≤ 0.001).*

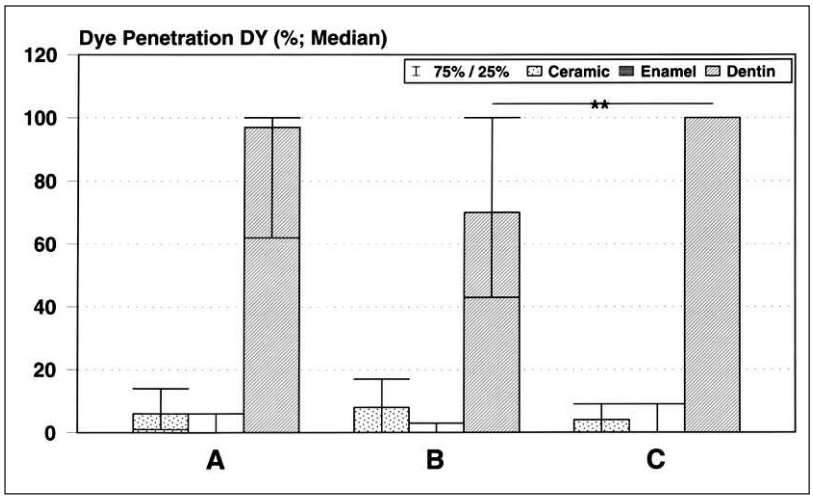


Figure 8: *Results: Dye penetration (%) with luting material Dyract/Prime&Bond NT at ceramic-, enamel- and dentin-interfaces for preparations A, B and C (n=10 specimens/sample; median and 25% to 75% quartiles). Bars indicate significant differences (*:p≤ 0.05; ** p≤ 0.01; ***:p≤ 0.001).*

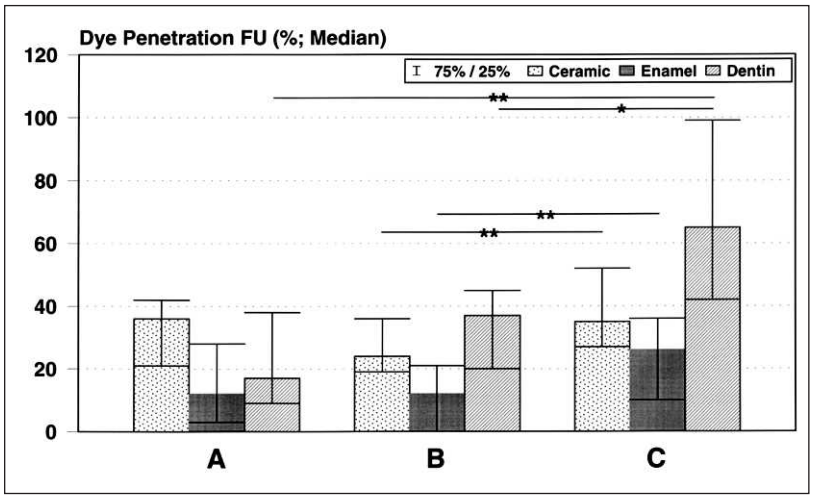


Figure 10: *Results: Dye penetration (%) with luting material Fuji Plus/GC Conditioner at ceramic-, enamel- and dentin-interfaces for preparations A, B and C (n=10 specimens/sample; median and 25% to 75% quartiles). Bars indicate significant differences (*:p≤ 0.05; ** p≤ 0.01; ***:p≤ 0.001).*

Preparation Type: Overall dye penetration for preparation A is 6% (median) (25% to 75% quartiles: 0% to 26%), for preparation B, 5% (0% to 24%) and for preparation C, 7% (0% to 31%). No statistically significant differences can be found between preparation types A, B and C, independent of the luting system and interface.

Luting Material: Overall dye penetration is 1% (0% to 9%) for VL, 0% (0% to 18%) for PA, 6% (0% to 23%) for DY and 26% (17% to 40%) for FU. Statistically significant differences can be found between luting systems independent of preparation type and interface, composite luting materials showing significantly less dye penetration than the compomer system ($p \leq 0.01$)

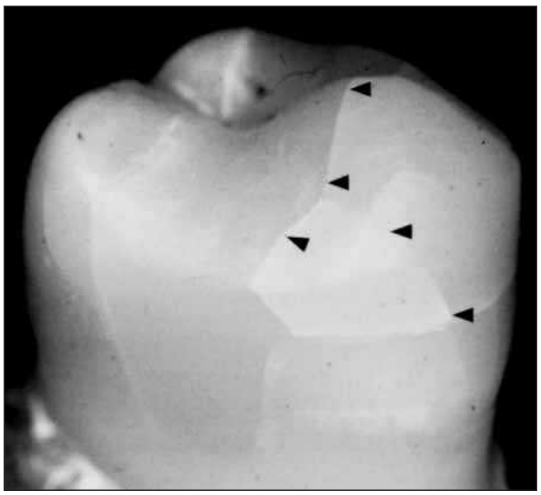


Figure 9: *Fracture of restoration inserted with RMGIC. Arrows indicate fracture lines.*

and RMGIC ($p \leq 0.0001$). No statistically significant difference can be determined between composite luting materials PA and VL.

Location of Restoration Margin: Overall dye penetration is 0% (0% to 10%) for the occlusal restoration margin, 4% (0% to 15%) for the vestibular restoration margin and 30% (4% to 68%) for the proximal restoration margin. Statistically significant differences can be found between locations of restoration margins—independent of preparation type and luting system; proximal restoration margins exhibit significantly higher marginal deterioration than occlusal and vestibular margins ($p \leq 0.000$), and vestibular restoration margins show significantly more dye penetration than occlusal margins ($p \leq 0.0005$).

DISCUSSION

Preparation of extensively damaged teeth results in the reduction of tooth stability and decreases fracture resistance due to the increased deflection of weakened cusps (Granath & Svensson, 1991). By means of adhesively bonding ceramic inlays to tooth structures, fracture resistance of the restored teeth can be enhanced (Haller & others, 1990; Lang, Schwan & Nolden, 1994). However, in a clinical investigation comparing extended ceramic inlay restorations with partial ceramic crowns, extended inlay restorations, especially in cavities with their proximal margins below the CEJ, showed a significant, time dependent increase in marginal deterioration (Lang, Schüler & Nolden, 1998). The authors conclude that the use of bonded partial crowns might be advisable in teeth with cavities located partially in dentin. Recently published data indicate that PCC may be a tissue conservative

approach to restoring extensively damaged teeth (Felden & others, 2000; van Dijken & others, 2001; Wagner & others, 2002). However, no preparation design standards for the ideal dentin-enamel-bonded PCC or for the influence of preparation design upon marginal integrity have been indicated in the literature.

In this investigation, preparation designs for PCC were chosen based on three general approaches for preparing PCC suggested in the literature. The non-functional cusps were not included in the PCC preparation and were left without coverage based on sufficient wall thickness of non-functional cusps (Mehl & others, 1996; 1998) and stabilization due to adhesive luting procedures (Haller & others, 1990; Lang & others, 1994). Functional cusps were covered, owing to the fact that in spite of initial stabilization due to adhesive luting procedures, continuous loading might cause a time dependent increase in marginal deterioration without cuspal coverage as described by Lang and others (1998). Cuspal coverage in preparation A followed suggestions for a traditional retentive cavity preparation modified to suit the ceramic as a restorative material (Jedynakiewicz & Martin, 1996), although this kind of preparation is thought to create a high amount of stress points in the ceramic restoration (Broderson, 1994). In preparation B, coverage of functional cusps was performed in a less invasive approach for the tooth tissues by mere horizontal reduction, neglecting the conventional means of retention (Kunzelmann, 1999) due to application of adhesive luting techniques. Preparation C represents the type of preparation described by van Dijken and others (2001), in which retention of the restoration relies mainly upon the adhesive bond between dentin, enamel, luting agent and ceramic and not on retentive elements in cavity geometry. Proximal margins were extended below the CEJ based upon reports of the adhesive restoration of extensively damaged teeth in the literature (Frankenberger & others, 2000; Thonemann & others, 1994; Tidehag & Gunne, 1995; van Dijken & others, 2001).

The clinical durability and fracture resistance of PCC will also depend upon the adhesive system and luting material used. In this study, two dual curing luting composites, a compomer and a resin-modified glass ionomer cement with corresponding adhesive systems, were chosen as luting agents based upon reports of currently used luting agents in the literature (Rosenstiel & others, 1998; Schmalz, Federlin & Reich, 1995). Dual curing luting agents are discussed controversially in the literature; lower conversion in the deeper parts of the cement layer are discussed as a shortcoming (Rueggeberg & Caughman, 1993). In a recent study on the clinical evaluation of ceramic inlays cemented with a dual cured or chemically cured resin composite luting agent, Sjögren and others (1998) reported no statistically significant differences between the two luting agents. These findings were confirmed by van Dijken and others (2001), who investigated the use of a dual cured and a chemically cured resin composite luting agent in restorations with extensive dentin/enamel bonded ceramic coverage. The authors could find no significant difference in the failure rate of the restorations between the two luting agents. RMGICs and compomers have been discussed and investigated as alternative luting materials for ceramic inlays *in vitro* and *in vivo* (Rosenstiel & others, 1998; Thonemann & others, 1995; van Dijken & others, 1999).

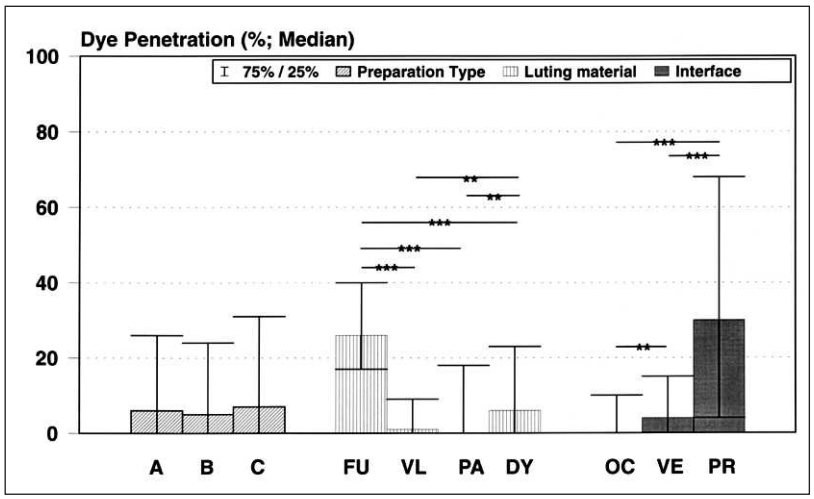


Figure 11: *Results: Dye penetration (%) summarized for preparation types A-C, luting materials (FU: Fuji Plus; VL: Variolink; PA: Panavia F; DY: Dyract) and location of restoration margins (oc: occlusal, enamel; ve: vestibular, enamel; pr: proximal, dentin) irrespective of all other parameters (median and 25% to 75% quartiles). Bars indicate significant differences (*:*p≤ 0.05; ** p≤ 0.01; ***:p≤ 0.001).*

With regard to the ceramic material/associated manufacturing process, disadvantages exhibited by the early, sintered dental ceramics or castable glass ceramics (Felden & others, 1998) were inhomogeneities and microporosities generated during sintering or extensive occlusal adjustment, which could initiate crack formation/propagation and result in fractures of the ceramic restoration (Seghi, Denry & Rosenstiel, 1995). In this investigation, machinable ceramic blocs and fabrication of the restorations with the Cerec 3 system (software 1.0) were chosen. The Cerec method has been reported to be particularly appropriate for the fabrication of large cuspal-replacement restorations, because of the nature of the ceramic material that it machines. The nature of the ceramic, combined with a fine surface finish, serves to minimize the wear on both the restoration and the antagonist teeth

(Datzmann, 1996). It thus enhances the suitability of the technique for replacement of occlusal surfaces (Datzmann, 1996; Jedynakiewicz & Martin, 1996). With the introduction of the Cerec 3 system, shortcomings of the Cerec 1 and 2 systems in terms of limitation in preparation design have been overcome; it allowed for fabrication of the PCC preparation designs described in this investigation (Bindl & Mörmann, 2003; Martin & Jedynakiewicz, 1996).

In this study, a TCML device was used that allowed for the simultaneous application of static load cycles and thermal stressing in order to simulate clinical conditions under the limitations of the protocol used (Roulet, 1994). In an *in vitro* study on marginal adaptation of adhesive ceramic inlays, Krejci, Lutz and Reimer (1993) showed that the initial marginal integrity in enamel and dentin deteriorated after thermocycling and mechanical loading. Chen and others (1999) demonstrated in an *in vitro* study that cyclic loading prior to fracture strength testing of all-ceramic crowns significantly reduced the fracture strengths of all-ceramic crowns.

The results of this study indicate that the choice of luting material is more relevant to the marginal adaptation of PCC restorations as it relates to microleakage after TCML than the preparation design, as long as the cavity dimensions meet the demands of the ceramic as a restorative material. Pooled dye penetration data showed no significant differences between preparation types A, B and C. Reports confirming these findings can be found in the literature. Burke (1996) showed no differences between the varying degrees of tooth preparation to enhance protection to fracture in an *in vitro* study. Van Dijken and others (2001) stated that no statistical differences were found among the four preparation types employed in their five-year follow-up of dentin/enamel bonded ceramic coverages.

Pooled dye penetration data for luting materials revealed significant differences between the two dual cure luting composites, Variolink II or Panavia F and the compomer material or RMGIC. RMGIC exhibited the most deleterious affect: 12 of the 30 PCCs luted with RMGIC fractured either prior to TCML after 24-hours storage or following TCML. Preparation type A revealed the highest number of fractures (n=6) compared to preparation types B (n=3) and C (n=3). Although RMGICs have been advocated for the insertion of ceramic inlays (Thonemann & others, 1995; van Dijken & others, 1999) anecdotal reports based on clinical observations have linked resin-modified glass ionomer luting agents with post cementation fracture of all-ceramic crowns. Leevailoj and others (1998) investigated the fracture incidence of all-ceramic crowns cemented with RMGIC luting materials and other luting agents. The authors could demonstrate restoration fractures occurring with two out of the three RMGICs they investigated. Feilzer and others (1995) observed a conversion of contraction stresses into expansion stresses in RMGICs, resulting in a build-up of compressive strength in the long-term. They indicated that it is of paramount importance that the expansion of RMGIC be limited and not grow to unacceptable values that might damage the restoration or tooth structure, as had occurred in this investigation.

The two dual cure luting agents exhibited dye penetration values in the same range, and lower overall values than the compomer, pooled dye penetration data showing an overall dye penetration of 1% (0% to 9%) for Variolink II/Excite and 0% (0% to 18%) for Panavia F/ED Primer. Regarding dye penetration at ceramic-, enamel- and dentin-luting composite interfaces separately, Panavia F/ED Primer showed a tendency toward the lowest dye penetration values in general, preparation type C revealed the lowest percentage of dye penetration at the proximal dentin-interface.

With all three preparation types, proximal restoration margins within dentin-cementum revealed severe dye penetration compared to enamel/and ceramic/luting composite interfaces. The data are in accordance with results reported by Hürzeler, Zimmermann and Mörmann (1990) in an investigation performed with respect to marginal adaptation of machined ceramic onlays *in vitro*. The authors reported that cervical restoration margins within dentin revealed significantly lower percentages of perfect margins than restoration margins within enamel. Sorensen and Munksgaard (1996) observed that it seems nearly impossible to completely eliminate gap formation at the cavity floor of resin-bonded ceramic inlays. No information on onlays is supplied in the literature. An alternative resin coating technique advocating the application of an adhesive system and a low viscosity microfilled resin to the prepared cavity walls prior to impression taking for indirect composite restorations has been shown to reduce gap formation at the internal dentin-restoration interface compared to non-coated specimens (Jayasooriya & others, 2003). This method may represent an approach for improving the early bond strength of resin cement to dentin and reduce gap formation/microleakage along the resin-dentin interface of indirect restorations. Van Dijken and others (2001) point out that there is a discrepancy between results in laboratory and clinical investigations regarding restoration margins within dentin. In contrast to the results of this investigation, they confirmed the sealing ability of dentin bonded ceramic restorations in a five-year follow up of PCCs.

CONCLUSIONS

Under the limitations of this study, the choice of luting material proved to be more relevant than the prepa-

ration design with adhesively bonded partial ceramic crowns (PCCs). Margins below the cemento-enamel junction are critical in spite of the subsequent application of adhesive luting techniques. RMGIC cannot be recommended as a luting material for CAD/CAM-machined feldspathic PCCs.

(Received 6 June 2003)

Acknowledgement

The authors express their thanks and appreciation to Prof Dr Loys J Nunez, Memphis, TN, USA, for his constructive criticism and advice regarding the manuscript.

References

Besek MJ, Mörmann WH & Lutz F (1996) Curing of Composites under CEREC Inlays in *CAD/CIM in Aesthetic Dentistry. CEREC 10 year Anniversary Symposium* (Editor WH Mörmann) Quintessence Publishing Co Inc Berlin 347-360.

Bindl A & Mörmann WH (2003) Clinical and SEM evaluation of all-ceramic chair-side CAD/CAM generated partial crowns *European Journal of Oral Sciences* **111(2)** 163-169.

Broderson SP (1994) Complete-crown and partial-coverage tooth preparation designs for bonded cast ceramic restorations *Quintessence International* **25(8)** 535-539.

Burke FJ (1996) Fracture resistance of teeth restored with dentin-bonded crowns: The effect of increased tooth preparation *Quintessence International* **27** 115-121.

Chen HY, Hickel R, Setcos JC & Kunzelmann KH (1999) Effects of surface finish and fatigue testing on the fracture strength of CAD-CAM and pressed-ceramic crowns *Journal of Prosthetic Dentistry* **82(4)** 468-475.

Datzmann G (1996) CEREC Vitablocs Mark II Machinable Ceramics in *CAD/CIM in Aesthetic Dentistry. CEREC 10 year Anniversary Symposium* (Editor WH Mörmann) Quintessence Publishing Co Inc Berlin 205-215.

Feilzer AJ, Kakaboura AI, de Gee AJ & Davidson CL (1995) The influence of water sorption on the development of setting shrinkage stress in traditional and resin-modified glass ionomer cements *Dental Materials* **11(3)** 186-190.

Felden A, Schmalz G, Federlin M & Hiller KA (1998) Retrospective clinical investigation and survival analysis on ceramic inlays and partial ceramic crowns: Results up to seven years *Clinical Oral Investigations* **2(4)** 161-167.

Felden A, Schmalz G & Hiller KA (2000) Retrospective clinical study and survival analysis on partial ceramic crowns: Results up to seven years *Clinical Oral Investigations* **4(4)** 199-205.

Frankenberger R, Petschelt A & Krämer N (2000) Leucite-reinforced glass ceramic inlays and onlays after six years: Clinical behavior *Operative Dentistry* **25(6)** 459-465.

Friedl KH, Hiller KA, Schmalz G & Bey B (1997) Clinical and quantitative margin analysis of feldspathic ceramic inlays at 4 years *Clinical Oral Investigations* **1(4)** 163-168.

Granath L & Svensson A (1991) Elastic outward bending of loaded buccal and lingual premolar walls in relation to cavity size and form *Scandinavian Journal of Dental Research* **99(1)** 1-7.

Haller B, Thull R, Klaiber B & Schmitz A (1990) [Höckerstabilisierung durch Adhäsivinlays in MOD Kavitäten] Ridge stabilization with adhesive inlays in MOD cavities *Deutsche Zahnärztliche Zeitschrift* **45(10)** 660-663.

Hansen S (2000) Preparations for Cerec 3: Where are the limits? *International Journal of Computerized Dentistry* **3(3)** 197-205.

Hürzeler M, Zimmermann E & Mörmann WH (1990) [Marginale Adaptation von maschinell hergestellten Onlays *in vitro*] Marginal adaptation of mechanically produced onlays *in vitro* *Schweizer Monatsschrift für Zahnmedizin* **100(6)** 715-720.

