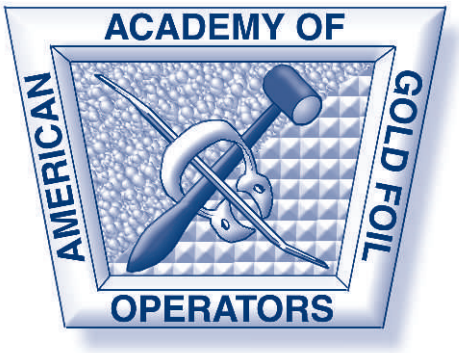
OPERATIVE DENTISTRY



may/june 2003 • volume 28 • number 3 • 213-324

(ISSN 0361-7734)

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

OPERATIVE DENTISTRY

MAY/JUNE 2003 • VOLUME 28 • NUMBER 3 • 213-324

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

Editor: Michael A Cochran
Editorial Assistant/Subscription Manager: Joan Matis
Editorial Associate: Karen E Wilczewski
Associate Editors: Bruce A Matis, Edward J DeSchepper and Richard B McCoy
Managing Editor: Timothy J Carlson
Assistant Managing Editors: Joel M Wagoner and Ronald K Harris

Editorial Board

Kinley K Adams
Maxwell H Anderson
Daniel J Armstrong
Steven R Armstrong
Tar-Chee Aw
Wayne W Barkmeier
Douglas M Barnes
Gardner Bassett
Mark W Beatty
Lars Bjørndal
Lawrence W Blank
Paul K Blaser
Murray R Bouschlicher
William W Brackett
James C Broome
K Birgitta Brown
William Browning
Paul A Brunton
Michael Burrow
Fred J Certosimo
Daniel CN Chan
David G Charlton
Gordon J Christensen
Kwok-hung Chung
N Blaine Cook
David Covey
Gerald E Denehy
Joseph B Dennison
Kim E Diefenderfer
E Steven Duke
Willaim J Dunn
Frederick C Eichmiller
Sigfus T Eliasson
Omar M El-Mowafy
John W Farah
Dennis J Fasbinder
Jack L Ferracane
Mark Fitzgerald
Kevin B Frazier
James C Gold
Carlos Gonzalez-Cabezas
Valeria V Gordan
Kevin M Gureckis
Mark S Hagge
Carl W Haveman
Van B Haywood
Charles B Hermesch
Harald O Heymann
Thomas J Hilton
Richard J Hoard
Barry W Holleron
Ronald C House
Poonam Jain
Gordon K Jones
Barry Katz
Robert C Keene
William P Kelsey, III
Edwina A M Kidd
George T Knight
Kelly R Kofford
Harold R Laswell
Mark A Latta
James S Lindemuth
Melvin R Lund
Dorothy McComb
Jonathan C Meiers
Georg Meyer
Ivar A Mjör
Michael P Molvar
B Keith Moore
Graham J Mount
David F Murchison
Ann-Marie Neme
Jennifer Neo
Jacques Nör
John W Osborne
Michael W Parker
Craig J Passon
Tilly Peters
Anne Peutzfeldt
Frank E Pink
T R Pitt Ford
Jeffrey A Platt
L Virginia Powell
James C Ragain
John W Reinhardt
Eduardo Reston
Philip J Rinaudo
André Ritter
J William Robbins
Frank T Robertello
Howard W Roberts
Boyd E Robinson
Clyde L Roggenkamp
William Rose
Jean-Francois Roulet
Frederick A Rueggeberg
Henry A St Germain, Jr
David C Sarrett
John W Shaner
Gregory E Smith
W Dan Sneed
Ivan Stangel
James M Strother
James B Summitt
Edward J Swift, Jr
William H Tate
Franklin R Tay
Choi Gait Toh
Peter T Triolo, Jr
Karen Troendle
Richard D Tucker
Marcos Vargas
Douglas Verhoef
Warren C Wagner
Joel M Wagoner
Charles W Wakefield
Steve W Wallace
Timothy F Watson
Nairn H F Wilson
Peter Yaman
Adrian U J Yap
Andrea G F Zandona

©2003 Operative Dentistry, Inc. • Printed in USA

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3**, 213-214

Editorial

The Second Mouse

There is a well-known saying that goes, "The early bird gets the worm." This is generally accepted as meaning that the first one to arrive, adopt, utilize, etc. is recognized as the most prepared and perceptive and is therefore rewarded. Whether we realize it or not, this seems to be the philosophy that drives much of the dental profession today. The idea that the newest products and techniques are better and more efficient than their older counterparts is pervasive in the popular press and in continuing education courses. In addition, we are made to feel guilty if we are not adding these things to our armamentarium as though we could not possibly be delivering optimum health care without utilizing all of these innovations. Finally, we know that many individuals tend to seek out what they perceive to be "modern, cutting-edge practices" and we don't want to fall behind in our ability to attract these new patients. The battle cry is "New is better and faster (and makes us more money)!!" and we respond like a huge flock of "early birds" fighting for our "worms."

I frequently find myself in this group. I direct a Graduate Operative Dentistry Program and feel obligated to expose my residents to the newest advances in technology. I suppose that I am also like many of you in that I am extremely "gadget" oriented. I love new equipment and materials. For years I have been in the category of "early adopter" in relation to my home electronic, photographic and computer equipment. However, I have learned over time that being the first to have something is often not the best position to be in and that this is particularly true in our profession as health care providers. New does not automatically correlate with better and, in fact, may not be as good as what we already have. The newest, most exciting items are often quickly replaced because they were introduced to the market before they were really perfected. New is almost never faster (at least initially) because there is a learning curve involved and, while we may embrace change, it usually takes considerable effort to alter our habits.

Therefore, let me suggest a modification to the lead sentence in this editorial…"The early bird gets the worm, but it is often the second mouse that gets the cheese." In my opinion, our patients will benefit from a slightly more judicious approach to health care. Waiting for some evidence that a new technique actually does provide reduced cost, chair-time or discomfort, or that a material offers greater longevity, biotolerance or cosmetic improvement than what you were using, is critical. How many of you have dental materials or equipment that you purchased (usually at considerable expense) because it was touted as the "newest and best," that is gathering dust on a back shelf because the reality did not match your expectations? How many of you have had patients return with failed restorations or complaints of sensitivity following treatment with the "very latest"? Do you feel that this contributed to either your clinic income or your patient rapport?

I am not taking the stance that all advances in technology are suspect or doomed to failure. I'm merely advising some restraint in making radical changes in your health care delivery. Dental manufacturers are in the business of making a profit, but they fully understand the need to provide quality products to maintain their reputation and consumer confidence. They also are well aware of the level of competition that exists in the marketplace and the constant demand from the dental profession for better materials and equipment. I believe that, for the most part, they do an outstanding job of providing us with what we want, but our demands and the competition can often lead to delivery of product before all appropriate evaluation is complete. Our practices then become the research facility and our patients the research subjects. When we report problems, we are usually presented with a new, improved, cutting-edge version to purchase, and the cycle continues.