Jayasooriya PR, Pereira PN, Nikaido T, Burrow MF & Tagami J (2003) The effect of a "resin coating" on the interfacial adaptation of composite inlays *Operative Dentistry* **28(1)** 28-35.

Jedynakiewicz NM & Martin N (1996) Procedures for Multi-Cusp Restorations in *CAD/CIM in Aesthetic Dentistry CEREC 10 year Anniversary Symposium* (Editor WH Mörmann) Quintessence Publishing Co Inc Berlin 143-152.

Krejci I, Lutz F & Reimer M (1993) Marginal adaptation and fit of adhesive ceramic inlays *Journal of Dentistry* **21(1)** 39-46.

Kunzelmann KH (1999) [Moderne Füllungstherapie mit keramischen Werkstoffen—Keramikinserts, -inlays und -teilkronen im Seitenzahnbereich] Ceramic restorations—State of the art of ceramic inserts, ceramic inlays and partial ceramic crowns *Bayerisches Zahnärzteblatt* **3** 17-22.

Lang H, Schüler N & Nolden R (1998) [Keramikinlay oder Keramikteilkrone?] Ceramic inlay or partial ceramic crown? *Deutsche Zahnärztliche Zeitschrift* **53(1)** 53-56.

Lang H, Schwan R & Nolden R (1994) [Die Verformung gefüllter Zähne] Deflection of restored teeth *Deutsche Zahnärztliche Zeitschrift* **49(10)** 812-815.

Leevailoj C, Platt JA, Cochran MA & Moore BK (1998) *In vitro* study of fracture incidence and compressive fracture load of all-ceramic crowns cemented with resin-modified glass ionomer and other luting agents *Journal of Prosthetic Dentistry* **80(6)** 699-707.

Martin N & Jedynakiewicz N (1996) Optimizing factors for extensive Cerec restorations in *CAD/CIM in Aesthetic Dentistry. CEREC 10 year Anniversary Symposium* (Editor WH Mörmann) Quintessence Publishing Co Inc Berlin 153-160.

Mehl A, Godescha P, Kunzelmann KH & Hickel R (1996) [Randspaltverhalten von Komposit- und Keramikinlays bei ausgedehnten Kavitäten] Marginal integrity of composite and ceramic inlays in large MOD cavities *Deutsche Zahnärztliche Zeitschrift* **51(11)** 701-704.

Mehl A, Pfeiffer A, Kremers L & Hickel R (1998) [Randständigkeit von Cerec-II-Inlay-Restaurationen bei ausgedehnten Kavitäten mit stark geschwächten Höckern] Marginal integrity of Cerec II inlays in extensive MOD cavities *Deutsche Zahnärztliche Zeitschrift* **53(1)** 57-60.

Pröbster L (2001) [Sind vollkeramische Kronen und Brücken wissenschaftlich anerkannt?] Are all ceramic crowns and bridges scientifically accepted? Gemeinsame Stellungnahme von DGZMK und DGZPW *Deutsche Zahnärztliche Zeitschrift* **56(10)** 575-576.

Reiss B & Walther W (2000) Clinical long-term results and 10-year Kaplan-Meier analysis of Cerec restorations *International Journal of Computerized Dentistry* **3(1)** 9-23.

Rosenstiel SF, Land MF & Crispin BJ (1998) Dental luting agents: A review of the current literature *Journal of Prosthetic Dentistry* **80(3)** 280-301.

Roulet JF (1997) Longevity of glass ceramic inlays and amalgam—results up to 6 years *Clinical Oral Investigations* **1(1)** 40-46.

Roulet JF (1994) Marginal integrity: Clinical significance *Journal of Dentistry* **22(Supplement 1)** S9-S12.

Rueggeberg FA & Caughman WF (1993) The influence of light exposure on polymerization of dual-cure resin cements *Operative Dentistry* **18(2)** 48-55.

Schmalz G, Federlin M & Reich E (1995) Effect of dimension of luting space and luting composite on marginal adaptation of a Class II ceramic inlay *Journal of Prosthetic Dentistry* **73(4)** 392-399.

Schmalz G & Geurtsen W (2001) [Keramik-Inlays und Veneers Wissenschaftliche Stellungnahme der DGZMK] Ceramic inlays and veneers. Scientific statement of the German Dental Association *Deutsche Zahnärztliche Zeitschrift* **56(6)** 347-348.

Seghi RR, Denry IL & Rosenstiel SF (1995) Relative fracture toughness and hardness of new dental ceramics *Journal of Prosthetic Dentistry* **74(2)** 145-150.

Sjögren G, Molin M & van Dijken JW (1998) A 5-year clinical evaluation of ceramic inlays (Cerec) cemented with a dual-cured or chemically cured resin composite luting agent *Acta Odontologica Scandinavica* **56(5)** 263-267.

Sorensen JA & Munksgaard EC (1996) Relative gap formation adjacent to ceramic inlays with combinations of resin cements and dentin bonding agents *Journal of Prosthetic Dentistry* **76(5)** 472-476.

Tidehag P & Gunne J (1995) A 2-year clinical follow-up study of IPS Empress ceramic inlays *International Journal of Prosthodontics* **8(5)** 456-60.

Thonemann B, Federlin M, Schmalz G & Hiller KA (1995) Resin-modified glass ionomers for luting posterior ceramic restorations *Dental Materials* **11(3)** 161-168.

Thonemann B, Schmalz G, Brandenstein S & Hiller KA (1994) [Randspaltverhalten von Keramikinlays mit Dentinadhäsiven *in vitro*] Marginal adaptation of ceramic inlays with dentin bonding agents *in vitro Deutsche Zahnärztliche Zeitschrift* **49(10)** 840-844.

Thonemann B, Federlin M, Schmalz G & Schams A (1997) Clinical evaluation of heat-pressed glass-ceramic inlays *in vivo*: 2-year results *Clinical Oral Investigations* **1(1)** 27-34.

van Dijken JW, Hasselrot L, Örmin A & Olofsson AL (2001) Restorations with extensive dentin/enamel-bonded ceramic coverage. A 5-year follow-up *European Journal of Oral Sciences* **109(4)** 222-229.

van Dijken JW, Örmin A & Olofsson AL (1999) Clinical performance of pressed ceramic inlays luted with resin-modified glass ionomer and autopolymerizing resin composite cements *Journal of Prosthetic Dentistry* **82(5)** 529-535.

Wagner J, Schmalz G & Hiller KA (2002) Partial crowns: Clinical performance and longevity of gold vs ceramic *Journal of Dental Research* **81(Special Issue A)** Abstract #2651 p 333.

©Operative Dentistry, 2004, **29-5,** 571-577

Analysis of the Degree of Conversion of LED and Halogen Lights Using Micro-Raman Spectroscopy

MS Soh • AUJ Yap
T Yu • ZX Shen

Clinical Relevance

The degree of conversion associated with LED curing lights was comparable to halogen lights.

SUMMARY

This study determined the degree of conversion of two LED (light-emitting diodes) (Elipar FreeLight [FL], 3M ESPE; GC e-Light [EL], GC), a high intensity (Elipar TriLight [TL], 3M ESPE) and a very high intensity (Astralis 10 [AS], Ivoclar Vivadent) halogen light. The degree of conversion of these lights was compared to a conventional halogen light (Max [MX] (control), Dentsply-Caulk). Ten different light curing regimens, including pulse (EL1), continuous (FL1, EL2, TL1), turbo (EL3, AS1) and soft-start (FL2, EL4, TL2) modes of various lights were also investigated. Composite specimens of dimensions 3 x 3 x 2 mm were cured with the 10 different light curing regimens investigated. Micro-Raman spectroscopy was used to determine the degree of conversion at the top and bottom surfaces of a composite restorative (Z100, [3M ESPE]) at 60 minutes post-light polymerization. Five specimens were made for each cure mode. The results were analyzed using ANOVA/Scheffe's post-hoc test and Independent Samples *t*-tests at significance level 0.05. The degree of conversion ranged from 55.98 ± 2.50 to 59.00 ± 2.76% for the top surface and 51.90 ± 3.36 to 57.28 ± 1.56% for the bottom surface. No significant difference in degree of conversion was observed for the 10 light curing regimens when compared to MX (control). The curing efficiency of LED lights was comparable to halogen lights regardless of curing modes.

MS Soh, BSc (Hons), research scholar, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

*Adrian UJ Yap, BDS, MSc, PhD, FAMS, FADM, FRSH, associate professor, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

T Yu, BSc, research scholar, Department of Physics, Faculty of Science, National University of Singapore, Republic of Singapore

ZX Shen, Bsc, PhD, associate professor, Department of Physics, Faculty of Science, National University of Singapore, Republic of Singapore

*Reprint request: 5 Lower Kent Ridge Road, Singapore 119074, Republic of Singapore, e-mail: rsdyapuj@nus.edu.sg

INTRODUCTION

The degree of polymerization in crosslinked polymeric matrix systems plays a potentially significant role in determining the ultimate physical and mechanical properties of the material (Ferracane & Greener, 1984). Inadequate polymerization results in inferior physico-mechanical properties, such as poor resistance to wear, poor color stability, secondary caries and adverse tissue reactions, increased rates of water sorption, solubility and early restoration failure (Vargas, Cobb & Schmit,

1998; Venhoven, de Gee & Davidson, 1993; Shortall, Wilson & Harrington, 1995; Pearson & Longman, 1989; Fan & others, 1987). While it is desirable for dental resin composites to achieve high levels of conversion, there is always a significant concentration of unreacted carbon double bonds remaining in the resin when cured. This is due to limitations in the mobility of reactive species imposed by the rapid formation of a cross-linked polymeric network (Ferracane, 1985). In addition, high levels of conversion also resulted in higher contraction strain rates during polymerization (Sakaguchi & Berge, 1998). This usually results in gaps around the cavity margins, resulting in microleakage, pulpal irritation, thermal sensitivity, recurrent caries and internal stresses (Uno & Asmussen, 1991; Feilzer, de Gee & Davidson, 1990).

Several analytical methods exist for the measurement of conversion in dental polymers. Differential scanning calorimetry provides a measure of methacrylate conversion based on the enthalpy of the exothermic polymerization process (Miyazaki & Horibe, 1988; Urabe, Wakasa & Yamaki, 1991). The extent of polymerization shrinkage has also been used to calculate conversion (Venhoven & others, 1993; Rueggeberg & Tamareselvy, 1995). However, the majority of analyses done to assign conversion in dental resins and composites have been based on the use of infrared spectroscopy which provides a direct measure of unreacted methacrylate groups. Fourier transform infrared spectroscopy (FTIR) has been proven to be a powerful and reliable technique used widely for detecting the C=C stretching vibrations directly before and after the curing of materials (Imazato & others, 1995; Sakaguchi & Berge, 1998; Ruyter & Øysæd, 1982; Eliades, Vougiouklakis & Caputo, 1987). As the polymerized specimens need to be pulverized, the procedure is time consuming when measuring the degree of conversion of bulk composites. In addition, the results obtained reflect the polymerization of a small portion of the specimen and may be inaccurate when curing of the specimen is disproportionate.

Studies (Lundin & Koch, 1992; Pianelli & others, 1999; Leloup & others, 2002) have shown that Raman spectroscopy, which involves scattering rather than absorption when compared to FTIR, may be an alternative spectroscopic method for the direct measurement of methacrylate conversion. The degree of conversion using the Raman technique is non-destructive and allows for measurement on the surfaces of the restorations to be performed *in vivo* and *in vitro* without any mechanical or chemical pretreatment, which may influence the results. In this method, the measurement of cure is made on a relative basis by comparing the vibration band of the residual unpolymerized methacrylate C=C band at 1640 cm^{-1} with the aromatic stretching band at 1610 cm^{-1}. Thus, Raman spectroscopy may be a more convenient, accurate technique than FTIR for determining the degree of conversion.

The aspect of polymerization under greatest control by the clinician is the application of the curing light (Sakaguchi & Berge, 1998). The use of halogen light curing units (LCUs) to polymerize dental composite has several drawbacks despite their popularity. The halogen bulbs (which have a limited effective lifetime of about 40-100 hours), reflector and filter degrade over time due to high operating temperatures and a significant quantity of heat produced during the curing cycles (Jandt & others, 2000). In view of the latter, using halogen curing lights requires intensive fan cooling. As the cooling air current enters and exits through slots in the casing, disinfection of the handpiece is incomplete and bacterial aerosol that is present in the patient's mouth may be dispersed. The aforementioned will reduce the effectiveness of polymerization in composite restoratives (Barghi, Berry & Hatton, 1994). To overcome the drawbacks of halogen lights, blue LED (light-emitting diodes) LCUs have been developed for polymerization of light-activated dental materials. LEDs have lifetimes of more than 10,000 hours and undergo little degradation of light output over time. They use junctions of doped semiconductors (p-n junctions) for the generation of light and, hence, require no filters to produce blue light and are resistant to shock and vibration. Their relatively low power consumption makes them suitable for portable use. The narrower spectral output of these blue LEDs of 440 to 490 nm fall within the CQ absorption spectrum (Mills, Jandt & Ashworth, 1999). When equal light energy was irradiated, the degree of conversion by LED was not significantly different from halogen lamps (Yoon & others, 2002). However, Knezevic and others (2001) and Tarle and others (2002) have shown that composites cured by LED LCUs resulted in a lower degree of conversion when compared to halogen LCUs.

The number of studies regarding the degree of conversion of LED lights is still limited and differences in findings have yet to be explained. Hence, the objective of this study was to determine the degree of conversion of composites cured with the various LED and halogen lights by non-destructive micro-Raman spectroscopy. The degree of conversion by the various pre-programmed light curing modes was also investigated and compared to standard continuous modes.

METHODS AND MATERIALS

A mini-filled resin composite (Z100; 3M ESPE, St Paul, MN, USA) of A2 shade, five LCUs (LED–Elipar Freelight [FL] and a GC e-light [EL]; a high intensity halogen light–Elipar Trilight [TL]; a very high intensity halogen light–Astralis 10 [AS] and a conventional halogen light–Max [MX], Dentsply-Caulk) were selected for this study. Pulse (EL1), continuous (FL1, EL2,

TL1), turbo (EL3, AS1) and soft-start (FL2, EL4, TL2) curing modes of the various lights were examined. A conventional continuous cure halogen LCU (Max) served as the control light source in this study. Details of the five LCUs and the 10 light curing regimens evaluated are listed in Table 1. The intensity of all curing lights was checked with a radiometer (Cure Rite, EFOS INC, Ontario, Canada) prior to use to ensure consistency in intensity output from the light source. Standard deviations ranging from 2.17 to 5.34 mW/cm^2 were obtained for the various lights.

The composite material was placed in customized acrylic molds with square cavities 2-mm deep and 3-mm wide/long confined between two opposing acetate strips (Hawe-Neos Dental, Bioggio, Switzerland). A glass slide was placed over the acetate strip and pressure was applied to extrude excess material. The composite specimens were then polymerized using the various curing lights and modes. Immediately after light polymerization, the acetate strips were discarded and the specimens stored in a light-proof container at room temperature of (25 ± 0.2)°C for one hour. Five specimens were prepared for each light curing mode.

Micro-Raman spectra of both unpolymerized and polymerized resins (top and bottom surfaces) were measured at room temperature in the backscattering geometry using Spex 1702/04 single-grating Raman spectrometer with an Olympus microscope attachment equipped with a liquid-nitrogen-cooled CCD detector. The instrumental resolution was ~ 0.7 cm^{-1}. The 632.8 nm lines of an He-Ne laser were used as the excitation source and the scattered laser light was rejected using a pair of super notch filters that allowed the Raman signal to reach the spectrograph. Typical Raman spectra were recorded with 10 mW laser power using a 100x microscope objective with NA 0.95. The Raman spectra were recorded in the region of 1580-1740 cm^{-1} with the following conditions: confocal hole: 200; irradiation time: 60 seconds; number of accumulations: 5. A standard baseline technique was used to calculate the degree of conversion. The degree of conversion was calculated using the following formula:

$$\text{Degree of conversion (\%)} = \left[1 - \frac{R_{polymerized}}{R_{unpolymerized}}\right] \times 100\%$$

where R = band height of C=C at 1640 cm^{-1}/band height of aromatic group at 1610 cm^{-1}.

The mean conversion ratio for the five specimens was calculated using the following formula: conversion ratio = degree of conversion of bottom surface/degree of conversion of top surface. All data obtained was subjected to one-way ANOVA/Scheffe's post-hoc tests and Independent Samples *t*-tests at significance level 0.05.

RESULTS

A representative diagram of the Raman spectra recorded for both the polymerized and unpolymerized specimens is shown in Figure 1. The mean degree of conversion and conversion ratio of the various light curing modes are shown in Table 2 and the results of statistical analysis are shown in Table 3 and 4.

No significant difference in the degree of conversion was observed when comparing the different light curing modes with the control (MX) and within the same lights. The degree of conversion of LED and halogen lights for the top surface ranged from 55.98 ± 2.50 to 58.10 ± 0.66% and 56.21 ± 1.08 to 59.78 ±

Table 1: *Details of the Light Curing Units (LCU) and the Various Curing Modes Evaluated*

LCU	Curing Modes	Curing Profiles		
Elipar FreeLight (LED)	Standard (FL1)	400 mW/cm^2 (40 seconds)		
3M ESPE, Seefeld, Germany	Exponential (FL2)	0-400 mW/cm^2 (12 seconds)	→	400 mW/cm^2 (28 seconds)
GC e-Light (LED)	Pulse Curing (EL1)	750 mW/cm^2 (10 pulses x 2 seconds)		
GC Europe, Leuven, Belgium	Standard (EL2)	350 mW/cm^2 (40 seconds)		
	Turbo (EL3)	600 mW/cm^2 (20 seconds)		
	Soft-start curing A (EL4)	0-600 mW/cm^2 (20 seconds)	→	600 mW/cm^2 (20 seconds)
Max (Halogen) Dentsply-Caulk, Milford, DE, USA	Standard (MX)	400 mW/cm^2 (40 seconds)		
Elipar TriLight (Halogen)	Standard (TL1)	800 mW/cm^2 (40 seconds)		
3M-ESPE, Seefeld, Germany	Exponential (TL2)	100-800 mW/cm^2 (15 seconds)	→	800 mW/cm^2 (25 seconds)
Astralis 10 (Halogen) Ivoclar-Vivadent, Liechtenstein, Austria	High Power (AS1)	1200 mW/cm^2 (10 seconds		

Curing profiles are based on manufacturers' information.

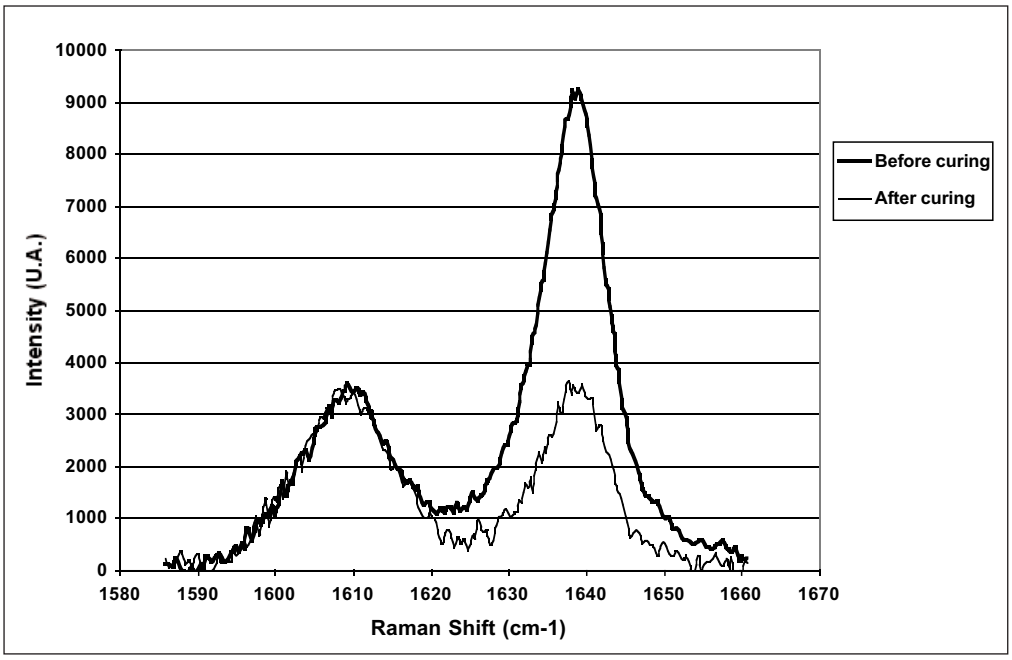


Figure 1. *Raman spectra of light-activated composie Z100.*

Table 2: *Mean Degree of Conversion of the Various Light Curing Modes*

Light Curing Mode	Top Surface	Bottom Surface	Conversion Ratio
FL1	55.98 (2.50)	53.49 (3.48)	0.96 (0.40)
FL2	58.10 (0.66)	57.20 (1.18)	0.98 (0.15)
EL1	56.88 (1.36)	53.17 (3.00)	0.93 (0.50)
EL2	56.85 (3.24)	53.94 (3.09)	0.95 (0.87)
EL3	56.03 (1.46)	51.90 (3.36)	0.93 (0.72)
EL4	57.80 (1.15)	54.10 (2.71)	0.94 (0.55)
MX	57.34 (1.00)	54.89 (1.36)	0.96 (0.27)
TL1	59.00 (2.76)	57.28 (1.56)	0.97 (0.56)
TL2	59.78 (1.27)	56.55 (1.58)	0.95 (0.02)
AS1	56.21 (1.08)	56.14 (1.51)	1.00 (0.38)

Standard deviations in parentheses.

Table 3: *Results of Statistical Analysis*

Variable	Differences
Top Surface	NS
Bottom Surface	NS
Conversion Ratio	NS

Results of one-way ANOVA/Scheffe's post-hoc test (p<0.05). NS denotes no significance.

1.27%, respectively. For the bottom surface, the degree of conversion of LED and halogen lights ranged from 51.90 ± 3.36 to 57.20 ± 1.18% and 54.89 ± 1.36 to 57.28 ± 1.56%, respectively. The degree of conversion at both the top and bottom surfaces of the specimens polymerized with soft-start curing regimens by LED LCUs was observed to be higher than the standard cure modes, that is, FL2 > FL1 and EL4 > EL2.

DISCUSSION

The curing of dental resins is important from a practical and fundamental viewpoint. Studies (Eliades & others, 1987; Ferracane, 1985; Ferracane & others, 1997; Asmussen & Peutzfeldt, 2003) have shown a direct correlation between the degree of conversion of the resins and the bulk properties, such as hardness, wear, polymerization shrinkage and tensile and compressive strength. Thus, it will be useful to have a quick, reliable method of determining the degree of cure of dental resins.

Micro-Raman spectroscopy, which is viewed as a conventional Raman spectroscopy with enhanced resolution bounded by the far-field diffraction limit, is an attractive technique for dental materials analysis since samples can be examined irrespective of thickness or form by simply illuminating them with a laser beam (Delhaye & Dhamelincourt, 1975; Rosasco, Etz & Cassatt, 1975). In principle, this allows qualitative and quantitative analysis of chemical and physical structure without sample modification. The ability to handle "difficult" samples is a key advantage over mid-infrared spectroscopy, which requires the preparation of thin films, KBr disks, Nujol mulls or solutions. This is indeed very important for morphology studies since sample preparation can easily perturb morphology.

Raman spectroscopy, such as infrared spectroscopy (IR), is a vibrational technique and, as such, is sensitive to the vibrational modes of molecules (Szymanski, 1967). In dental resins, the vibrational bands of interest are typically the C=C double bond, the C=O vibration, the aromatic ring of the monomers and crosslinked networks. For highly symmetrical molecules, the quantum mechanical selection rules determine which modes of vibration will be IR or Raman active. For the unsymmetrical monomers and polymers used in dental resins, most vibrations have both infrared and Raman activity. However, there are still two important differences between the IR and Raman

spectroscopy of these systems: (1) IR spectroscopy is an absorption technique; whereas, Raman spectroscopy is a scattering method and (2) intensities in IR measurements are determined by changes in the dipole moments of the vibrations; whereas, for Raman measurements, the relevant quantity is the change in the polarizability tensor. These differences affect both the method of obtaining data from samples and the parameters necessary for calibration curves (Shin & others, 1993). In Raman scattering, the relevant quantity is the Raman scattering cross section of the band of interest that depends on the intensity of the incoming light and the polarizability tensor of the particular vibration (Hendra, Jones & Warnes, 1991). Thus, it is always useful to have one band whose intensity can act as an internal standard.

Table 3: *Comparison of Mean Degree of Conversion Between Curing Modes for LCU That Offer Different Polymerization Regimens*

Variable	LCU	Differences
KHN Top	Elipar FreeLight	NS
	GC e-Light	NS
	Elipar TriLight	NS
KHN Bottom	Elipar FreeLight	NS
	GC e-Light	NS
	Elipar TriLight	NS
Coversion ratio	Elipar FreeLight	NS
	GC e-Light	NS
	Elipar TriLight	NS

Results of One-way ANOVA/Scheffe's post-hoc test or Independent Samples t-test ($p<0.05$). NS denotes no statistical significance.

When light energy was supplied to activate composites for polymerization to occur, the C=C vibration decreased with respect to the aromatic group mode after polymerization (Figure 1). In this study, the aromatic group that remains unchanged before and after curing was identified as the internal reference. The carbonyl (C=O) group, which has a characteristic frequency ranging from 1600 to 1800 cm^{-1}, was ruled out as the internal reference as the exact location of the C=O frequency varies depending on the atoms attached to the carbonyl. Electron donating groups, electron withdrawing groups, resonance effects and hydrogen bonding all cause the force constant of the C=O bond to vary and therefore the frequency of the carbonyl absorption to change. In a conjugated system, the C=C frequencies may fall near aromatic bands. The carbonyl bands, which absorb strongly in IR, can also obscure the original position of C=C vibrations, making it very difficult to interpret. Usually, the C=C stretch band has a more distinctive Raman band that is high in intensity and not disturbed by the weaker infrared intensity of the C=O stretch band. As conjugated double bonds are more sensitive to Raman spectroscopy than infrared, any change occurring within the double bond can be predicted more precisely by using this technique (Rehman, Harper & Bonfield, 1996).

The mini-filled resin composites, Z100 of A2 shade, were selected for this study to minimize the effects of colorants on light polymerization (Bayne, Heymann & Swift, 1994). Factors influencing the transmission of light include thickness of the restorative material, the presence and size of filler particles and the distance of the light tip to the restoration surface (Tate, Porter & Dosch, 1999). As these factors were all standardized in this study, any differences in the degree of conversion may be attributed to the light-curing regimen. Two-mm thick composite specimens were used to ensure uniform and maximum polymerization (Yap, 2000). As a minimum intensity of 400 mW/cm^2 has been suggested for routine polymerization (Tate & others, 1999; Rueggeberg, Caughman & Curtis, 1994), this light intensity (Max polymerization unit), together with the manufacturer's recommended cure time of 40 seconds, was used as control in this study. Raman spectra were recorded at one hour post light irradiation, as the degree of conversion shows a gradual increase after light exposure and maximum hardness was attained after the first hour of polymerization (Pilo & Cardash, 1992). In addition, the post-gel shrinkage of composites occurred most rapidly during the first hour of post-light irradiation (Yap & others, 2000).

The degree of composite cure is proportional to the amount of light to which they are exposed. The degree of conversion was found to be higher on the top surfaces than the bottom surfaces. However, top surface hardness is not an adequate clinical indicator of an adequately polymerized composite restoration, because even a very poor light source may produce a well-cured surface that conceals inadequate or unpolymerized resin in the deeper parts of the cavity (Hansen & Asmussen, 1993). At the bottom surfaces, no significant difference in the degree of conversion was found among all light curing regimens and the control (MX). All light curing regimens achieved a degree of conversion greater than 51% for both surfaces. The results corroborated well with earlier studies where the degree of conversion ranged from 43.5% to 73.8% (Pianelli & others, 1999; Yoon & others, 2002; Chung & Greener, 1988). However, it must be noted that the degree of conversion does not indicate the degree of polymerization of Bis-GMA or TEGDMA itself but indicates the conversion of aliphatic C=C bond in the methyl methacrylate group into a C-C bond. The 51% of degree of conversion does not mean that 49% of the monomer remains but indicates that 49% of C=C bonds remain.

The conversion ratio obtained in this study was found to be greater than 0.9 for all light curing regimens. For an adequately cured composite, the hardness gradient should not exceed 10% to 20% (the conversion ratio should be greater than 0.8) (Pilo & Cardash, 1992). The difference in conversion ratio can be attributed to light scattering and absorption as light passed through the bulk of the composite (Ruyter & Øysæd, 1982). This scattering of light accounted for minor differences in the degree of conversion between the top and bottom surfaces of the 2-mm composites evaluated in this study.

The use of a high intensity light source to improve composite properties has recently been introduced. High intensity lights provide higher values of the degree of conversion and superior mechanical and physical properties but produce higher contraction strain rates during the polymerization of composites (Uno & Asmussen, 1991). The use of very high light intensities for short durations (turbo cure) has also been developed. These curing regimens were established primarily to reduce clinical time and were shown not to increase polymerization stresses if the total light energy density (intensity x time) is maintained (Yap, Wong & Siow, 2003). The use of high intensity (TL) and very high intensity (AS) lights in this study did not result in a degree of conversion that was significantly different from the control (MX) for both the top and bottom surfaces of the specimens evaluated. This illustrated the potential use of high intensity lights or turbo cure mode for dental restorations. However, it must be noted that the application of high intensity lights resulted in an increase in temperature that may be damaging to the pulp (Yap & Soh, 2003).

From the results obtained in this study, the degree of conversion by LED was not significantly different from halogen LCU. The results suggest that comparable composite cure may be achieved with LED curing lights and regimens. Given the inherent advantages of the LED principle and swift progress in semiconductor technology, LED LCUs appear to have greater potential in future clinical applications compared to halogen LCUs.

CONCLUSIONS

Under the conditions of this *in vitro* study:

1. Micro-Raman spectroscopy is an easy, effective technique for measuring degree of conversion.
2. The degree of conversion was found to be independent of curing lights and light curing regimens.
3. The degree of conversion at both the top and bottom surfaces by LED, high intensity and very high intensity lights was found to be comparable to the conventional halogen light.

(Received 6 June 2003)

References

Asmussen E & Peutzfeldt A (2003) Light-emitting diode curing: Influence on selected properties of resin composites *Quintessence International* **34(1)** 71-75.

Barghi N, Berry T & Hatton C (1994) Evaluating intensity output of curing lights in private dental offices *Journal of the American Dental Association* **125(7)** 992-996.

Bayne SC, Heymann HO & Swift EJ Jr (1994) Update on dental composite restorations *Journal of the American Dental Association* **125(6)** 687-701.

Chung K & Greener EH (1988) Degree of conversion of seven visible light-cured posterior composites *Journal of Oral Rehabilitation* **15(6)** 555-560.

Delhaye M & Dhamelincourt P (1975) Raman microprobe and microscope with laser excitation *Journal of Raman Spectroscopy* **3** 33-43.

Eliades GC, Vougiouklakis GJ & Caputo AA (1987) Degree of double bond conversion in light-cured composites *Dental Materials* **3(1)** 19-25.

Fan PL, Wozniak WT, Reyes WD & Stanford JW (1987) Irradiance of visible light-curing units and voltage variation effects *Journal of the American Dental Association* **115(3)** 442-445.

Feilzer AJ, de Gee AJ & Davidson CL (1990) Quantitative determination of stress reduction by flow in composite restorations *Dental Materials* **6(3)** 167-171.

Ferracane JL (1985) Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins *Dental Materials* **1(1)** 11-14.

Ferracane JL & Greener EH (1984) Fourier transform infrared analysis of degree of polymerization in unfilled resins-methods comparison *Journal of Dental Research* **63(8)** 1093-1095.

Ferracane JL, Mitchem JC, Condon JR & Todd R (1997) Wear and marginal breakdown of composites with various degrees of cure *Journal of Dental Research* **76(8)** 1508-1516.

Hansen EK & Asmussen E (1993) Correlation between depth of cure and surface hardness of a light-activated resin *Scandinavian Journal of Dental Research* **101(1)** 62-64.

Hendra P, Jones C & Warnes G (1991) *Fourier Transform Raman Spectroscopy: Instrumentation and Chemical Applications* New York Ellis Harwood.

Imazato S, Tarumi H, Kobayashi K, Hiraguri H, Oda K & Tsuchitani Y (1995) Relationship between the degree of conversion and internal discoloration of light-activated composite *Dental Materials Journal* **14(1)** 23-30.

Jandt KD, Mills RW, Blackwell GB & Ashworth SH (2000) Depth of cure and compressive strength of dental composites cured with blue light emitting diodes (LEDs) *Dental Materials* **16(1)** 41-47.

Knezevic A, Tarle Z, Meniga A, Sutalo J, Pichler G & Ristic M (2001) Degree of conversion and temperature rise during polymerization of composite resin samples with blue diodes *Journal of Oral Rehabilitation* **28(6)** 586-591.

Leloup G, Holvoet PE, Bebelman S & Devaux J (2002) Raman scattering determination of the depth of cure of light-activated composites: Influence of different clinically relevant parameters *Journal of Oral Rehabilitation* **29(6)** 510-515.

Lundin SA & Koch G (1992) Cure profiles of visible-light-cured class II composite restorations *in vivo* and *in vitro Dental Materials* **8(1)** 7-9.

Mills RW, Jandt KD & Ashworth SH (1999) Dental composite depth of cure with halogen and blue light emitting diode technology *British Dental Journal* **186(8)** 388-391.

Miyazaki K & Horibe TJ (1988) Polymerization of multifunctional methacrylates and acrylates *Journal of Biomedical Materials Research* **22(11)** 1011-1022.

Pearson GJ & Longman CM (1989) Water sorption and solubility of resin-based materials following inadequate polymerization by a visible-light curing system *Journal of Oral Rehabilitation* **16(1)** 57-61.

Pianelli C, Devaux J, Bebelman S & Leloup G (1999) The micro-Raman spectroscopy, a useful tool to determine the degree of conversion of light-activated composite resins *Journal of Biomedical Materials Research (Applied Biomaterials)* **48(5)** 675-681.

Pilo R & Cardash HS (1992) Post-irradiation polymerization of different anterior and posterior visible light-activated resin composites *Dental Materials* **8(5)** 299-304.

Rehman I, Harper EJ & Bonfield W (1996) *In situ* analysis of the degree of polymerization of bone cement by using FT-Raman spectroscopy *Biomaterials* **17(16)** 1615-1619.

Rosasco GJ, Etz ES & Cassatt WA (1975) The analysis of discrete fine particles by Raman spectroscopy *Applied Spectroscopy* **29** 396-404.

Rueggeberg F & Tamareselvy K (1995) Resin cure determination by polymerization shrinkage *Dental Materials* **11(4)** 265-268.

Rueggeberg FA, Caughman WF & Curtis JW Jr (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19(1)** 26-32.

Ruyter IE & Øysæd H (1982) Conversion in different depths of ultraviolet and visible light activated composite materials *Acta Odontologica Scandinavica* **40(3)** 179-192.

Sakaguchi RL & Berge HX (1998) Reduced light energy density decreases post-gel contraction while maintaining the degree of conversion in composites *Journal of Dentistry* **26(8)** 695-700.

Shin WS, Li XF, Schwartz B, Wunder SL & Baran GR (1993) Determination of the degree of cure of dental resins using Raman and FT-Raman spectroscopy *Dental Materials* **9(5)** 317-324.

Shortall AC, Wilson HJ & Harrington E (1995) Depth of cure of radiation-activated composite restoratives—Influence of shade and opacity *Journal of Oral Rehabilitation* **22(5)** 337-342.

Szymanski HA (1967) *Raman Spectroscopy: Theory and Practice* New York Plenum Press.

Tarle Z, Meniga A, Knezevic A, Sutalo J, Ristic M & Pichler G (2002) Composite conversion and temperature rise using a conventional, plasma arc, and an experimental blue LED curing unit *Journal of Oral Rehabilitation* **29(7)** 662-667.

Tate WH, Porter KH & Dosch RO (1999) Successful photocuring: Don't restore without it *Operative Dentistry* **24(2)** 109-114.

Uno S & Asmussen E (1991) Marginal adaptation of a restorative resin polymerized at reduced rate *Scandinavian Journal of Dental Research* **99(5)** 440-444.

Urabe H, Wakasa K & Yamaki M (1991) Cure performance of multifunctional monomers to photo-initiators: A thermoanalytical study on bis-GMA- based resins *Journal of Materials Science* **26** 3185-3190.

Vargas MA, Cobb DS & Schmit JL (1998) Polymerization of composite resins: Argon laser vs conventional light *Operative Dentistry* **23(2)** 87-93.

Venhoven BA, de Gee AJ & Davidson CL (1993) Polymerization contraction and conversion of light-curing bisGMA-based methacrylate resins *Biomaterials* **14(11)** 871-875.

Yap AU & Soh MS (2003) Thermal emission by different light-curing units *Operative Dentistry* **28(3)** 260-266.

Yap AU (2000) Effectiveness of polymerization in composite restoratives claiming bulk placement: Impact of cavity depth and exposure time *Operative Dentistry* **25(2)** 113-120.

Yap AU, Wang HB, Siow KS & Gan LM (2000) Polymerization shrinkage of visible-light-cured composites *Operative Dentistry* **25(2)** 98-103.

Yap AU, Wong NY & Siow KS (2003) Composite cure and shrinkage associated with high intensity curing light *Operative Dentistry* **28(4)** 357-364.

Yoon TH, Lee YK, Lim BS & Kim CW (2002) Degree of polymerization of resin composites by different light sources *Journal of Oral Rehabilitation* **29(12)** 1165-1173.

©Operative Dentistry, 2004, **29-5,** 578-585

Demineralization Inhibition of Direct Tooth-colored Restorative Materials

EH Gonzalez • AUJ Yap • SCY Hsu

Clinical Relevance

At the margins of restorations, the caries inhibition effect of glass ionomers and giomers was significantly better than compomers and composites.

SUMMARY

This study compared the demineralization inhibition properties of fluoride releasing tooth-colored restorative materials. Materials evaluated included a giomer (Reactmer, Shofu [RM]), a conventional glass ionomer (Fuji II, GC [FJ]), a resin modified glass ionomer (Fuji II LC, GC [FL]) and a compomer (Dyract AP, Dentsply [DY]). A non-fluoride releasing composite (Spectrum TPH, Dentsply [SP]) was used for comparison. Class V preparations on buccal and palatal/lingual were made at the CEJ of 75 freshly extracted molars. The teeth were randomly divided into five groups of 15 and restored with the various materials. The occlusal half of each restoration was in enamel, while the gingival half was in dentin. The restored teeth were stored in distilled water at 37°C for two weeks and subjected to artificial caries challenge (18 hours demineralization [pH 5.0] followed by six hours of remineralization [pH 7.0]) for three days. Sections of 130 ± 20 µm were examined with a polarized light microscope, and outer lesion depth [OLD] and wall area [WA] lesion/inhibition measurements were made using image analysis software. All data were subjected to statistical analyses at 0.05 significance level. For the various materials, OLD ranged from 54.55 to 65.86 µm and 124.68 to 145.97 µm in enamel and dentin, respectively. WA ranged from -2356.13 to 1398.20 µm² and -3011.73 to 5095.80 µm² (positive values indicate wall inhibition, negative values indicate wall lesion) in enamel and dentin, respectively. Results of ANOVA/Scheffe's post-hoc test ($p<0.05$) were as follows: Enamel OLD—no significant difference between materials; Dentin OLD—SP > FJ, FL & RM; Enamel WA inhibition—FJ, FL & RM > DY & SP and Dentin WA inhibition—FJ > FL > RM > DY > SP. The demineralization inhibition effect of giomers, conventional and resin-modified glass ionomer cements appear to be more evident at the margins of restorations.