One other problem related to the "early bird" syndrome is our strange compulsion to always replace the old with the new rather than providing treatment alternatives to our patients. Cavity preparations with airborne abrasives, lasers and ultrasonic diamond instruments are wonderful alternatives for certain situations, but have certainly not replaced the con-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

ventional rotary handpiece. Advances in caries detection are showing tremendous promise but, despite the trend toward conservatism and remineralization, can be misused and lead to surgical over-treatment. In addition, consumer demand seems to have affected our judgment in relation to appearance versus health and nature. The current obsession with youthful appearance and cosmetic improvements has resulted in the need for tooth-colored restorative materials that are much higher in value than the natural human dentition, aggressive restorative therapy where bleaching would solve the problem and finished cases that are "white and straight" with compromised, unhealthy supporting tissues.

The need for beneficial change is always important and we must constantly reevaluate our procedures and materials based on experience and evidence. We should embrace advances in technology once they have been shown to offer real advantages to our patients and improvements in our ability to deliver health care. We should also be able to offer our patients multiple treatment alternatives rather than limiting their choices (does the phrase "metal-free practice" ring a bell?) since we have yet to find the single therapy that provides the best initial and long-term results in all situations. We need to use a little more common sense and not be carried away by advertisements and testimonials (I'm still waiting for more high-definition television offerings to make my expensive home theater truly worth the investment).

There are many traps and pitfalls to be considered when you aspire to be the early bird. I'm positive that the first mouse that went for the cheese would support this statement, but unfortunately he's unavailable for comment due to his sudden demise. However, his colleague (the second mouse) is in complete agreement…and is thoroughly enjoying his cheese.

Michael A Cochran
Editor

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3,** 215-235

Buonocore Memorial Lecture

Adhesion to Enamel and Dentin: Current Status and Future Challenges

Michael Buonocore

B Van Meerbeek • J De Munck • Y Yoshida
S Inoue • M Vargas • P Vijay
K Van Landuyt • P Lambrechts • G Vanherle

Bart Van Meerbeek

SUMMARY

Bonding to tooth tissue can be achieved through an "etch&rinse," "self-etch" or "glass-ionomer" approach. In this paper, the basic bonding mechanism to enamel and dentin of these three approaches is demonstrated by means of ultramorphological and chemical characterization of tooth-biomaterial interfacial interactions. Furthermore, bond-strength testing and measurement of marginal-sealing effectiveness (the two most commonly employed methodologies to determine "bonding effectiveness" in the laboratory) are evaluated upon their value and relevance in predicting clinical performance. A new dynamic methodology to test biomaterial-tooth bonds in a fatigue mode is introduced with a recently developed micro-rotary fatigue-testing device. Eventually, today's adhesives will be critically weighted upon their performance in diverse laboratory studies and clinical trials. Special attention has been given to the benefits/drawbacks of an etch&rinse versus a self-etch approach and the long-term performance of these adhesives. Correlating data gathered in the laboratory with clinical results clearly showed that laboratory research CAN predict clinical effectiveness. Although there is a tendency to simplify bonding procedures, the data presented confirm that conventional three-step etch&rinse adhesives still perform most

*Bart Van Meerbeek, DDS, PhD, professor, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-facial Surgery, Catholic University of Leuven, Belgium

Jan De Munck, DDS, PhD-student, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-facial Surgery, Catholic University of Leuven, Belgium

Yasuhiro Yoshida, DDS, PhD, associate professor, Department of Biomaterials, Okayama University Graduate School of Medicine and Dentistry, Japan

Satoshi Inoue, DDS, PhD, lecturer, Division for General Dentistry, Hokkaido University Dental Hospital, Japan

Marcos Vargas, DDS, MS, associate professor, Department of Operative Dentistry, University of Iowa, USA

Padmini Vijay, DDS, MS-student, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-facial Surgery, Catholic University of Leuven, Belgium

Kirsten Van Landuyt, DDS, PhD-student, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-facial Surgery, Catholic University of Leuven, Belgium

Paul Lambrechts, DDS, PhD, full professor, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-facial Surgery, Catholic University of Leuven, Belgium

Guido Vanherle, MD, DDS, emeritus full professor, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-facial Surgery, Catholic University of Leuven, Belgium

*Reprint request: Catholic University of Leuven, Kapucijnenvoer 7, B-3000, Leuven, Belgium; e-mail: bart.vanmeerbeek@med.kuleuven.ac.be

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

favorably and are most reliable in the long-term. Nevertheless, a self-etch approach may have the best future perspective. Clinically, when adhesives no longer require an "etch&rinse" step, the application time, and probably more importantly, the technique-sensitivity are substantially reduced. Especially "mild," two-step self-etch adhesives that bond through a combined micromechanical and chemical interaction with tooth tissue closely approach conventional three-step systems in bonding performance.

INTRODUCTION

Adhesive dentistry evolves rapidly. Two main incentives drive this evolution. Adhesive techniques combined with using tooth-colored restorative materials are frequently requested by patients. They want us to restore their teeth not only anatomically and functionally, but also esthetically and, thus, nearly invisibly. From our perspective, today's operative dentistry should primarily involve "minimally invasive" (Degrange & Roulet, 1997) or "minimum intervention" (Tyas, Anusavice & Frencken, 2000) care. This means that only the lost or diseased tooth tissue is replaced by the restorative material that is directly bonded to the remaining sound tissue. Also, the more recent approach of promoting "maintenance and repair" (Bouschlicher, Reinhardt & Vargas, 1997; Denehy, Bouschlicher & Vargas, 1998; Roeters, 2000; Wilson, Setcos & Brunton, 2001), rather than replacing entire restorations (exhibiting marginal discolorations and/or defects) has further boosted the use of adhesive techniques in diverse applications of everyday clinical practice.

Major drawbacks of this approach are that adhesive techniques still accompany a higher placement complexity and technique sensitivity (risk of making manipulation errors). Also, though the retention of adhesive restorations for a reasonable time is no longer a clinical problem, maintaining the margins of adhesive restorations sealed against leakage phenomena remains the major factor that shortens clinical longevity.

The fundamental principle of adhesion to tooth substrate is based upon an exchange process by which inorganic tooth material is exchanged for synthetic resin (Van Meerbeek & others, 2001a). This process involves two phases. One phase consists of removing calcium phosphates by which microporosities are exposed at both the enamel and dentin tooth surface. The other so-called hybridization phase involves infiltration and subsequent *in situ* polymerization of resin within the created surface microporosities. This results is micro-mechanical interlocking that is primarily based on mechanisms of diffusion. While micro-mechanical interlocking is believed to be a prerequisite to achieving good bonding within clinical circumstances, the potential benefit of additional chemical interaction between functional monomers and tooth substrate components has recently gained new attention. This paper sketches the current status of adhesives in terms of bonding effectiveness measured in the laboratory and in clinical practice. Special attention is given to the potential roles of both micro-mechanical and chemical bonding mechanisms through correlating morphologic and chemical interfacial characteristics of tooth-biomaterial interactions using diverse kinds of adhesives.

MECHANISMS OF ADHESION TO ENAMEL AND DENTIN

Using contemporary adhesives, the substance exchange between biomaterial and tooth tissue is carried out in one, two or three clinical application steps, respectively. Besides the number of application steps, adhesives can further be classified based on the underlying adhesion strategy in *"etch&rinse," "self-etch"* and *"(resin-modified) glass-ionomer* adhesives" (Van Meerbeek & others, 2001a) (Figure 1). The degree of substance exchange substantially differs among these adhesives. In general, the exchange intensity induced by etch&rinse adhesives exceeds that of self-etch adhesives, though among the latter, systems that rather intensively interact with tooth tissue also exist, even when applied in only a single step.

Etch&Rinse Approach

This adhesion strategy involves at least two steps and, in its most conventional form, three steps with successive application of the *conditioner* or acid etchant, followed by the *primer* or adhesion promoting agent, and eventually, application of the actual bonding agent or *adhesive resin* (Figure 1). The simplified two-step version combines the second and third step but still follows a separate *"etch&rinse"* phase.

This etch&rinse technique is still the most effective approach to achieving efficient and stable bonding to enamel and basically only requires two steps. Selective dissolution of hydroxyapatite crystals through etching (commonly with a 30-40% phosphoric-acid gel) is followed by *in situ* polymerization of resin that is readily absorbed by capillary attraction within the created etch pits, thereby, enveloping individually exposed hydroxyapatite crystals (Figure 2). Two types of resin tags interlock within the etch-pits. "Macro"-tags fill the space surrounding the enamel prisms (Figure 2a), while numerous "micro"-tags result from resin infiltration/polymerization within the tiny etch-pits at the cores of the etched enamel prisms (Figure 2b). The latter are especially thought to contribute the most with regard to retention to enamel.

At dentin, this phosphoric-acid treatment exposes a microporous network of collagen that is nearly totally deprived of hydroxyapatite (Figures 3 and 4). High-resolution transmission electron microscopy (TEM) and

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

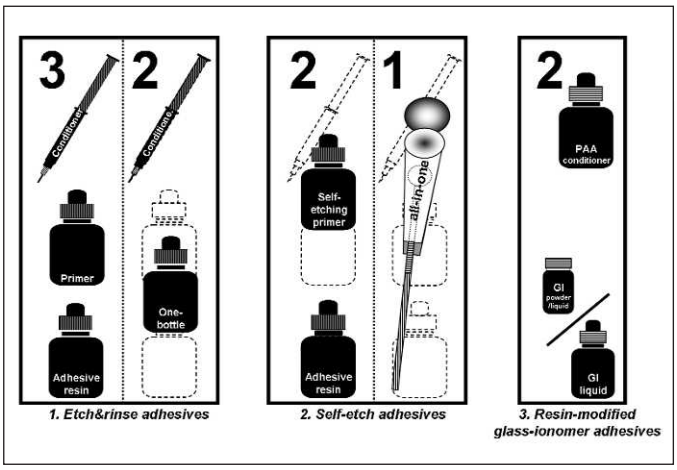


Figure 1. *Classification of contemporary adhesives following adhesion strategy and number of clinical application steps.*

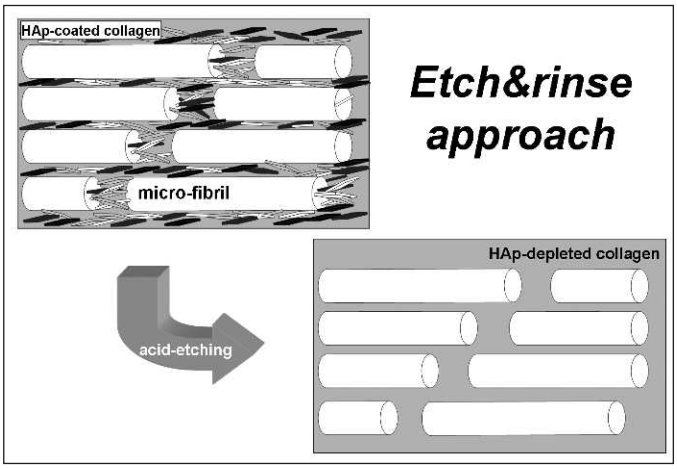


Figure 3. *Schematic drawing presenting the effect of an etch&rinse approach on the hydroxyapatite-collagen arrangement.*

chemical surface analysis by energy dispersive X-ray spectroscopy (EDXS) and X-ray photoelectron spectroscopy (XPS) have confirmed that nearly all calcium phosphates were removed or at least became under detection limit (Figure 5) (Van Meerbeek & others, 1996; Yoshida & Van Meerbeek, 2002). As a result, the primary bonding mechanism of etch&rinse adhesives to dentin is primarily diffusion-based and depends on hybridization or infiltration of resin within the exposed collagen fibril scaffold, which should be as complete as possible (Figure 6). True chemical bonding is rather unlikely, because the functional groups of monomers may have only weak affinity to the *"hydroxyapatite-depleted"* collagen. Such challenging monomer-collagen interaction might be the principle reason for what has been documented as manifesting in the form of *"nanoleakage"* phenomena (Sano & others, 1994b, 1995).

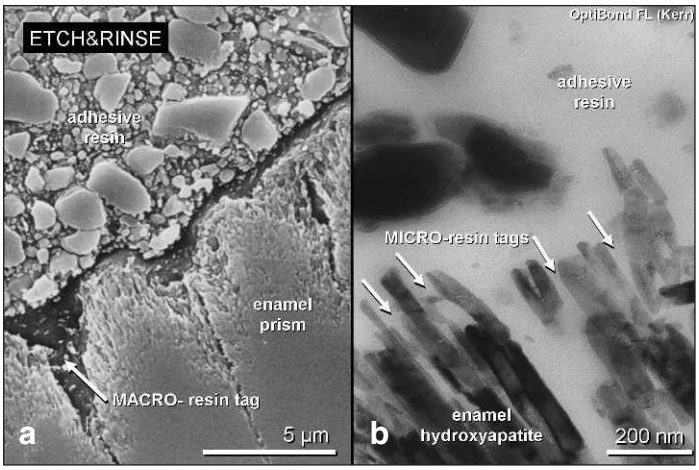


Figure 2 a, b. *Field-emission scanning electron microscopy (Fe-SEM) photomicrograph (a, left) and transmission electron microscopy (TEM) photomicrograph (b, right) of the enamel-resin interface illustrating the occurrence of micro-mechanical interlocking of resin within the etch-pits created through conventional phosphoric-acid etching. "Macro"-tags represent the infiltration and in situ polymerization of resin in between adjacent enamel prisms, whereas "micro"-tags probably contribute most to the eventual bonding effectiveness by enveloping individual hydroxyapatite crystals at the enamel prism cores.*