Edelmiro de Hoyos Gonzalez, BDS, research scholar, Department of Restorative Dentistry, National University of Singapore, Republic of Singapore

*Adrian UJ Yap, BDS, MSc, PhD, FAMS, FADM, FRSH, associate professor, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

Stephen CY Hsu, DDS, MS, PhD, associate professor, Department of Preventive Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

*Reprint request: 5 Lower Kent Ridge Road, Singapore 119074, Republic of Singapore, e-mail: rsdyapuj@nus.edu.sg

INTRODUCTION

Recurrent or secondary caries is one of the major reasons for dental restoration failure (Kidd, Toffenetti & Mjör, 1992; Mjör, 1985). By definition, it is found at the tooth-restoration interface and is, in general, the result of microleakage (Arends, Dijkman & Dijkman, 1995). Microleakage is defined as the clinically undetectable passage of bacteria and fluids between cavity walls and restorative materials. The loss of marginal integrity provides potential pathways for reinfection, as cariogenic bacteria can penetrate into the underlying dentin through these defects (Brännström & Nordenvall, 1978). These micro-organisms are responsible for the demineralization of adjacent dentin and/or enamel via a chemical process presumed to be similar to that in primary caries (Arends & others, 1995). As the marginal seal of tooth-colored restoratives to tooth substrates is still not perfect (Sjodin, Uusitalo & Van Dijken, 1996; Yap, Lim & Neo, 1995), antibacterial and fluoride releasing properties are desirable.

Glass ionomer cements were introduced to the dental profession in the early 1970s (Wilson & Kent, 1972). Their favorable adhesive and fluoride-releasing properties have led to their widespread use as luting, lining and restorative materials (Sidhu & Watson, 1995). The disadvantages of these cements, however, include sensitivity to moisture, low initial mechanical properties and inferior translucency compared to resin composites. Hybrid materials combining the technologies of glass ionomers and resin composites were subsequently developed to help overcome the problems of conventional glass ionomer cements (GIC) and maintain their clinical advantages. Examples of these hybrid materials include resin-modified glass ionomer cements (RMGIC) and compomers (polyacid-modified resin composites). Recently, a new category of hybrid material was presented to the dental profession. Known as giomers, they employ the use of pre-reacted glass ionomer (PRG) technology to form a stable phase of glass ionomer in the restorative. Unlike compomers, fluoro-alumino silicate glass is reacted with polyacrylic acid prior to inclusion into the urethane resin. The manufacturer's claims include fluoride release, fluoride recharge, biocompatibility, smooth surface finish, excellent aesthetics and clinical stability. Like compomers, giomers are light polymerized and require the use of bonding systems for adhesion to tooth structure.

A number of studies have used *in vitro* methods to produce artificial caries lesions in enamel and dentin. They incorporated the use of acidified gels (Attar & Onen, 2002; Dionysopoulos & others, 1998; Dunne & others, 1996; Hicks & Flaitz, 2000; Millar, Abiden & Nicholson, 1998; Tam, Chan & Yim, 1997), buffered solutions (Donly & Grandgenett, 1998; Heilman & others, 1997) and incubation with natural plaque (Gilmour, Edmunds & Newcombe, 1997; Hsu & others, 1998; Itota & others, 2001; Nagamine & others, 1997; Torii & others, 2001). These *in vitro* methods have the ability to demineralize and remineralize tooth structure around restorations and determine if restorative materials will decrease tooth demineralization (Donly, 1994; Erickson & Glasspoole, 1995; Featherstone, 1996; Wefel, Heilman & Jordan, 1995). Although the enamel and/or dentin demineralization inhibition effects of glass ionomers and compomers have been widely reported, no literature is available regarding the demineralization inhibition effect of giomers.

This study determined the enamel and dentin demineralization inhibition properties of the continuum of direct tooth-colored restoratives including the recently introduced giomers. In addition, demineralization inhibition offered by the various materials was also compared adjacent to and at a distance from the restorations in an attempt to define the role of the materials in the continuum.

METHODS AND MATERIALS

Tables 1 and 2 summarize the materials and bonding/coating agents used in this study. They included a conventional glass ionomer cement (Fuji II Cap [FJ]), a resin-modified glass ionomer cement (Fuji II LC [FL]), a giomer (Reactmer [RM]), a compomer (Dyract AP [DY]) and a non-fluoride releasing resin composite (Spectrum TPH [SP]) control. Seventy-five freshly extracted human third molars were randomly divided into five groups of 15 teeth. The teeth were free from caries, structural defects and extraction flaws. This was assessed under a stereomicroscope [Olympus SZ40, Tokyo, Japan]) at 10x magnification. Immediately following extraction, the teeth were placed in 10% formaline-saline solution for 10 minutes, cleaned and stored in distilled water at 4°C. Buccal and palatal/lingual Class V cavities (2-mm deep, 4-mm long [mesio-distal] and 3-mm wide [occlusal-gingival]) were carefully prepared at the CEJ of each tooth by a single operator. The cavities were prepared by means of a high-speed handpiece with a #330 carbide bur and a #1311 diamond bur (Shofu, Kyoto, Japan) under water spray. Standardization of the cavities was ensured by measurement with a digital caliper (Fowler Ultra-cal Mark III, Sylvac, Sweden). Conditioners, coating and priming materials were thoroughly hand-shaked prior to use as indicated in the manufacturer's instructions. One hundred and fifty restorations were placed in one increment following manufacturer's instructions (Table 3) with the aid of transparent cervical matrices (Hawe-Neos Dental 721; Gentilino, Switzerland). All restorations were gross finished 10 minutes after placement using a high-speed handpiece and eight flute tungsten carbide burs (Robot Carbide SH134; Shofu, Kyoto, Japan) under water spray. FJ and FL restorations were covered with a layer of unfilled resin (FujiCoat) and

Table 1: *Technical Profiles and Manufacturers of the Materials Evaluated*

Material	Manufacturer	Chemical Composition		Color	Lot #
		Powder:	**Liquid:**		
Fuji II Capsule	GC Corp Tokyo, Japan	FASG PAA	PAA TA Water	Shade A2	0007186
		Powder:	**Liquid:**		
Fuji II LC Capsule	GC Corp Tokyo, Japan	FASG Pigments	PAA Water HEMA CQ	Shade A2	9912202
		Resin:	**Fillers:**		
Reactmer Paste	Shofu Inc, Kyoto, Japan	FASG UDMA HEMA Photo Initiator	F-PRG, Silica, Aerosil silica Glass fillers	Shade A2	100102
		Resin:	**Fillers:**		
Dyract AP	Dentsply-DeTrey Konstanz, Germany	UDMA TCB	Strontium-fluoro-Silicate glass	Shade A2	0003001521
		Resin:	**Fillers:**		
Spectrum TPH	Dentsply-DeTrey	BisGMA-adduct Bis-EMA TEGDMA	Barium aluminum-Borosilicate, Silica	Shade A2	0006000747

CQ: Camphorquinone
Bis-EMA: Ethoxylated bisphenol-A-glycidyl methacrylate
BisGMA: Bisphenol-A-glycidyl methacrylate
BisGMA -adduct: Adduct of 2,2-Bis[4-2-hydroxy-3-methacry loyloxpropoxy)-phenyl]propane with hexamethylene diisocyanate
FASG: Fluoroaluminosilicate-glass
HEMA: Hydroxyethyl methacrylate
TA : Tartaric Acid
PAA: Poly Acrylic Acid
TEGDMA: Triethylene glycol dimethacrylate
TCB: Reaction product butane tetracarboxylic acid and HEMA
UDMA: Urethane dimethacrylate

Table 2: *Technical Profiles and Manufacturers of the Bonding/Coating Agents*

Material	Manufacturer	Chemical Composition	Lot #
GC Cavity Conditioner	GC Corp Tokyo, Japan	20% PAA 3% Aluminum Chloride Hexahydrate	0006301
GC Fuji Coat LC	GC Corp Tokyo, Japan	Methyl metacrylate	9911021
Reactmer Bond A & B	Shofu Inc, Kyoto, Japan	F-PRG FASG NI Water Acetone	
		4-AET 4-AETA HEMA UDMA Photo Initiator	0901
Conditioner 36	Dentsply-De Trey, Konstanz, Germany	Phosphoric Acid 36%	9904000621
Prime & Bond NT	Dentsply-De Trey, Konstanz, Germany PENTA	PENTA UDMA Nano F Acetone *Resins	0001000767

4-AET: 4-Acryloxyethyltrimellitic acid
4-AETA: 4-Acryloxyethyltrimellitate anhydride
FASG: Fluoroaluminosilicate-glass
F-PRG: Full reaction type pre-reacted glass-ionomer filler
HEMA: Hydroxyetylmetacrylate
Nano F: Nano-filler initiators
NI: New Initiators
PENTA: phosphonated penta-acrylate ester
PAA: Poly Acrylic Acid
**Resins: Resin R5-62-1, T-resin, D-resin*
UDMA: Urethane dimethacrylate

light-cured for 15 seconds after gross finishing. The restored teeth were then stored in distilled water at 37°C for one week and finished/polished with 10 strokes of coarse, medium, fine and extra-fine Sof-lex discs (3M Dental Products, St Paul, MN, USA), at 10,000 rpm. Special care was taken to ensure that no overhangs were present at the enamel and dentin margins of the restorations. After treatment with Sof-lex discs, the restored teeth were returned to distilled water at 37°C for an additional week. The crowns of the restored teeth were separated from the roots and bisected mesio-distally. The tooth fragments were coated with two layers of acid-resistant nail varnish, except for a zone approximately 1-mm wide around the restorations (Donly & Grandgenett, 1998). Half of the restorations in each group were randomly selected for this study (n=75), while the remaining half was used for an extended study.

Artificial recurrent caries were produced around restorations by a three-day pH-cycling regimen beginning with the demineralization phase. The specimens were immersed in demineralizing solution (acetic acid buffer with 2.2 mM calcium ($CaCl_2$), 2.2 mM phosphate (NaH_2 PO_4), 0.05 M acetic acid) for 18 hours at pH 5.0 (Konishi & others, 1999), fol-

Table 3: *Tooth Restoration Procedure*

Fuji II Material 1	**Fuji II LC Material 2**	**Reactmer Material 3**	**Dyract AP Material 4**	**Spectrum TPH Material 5**
GC Cavity Conditioner (10 seconds)	GC Cavity Conditioner (10 seconds)	Reactmer Bond (20 seconds)	Conditioner 36 (20 seconds)	Conditioner 36 (20 seconds)
↓	↓	↓	↓	↓
Rinse (5 seconds) & Dry (3 seconds)	Rinse (5 seconds) & Dry (3 seconds)	Dry (3 seconds)	Rinse (20 seconds) & Dry (3 seconds)	Rinse (20 seconds) & Dry (3 seconds)
↓	↓	↓	↓	↓
Fuji II Caps	Fuji II LC Caps	Light Cure 20 seconds	Prime & Bond NT (20 seconds)	Prime & Bond NT (20 seconds)
↓	↓	↓	↓	↓
ST (4 minutes)	Light Cure 20 seconds	Reactmer Paste	Dry (5 seconds)	Dry (5 seconds)
↓	↓	↓	↓	↓
Gross finishing w/Tungsten carbide 8 flute bur	Gross finishing w/Tungsten carbide 8 flute bur	Light Cure 30 seconds	Light Cure 10 seconds	Light Cure 10 seconds
↓	↓	↓	↓	↓
GC Fuji Coat	GC Fuji Coat	Gross finishing w/Tungsten carbide 8 flute bur	Dyract AP	Spectrum TPH
↓	↓	↓	↓	↓
Light Cure 15 seconds	Light Cure 15 seconds	Stored 1 week in distilled water*	Light Cure 40 seconds	Light Cure 20 seconds
↓	↓	↓	↓	↓
Stored 1 week in distilled water**	Stored 1 week in distilled water**	Finish/polish (w/Sof-lex*) Stored 1 week in distilled water**	Gross finishing w/Tungsten carbide 8 flute bur Stored 1 week in distilled water**	Gross finishing w/Tungsten carbide 8 flute bur Stored 1 week in distilled water**
↓	↓		↓	↓
Finish/Polish (w/Sof-lex*)	Finish/Polish (w/Sof-lex*)		Finish/Polish (w/Sof-lex*)	Finish/Polish (w/Sof-lex*)
↓	↓		↓	↓
Stored 1 week in distilled water**	Stored 1 week in distilled water**		Stored 1 week in distilled water**	Stored 1 week distilled water**

**3M Dental Products, St Paul, MN, USA, **10 ml with distilled water at 37°C, changed every 24 hours. Above instructions were obtained from manufacturer booklets. ST=Setting Time.*

lowed by six hours of remineralizing solution (1.5 mM of calcium, 0.9 mM of phosphate, 0.15 M KCl), at pH 7.0 to obtain caries-like demineralization around restorations each day (Damato, Strang & Stephen, 1988). All solutions were constantly stirred at 132 rpm at 37°C with a Data Plate Hot Plate/Stirrer (Model 735, Barnstead-Thermolyne, Iowa, USA). A five-minute wash in deionized water was conducted between the demineralizing and remineralizing phases and at the end of the process. The specimens were then stored in distilled water at 37°C before being sectioned.

Longitudinal sections of 130 ± 20 µm in thickness were obtained with a Silverstone/Taylor hard-tissue microtome (Series 1000 Deluxe, Sci Fab, Colorado, USA). The enamel/dentin margins were photographed in an imbibition media of distilled water with a digital color video camera (Sony ExwaveHAD SSC-DC58AP, Sony Co, Tokyo, Japan) attached to a polarized light microscope (Olympus BX51, Tokyo, Japan). Photomicrographs were traced using image analysis software (Microimage v4.0; Olympus Optical Co Europa GMBH, Hamburg, Germany) and outer lesion depths [OLD]/wall area [WA] lesions or inhibition were measured according to Hsu and others (1998) (Figure 1).

All data were subjected to statistical analysis at a significance level of 0.05. Paired Samples *t*-test was used to compare OLD and WA values between dentin and enamel. One-way ANOVA and Scheffe's post-hoc tests were performed to compare mean OLD and WA values between materials. Non-parametric Kruskal-Wallis and Mann Whitney U tests were used to compare wall lesion patterns between tissues and materials.

RESULTS

The mean OLD and WA lesion/inhibition values of the various materials are shown in Table 4. Mean OLD ranged from 54.55 to 65.86 µm and 124.68 to 145.97 µm in enamel and dentin, respectively. Mean WA ranged from -2356.13 to 1398.20 µm^2 and -3011.73 to 5095.80 µm^2 in enamel and dentin, respectively. For WA, positive (+) values indicate wall inhibition, while negative (-) values indicate wall lesion. The results of statistical analyses are shown in Tables 5 and 7. Representative photomicrographs associated with the various materials are shown in Figure 2 (a-h). Table 6 shows the frequency distribution of wall lesion/inhibition patterns. Specimens restored with Fuji II showed 100% enamel wall inhibition, while those restored with Spectrum TPH demonstrated 100% dentin wall lesion. Specimens

restored with Fuji II LC and Reactmer showed a mixture of wall inhibitions and "no effects," while those treated with Dyract showed a mixture of wall lesions and "no effects" in enamel and dentin.

DISCUSSION

Materials evaluated in this study represent the entire continuum of direct aesthetic restorative materials currently available to the dental practitioner. Although the experimental setup does not accurately simulate the clinical situation, it gives an indication of caries demineralization inhibition of the materials evaluated. The lack of a pellicle or biofilm of precipitated proteins from gingival fluid, saliva or dentinal fluid on surfaces and in crevices *in vivo* is probably the main difference from the *in vitro* situation (Mjör & Toffenetti, 2000). Deionized water was selected as more fluoride is released in deionized water than in artificial saliva (El-Mallakh & Sarkar, 1990). Fluoride plays an important role in demineralization inhibition, because it can have a topical effect on tooth structures (Donly & Grandgenett, 1998). Based on OLD and WA inhibition results, the beneficial effects of fluoride releasing material appear greater in dentin than enamel. A possible explanation could be that enamel is more resistant to acid attack and has a smaller capacity to take up fluoride than dentin due to its difference in microstructure (Retief & others, 1984; Tveit & Hals, 1980; Weatherell & others, 1983). Another possible reason could be that dentin demineralization is faster than that of enamel. Dentin has been shown to be more vulnerable to acid attack than enamel (Marshall & others, 1989; Phankosol & others, 1985). Therefore, at a lower pH, the dentin structure is demineralized before the enamel structure and the available fluoride will be absorbed more rapidly into dentin than enamel. This phenomenon may explain why the inhibition effect of fluoride on demineralization was usually greater on the dentin surface than enamel, especially if the local concentration of fluoride was low. Specimens restored with Spectrum TPH showed less wall lesion in enamel than in dentin. This may be attributed to the excellent seal offered by the acid etching of enamel.

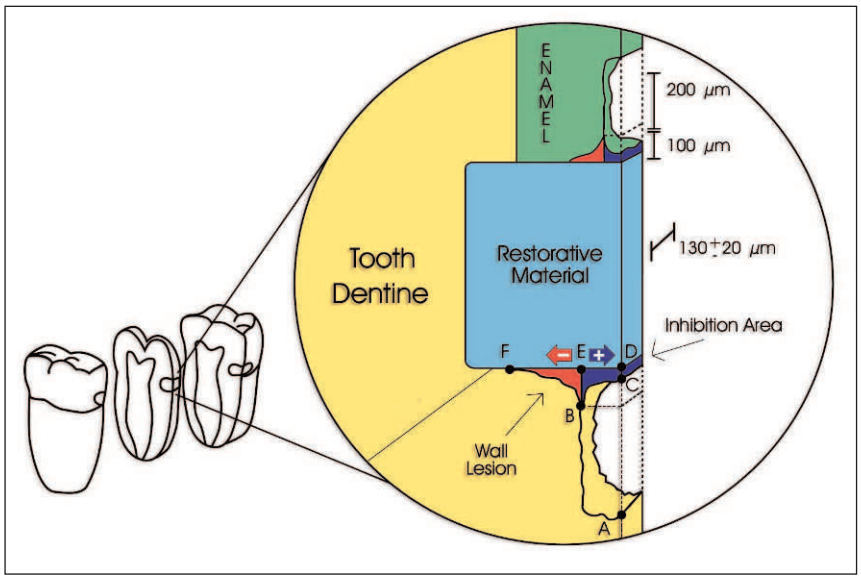


Figure 1. *Lesion measurement and data collection.*
OLD was determined by measuring a lesion area of 200 μm in length and dividing it by 200. WA lesion/inhibition was computed based on a 100 μm area adjacent to the restoration margins. Three major patterns of demineralization along cavity walls were evaluated with polarized light microscopy in both enamel and dentin. Three types of lesions along the cavity wall can be categorized based on three different directions that the inner border of demineralization may take toward the cavity wall (DEF line). If the inner border of demineralization curved upward (ABC line), the effect was an "inhibition" represented by the BCDE area with a positive value (+). If the inner border of the demineralization extended straight to the cavity wall at 90° (ABE line), the lesion exhibited "no effect." If the inner border of demineralization curved downward (ABF line), the effect was a "lesion" represented by the BEF triangular area with a negative value (-).