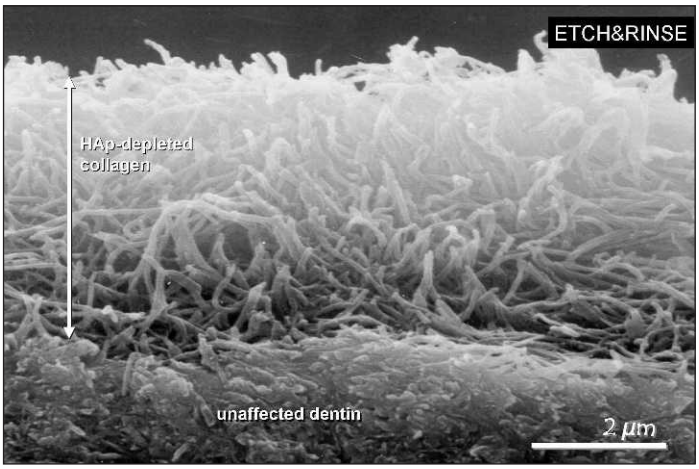


Figure 4. *Fe-SEM photomicrograph (image from M Vargas) of dentin etched for 15 seconds with 35% phosphoric acid (Ultra-Etch, Ultradent). Note the demineralization effect with exposure of collagen up to a depth of 4-5 µm. The transition of the exposed collagen fibril network towards the underlying unaffected dentin is very abrupt. Collagen fibrils were nearly completely deprived from hydroxyapatite.*

Most critical in the etch&rinse approach is the priming step. When an acetone-based adhesive is used, the highly technique-sensitive *"wet-bonding"* technique is mandatory (Tay & others, 1996). Otherwise, gentle post-conditioning air-drying of acid-etch dentin (and enamel) following a *"dry-bonding"* technique still guarantees effective bonding when a water/ethanol-based adhesive is used (Van Meerbeek & others, 1996, 1998c).

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

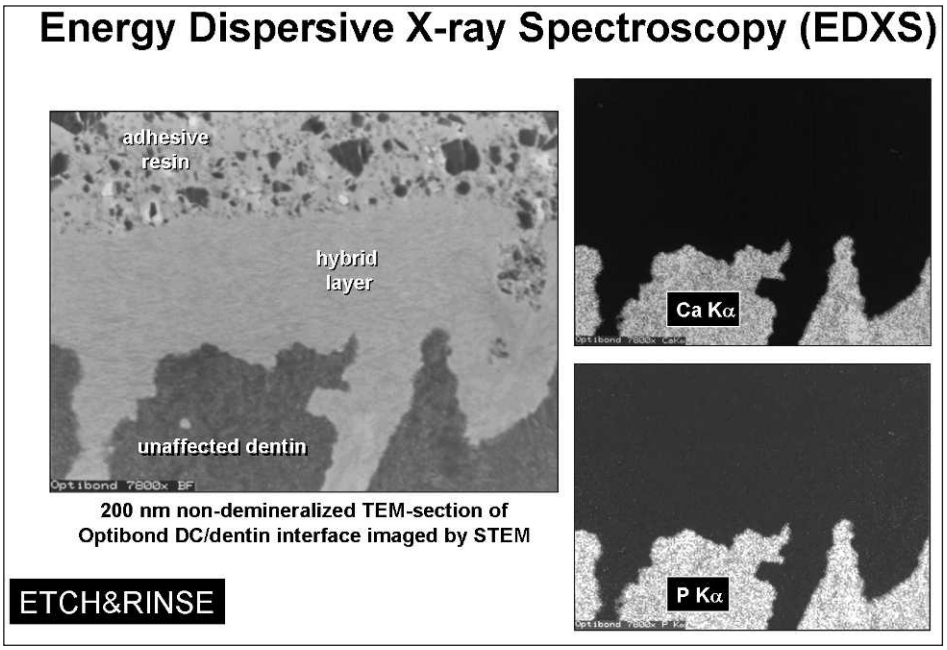


Figure 5. *TEM photomicrograph (left) showing an unstained, non-demineralized 200-nm section through the resin-dentin interface produced by Optibond Dual-Cure (Kerr). The hybrid layer clearly does not contain any hydroxyapatite (HAp), which would have appeared electron dense (dark gray to black) as within the unaffected dentin underneath. EDXS surface mapping (top right) confirmed that calcium was below detection limit, while only a scarce amount of phosphorus could be detected (bottom right). The latter may also originate from the phosphate-based monomer GPDM (glycerophosphoric acid dimethacrylate), which is a basic constituent of the Optibond Dual-Cure primer.*

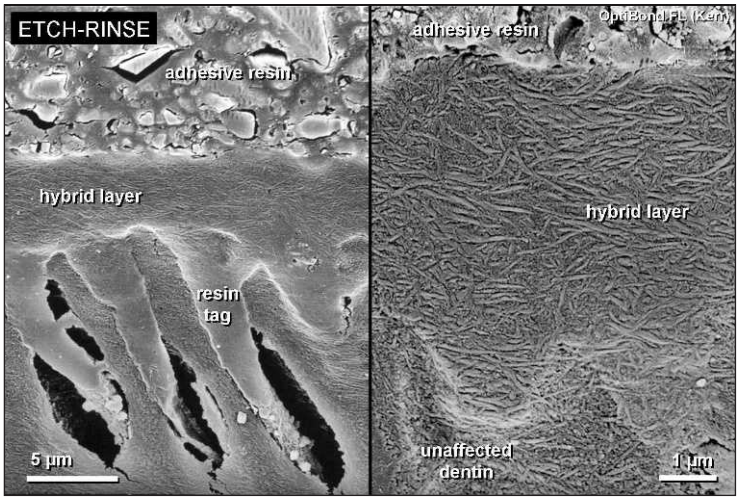


Figure 6. *Fe-SEM photomicrographs of a diamond-knife sectioned resin-dentin interface produced by the three-step etch&rinse adhesive Optibond FL (Kerr). Using this sample preparation methodology, fewer artifacts are produced and the interfacial ultra-structure is better preserved. Whereas resin-tags seal the opened dentin tubules, a 4-5 µm thick hybrid layer is formed that consists of a loosely organized arrangement of collagen fibrils interspersed by resin that infiltrated in the exposed collagen network.*

Glass-Ionomer Approach

Glass-ionomers remain as the only materials that are *self-adhesive* to tooth tissue, in principle, without any surface pre-treatment (Figure 7). Although this is certainly true, pre-treatment with a weak polyalkenoic-acid conditioner significantly improves bonding efficiency (Inoue & others, 2001a). Hence, this glass-ionomer approach can be achieved following a one- or two-step application procedure (Figure 1). The additional conditioning step becomes more important, especially when coarse cutting diamonds are used and, consequently, thicker and more compact smear layers are produced. In general, such a polyalkenoic-acid conditioner is applied for 10-to-20 seconds and gently rinsed off, followed by gently air-drying without dehydrating the surface (Figures 7 and 8a). The increase in bonding efficiency must be partially attributed to (1) a "cleaning" effect, by which loose cutting debris is removed, (2) a partial "demineralization" effect, by which the surface area is increased and microporosities for micromechanical interlocking or hybridization are exposed, but also in part to (3) chemical interaction of polyalkenoic acid with residual hydroxyapatite (see below). A network of *"hydroxyapatite-coated"* collagen fibrils interspersed by pores is typically exposed to a depth no deeper than 1 µm. TEM and XPS have demonstrated that (depending on the product) this polyalkenoic acid conditioner cannot be completely rinsed off (Van Meerbeek & others, 1998b, 2001b). An up to 0.5 µm thick layer, often referred to as "gel phase," remains attached to the tooth surface despite the conditioner being rinsed off (Figure 8b).