In enamel, no significant differences in outer lesion depths were seen between materials. Contradictory results regarding enamel/dentin outer lesion depths have been reported. This may be due to differences in the composition of materials and the artificial caries model used. The authors' findings corroborated that of Dunne and others (1996), who reported no significant difference between the outer lesion depths of conventional and resin-modified glass ionomers in enamel and dentin. Millar and others (1998) also found no significant difference in enamel surface lesion depths between glass ionomers and compomers. In this study, Spectrum TPH had significantly greater dentin outer lesion depth than

Table 4: *Mean Outer Lesion Depths (OLD) and Wall Lesion/Inhibition Area (WA) of the Various Materials*

Material	OLD - μm (SD)		WA - μm²(SD)	
	Enamel	Dentin	Enamel	Dentin
Fuji II	54.99 (6.83)	128.81 (15.36)	1398.20 (505.20)	5095.80 (1574.64)
Fuji II LC	54.55 (10.66)	124.68 (10.20)	1283.73 (636.43)	2903.33 (598.19)
Reactmer	56.04 (12.96)	129.02 (15.50)	689.53 (460.24)	1691.53 (1128.13)
Dyract	58.30 (12.53)	134.35 (18.32)	-1399.33 (1508.89)	-327.20 (549.50)
Spectrum TPH	65.86 (16.87)	145.97 (14.67)	-2356.13 (968.46)	-3011.73 (566.84)

Standard deviations in parentheses. For WA, positive values (+) indicates wall inhibition, negative values (-) indicates wall lesion.

Fuji II, Fuji II LC and Reactmer. This finding was in agreement with Nagamine and others (1997), who evaluated the caries inhibitory effect of three RMGICs, one GIC and a resin composite in dentin. They found no significant difference in OLD between GICs and RMGICs and significant differences between glass ionomers and composites. Attar and Onen (2002), however, found significant differences in enamel and no significant differences in dentin OLD between a conventional glass ionomer and two compomers. As with this study, deeper lesions were found in dentin than enamel. This study's findings also corroborated those of Torii and others (2001), who found no difference in dentin OLD between compomers and non-fluoride releasing resin composites.

In this study, conventional and resin-modified glass ionomers and giomer restorations exhibited significantly more enamel/dentin wall inhibition than compomer and non-fluoride resin composite restorations. This is not surprising since conventional and resin-modified glass ionomers have been shown to inhibit *in vitro* demineralization adjacent to restoration margins (Attar & Onen, 2002; Gilmour & others, 1997; Hicks & Flaitz, 2000; Torii & others, 2001). Tam and others (1997) also observed the presence of narrow zones of non-carious dentin between the margin of the restoration and the body of dentin decay. In an ultrastructural study, Tay and others (2001) demonstrated that glass ionomer phases were readily observed in these materials, while no evidence of a glass ionomer phase were noted in the compomer after 24 hours of aging. Compomers behaved more like resin composite (Meyer, Cattani-Lorente & Dupuis, 1998; Tay & others, 2001), showing more wall lesion patterns, while giomers behaved more like resin-modified glass ionomers (Tay & others, 2001), showing more wall inhibition patterns. Moreover, Xu and others (2000) reported that pre-reacted GIC powder incorporated into ceramic-whisker-containing experimental composites has a cumulative fluoride release of about 20% of the original GIC. Tay and others (2001) proposed that this decrease might be partially attributed to the presence of silane coupling in the pre-reacted fillers versus non-silanized glass particles in the original GIC. This may explain the smaller mean WA inhibition areas in giomers compared to those seen in glass ionomers. Dyract generally lacked the inhibitory beneficial properties of glass ionomers, resulting in a high frequency of wall lesions adjacent to restoration margins (Donly & Grandgenett, 1998; Millar & others, 1998; Torii & others, 2001). The development of wall lesions along the enamel and/or dentin restoration interface of resin composites had been also widely reported (Attar & Onen, 2002; Dionysopoulos & others, 1998; Hicks & Flaitz, 2000; Nagamine & others, 1997).

It is important to note that differences across materials may be partly due to differences in polymerization shrinkage stress, as the materials were placed and cured in one increment.

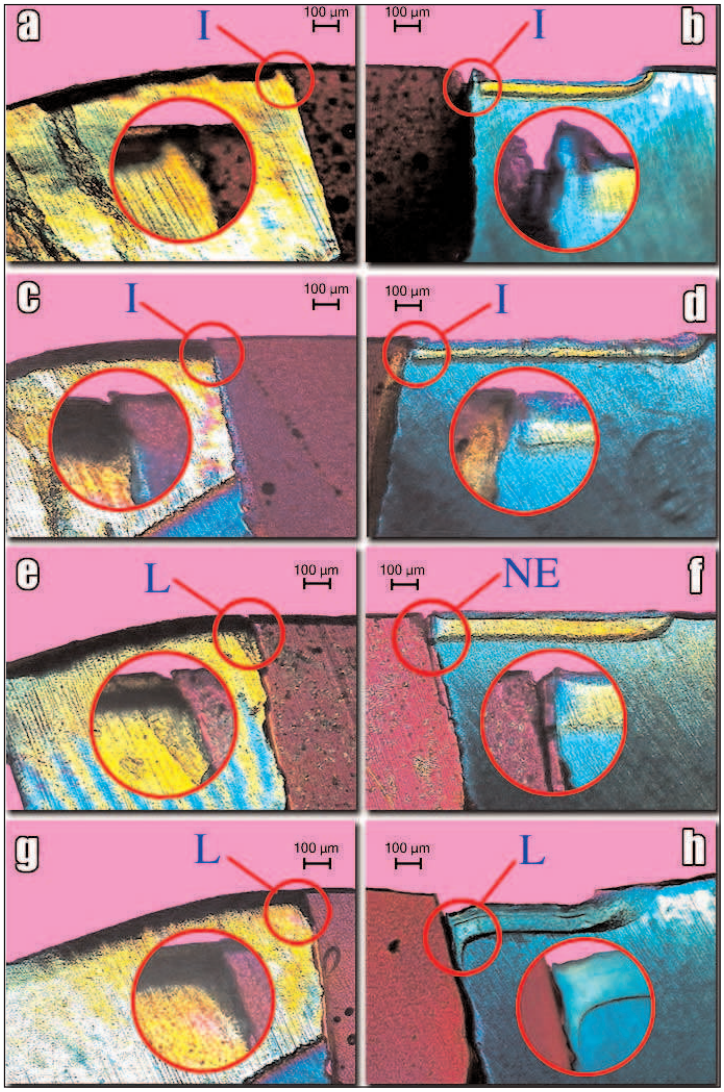


Figure 2. *PLM pictures of materials at 40x of: a) Fuji II and Fuji II LC in enamel, b) Fuji II and Fuji II LC in dentin, c) Reactmer in enamel, d) Reactmer in dentin, e) Dyract in enamel, f) Dyract in dentin, g) Spectrum TPH resin in enamel, h) Spectrum TPH resin in dentin. Restoration margin pictures at 100x are shown in circles. Note the presence of wall inhibition areas (I), no effect lesions (NE) and wall lesions areas (L).*

Table 5: *Comparison of Means (OLD & WA) Between Tissues and Materials*

Variable		OLD - µm	WA Inhibition - µm²
Materials*	FJ, FL, RM, DY	Dentin > Enamel	Dentin > Enamel
	SP	Dentin > Enamel	Enamel > Dentin
Tissues**	Enamel	NS	FJ, FL, RM > DY, SP
	Dentin	SP > FJ, FL, RM	FJ > FL > RM > DY > SP

Results of Paired Samples t-Test and one-way ANOVA/post-hoc Scheffe's tests** ($p<0.05$). > indicates significantly greater OLD lesion depths and significantly greater WA inhibition. NS indicates no statistical significance between materials. FJ= Fuji II; FL= Fuji II LC; RM= Reactmer; DY= Dyract AP; SP=Spectrum TPH.*

Table 6: *Frequency of Wall Lesion/Inhibition Patterns*

Tissue	Material	Frequencies			Total
		Inhibition	No Effect	Lesion	
Enamel	Fuji II	15			15
	Fuji II LC	13	2		15
	Reactmer	12	3		15
	Dyract		5	10	15
	Spectrum TPH			15	15
	Total	40	10	25	75
Dentin	Fuji II	15			15
	Fuji II LC	15			15
	Reactmer	12	3		15
	Dyract	1	9	5	15
	Spectrum TPH			15	15
	Total	43	12	20	75

Results of descriptive statistics—crosstabs.

Table 7: *Comparison of Wall Area Patterns*

Variable	Patterns	
Materials	All	Dentin > Enamel
Tissues	Enamel	SP, DY > FJ, FL, RM
	Dentin	SP > DY > FJ, FL, RM

Results of Kruskall Wallis and Mann Whitney U tests ($p<0.05$). > indicates significantly higher frequency of wall lesions. FJ= Fuji II; FL= Fuji II LC; RM= Reactmer; DY= Dyract AP; SP=Spectrum TPH.

The results of this study showed that the wall area demineralization inhibition of giomers and glass ionomers is better than compomer and composite materials, creating inhibition areas adjacent to restoration margins. Although the demineralization inhibition potential of giomers is similar to GICs in enamel, it is significantly poorer at the dentin margins of restorations. Due to the limitations of this *in vitro* study, it is difficult to extrapolate a definitive conclusion regarding the demineralization inhibition effect of giomers in the clinical situation. As the long-term fluoride release of giomers is questionable (Yap & others, 2002), the effect of aging on caries inhibition by giomers also warrants investigation.

CONCLUSIONS

Under the conditions of this *in vitro* study:

1. Dentin is more susceptible to demineralization than enamel.
2. At the margins of the restorations, the demineralization inhibition effect of all materials was significantly greater in dentin than in enamel with the exception of the composite material.
3. At the enamel margins, demineralization inhibition by glass ionomer cements and giomer were significantly greater than compomer and composite.
4. At the dentin margins, significant differences in demineralization inhibition were observed between materials in the following order: conventional glass ionomer > resin-modified glass ionomer > giomer > compomer > composite.
5. The demineralization inhibition effect of giomers and conventional and resin-modified glass ionomer cements appear to be more evident at the margins of restorations.

(Received 6 June 2003)

References

Arends J, Dijkman G & Dijkman A (1995) Review of fluoride release and secondary caries reduction by fluoridating composites *Advances in Dental Research* **9(4)** 367-376.

Attar N & Onen A (2002) Artificial formed caries-like lesions around esthetic restorative materials *Journal of Clinical Pediatric Dentistry* **26(3)** 289-296.

Brännström M & Nordenvall KJ (1978) Bacterial penetration, pulpal reaction and the inner surface of Concise enamel bond. Composite fillings in etched and unetched cavities *Journal of Dental Research* **57(1)** 3-10.

Damato FA, Strang R & Stephen KW (1988) Comparison of solution- and gel-prepared enamel lesions—and *in vitro* pH-cycling study *Journal of Dental Research* **67(8)** 1122-1125.

Dionysopoulos P, Kotsanos N, Papadogiannis Y & Konstantinidis A (1998) Artificial secondary caries around two new F-containing restoratives *Operative Dentistry* **23(2)** 81-86.

Donly KJ (1994) Enamel and dentin demineralization inhibition of fluoride-releasing materials *American Journal of Dentistry* **7(5)** 275-278.

Donly KJ & Grandgenett C (1998) Dentin demineralization inhibition at restoration margins of Vitremer, Dyract and Compoglass *American Journal of Dentistry* **11(5)** 245-248.

Dunne SM, Goolnik JS, Millar BJ & Seddon RP (1996) Caries inhibition by a resin-modified and a conventional glass ionomer cement, *in vitro Journal of Dentistry* **24(1-2)** 91-94.

El-Mallakh BF & Sarkar NK (1990) Fluoride release from glass-ionomer cements in de-ionized water and artificial saliva *Dental Materials* **6(2)** 118-122.

Erickson RL & Glasspoole EA (1995) Model investigations of caries inhibition by fluoride-releasing dental materials *Advances in Dental Research* **9(3)** 315-323; discussion 324-331.

Featherstone JD (1996) Modeling the caries-inhibitory effects of dental materials *Dental Materials* **12(3)** 194-197.

Gilmour AS, Edmunds DH & Newcombe RG (1997) Prevalence and depth of artificial caries-like lesions adjacent to cavities prepared in roots and restored with a glass ionomer or a dentin-bonded composite material *Journal of Dental Research* **76(12)** 1854-1861.

Heilman JR, Jordan TH, Warwick R & Wefel JS (1997) Remineralization of root surfaces demineralized in solutions of differing fluoride levels *Caries Research* **31(6)** 423-428.

Hicks MJ & Flaitz CM (2000) Resin-modified glass-ionomer restorations and *in vitro* secondary caries formation in coronal enamel *Quintessence International* **31(8)** 570-578.

Hsu CY, Donly KJ, Drake DR & Wefel JS (1998) Effects of aged fluoride-containing restorative materials on recurrent root caries *Journal of Dental Research* **77(2)** 418-425.

Itota T, Nakabo S, Iwai Y, Konishi N, Nagamine M, Torii Y & Yoshiyama M (2001) Effect of adhesives on the inhibition of secondary caries around compomer restorations *Operative Dentistry* **26(5)** 445-450.

Kidd EA, Toffenetti F & Mjör IA (1992) Secondary caries *International Dental Journal* **42(3)** 127-138.

Konishi N, Fried D, Staninec M & Featherstone JD (1999) Artificial caries removal and inhibition of artificial secondary caries by pulsed CO2 laser irradiation *American Journal of Dentistry* **12(5)** 213-216.

Marshall GW Jr, Staninec M, Torii Y & Marshall SJ (1989) Comparison of backscattered scanning electron microscopy and microradiography of secondary caries *Scanning Microscopy* **3(4)** 1043-1049; discussion 1049-1050.

Meyer JM, Cattani-Lorente MA & Dupuis V (1998) Compomers: Between glass-ionomer cements and composites *Biomaterials* **19(6)** 529-539.

Millar BJ, Abiden F & Nicholson JW (1998) *In vitro* caries inhibition by polyacid-modified composite resins ("compomers") *Journal of Dentistry* **26(2)** 133-136.

Mjör IA (1985) Frequency of secondary caries at various anatomical locations *Operative Dentistry* **10(3)** 88-92.

Mjör IA & Toffenetti F (2000) Secondary caries: A literature review with case reports *Quintessence International* **31(3)** 165-179.

Nagamine M, Itota T, Torii Y, Irie M, Staninec M & Inoue K (1997) Effect of resin-modified glass ionomer cements on secondary caries *American Journal of Dentistry* **10(4)** 173-178.

Phankosol P, Ettinger RL, Hicks MJ & Wefel JS (1985) Depth of penetration of *in vitro* root surface lesions *Journal of Dental Research* **64(6)** 897-899.

Retief DH, Bradley EL, Denton JC & Switzer P (1984) Enamel and cementum fluoride uptake from a glass ionomer cement *Caries Research* **18(3)** 250-257.

Sidhu SK & Watson TF (1995) Resin-modified glass ionomer materials. A status report for the *American Journal of Dentistry* **8(1)** 59-67.

Sjodin L, Uusitalo M & Van Dijken J (1996) Resin-modified glass ionomer cements. *In vitro* microleakage in direct Class V and Class II sandwich restorations *Swedish Dental Journal* **20(3)** 77-86.

Tam LE, Chan GP & Yim D (1997) *In vitro* caries inhibition effects by conventional and resin-modified glass-ionomer restorations *Operative Dentistry* **22(1)** 4-14.

Tay FR, Pashley EL, Huang C, Hashimoto M, Sano H, Smales RJ & Pashley DH (2001) The glass-ionomer phase in resin-based restorative materials *Journal of Dental Research* **80(9)** 1808-1812.

Torii Y, Itota T, Okamoto M, Nakabo S, Nagamine M & Inoue K (2001) Inhibition of artificial secondary caries in root by fluoride-releasing restorative materials *Operative Dentistry* **26(1)** 36-43.

Tveit AB & Hals E (1980) Inhibitory effect of a fluoride-containing amalgam on development of cavity wall lesions *in vitro* *Acta Odontologica Scandinavica* **38(1)** 29-39.

Weatherell JA, Robinson C, Schaper R & Kunzel W (1983) Distribution of fluoride in clinically sound enamel surfaces of permanent upper incisors *Caries Research* **17(2)** 118-124.

Wefel JS, Heilman JR & Jordan TH (1995) Comparisons of *in vitro* root caries models *Caries Research* **29(3)** 204-209.

Wilson AD & Kent BE (1972) A new translucent cement for dentistry. The glass ionomer cement *British Dental Journal* **132(4)** 133-135.

Xu HH, Eichmiller FC, Antonucci JM, Schumacher GE & Ives LK (2000) Dental resin composites containing ceramic whiskers and precured glass ionomer particles *Dental Materials* **16(5)** 356-363.

Yap AU, Lim CC & Neo JC (1995) Marginal sealing ability of three cervical restorative systems *Quintessence International* **26(11)** 817-820.

Yap AU, Tham SY, Zhu LY & Lee HK (2002) Short-term fluoride release from various aesthetic restorative materials *Operative Dentistry* **27(3)** 259-265.

©Operative Dentistry, 2004, **29-5,** 586-591

Effects of pH on the Surface Texture of Glass Ionomer Based/Containing Restorative Materials

MA Mohamed-Tahir • AUJ Yap

Clinical Relevance

The surface texture of all glass-ionomer based/containing restorative materials was significantly affected by acids of low pH. The significant increase in surface roughness of highly viscous glass ionomer cements after exposure to acids may render them more susceptible to bacterial adhesion and possible clinical failure.

SUMMARY

This study determined the effect of pH on the surface texture of commonly used posterior glass-ionomer based/containing restorative materials. The materials evaluated included a compomer (Dyract AP), a giomer (Beautifil) and two highly viscous glass ionomer cements (Fuji IX and Ketac Molar). A resin composite (Esthet-X) was used for comparison. Forty-two specimens (3-mm wide x 3-mm long x 2-mm deep) were made for each material. The specimens were divided into six groups and conditioned in the following solutions at 37°C for one week: Citric acid (pH 2, 3, 4, 5 and 6) and distilled water (pH 7). After conditioning, the surface roughness (Ra, µm) of each specimen was measured using a surface profilometer (Surftest, Mitutoyo Corp, Tokyo, Japan). Data was analyzed using one-way ANOVA and Scheffe's test at a significance level of 0.05. The effects of pH on the surface texture of glass-ionomer based/containing restoratives were material dependent. Ra values ranged from 0.02 µm to 0.15 µm and 0.03 µm to 4.40 µm for pH 7 and 2, respectively. With the exception of the composite, the surface roughness of all materials evaluated was significantly affected by acids of low pH. The surface texture of highly viscous glass ionomer cements deteriorated significantly when conditioned in solutions of low pH, which makes them more susceptible to clinical failure.

*MA Mohamed-Tahir, BDS, MSc, assistant professor, Department of Preventive Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

AUJ Yap, BDS, MSc, PhD, FAMS, FADM, FRSH, associate professor, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

*Reprint request: 5 Lower Kent Ridge Road, Singapore 119074, Republic of Singapore; e-mail: rashid@nus.edu.sg

INTRODUCTION

The prevalence of tooth surface loss has increased significantly. It was reported that 41% of children in the United States and 37% of children in the United Kingdom ranging in age from 11 to 13 years had dental erosion (Deery & others, 2000). In randomly selected adult subjects between 26 and 30 years of age, 7.7% had facial erosive lesions into dentin and 29.9% had occlusal tooth wear into dentin. For subjects between 46 and 50

years of age, 13.2% showed facial erosive lesions into dentin and 42.6% had occlusal erosion into dentin (Lussi & others, 1991). Dental erosion is defined as the irreversible loss of dental hard tissue by a chemical process without the involvement of micro-organisms (Eccles, 1979) and is due to either extrinsic or intrinsic acid sources. Intrinsic acids are produced by recurrent vomiting in patients with anorexia, bulimia or gastroesophageal reflux (Meurman & ten Cate, 1996) and extrinsic acids are derived from the environment, medications, lifestyle and diet (Zero, 1996). The over-consumption of dietary acids in the form of soft drinks has been linked to tooth surface loss (Milosevic, Lennon & Fear, 1997). It was reported that in the year 2002, Americans consumed approximately 53 gallons of soft drinks per person per year (National Soft Drinks Association, 2002) and there is an increasing trend towards increased consumption of fruit drinks in children (Park & others, 2002).

Exposure to either extrinsic or intrinsic acids may adversely affect dental restorations, as the low pH level may cause erosion of the glass-ionomer based/containing materials and leaching of the principle matrix forming substances (Yip, Peng & Smales, 2001; Abu-bakr & others, 2000; Taggart & Pearson, 1991; Smith & Martin, 1990; Smith 1988). Studies that looked at the effects of low-pH drinks on the properties of restorative materials showed that composite, resin-modified (RM-GIC) and conventional glass ionomer cements (GIC) were more resistant to varying pH levels than dental enamel (Shabanian & Richards, 2002). However, acid resistance of conventional GIC was consistently less than RM-GIC and composite. The surface roughness of direct tooth-colored restorative materials is significantly affected by conditioning in different food simulating liquids, including acids (Yap, Low & Ong, 2000). Abu-bakr and others (2000), who compared the effects of various immersion media on the mechanical properties and surface texture of composite, compomer and RM-GIC, found that all the materials had increased solubility and SEM changes in surface texture when immersed in low pH-level soft drinks (orange juice and cola) as compared to deionized water and whiskey. The SEM revealed that the surface of the composite was slightly affected, whereas, the surfaces of the compomer and RM-GIC showed surface loss with marked crack formation. These findings suggest that some tooth-colored restorative materials, especially glass-ionomer based/containing ones, might not be suitable for use in patients with gastric regurgitation or those who consume a high amount of low-pH drinks.

The surface texture of tooth-colored restorative materials affects the lifespan of the restorations. The presence of irregularities on the surface of materials may influence appearance, plaque retention, surface discoloration and gingival irritation (Shintani & others, 1985; Dunkin & Chambers, 1983; Chan, Fuller & Hormati, 1980; Weitman & Eames, 1975; Larato, 1972). The increasing use of glass-ionomer based/containing restorative materials and the recent trend toward increased consumption of acidic drinks necessitates studying the effects of pH level on these restorative materials. This study determined the effect of pH on the surface texture of glass-ionomer based/containing restorative materials used for the restorations of posterior teeth.

METHODS AND MATERIALS

Table 1 lists the restorative materials evaluated in this study. They include a compomer (Dyract AP), a giomer (Beautifil) and two highly viscous glass ionomer cements (Fuji IX and KetacMolar). A resin composite (Esthet-X) was used for comparison. The materials were placed in the rectangular recesses (3-mm long x 3-mm wide x 2-mm deep) of customized acrylic molds and were covered with acetate strips (Hawe Neos Dental, Bioggio, Switzerland). A glass slide was placed over the acetate strip and pressure was applied to extrude the excess material. The light-cured restorative materials were then light-polymerized according to the manufacturers' cure-times through the glass slide using an Elipar Trilight curing light (3M-ESPE, St Paul, MN, USA). The intensity of the light source was checked with a built-in radiometer before starting the experiment and a constant output of 850 mW/cm^2 was ensured. The two highly viscous glass ionomer cements in capsulated form were activated/mixed according to the manufacturers' directions. Immediately after setting, the acetate strips were discarded and the speci-

Table 1: *Restorative Materials Evaluated in this Study*

Material	Category	Manufacturer	Batch #	Shade/ Cure Time
Esthet-X	Composite	Dentsply–De Trey, Konstanz, Germany	0210101	A2 20 seconds
Dyract AP	Compomer	Dentsply–De Trey, Konstanz, Germany	0207001423	A2 40 seconds
Fuji IX (Capsulated)	Glass ionomer cement	GC Corporation, Tokyo, Japan	0207152	A2 (Not applicable)
KetacMolar	Glass ionomer cement	3M–ESPE St Paul, MN, USA	130929	A2 (Not applicable)
Beautifil	Giomer	Shofu Inc, Kyoto, Japan	110262	A2 20 seconds

Table 2: *Mean Surface Roughness, Ra Values (µm) and Standard Deviations at Different pH*

	Esthet X	Dyract AP	Fuji IX	Ketac Molar	Beautifil
pH 2	0.03 (0.01)	0.04 (0.01)	4.40 (0.21)	1.82 (1.71)	0.13 (0.02)
pH 3	0.03 (0.01)	0.06 (0.01)	0.76 (0.16)	0.19 (0.04)	0.13 (0.05)
pH 4	0.04 (0.01)	0.05 (0.01)	0.15 (0.05)	0.25 (0.09)	0.05 (0.00)
pH 5	0.03 (0.01)	0.04 (0.01)	0.14 (0.03)	0.26 (0.13)	0.04 (0.01)
pH 6	0.04 (0.01)	0.03 (0.01)	0.14 (0.03)	0.19 (0.07)	0.03 (0.01)
pH 7	0.02 (0.01)	0.04 (0.01)	0.15 (0.03)	0.08 (0.02)	0.05 (0.01)

Standard deviations in parenthesis.

Table 3: *Results of Statistical Analysis*

	Comparison Between pH
Materials	**Differences**
Esthet X	No statistically significant difference
Dyract AP	pH3 > pH5, pH6 pH4 > pH6
Fuji IX	pH2 > pH3 > pH4, pH5, pH6, pH7
Ketac Molar	pH2 > pH3, pH4, pH5, pH6, pH7
Beautifil	pH2, pH3 > pH4, pH5, pH6, pH7

> indicates statistically significant difference. Results of one-way ANOVA/Scheffe's test (p<0.05).

Table 4: *Results of Statistical Analysis*

	Comparison Between Materials
PH	**Differences**
pH 2	Fuji IX > Ketac Molar > Beautifil, Dyract AP, Esthet X
pH 3	Fuji IX > Ketac Molar > Dyract AP, Esthet X Fuji IX > Beautifil
pH 4	Ketac Molar > Fuji IX > Beautifil, Dyract AP, Esthet X
pH 5	Ketac Molar > Fuji IX > Esthet X Ketac Molar > Beautifil, Dyract AP
pH 6	Ketac Molar, Fuji IX > Esthet X, Beautifil, Dyract AP
pH 7	Fuji IX > Ketac Molar > Dyract AP, Esthet X Fuji IX > Beautifil

> indicates statistically significant difference. Results of one-way ANOVA/Scheffe's test (p<0.05).

mens stored in deionized distilled water for two weeks at 37°C.

Forty-two specimens were made for each restorative material. They were randomly divided into six groups of seven specimens and conditioned in citric acid solutions of differing pH (pH 2, 3, 4, 5 and 6) levels at 37°C for one week. The pH of the citric acid solutions was adjusted by adding distilled water that was also used as pH 7. A sample size of 7 was adopted based on previous studies (Yap & others, 2000; Yap & Mok, 2002). The pH of the solutions was measured using a pH meter (MP 220; Mettler-Toledo GmbH, CH-8603 Schwerzenbach, Germany). After the one-week conditioning period, the samples were lightly rinsed with water and gently dabbed dry with absorbent paper. Surface roughness was then determined using a profilometer (Surftest; Mitutoyo Corp, Tokyo, Japan) with a probe diameter of 5 µm. Ra values for each specimen were taken across the center of each specimen over a standard length of 0.25 mm X 4. Ra value is the arithmetic mean of the departures of the roughness profile from the mean line calculated by the computer. All statistical analysis was carried out at significance level 0.05. Two-way ANOVA was used to determine material-pH interactions. One-way ANOVA and Scheffe's post-hoc tests were used to compare the surface roughness of the materials after conditioning in acids of different pH.

RESULTS

Table 2 and Figure 1 show the mean Ra values for the different materials at different pH levels. The results of statistical analysis are reflected in Tables 3 and 4.

The effects of pH on surface roughness were material dependent. Ra values ranged from 0.02 to 0.15 µm and 0.03 to 4.40 µm for pH 7 and 2, respectively. With the exception of Esthet-X, significant differences in Ra values were observed between pH. For Dyract AP, specimens conditioned at pH 3 were significantly rougher than those conditioned in pH 5 and 6. In addition, specimens conditioned at pH 4 were significantly rougher than those stored at pH 6. For Fuji IX and Beautifil, conditioning at pH 2 and 3 resulted in significantly rougher surfaces than those conditioned in pH 4 to 7. For Ketac Molar, significantly greater Ra values were observed between specimens conditioned in pH 2 compared to pH 3 to 7.

The materials exhibited different degrees of surface degradation at different pH levels. At pH 7 and 3, Fuji IX and Ketac Molar were significantly rougher than Dyract AP and Esthet-X but only Fuji IX was significantly rougher than Beautifil. At pH 6 and 2, Fuji IX and Ketac Molar were significantly rougher than all the other materials. However, Ketac Molar was the roughest material at pH 5, with significantly higher Ra values than Beautifil and Dyract AP. Ketac Molar and Fuji IX were also significantly rougher than Esthet-X. At pH 4, Ketac Molar and Fuji IX were significantly rougher than Beautifil, Dyract AP and Esthet-X.

DISCUSSION

Citric acid was chosen as the erosive medium, as it is the most common acid found in fruit juices and drinks and is often added to foodstuff. In addition, it is commonly used in *in vitro* studies (Barbour & others, 2003; Eisenburger, Addy & Roßbach, 2003; Vanuspong,

Eisenburger & Addy, 2002). The different pH levels investigated reflect the levels of dietary acid, between pH 2 to 7, that the restorative materials would be subjected to intra-orally, for example, white bread pH 5–6.2, apples pH 3.3–4 and lemon juice pH 2–2.6 (US Food and Drug Administration, 2000). The conditioning was delayed for two weeks to allow for post-irradiation hardening of the composites and maturation of the glass ionomer cements (Wan, Yap & Hastings, 1999; Yap, 1997; Leung, Adishian & Fan, 1985). Compomers or polyacid-modified composites (Dyract AP) are materials that contain either or both of the essential components of glass ionomer cement but at levels insufficient to promote the acid-base cure reaction in the dark (McLean, Nicholson & Wilson, 1994). Giomers (Beautifil) are a new class of glass ionomer containing restorative material. They contain the essential components of glass ionomer cements but cannot be classified as compomers, as the acid-base reaction has already occurred. Therefore, a new term, pre-reacted glass ionomer (PRG) composite, is used to describe the material. The glass-ionomer based restorative materials evaluated (Fuji IX and Ketac Molar) are high powder: liquid ratio highly viscous cements.

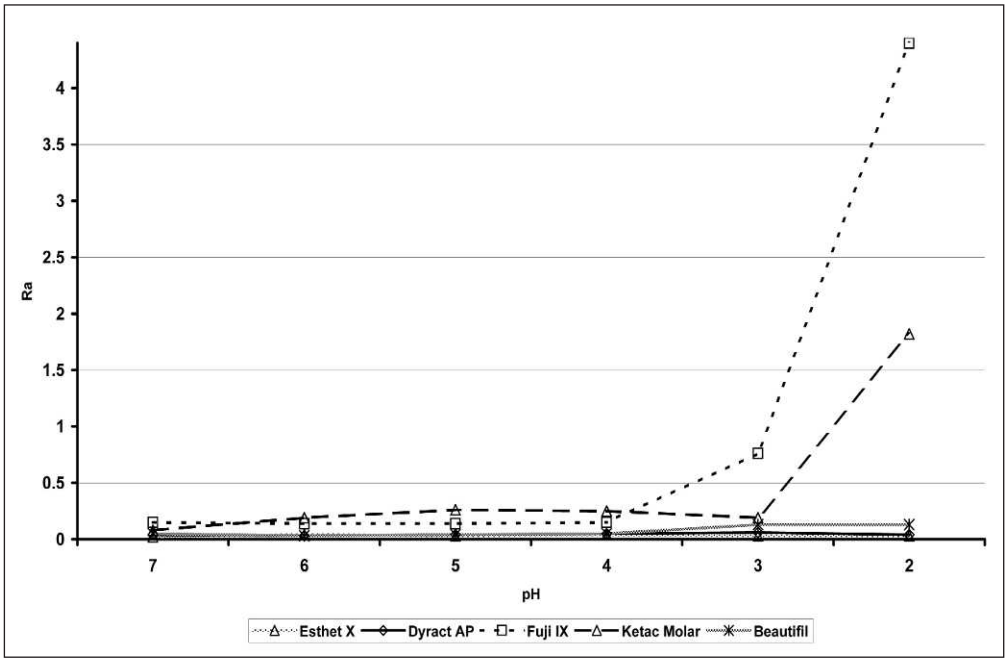


Figure 1. *Mean surface roughness at different pH.*

The effect of pH on surface roughness was material dependent and only the composite material (Esthet-X) was not significantly affected by varying pH levels. The compomer (Dyract AP) demonstrated significantly lower surface roughness than other glass-ionomer based restorative materials (Fuji IX and Ketac Molar) after conditioning in the acids but the roughness was not statistically significantly different from that of Esthet-X at all pH levels. In fact, the surface roughness of Dyract AP has been reported to more closely approximate resin composites (Gladys & others, 1997). The giomer (Beautifil) exhibited surface degradation at levels between the compomer and the highly viscous GIC when conditioned at varying pH levels. It was found that the surface roughness of Beautifil was not significantly different from Esthet-X or Dyract AP at all pH levels but showed roughness close to that of Ketac Molar at pH 3 and 7 and Fuji IX at pH 5. For all materials tested, the smoothest surface texture was found when they were conditioned in deionized distilled water with a neutral pH 7, even though the mean Ra values differed between the materials, with Fuji IX and Ketac Molar being significantly rougher than the rest. All the materials tested can be considered biphasic, with one phase embedded in the other. Glass ionomers consist of glass particles in a hydrogel matrix and composites consist of glass filler particles in a polymer resin (Yap & Mok, 2002). In acidic solutions, H^+ ions of citric acid diffused into the glass ionomer components and replaced metal cations in the matrix. These free cations would diffuse outward and be released from the surface. As the metal cations in the matrix decreased, more would be extracted from the surrounding glass particles, causing them to dissolve (Fukazawa, Matsuya & Yamane, 1990). Consequently, the material would present a rough surface with voids and protruded, undissolved glass particles. Prolonged exposure of these glass-ionomer based/containing materials to acids would result in higher Ra values recorded by the profilometer.

It is believed that a threshold surface roughness for bacterial retention (Ra = 0.2 μm) exists below which no further reduction in bacterial accumulation can be expected. An increase in surface roughness above this threshold roughness, however, will result in a simultaneous increase in plaque accumulation, thereby, increasing the risk for both caries and periodontal inflammation (Bollen, Lambrechts & Quirynen, 1997). The materials with Ra values above the 0.2 μm threshold are Fuji IX (pH 2 and 3) and Ketac Molar (pH 2, 4 and 5) when conditioned in acids. This deterioration in the surface texture is more likely to cause increased bacterial adhesion, besides causing a clinically rough and dull surface. Therefore, careful selection of posterior

restorative material is important in patients with a history of gastric reflux, eating disorders or high consumption of dietary acids. From the results of this study, it is hypothesized that there is a critical pH unique to individual materials at which the surface texture was significantly affected. This requires further investigation.

CONCLUSIONS

Under the conditions of this *in vitro* study:

1. The effects of pH on the surface texture of glass-ionomer based/containing restoratives are material dependent.
2. With the exception of composites, the surface roughness of all materials evaluated was significantly affected by acids of low pH.
3. The surface texture of highly viscous glass ionomer cements deteriorated significantly when conditioned in solutions of low pH, which makes them susceptible to bacterial adhesion.

(Received 6 June 2003)

References

Abu-bakr N, Han L, Okamoto A & Iwaku M (2000) Changes in the mechanical properties and surface texture of compomer immersed in various media *Journal of Prosthetic Dentistry* **84(4)** 444-452.

Barbour ME, Parker DM, Allen GC & Jandt KD (2003) Human enamel dissolution in citric acid as a function of pH in the range 2.30<pH<6.30–a nanoindentation study *European Journal of Oral Science* **111(3)** 258-262.

Bollen CM, Lambrechts P & Quirynen M (1997) Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: A review of the literature *Dental Materials* **13(4)** 258-269.

Chan KC, Fuller JL & Hormati AA (1980) The ability of foods to stain two composite resins *Journal of Prosthetic Dentistry* **43(5)** 542-545.

Deery C, Wagner ML, Longbottom C, Simon R & Nugent ZJ (2000) The prevalence of dental erosion in a United States and a United Kingdom sample of adolescents *Pediatric Dentistry* **22(6)** 505-510.

Dunkin RT & Chambers DW (1983) Gingival response to Class V composite resin restorations *Journal of the American Dental Association* **106(4)** 482-484.

Eccles JD (1979) Dental erosion of non-industrial origin: A clinical survey and classification *Journal of Prosthetic Dentistry* **42(6)** 649-653.

Eisenburger M, Addy M & Roßbach A (2003) Acidic solubility of luting cements *Journal of Dentistry* **31(2)** 137-142.

Fukazawa M, Matsuya S & Yamane M (1990) The mechanism for erosion of glass-ionomer cements in organic-acid buffer solutions *Journal of Dental Research* **69(5)** 1175-1179.

Gladys S, Van Meerbeek B, Braem M, Lambrechts P & Vanherle G (1997) Comparative physico-mechanical characterization of new hybrid restorative materials with conventional glass-ionomer and resin composite restorative materials *Journal of Dental Research* **76(4)** 883-894.

Larato DC (1972) Influence of a composite resin restoration on the gingival *Journal of Prosthetic Dentistry* **28(4)** 402-404.

Leung RL, Adishian SR & Fan PL (1985) Post-irradiation comparison of photo activated composite resins *Journal of Prosthetic Dentistry* **54(5)** 645-649.

Lussi A, Schaffner M, Hotz P & Suter P (1991) Dental erosion in a population of Swiss adults *Community Dentistry and Oral Epidemiology* **19(5)** 286-290.

McLean JW, Nicholson JW & Wilson AD (1994) Proposed nomenclature for glass ionomer dental cements and related materials *Quintessence International* **25(9)** 587-589.

Meurman JH & ten Cate JM (1996) Pathogenesis and modifying factors of dental erosion *European Journal of Oral Science* **104(2)** 199-206.

Milosevic A, Lennon MA & Fear SC (1997) Risk factors associated with tooth wear in teenagers: A case control study *Community Dental Health* **14(3)** 143-147.

National Soft Drink Association (2002) Soft drinks facts http://www.nsda.org/softdrinks/History/funfacts.html accessed 10 May 2003.

Park YK, Meier ER, Bianchi P & Song WO (2002) Trends in children's consumption of beverages: 1987 to 1998 *Family Economics and Nutrition Review* **14(2)** 69-79.

Shabanian M & Richards LC (2002) *In vitro* wear rates of materials under different loads and varying pH *Journal of Prosthetic Dentistry* **87(6)** 650-656.

Shintani H, Satou J, Satou N, Hayashihara H & Inoue T (1985) Effects of various finishing methods on staining and accumulation of Streptococcus mutans HS-6 on composite resins *Dental Materials* **1(6)** 225-227.

Smith ED & Martin FE (1990) Acid etching of a glass ionomer cement base: SEM study *Australian Dental Journal* **35(3)** 236-240.

Smith GE (1988) Surface deterioration of glass ionomer cement during acid etching: An SEM evaluation *Operative Dentistry* **13(1)** 3-7.

Taggart SE & Pearson GJ (1991) The effect of etching on glass polyalkenoate cements *Journal of Oral Rehabilitation* **18(1)** 31-42.

US Food and Drug Administration (2000) Center for Food Safety and Applied Nutrition. Approximate pH of Foods and Food Products http://www.cfsan.fda.gov/~comm/lacf-phs.html accessed 9 May 2003.

Vanuspong W, Eisenburger M & Addy M (2002) Cervical tooth wear and sensitivity: Erosion, softening and rehardening of dentine; effects of pH, time and ultrasonication *Journal of Clinical Periodontology* **29(4)** 351-357.