The actual auto-adhesion of glass ionomers to tooth tissue has recently been determined to be twofold. *Micromechanical interlocking* is achieved by shallow hybridization of the micro-porous, hydroxyapatite-coated collagen fibril network (Figure 8) (Van Meerbeek & others, 1998b, 2001b; Tay & others, 2001; Yip & others, 2001). In this respect, glass ionomers can be considered as adhering to tooth tissue through a "mild" self-etch approach (see below). The basic difference with the resin-based self-etch approach is that glass ionomers are self-etching through the use of a relatively high molecular weight (8,000-15,000) polycarboxyl-based polymer. Resin-based self-etch adhesives make use of acidic low-mollecular weight monomers.

As the second component of the self-adhesion mechanism, true primary chemical bonding occurs through

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

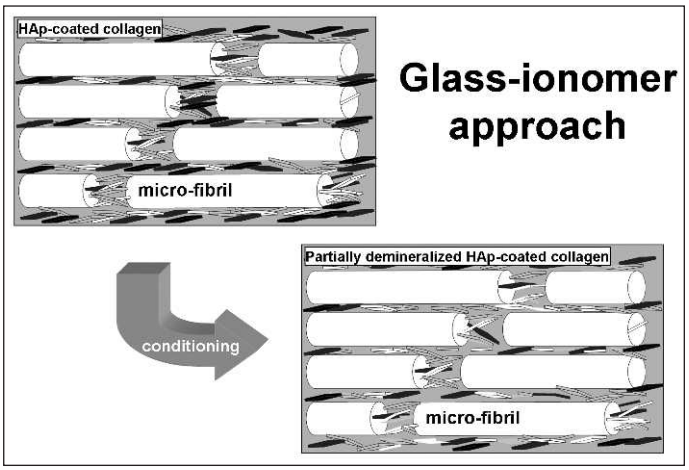


Figure 7. *Schematic drawing illustrating the effect of the glass-ionomer approach on the hydroxyapatite-collagen arrangement. Pre-treatment with a weak polyalkenoic acid conditioner only partially demineralizes dentin, exposing micro-porosities for micro-mechanical interaction and leaving hydroxyapatite crystals attached to individual collagen fibrils as receptors for additional chemical bonding.*

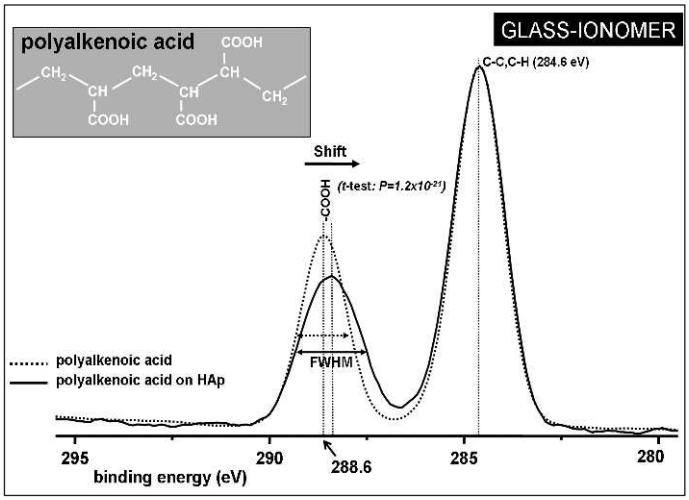


Figure 9. *XPS narrow-scan spectra of the C 1s region of the acrylic/maleic polyalkenoic acid co-polymer and of the polyalkenoic acid applied on hydroxyapatite (Yoshida & others, 2000). Interaction of the polyalkenoic acid with hydroxyapatite resulted in a significant shift of the peak representing the carboxyl groups (-COO-) to a lower binding energy, suggesting the formation of an ionic bond to hydroxyapatite as schematically explained in Figure 10.*

forming ionic bonds between the carboxyl groups of the polyalkenoic acid and calcium of hydroxyapatite that remains around the exposed surface collagen (Figures 9-11). This was proven for polyalkenoic acids applied to hydroxyapatite (Yoshida & others, 2000), but also to enamel and dentin (Fukuda & others, 2003). The application of a polyalkenoic-acid to synthetic hydroxyapatite (and dentin/enamel) produced a significant shift in the carboxyl (-COOH) peak to a lower binding energy, indicating that the carboxyl functional group

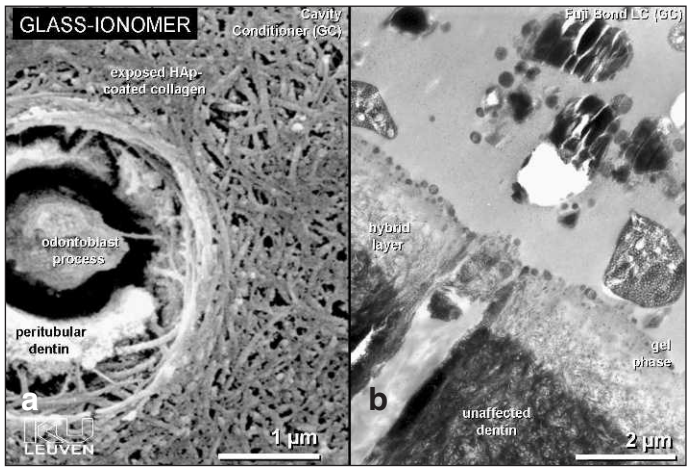


Figure 8. *(a) Fe-SEM photomicrograph (left) illustrating the effect of a polyalkenoic acid conditioner (Cavity Conditioner, GC) that was applied for 10 seconds on dentin covered by a smear layer. Although intertubular dentinal collagen was exposed, hydroxyapatite was not completely removed and remained as receptors for additional chemical interaction. Micropores were created to enable micro-mechanical interlocking through hybridization. (b) TEM photomicrograph (right) of an unstained, non-demineralized section through the glass-ionomer-dentin interface illustrating the twofold structural appearance of a glass-ionomer-dentin interface resulting from the application of the resin-modified glass-ionomer adhesive Fuji Bond LC (GC). On top of the hybrid layer, an amorphous, gray "gel-phase" represents the morphologic manifestation of the reaction product formed through interaction of the polyalkenoic acid with calcium that was extracted from the dentin surface.*

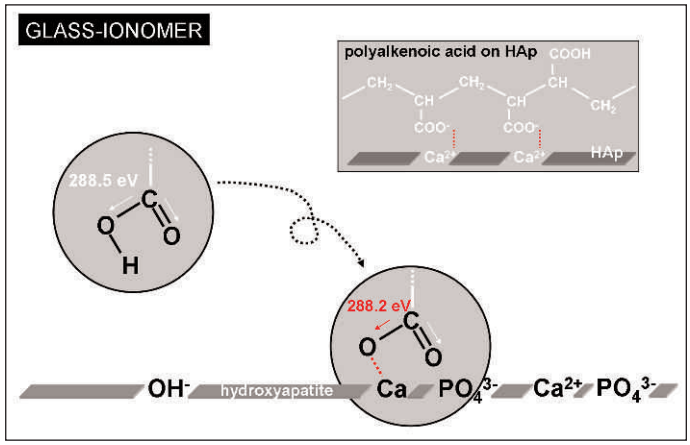


Figure 10. *Schematic explaining the interpretation of the detected shift of the carboxyl peak in the XPS spectrum of the polyalkenoic acid bonded to hydroxyapatite in Figure 9. The binding energy of C 1s of the carboxyl group is about 288.5 eV as a cumulative result of both oxygen atoms pulling at the carbon atom (top circle). When the carboxyl group forms an ionic bond to calcium of hydroxyapatite, oxygen will pull less intense to carbon leading to a reduction of the binding energy.*

interacted with the hydroxyapatite surface (Figure 9). This interaction was relatively strong, as this peak shift was recorded after ultrasonically rinsing off the polyalkenoic acid solution. This shifted peak at the XPS spectrum in Figure 9 represents the binding ener-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

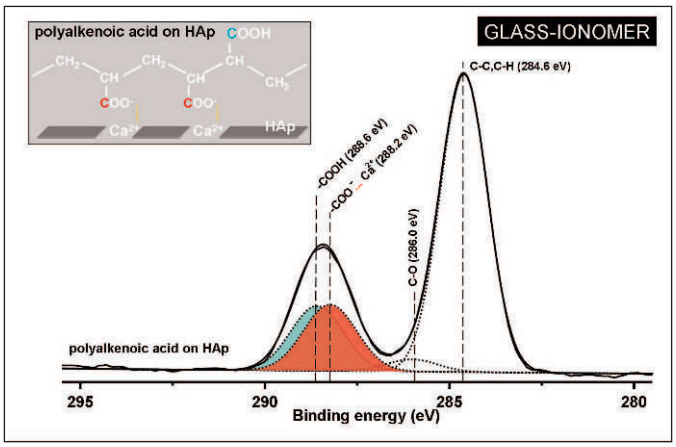


Figure 11. *Deconvolution of the shifted carboxyl peak in Figure 9 disclosed a peak at 288.6 eV that represents unreacted carboxyl groups, and a peak at 288.2 ev that results from carboxyl groups that bonded to calcium of hydroxyapatite (Yoshida & others, 2000).*

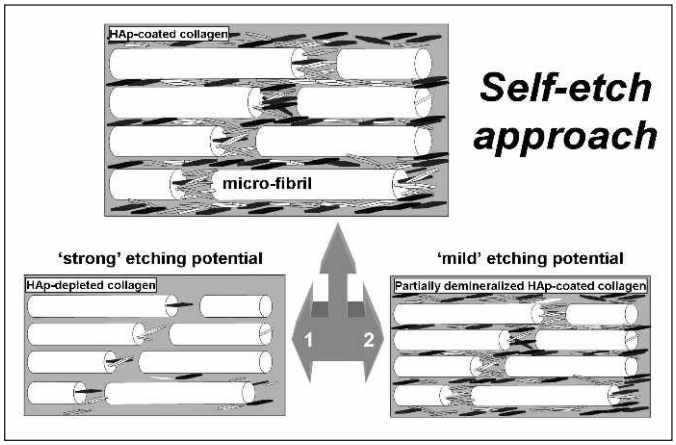


Figure 13. *Schematic drawing showing the effect of a self-etch approach on the hydroxyapatite-collagen arrangement. In case of a "strong" self-etch potential, dentin is nearly completely demineralized, exposing a hydroxyapatite-depleted collagen network that resembles that exposed by a total-etch approach. In case of a "mild" self-etch approach, dentin is only partially demineralized, by which residual hydroxyapatite crystals remain around individual collagen fibrils and provide sites for the functional groups of the monomers to chemically react with.*

gy of the C atom (C 1s) of the carboxyl group, being 288.6 eV for the unreacted polyalkenoic acid itself. This binding energy results from two oxygen atoms that pull on the carbon atom. As explained in Figure 10, when one oxygen atom of the carboxyl functional group of the polyalkenoic acid reacts chemically with calcium of hydroxyapatite, it consumes energy to form an ionic bond. Consequently, its pull to the carbon atom of the carboxyl group is less intense, thus, reducing its binding energy to 288.2 eV. However, the carboxyl peak in Figure 9 did not shift entirely to 288.2 eV, indicating that not all carboxyl groups interacted with hydroxyapatite. In fact, deconvolution disclosed that the shifted peak consists of two sub-peaks (Figure 11),

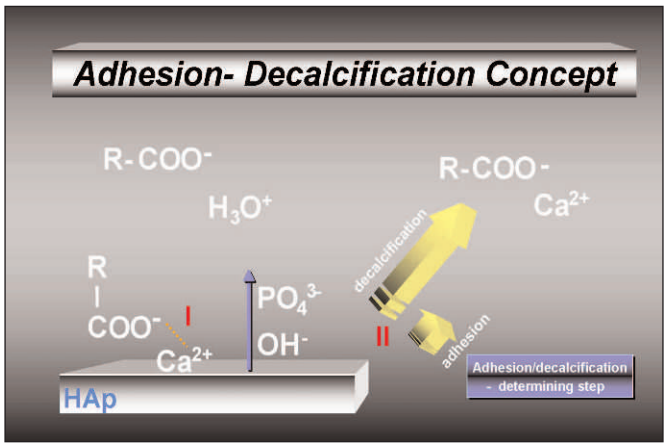


Figure 12. *Schematic drawing presenting the "Adhesion-Demineralization model" that explains why molecules that contain functional carboxyl groups either adhere to or decalcify hydroxyapatite tissues (Yoshida & others, 2001). After a first step involving adhesion to hydroxyapatite, molecules will remain attached to the hydroxyapatite surface depending on the solubility of the calcium salt in the own solution. The latter second phase is the adhesion/decalcification determining step.*

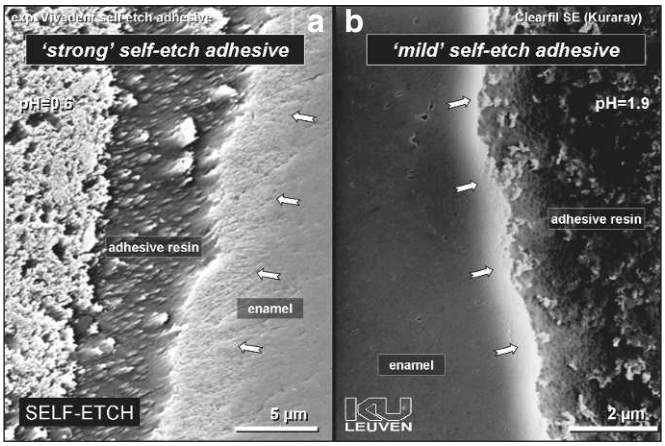


Figure 14. *Fe-SEM photomicrographs of resin-enamel interfaces when either a "strong" self-etch adhesive (left/a) or a "mild" self-etch adhesive (right/b) was applied. Depending on the acidity of the self-etching primer, the strong self-etch adhesive relatively intensively interacted with enamel up to a depth of 4-5 µm, whereas, hardly morphologic signs of interaction could be observed when the mild self-etch adhesive was applied.*

representing carboxyl groups that interacted with hydroxyapatite (sub-peak at 288.2 eV) and those that did not (sub-peak at 288.6 eV). It was also demonstrated that the actual molecular formula of the polyalkenoic acid significantly influences the chemical bonding potential (Yoshida & others, 2000; Fukuda & others, 2003). XPS clearly showed that a polyalkenoic acid based upon 10:1 acrylic/maleic acid units has about two-thirds of its carboxyl groups bonded to hydroxyapatite versus only half of the carboxyl groups of pure polyacrylic acid (Yoshida & others, 2000; Fukuda & others, 2003). Based on these XPS data