Wan AC, Yap AU & Hastings GW (1999) Acid-base complex reactions in resin-modified and conventional glass ionomer cements *Journal of Biomedical Materials Research (Applied Biomaterials)* **48(5)** 700-704.

Weitman RT & Eames WB (1975) Plaque accumulation on composite surface after various finishing procedures *Journal of the American Dental Association* **91(1)** 101-106.

Yap AU & Mok BY (2002) Surface finish of a new hybrid aesthetic restorative material *Operative Dentistry* **27(2)** 161-166.

Yap AU (1997) Post-irradiation hardness of resin-modified glass ionomer cements and a polyacid-modified composite *Journal of Materials Science: Materials in Medicine* **8** 413-416.

Yap AU, Low JS & Ong LF (2000) Effect of food-simulating liquids on surface characteristics of composite and polyacid-modified composite restoratives *Operative Dentistry* **25(3)** 170-176.

Yip KH, Peng D & Smales RJ (2001) Effects of APF gel on the physical structure of compomers and glass ionomer cements *Operative Dentistry* **26(3)** 231-238.

Zero DT (1996) Etiology of dental erosion—extrinsic factors *European Journal of Oral Science* **104(2(2)** 162-177.

©Operative Dentistry, 2004, **29-5**, 592-598

Stability of Dentin Bond Strengths Using Different Bonding Techniques After 12 Months: Total-etch, Deproteinization and Self-etching

LAF Pimenta • CM Amaral
AKB Bedran de Castro • AV Ritter

Clinical Relevance

The bond strength of composite restorations to dentin remains stable after 12 months. When compared to a self-etching and a collagen removal technique, total-etch bonding provided the best results.

SUMMARY

This study evaluated the influence of (1) different dentin treatments and (2) storage time on dentin shear bond strengths. Two hundred and twenty-five bovine incisors were collected, ground to expose a flat dentin surface and randomly divided into three groups according to dentin surface treatment: (1) Total etch + Single Bond; (2) Total etch + collagen depletion with 10% NaOCl + Single Bond and (3) No etch + experimental self-etching adhesive. Composite was applied to the treated surfaces using a 3 x 5 mm cylindrical Teflon matrix and was light-cured. Each group was further stratified in five subgroups according to storage time in water at 37°C: 1 day, 7 days, 30 days, 6 months and 12 months. Shear bond strengths (SBS) were determined and expressed in MPa. Data was analyzed for statistical significance with two-way ANOVA and Tukey's test (p=0.05). No interaction was observed between surface treatment and storage time. Storage time did not significantly affect bond strengths. Statistically significant differences were observed among the different surface treatments. Single Bond applied after total-etch presented higher mean SBS when compared to the other surface treatment methods. Collagen removal negatively influenced SBS, and the experimental self-etching adhesive presented intermediate values.

*Luiz André Freire Pimenta, DDS, MS, PhD, associate professor, Department of Restorative Dentistry, School of Dentistry of Piracicaba, University of Campinas (UNICAMP), Piracicaba, Brazil

Cristiane Mariote Amaral, DDS, MS, graduate student, Department of Restorative Dentistry, School of Dentistry of Piracicaba, University of Campinas (UNICAMP), Piracicaba, Brazil

Ana Karina Barbieri Bedran de Castro, DDS, MS, graduate student, Department of Restorative Dentistry, School of Dentistry of Piracicaba, University of Campinas (UNICAMP), Piracicaba, Brazil

André Vicente Ritter, DDS, MS, assistant professor, Department of Operative Dentistry, University of North Carolina at Chapel Hill, Chapel Hill, NC, USA

*Reprint request: Av Limeira, 901 – Areião, Piracicaba - SP, Brazil; e-mail: lpimenta@fop.unicamp.br

INTRODUCTION

Bonding to enamel via acid etching is considered a durable, predictable clinical procedure. The acid-etch technique (Buonocore, 1955) relies on micromechanical

retention created by enamel acid etching and the subsequent penetration of a blend of polymerizable monomers into the interprismatic spaces to form resin tags (Gwinnett & Matsui, 1967). In contrast, bonding to dentin is more complicated due to its wet tubular structure and organic content. Several current dentin adhesives rely on the permeation of hydrophilic monomers into acid-etched dentin (Van Meerbeek & others, 1992). High-vapor pressure organic solvents, such as acetone or ethanol, are used to carry these monomers into intimate contact with the dense filigree of collagen fibers, resulting in a nano-stream of resin within the spaces formed by the intimacy of neighbor fibrils (Nakabayashi, Ashizawa & Nakamura, 1992). The entanglement of resin with collagen and residual hydroxyapatite crystals has been named the "hybrid layer" or "resin-dentin interdiffusion zone" (Nakabayashi, Kojima & Masuhara, 1982; Van Meerbeek & others, 1992). Such structure is therefore an area of resin-modified demineralized dentin that results in bond strengths comparable to enamel, particularly when acid-etched dentin is left visibly moist (Kanca, 1992a; 1992b; 1996).

The main limitation imposed by the total-etch, wet-bonding technique is derived from the observation that the degree of wetness of the substrate might influence the dentin bond strengths (Kanca, 1992a; Tay & others, 1996). If the substrate is dried after acid etching and rinsing, the demineralized collagen might collapse and form a relatively impermeable film capable of preventing the adhesive resin from infiltrating into the substrate. On the other hand, if the substrate is overwet, the excess water can also interfere with the action of the adhesive resin (Tay, Gwinnett & Wei, 1997; Perdigão & others, 1998). Re-wetting techniques have been reported and seem to effectively solve this problem, although they call for an additional procedural step in the clinical sequence (Gwinnett, 1994). The potential for over-rewetting still remains but has not been studied in detail.

Another limitation of the total-etch technique is its potential for incomplete penetration of demineralized dentin by adhesive resin (Nakabayashi & others, 1992). Reports have shown that leakage can occur below the hybridized layer (Sano & others, 1995; Okuda & others, 2001). Additionally, even though this phenomenon has been scarcely studied, the acid-exposed, non-infiltrated collagen fibers can undergo hydrolytic degradation when exposed to fluids for long periods (Nakabayashi, Ashizawa & Nakamura, 1992; Hashimoto & others, 2002b).

Due to the limitations imposed by the total-etch technique, alternative dentin bonding strategies have been proposed (Wakabayashi & others, 1994; Chigira & others, 1994). One strategy involves complete deproteinization of the etched dentin, which would eliminate the problem of handling the delicate collagen network exposed by the acid, and would also allow for intimate contact between the adhesive resin and dentin substrate (Wakabayashi & others, 1994; Toledano & others, 1999; Saboia, Rodrigues & Pimenta, 2000). A "reverse hybrid-layer" has been shown to form when this technique is applied in sound dentin, and bond strength assays using this technique have shown system dependent high bond strength values (Gwinnett & others, 1996; Vargas, Cobb & Armstrong, 1997; Saboia & others, 2000).

Another strategy to eliminating the acid-etching step is using self-etching acidic primers and adhesives (Chigira & others, 1994; Itou & others, 2001; Koibuchi, Yasuda & Nakabayashi, 2001). Self-etching primers are two-step products, where an acidic primer is first applied followed by application of the adhesive. Self-etching primers-adhesives, on the other hand, are one-step products where etching, priming and bonding are accomplished simultaneously. Controversial results have been observed when these products are used, and low bond-strengths to uncut enamel have been reported (Hara & others, 1999; Toledano & others, 2001; Ibarra & others, 2002).

Although the deproteinization technique is promising, the self-etching technique offers the additional benefit of reducing the number of clinical steps necessary to complete the bonding sequence. Until an efficient self-adhering direct restorative material is developed, a one-step self-etching adhesive with low sensitivity to substrate variability would be welcome.

This *in vitro* study evaluated the stability of dentin bond strengths over time when a resin-based restorative composite was applied to dentin using different bonding techniques: total-etch technique, deproteinization technique and self-etching adhesive technique. The hypotheses tested in this study were 1) the deproteinization technique provides bond strengths equivalent to or better than bond strengths generated by the total-etch technique; 2) the experimental self-etching adhesive tested provides bond strengths equivalent to or better than bond strengths generated by the total-etch technique and the deproteinization technique and 3) storage time does not affect bond strengths independent of the technique.

METHODS AND MATERIALS

Two hundred and twenty-five recently extracted bovine incisors were used in this study. In each specimen, the root and part of the crown were cut-off with a double-faced diamond disk (KG Sorensen, Barueri, SP 06465-130, Brazil). A 5 x 5-mm crown fragment of the buccal surface was included in a 1/2 inch PVC ring with self-curing polystyrene resin (Cromex, Piracicaba, São

Paulo, Brazil). The specimens were ground on a water-cooled mechanical grinder (Maxigrind, Solotest, São Paulo, Brazil) using 320, 400 and 600-grit Al_2O_3 abrasive paper (Saint-Gobain Abrasivos Ltda, Guarulhos, Brazil) to obtain flat standardized dentin surfaces.

After polishing, the specimens were randomly assigned to 15 groups (n=15) for bonding with the three different surface treatments and five different storage times tested (Table 1). Before surface treatment, the bonding area of each specimen was demarcated with a piece of vinyl tape in which a 3-mm diameter hole had been punched over the dentin surface. The surface treatments tested where (1) a total-etch, wet bonding technique using an ethanol- and water-based adhesive (Single Bond, 3M ESPE, St Paul, MN, USA), (2) a total-etch, collagen removal technique using Single Bond (3M ESPE) or (3) a self-etch technique using a two component experimental self-etching adhesive (EXL 547, 3M ESPE). Adhesives were applied according to manufacturers' instructions, as follows.

Table 1: *Summary of the Experimental Procedures*

Bonding Technique	Bonding Agent	Storage Time	Mean (MPa)	N
Total-etch	Single Bond	1 day	15.4 (4.4)	15
		7 days	17.4 (4.4)	15
		30 days	15.3 (3.9)	15
		6 months	16.5 (5.2)	15
		12 months	17.2 (4.1)	15
Total-etch + Deproteinization	Single Bond	1 day	10.9 (3.4)	15
		7 days	8.9 (1.9)	15
		30 days	10.4 (3.6)	15
		6 months	10.8 (3.3)	15
		12 months	12.9 (5.6)	15
Self-etching adhesive	Experimental EXL 547	1 day	16.4 (3.7)	15
		7 days	14.0 (5.0)	15
		30 days	13.1 (5.8)	15
		6 months	13.6 (3.3)	15
		12 months	13.3 (4.0)	15

Shear bond strength values (Mean, ± standard deviation) in MPa for each bonding technique and storage times.

Table 2: *Analysis of Variance (two-way ANOVA) Summary of All Effects*

	df Effect	MS Effect	MS Error	F	p-level
Treatments	2	539.7004	18.33841	29.43005	0.000001*
Storage Time	4	16.3721	18.33841	0.89278	0.469293
Treat X St Time	8	24.9615	18.33841	1.36116	0.215849

** Statistical significant difference (p<0.05).*

Total-etch + Single Bond (SB) (3M ESPE): 37% phosphoric acid gel (3M ESPE) was applied for 15 seconds, then rinsed for 15 seconds. The etched area was gently dried with absorbing paper and two consecutive layers of adhesive were applied, air dried for three seconds and light-cured (Optilux 500, Demetrom/Kerr, Danbury, CT, USA) for 10 seconds.

Total-etch + Deproteinization + Single Bond (SBHC): 37% phosphoric acid gel (3M ESPE) was applied for 15 seconds, then rinsed for 15 seconds. The etched area was gently dried with absorbing paper and 10% NaOCl was applied for 60 seconds and washed for 30 seconds. The etched, deproteinized area was gently dried with absorbing paper and two consecutive layers of adhesive were applied, air dried for three seconds and light-cured (Optilux 500) for 10 seconds.

EXL 547 Experimental Self-etching Adhesive (SE) (3M ESPE): one drop of liquid A and one drop of liquid B were mixed thoroughly on a mixing block with a brush tip for 10 seconds. A generous quantity of self-etching adhesive was applied and rubbed continually for 20 seconds, followed by air drying for three seconds and light-curing for 10 seconds.

Table 3: *Tukey HSD for Unequal N (Spjotvoll/Stoline test), Probabilities for Post-hoc Tests for the Different Techniques Independent of the Storage-time*

Treatment*	Mean	*p*<0.05**
SB	16.3	a
SE	14.1	b
SBHC	10.8	c

**SB: total-etch; SBHC: total-etch + deproteinization; SE: self-etch adhesive.*
***Statistical differences expressed by different letters when compared by Tukey test (p<0.05).*

A 3 x 5-mm cylindrical Teflon mold was placed against the specimen to receive the resin-based composite (Filtek Z250, shade A1, 3M ESPE). The composite was inserted in two 2.5-mm increments, each increment being light cured for 40 seconds. After removing the mold, the composite was light cured for an additional 20 seconds in each side of the resin composite cylinder. The light intensity was monitored throughout the experiment using a curing radiometer (Demetrom, Danbury, CT, USA) that ranged from 570 to 580 mW/cm^2. The restorative procedures were done following a random

sequence. The specimens were stored in deionized distilled water (pH = 7) at 37°C for 1 day, 7 days, 30 days, 6 months or 12 months. During the storage period, the water was replaced every 15 days.

The specimens were tested in shear in a universal testing machine (EMIC Ltda, São José dos Pinhais, Brazil) at a cross-head speed of 0.5 mm/minute. The shear bond strength (SBS) of each specimen was noted in MPa. Data was submitted to split-plot ANOVA and Tukey tests (α=0.05) to test for significant differences.

Debonded specimens were examined under the scanning electron microscope (SEM) at 500x magnification to determine the fracture mode (adhesive vs cohesive vs mixed).

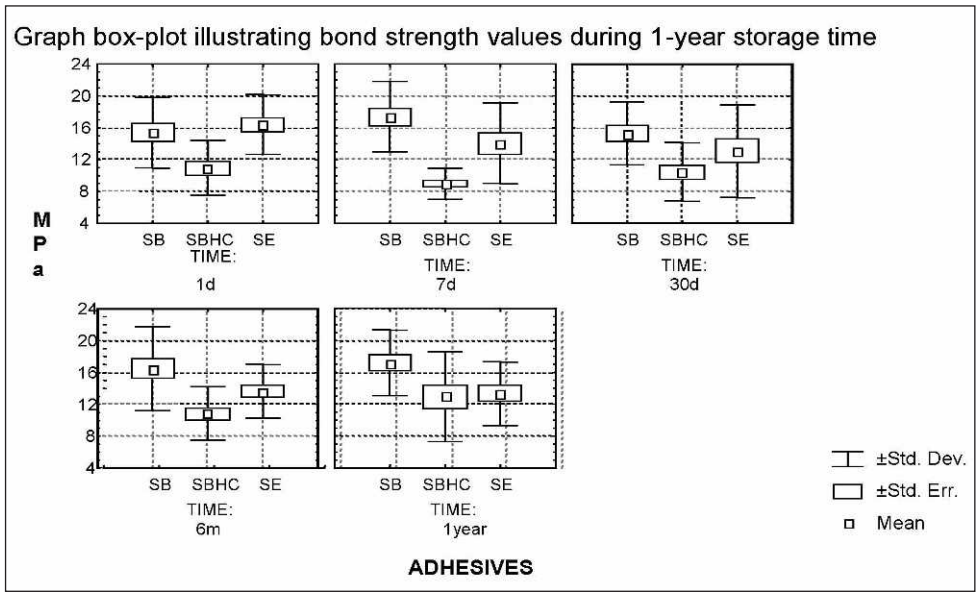


Figure 1. *Graphic of box plot of the means (+/-SD) of shear bond strength of the bonding techniques in different storage-time (SB: total-etch; SBHC: total-etch + deproteinization; SE: self-etch adhesive).*

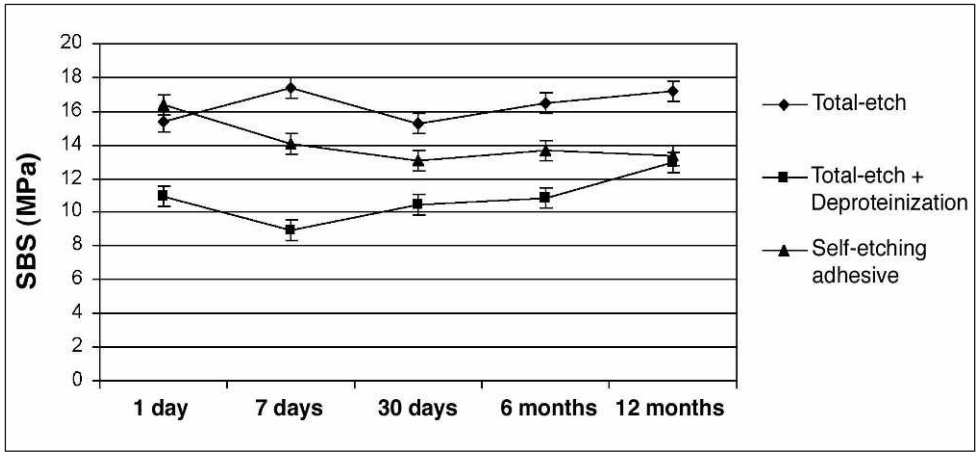


Figure 2. *Graphic of shear bond strength of each bonding technique in function of storage.*

RESULTS

The results are summarized in Tables 1 and 2. Mean SBS ranged from 8.9 MPa (SBHC at seven days) to 17.2 MPa (SB at 12 months). There was no significant effect (p=0.46) of the storage time for all bonding techniques and no interaction was observed between the two factors (p=0.21). All bonding techniques had adhesion stability up to 12 months after bonding but presented different SBS (Figure 1). ANOVA showed statistically significant differences among surface treatments (p<0.001). When surface treatment was analyzed independently of the storage time, Tukey's post-hoc test showed that SB presented a mean SBS significantly higher than SE and SBHC (16.3 MPa vs 14.1 MPa and 10.8 MPa, respectively). SBHC presented a mean SBS significantly lower than SE and SB, independent of the times (Table 3).

The examination of debonded samples under SEM showed that the fractures were predominantly adhesive in nature. A few cohesive fractures in dentin were found for SB and SBHC, all the fractures were adhesive.

DISCUSSION

One of the goals in adhesive dentistry is to develop materials that can replace dental structure in form, function and esthetics. It is clinically important to enhance the adhesion between dentin and restorative resin, leading to better retention of restorations, preventing marginal leakage and, consequently, reducing the incidence of secondary caries (Wakabayashi & others, 1994; Kitasako & others, 2001). The stability of the bond between composites and dentin is of critical importance for the longevity of restorations (Gwinnett & Yu, 1995; Burrow, Satoh & Tagami, 1996; Kato & Nakabayashi, 1998; Hashimoto & others, 2000, 2002a; Kitasako & others, 2001). Thus, studies on the longevity of bonded restorations and the degradation of resin-dentin bonds have been conducted to investigate the durability of the dentin bonding mechanism (Alhadainy & Abdalla, 1996; Tyas, 1996).

Studies report a significant reduction in the resin-dentin bond strength after water storage (Burrow &

others, 1996; Gwinnett & Yu, 1995; Kitasako & others, 2001). It has been speculated that the remaining exposed collagen fibrils at the base of the hybrid layer are susceptible to hydrolytic degradation over time, leading to a reduction in bond strength (Kato & Nakabayashi, 1998; Hashimoto & others, 2000; Miyazaki, Onose & Moore, 2002). In this study, bond strengths were not affected by 12-month storage (Table 1 and Figure 2), which is in agreement with other studies (De Munck & others, 2003; Giannini & others, 2003). When Single Bond was applied according to manufacturers' instructions, maintaining the wet collagen layer after acid etching, SBS values were maintained after 12 months, indicating that this adhesive may have adequately enveloped the collagen fibrils, preventing their degradation.