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

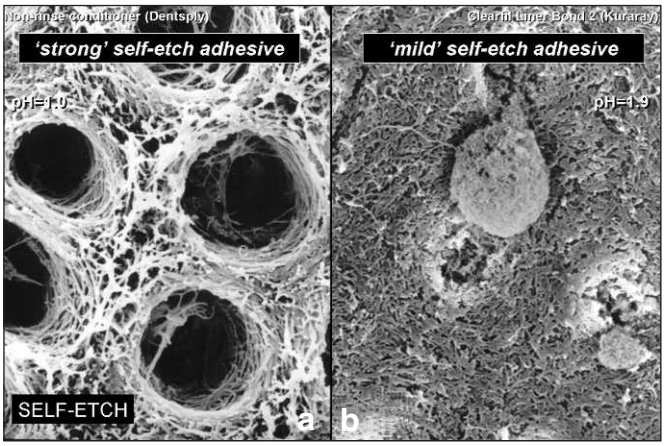


Figure 15. *Fe-SEM photomicrographs of dentin either treated (left/a) with the strong self-etching primer Non-Rinse Conditioner (Dentsply) or (right/b, image taken by J Perdigão) with the mild self-etching primer of Clearfil Liner Bond 2 (Kuraray). Non-Rinse Conditioner clearly opened the dentin tubules and exposed a micro-porous collagen fibril network similar to the effect of an etch&rinse approach using phosphoric acid. However, Clearfil Liner Bond 2 primer interacted clearly less intense with some exposure of collagen, while most tubules remained occluded.*

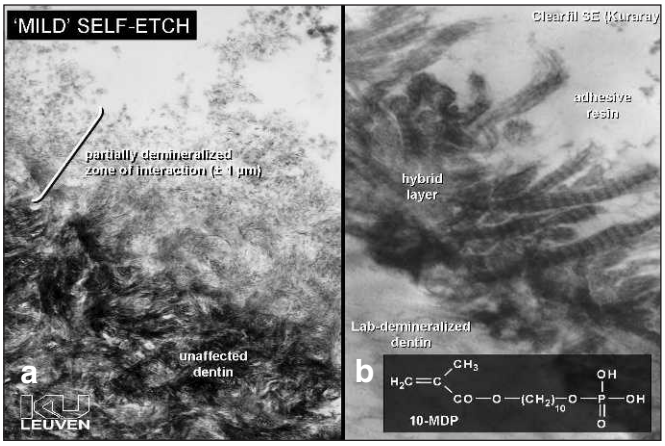


Figure 17. *(a) TEM photomicrograph (left) of an unstained, non-demineralized section through the resin-dentin interface produced by Clearfil SE (Kuraray). Note that dentin was only partially demineralized for about 1 µm deep, leaving hydroxyapatite crystals within the hybrid layer. (b) TEM photomicrograph of a stained, demineralized section through the resin-dentin interface produced by Clearfil SE (Kuraray). Note the formation of a 1-µm thick hybrid layer with a typical shag-carpet appearance at the transition to the adhesive resin and individual cross-banded collagen fibrils separated by electron lucent interfibrillar spaces. The chemical formula of the functional monomer 10-MDP is presented in the insert.*

(Yoshida & others, 2000, 2001; Yoshioka & others, 2002), the authors proposed an "Adhesion-Decalcification model" (AD-model) that explains why certain acids adhere to tooth tissue more than they decalcify it (Figure 12). This largely depends on the solubility of the formed calcium salt at the hydroxyapatite surface in its own acidic solution. The more soluble the calcium salts of the acids (or the adhesive

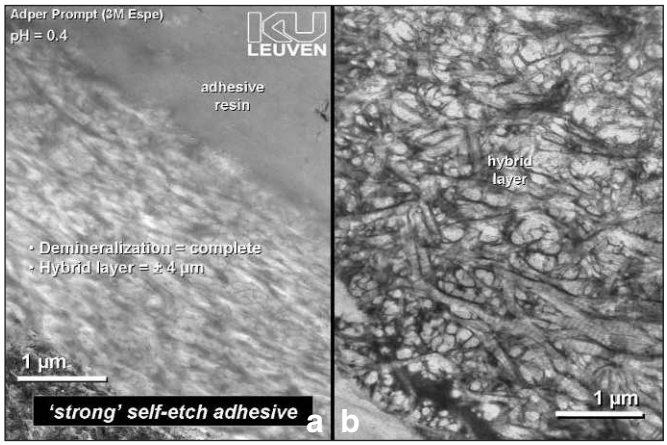


Figure 16. *TEM photomicrographs of an unstained non-demineralized (left/a) and stained demineralized (right/b) section through the resin-dentin interface produced by the strong one-step self-etch adhesive Adper Prompt (3M ESPE). Note that dentin was rather deeply demineralized up to about 3 µm. All hydroxyapatite around collagen was dissolved and the demineralization front stopped abruptly. A rather thick hybrid layer of about 3 µm was formed and resembles a hybrid layer as it would typically be produced following a etch&rinse approach. The typical phosphate-based composition of the adhesive resulted in a strong pick-up of heavy metal stain, by which the infiltration of the electron-dense resin within the hydroxyapatite-depleted collagen can be clearly detected. Some phase separation between electron lucent hydrophilic and electron dense hydrophobic adhesive components can be observed within the adhesive resin layer on top of the hybrid layer.*