Regarding the collagen removal technique, the results allow the authors to infer that the ethanol- and water-based adhesive used did not diffuse well into the etched and collagen depleted dentin. Intertubular porosities created by NaOCl treatment could not be filled with adhesive monomers, which could explain the low SBS values obtained on SBHC-treated specimens (Jacobsen & Söderhold, 1995; Saboia & others, 2000). These results are consistent with those of microleakage evaluation after one-year storage, where Single Bond presented no significant differences when the collagen removal technique was applied (Saboia, Pimenta & Ambrosano, 2002). Vichi, Ferrari and Davidson (1997) also reported a statistically significant increase in microleakage for a water-based adhesive system (Scotchbond MP Plus, 3M ESPE) when applied to collagen-depleted dentin.

Collagen removal by NaOCl treatment increases the surface roughness of dentin and its wettability (Toledano & others, 1999) and transforms the dentin into a porous structure with multiple irregularities that allow for good mechanical retention (Vargas & others, 1997). In spite of such morphological aspects, in the results of this study, the collagen removal technique resulted in the lowest SBS values (Table 3, Figure 1), which might indicate that the results are system-dependent (Vargas & others, 1997). Despite these low values, this technique promoted a stable interface over time, because it is essentially made of minerals (Toledano & others, 1999) that could explain the maintenance of SBS values presented in the results.

The results obtained in this study using the experimental self-etching adhesive support the hypothesis that collagen fibrils were involved by the adhesive's resin monomers concomitantly to dentin demineralization (Itou & others, 2001), presenting intermediary SBS results after storage (Table 3). The statistical analysis for this sample revealed no reduction in mean SBS values after 12-month storage in water.

In addition to degrading the unprotected collagen layer, water might play a role in weakening the properties of adhesive monomers through water sorption (Gwinnett & Yu, 1995). However, the adhesives tested formed hybrid layers that resist water degradation, considering that in 12 months storage, the results remained stable except for a numerical decrease in SBS values for the experimental self-etching adhesive (Figure 2).

When performance of the different techniques tested in this study is followed over time (Figure 2), it is interesting to note that, at day one, the mean SBS for the total-etch specimens (15.4 MPa) was similar to the mean SBS for the specimens treated with the self-etching adhesive (16.4 MPa). At the end of the experiment, however, mean SBS for the specimens treated with the self-etching specimens were reduced numerically to 13.3 MPa, while the mean SBS for the total-etch specimens remained stable at 17.2 MPa. Even though those differences are not statistically significant, this tendency clearly highlights the importance of conducting long-term bonding studies to test the performance of adhesive techniques up to 12 months or longer. Many reports of bond strength studies consider only 24-hour data, which, in this example, would have inferred a very different conclusion, that is, that total-etch and self-etching techniques perform equally well, which might not be the case if long-term data is factored into the analyses.

Another interesting tendency noted was that, overall, the performance (measured as SBS) of the experimental self-etching adhesive decreased steadily over time, whereas, performance of the ethanol- and water-based total-etch adhesive (with or without collagen removal) remained relatively stable. This observation might indicate some form of hydrolytic degradation of the adhesive itself or the adhesive interface. Based on the data collected in this study, however, it is not possible to speculate on the cause of this tendency.

All groups presented similar fracture pattern. Evaluation of the fractured interface of SBHC demonstrated that 100% of the fractures were adhesive. These data reinforced the observation that the adhesion of the ethanol- and water-based adhesive system in collagen-depleted dentin was not well-established (Vichi & others, 1997; Saboia & others, 2000, 2002).

The results of this study demonstrate that for both one-day and 12-month post-bonding, the use of an adhesive system based on phosphoric acid etching could be considered as the first choice. However, the self-etching adhesive also presented good, stable results after 12-month storage in water. The collagen removal technique, the other evaluated alternative to improve bond strength, presented poor results and should not be used in conjunction with the tested adhesive system.

CONCLUSIONS

In conclusion, the hypotheses advanced were only partially confirmed:

- The deproteinization technique did not provide bond strengths equivalent to or better than the bond strengths generated by the total-etch technique.
- The experimental self-etching adhesive tested did not provide bond strengths of equivalent or better quality compared to the bond strengths generated by the total-etch technique.
- Storage time up to one year did not affect bond strengths.

(Received 6 June 2003)

Acknowledgement

The authors acknowledge CNPq (National Council for Scientific and Technological Development - Brazil) for its financial support (#300924/1997-6) and 3M/ESPE for providing the adhesive systems.

References

Alhadainy HA & Abdalla AJ (1996) 2-year clinical evaluation of dentin bonding systems *American Journal of Dentistry* **9(2)** 77-79.

Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34(6)** 849-853.

Burrow MF, Satoh M & Tagami J (1996) Dentin bond durability after three years using a dentin bonding agent with and without priming *Dental Materials* **12(5)** 302-307.

Chigira H, Yukitani W, Hasegawa T, Manabe A, Itoh K, Hayakawa T, Debari K, Wakumoto S & Hisamitsu H (1994) Self-etching dentin primers containing phenyl-P *Journal of Dental Research* **73(5)** 1088-1095.

De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, Lambrechts P & Vanherle G (2003) Four-year water degradation of total-etch adhesives bonded to dentin *Journal of Dental Research* **82(2)** 136-140.

Giannini M, Seixas CA, Reis AF & Pimenta LA (2003) Six-month storage time evaluation of one-bottle adhesive systems to dentin *Journal of Esthetic and Restorative Dentistry* **15(1)** 43-48; discussion 49.

Gwinnett AJ (1994) Dentin bond strength after air-drying and re-wetting *American Journal of Dentistry* **7(3)** 144-148.

Gwinnett AJ & Matsui A (1967) A study of enamel adhesives. The physical relationship between enamel and adhesive *Archives of Oral Biology* **12(12)** 1615-1620.

Gwinnett AJ & Yu S (1995) Effect of long-term water storage on dentin bonding *American Journal of Dentistry* **8(2)** 109-111.

Gwinnett JA, Tay FR, Pang KM & Wei SH (1996) Quantitative contribution of the collagen network in dentin hybridization *American Journal of Dentistry* **9(4)** 140-144.

Hara AT, Amaral CM, Pimenta LA & Sinhoreti MA (1999) Shear bond strength of hydrophilic adhesive systems to enamel *American Journal of Dentistry* **12(4)** 181-184.

Hashimoto M, Ohno H, Kaga M, Endo K, Sano H & Oguchi H (2000) *In vivo* degradation of resin-dentin bonds in humans over 1 to 3 years *Journal of Dental Research* **79(6)** 1385-1391.

Hashimoto M, Ohno H, Sano H, Tay FR, Kaga M, Kudou Y, Oguchi H, Araki Y & Kubota M (2002a) Micromorphological changes in resin-dentin bonds after 1 year of water storage *Journal of Biomedical Materials Research* **63(3)** 306-311.

Hashimoto M, Ohno H, Kaga M, Sano H, Tay FR, Oguchi H, Araki Y & Kubota M (2002b) Over-etching effects on microtensile bond strength and failure patterns for two dentin bonding systems *Journal of Dentistry* **30(2-3)** 99-105.

Ibarra G, Vargas MA, Armstrong SR & Cobbb DS (2002) Microtensile bond strength of self-etching adhesives to ground and unground enamel *Journal of Adhesive Dentistry* **4(2)** 115-124.

Itou K, Torii Y, Takimura T, Chikami K, Ishikawa K & Suzuki K (2001) Effect of priming time on tensile bond strength to bovine teeth and morphologic structure of interfaces created by self-etching primers *International Journal of Prosthodontics* **14(3)** 225-230.

Jacobsen T & Söderhold KJ (1995) Some effects of water on dentin bonding *Dental Materials* **11(2)** 132-136.

Kanca J 3rd (1992a) Resin bonding to wet substrate. I Bonding to dentin *Quintessence International* **23(1)** 39-41.

Kanca J 3rd (1992b) Effect of resin primer solvents and surface wetness on resin composite bond strength to dentin *American Journal of Dentistry* **5(4)** 213-215.

Kanca J 3rd (1996) Wet bonding: Effect of drying time and distance *American Journal of Dentistry* **9(6)** 273-276.

Kato G & Nakabayashi N (1998) The durability of adhesion to phosphoric acid etched, wet dentin substrates *Dental Materials* **14(5)** 347-352.

Kitasako Y, Burrow MF, Katahira N, Nikaido T & Tagami J (2001) Shear bond strengths of three resin cements to dentine over 3 years *in vitro Journal of Dentistry* **29(2)** 139-144.

Koibuchi H, Yasuda N & Nakabayashi N (2001) Bonding to dentin with a self-etching primer: The effect of smear layers *Dental Materials* **17(2)** 122-126.

Miyazaki M, Onose H & Moore BK (2002) Analysis of the dentin-resin interface by use of laser Raman spectroscopy *Dental Materials* **18(8)** 576-580.

Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials Research* **16(3)** 265-273.

Nakabayashi N & Takarada K (1992) Effect of HEMA on bonding to dentin *Dental Materials* **8(2)** 125-130.

Nakabayashi N, Ashizawa M & Nakamura M (1992) Identification of a resin-dentin hybrid layer in vital human dentin created *in vivo*: Durable bonding to vital dentin *Quintessence International* **23(2)** 135-141.

Okuda M, Pereira PN, Nakajima M & Tagami J (2001) Relationship between nanoleakage and long-term durability of dentin bonds *Operative Dentistry* **26(5)** 482-490.

Perdigão J, Swift EJ Jr, Heymann HO & Malek MA (1998) The effect of a re-wetting agent on the performance of acetone-based dentin adhesives *American Journal of Dentistry* **11(5)** 207-213.

Saboia VP, Rodrigues AL & Pimenta, LA (2000) Effect of collagen removal on shear bond strength of two single-bottle adhesive systems *Operative Dentistry* **25(5)** 395-400.

Saboia VP, Pimenta LA & Ambrosano GM (2002) Effect of collagen removal on microleakage of resin composite restorations *Operative Dentistry* **27(1)** 38-43.

Sano H, Takatsu T, Ciucchi B, Horner JA, Matthews WG & Pashley DH (1995) Nanoleakage: Leakage within the hybrid layer *Operative Dentistry* **20(1)** 18-25.

Tay FR, Gwinnett AJ, Pang KM & Wei SH (1996) Resin permeation into acid-conditioned, moist, and dry dentin: A paradigm using water-free adhesive primers *Journal of Dental Research* **75(4)** 1034-1044.

Tay FR, Gwinnett AJ & Wei SH (1997) Ultrastructure of the resin-dentin interface following reversible and irreversible rewetting *American Journal of Dentistry* **10(2)** 77-82.

Toledano M, Osorio R, Perdigão J, Rosales JI, Thompson JY & Cabrerizo-Vilchez MA (1999) Effect of acid etching and collagen removal on dentin wettability and roughness *Journal of Biomedical Materials Research* **47(2)** 198-203.

Toledano M, Osorio R, de Leonardi G, Rosales-Leal JI, Ceballos L & Cabrerizo-Vilchez MA (2001) Influence of self-etching primer on the resin adhesion to enamel and dentin *American Journal of Dentistry* **14(4)** 205-210.

Tyas MJ (1996) Clinical evaluation of five adhesive systems: Three-year results *International Dental Journal* **46(1)** 10-14.

Van Meerbeek B, Inokoshi S, Braem M, Lambrechts P & Vanherle G (1992) Morphological aspects of the resin-dentin interdiffusion zone with different dentin adhesive systems *Journal of Dental Research* **71(8)** 1530-1540.

Van Meerbeek B, Perdigão J, Lambrechts P & Vanherle G (1998) The clinical performance of adhesives *Journal of Dentistry* **26(1)** 1-20.

Vargas MA, Cobb DS & Armstrong SR (1997) Resin-dentin shear bond strength and interfacial ultrastructure with and without a hybrid layer *Operative Dentistry* **22(4)** 159-166.

Vichi A, Ferrari M & Davidson CL (1997) *In vivo* leakage of an adhesive system with and without NaOCl as pretreatment *Journal of Dental Research* **76** IADR Abstract #3077 p 398.

Wakabayashi Y, Kondou Y, Suzuki K, Yatani H & Yamashita A (1994) Effect of dissolution of collagen on adhesion to dentin *International Journal of Prosthodontics* **7(4)** 302-306.

Departments

Classifieds: Faculty Positions

School of Dentistry
Oregon Health & Science University

The School of Dentistry at the Oregon Health & Science University is seeking an energetic, motivated and qualified individual for a full-time position at the level of assistant/associate professor in the Department of Restorative Dentistry, Division of Operative Dentistry. Experience in teaching, research, service and patient care, as well as excellent interpersonal and communication skills are preferred. Specific teaching responsibilities will include participation at the pre-doctoral level in both the clinical and pre-clinical curriculum, with the opportunity to serve as course director. Candidates should possess a DMD/DDS degree. One day per week (0.2 FTE) will be available for participation in the Faculty Dental Practice. Collaboration in research opportunities is available and encouraged. Salary will be determined by credentials and experience. OHSU is an Equal Opportunity institution. Interested candidates should submit a letter, curriculum vitae and references to:

Dr Jack Ferracane, PhD
Chair, Restorative Dentistry
OSHU School of Dentistry
611 SW Campus Drive
Portland, OR 97239-3097
(Ferracan@ohsu.edu)
(503) 494-4327

Operative Dentistry Home Page

We hope all our readers will take advantage of the information available by accessing our Internet home page. Our address is: **http://www.jopdent.org/**

The home page contains a search engine and buttons that, hopefully, will lead you to answers to any questions you may have related to ***Operative Dentistry***. These are:

Journal: Leads to information on the Editorial Staff and Editorial Board; a complete index of journal volumes; a compilation of direct gold references; highlights of the current, next, and future issues, as well as a more detailed look at published Editorials and Clinical Pearls.

Subscribe: Leads to complete information on subscription rates; purchasing back issues, reprints, and bound volumes; and subscription and change of address forms.

Affiliates: Provides links to the American Academy of Gold Foil Operators, the Academy of Operative Dentistry, the AADS-Operative Section, and our Corporate Sponsors. In addition, membership applications for the journal's parent academies are available for downloading.

News: Announcements of interest to our readers, including meeting information, advertised faculty positions, and upcoming CE courses.

Authors: Complete instructions for contributors to the journal.

Reviewers: Link for our Editorial Board to submit manuscript reviews electronically.

Corporate Sponsorship

Operative Dentistry invites dental manufacturers that share our commitment to timely publication of research and clinical articles relevant to the discipline of restorative dentistry to become Corporate Sponsors of the journal. ***Operative Dentistry*** is distributed in 54 countries with more than 1,800 subscribers. The cost of sponsorship is US $3,000.00 per year (January through December). Sponsors will be recognized by having their company logo displayed on a special page in each issue of the journal for the duration of their sponsorship.

A complimentary subscription to ***Operative Dentistry*** will be sent to the corporate contact person, and the sponsor's logo will appear in the Corporate Sponsors section of our web page, where it will act as a link to their company's website. Interested parties should contact the Editor at ***Operative Dentistry***, 1121 West Michigan Street, Indianapolis, IN 46202-5186; phone: (317) 278-4800; fax: (317) 278-4900; e-mail: editor@jopdent,org; URL: http://www.jopdent.org/

Instructions to Contributors

Correspondence

Send manuscripts and correspondence regarding manuscripts to Dr Michael A Cochran, Editor, *Operative Dentistry*, Indiana University School of Dentistry, Room S411, 1121 W Michigan St, Indpls, IN 46202-5186; phone (317) 278-4800; fax (317) 278-4900; e-mail: editor@jopdent.org; URL: http://www.jopdent.org/.

Exclusive Publication

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

Manuscripts

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

❒ Submit a computer disk and identify the operating system (Macintosh or IBM-compatible) and the word processing program used.

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

❒ Supply complete names, degrees, titles and affiliations for all authors (include addresses that are different from the corresponding author's).

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

❒ Research (clinical and laboratory) papers MUST include a one sentence Clinical Relevance statement, as well as a Summary, Introduction, Methods and Materials, Results, Discussion and Conclusions section. Funding other than material supply must be stated.

❒ Clinical Technique/Case Report papers should contain at least the following: Purpose, Description of Technique or Solution, along with materials and potential problems and a Summary outlining advantages and disadvantages.

❒ Type double-spaced, including references, and leave margins of at least 3 cm (1 inch). Spelling should conform to the *American Heritage Dictionary of the English Language*. SI (Système International) units are preferred for scientific measurement, but traditional units are acceptable.

❒ The editor reserves the right to make literary corrections.

Illustrations

Please do NOT submit any illustrations or graphs in Microsoft Power Point or Word format. They will not be accepted.

❒ Submit four copies of each illustration.

❒ Line drawings should be in India ink or its equivalent on heavy white paper, card or tracing velum. All lettering must be of professional quality, legible against its background and remain proportionally legible if reduced. Type legends on separate sheets.

❒ Photographs should be on glossy paper with a maximum size of 15x20 cm (6x8 inches). For best reproduction, a print should be one-third larger than its reproduced size.

❒ On the back of each illustration indicate lightly in pencil the top and the number of the figure ONLY (no names). Where relevant, state staining technique(s) and the magnification of the prints. Obtain written consent from holders of copyright to republish any illustrations published elsewhere.

❒ Illustrations may also be supplied on floppy disk, Zip disk or CD as TIFF files with a minimum resolution of 300 dpi (dots per inch) for grayscale and 1200 dpi for color.

❒ Photographs become the property of *Operative Dentistry*.

Tables and Graphs

❒ Submit tables and graphs on sheets separate from the text.

❒ Graphs are to be submitted with any lettering proportional to their size, with their horizontal and vertical axes values displayed.

❒ Data for constructing graphs MUST be provided with the manuscript in a spreadsheet (Excel) or word processing format on computer disk.

❒ Graphs may be supplied on floppy disk, Zip disk or CD as TIFF files with a minimum resolution of 300 dpi. or as Microsoft Excel files.

References

❒ References must be arranged in alphabetical order by authors' names at the end of the article, with the year of publication placed in parentheses immediately after the author's name. This is followed by the full journal title (no abbreviations and in italics), the full subject title, volume and issue number and first and last pages.

❒ In the text, cite references by giving the author and, in parentheses, the date: Smith (1975) found…; or, by placing both name and date in parentheses: It was found…(Smith & Brown, 1975; Jones, 1974).

❒ When an article being cited has three authors, include the names of all of the authors the first time the article is cited; subsequently, use the form (Brown & others, 1975). Four or more authors should always be cited in the text as (Jones & others, 1975). In the References section, always list all the authors.

❒ If reference is made to more than one article by the same author and is published in the same year, the articles should be identified by a letter (a, b) following the date, both in the text and in the list of references.

❒ Book titles should be followed by the publication address and the name of the publisher.

Reprints

Reprints of any article, report or letter can be ordered through the editorial office.

OPERATIVE DENTISTRY
CORPORATE SPONSORS

These Dental Manufacturers have joined Operative Dentistry in our commitment to publish quality dental literature in a timely manner. We thank them for their support.

DISCUS DENTAL®

SDI
SOUTHERN DENTAL INDUSTRIES

GC
GC America Inc.

DENTSPLY
CAULK

BISCO
Specialist In Adhesive and Composite Technology
Bringing Science to the Art of Dentistry™

ULTRADENT

3M ESPE

Den-Mat®

ivoclar vivadent

sds Kerr
SYBRON DENTAL SPECIALTIES

Please view the Corporate Sponsors Page at our website (http://www.jopdent.org) for direct links to these companies.

SEPTEMBER/OCTOBER 2004 • VOLUME 29 • NUMBER 5 • 481-600

©OPERATIVE DENTISTRY, Inc.

BUONOCORE MEMORIAL LECTURE

CLINICAL RESEARCH

LABORATORY RESEARCH

DEPARTMENTS

10-9385
Operative Dentistry
Indiana University School of Dentistry, Rm S411
1121 West Michigan Street
Indianapolis, IN 46202-5186 USA

Periodicals