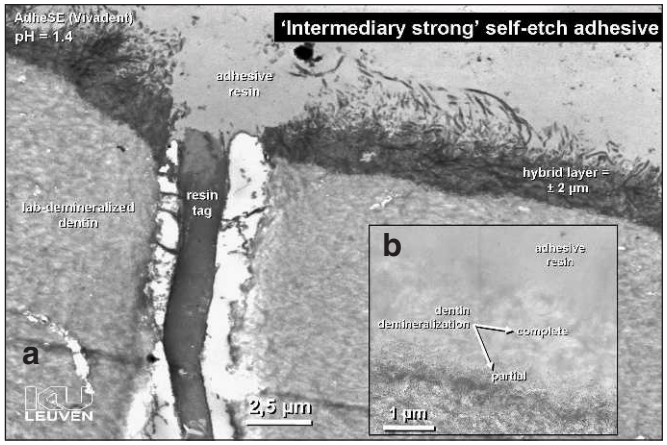


Figure 18. *TEM photomicrographs of the resin-dentin interface produced by the "intermediary strong" self-etch adhesive AdheSE (Vivadent). A relatively thick hybrid layer of about 2 µm can be observed on the photomicrograph representing a stained, demineralized section. The insert shows an unstained, non-demineralized TEM section, on which can be seen that the top 1.5-2 µm of the hybrid layer does not contain any residual hydroxyapatite crystals. The 0.5-1 µm layer at the hybrid layer base still contains residual hydroxyapatite and forms a rather gradual transition to the underlying affected dentin.*

monomer/polymer), the less it will adhere to the mineral substrate. As the calcium salts of polyalkenoic acids could hardly be dissolved, they have an adequate chemical bonding potential to hydroxyapatite-based tissues.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

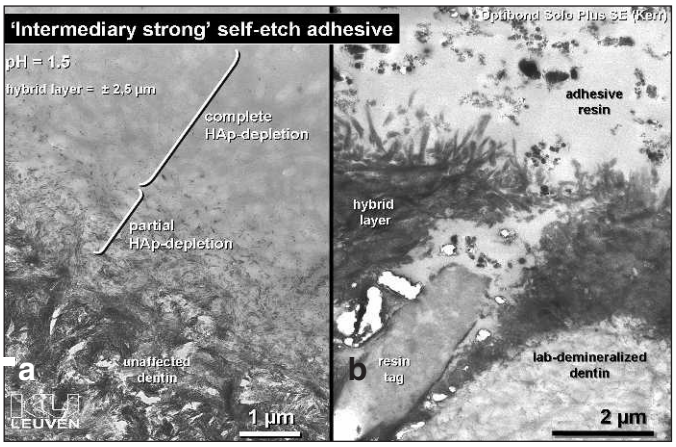


Figure 19. *TEM photomicrographs of an unstained, non-demineralized section (left/a) and of a stained, demineralized section (right/b) through the resin-dentin interface produced by the "intermediary strong" self-etch adhesive OptiBond Solo Plus Self-etch (Kerr). The hybrid layer clearly has two zones, without hydroxyapatite at the hybrid layer top and containing residual hydrxoxyapatite at the hybrid layer base. Staining disclosed a homogenous hybrid layer of 2.5 µm with a typical shag-carpet appearance at the transition to the adhesive.*

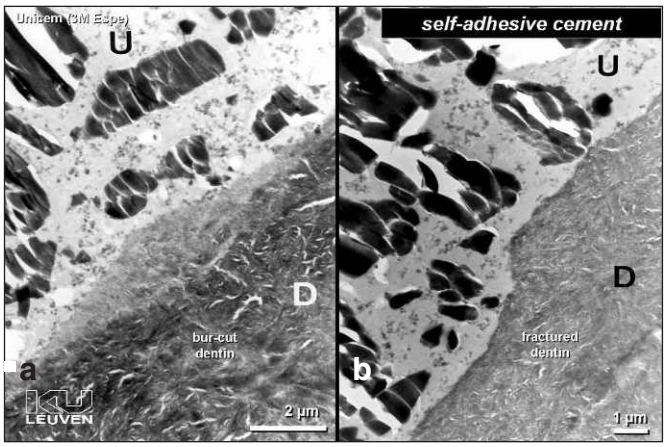


Figure 20. *Unstained, non-demineralized TEM sections representing the cement-dentin interface when either the self-adhesive luting material Unicem (3M ESPE) was applied to bur-cut dentin (left) or when it was applied to smear layer-free, fractured dentin (right). When applied to bur-cut dentin, the gray intermediary zone probably represents the partial infiltration of Unicem components within smear deposited by the diamond on the dentin surface. When Unicem was applied to fractured dentin, Unicem clearly appeared to have interacted only very superficially.*

Typical of some glass ionomers is the morphologic manifestation of a "gel-phase" at the interface, as was shown correlatively by transmission electron microscopy (Figure 8) and atomic force microscopy (Van Meerbeek & others, 1998b, 2001b; Yoshida & others, 1999). Correlating TEM and XPS data elucidated that this gel phase represents the formation of a calcium polycarboxylate salt resulting from either the polyalkenoic acid conditioner or the glass ionomer material itself (Van Meerbeek & others, 2001b). This phase has been shown to be stable and strong, intermediary between the shallow 0.5-1 µm hybrid layer and the glass-ionomer matrix. In microtensile bond strength testing, the interface typically fractured well above the gel phase within the matrix of the glass-ionomer material (Van Meerbeek & others, 2001b). AFM surface analysis confirmed that this gel phase is stronger than the actual glass-ionomer matrix (Van Meerbeek & others, 2001b). The actual function and contribution of this phase to the bond integrity needs to be further elucidated.

Self-Etch Approach

Probably, in regard to user-friendliness and technique-sensitivity, clinically, the most promising approach is self-etch. It no longer needs an "etch&rinse" phase, which not only lessens clinical application time, but also significantly reduces technique-sensitivity or the risk of making errors during application and manipulation. Another important advantage of the self-etch approach is that infiltration of resin occurs simultaneously with the self-etching process, by which the risk of discrepancy between both processes is low or non-existent. However, little is known about the long-term effects of incorporating dissolved hydroxyapatite crystals and residual smear layer remnants within the bond. How much of the primer/adhesive solvent is kept within the interfacial structure should also be investigated. Such solvent surplus will directly weaken the bond integrity, provide channels for nanoleakage or may affect polymerization of the infiltrated monomers. The resultant interfacial structure also becomes more hydrophilic and, thus, more prone to hydrolytic degradation (Tay & others, 2002a; Tay, Pashley & Yoshiyama, 2002b).

A self-etch approach involves either a two- or one-step application procedure (Figure 1). The self-etch effect should be ascribed to monomers to which one or more carboxylic or phosphate acid groups are grafted (Van Meerbeek & others, 2001a). Depending on etching aggressiveness, they can be subdivided into "strong" and "mild" self-etch adhesives (Figure 13).

"Strong" self-etch adhesives usually have a pH of 1 or below (Table 1). This high acidity results in rather deep demineralization effects. At enamel, the resulting acid-etch pattern resembles a phosphoric-acid treatment following an etch&rinse approach (Figure 14a) (Inoue & others, 2000; Pashley & Tay, 2001). At dentin, collagen is exposed and nearly all hydroxyapatite is dissolved (Figures 15a and 16). Consequently, the underlying bonding mechanism of "strong" self-etch adhesives is primarily diffusion-based, similar to the etch&rinse approach. Such low-pH self-etch adhesives have often been documented with rather low bond strength values, especially at dentin, and quite a high

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 1: *Acidity (pH) of Diverse Adhesive Solutions*

Adhesive	Classification	pH primer*
Adper Prompt L-Pop (3M ESPE)	One-step self-etch	0.4
Prompt L-Pop 2 (3M ESPE)	One-step self-etch	0.8
Xeno III (Dentsply)	One-step self-etch	1.4
i-Bond (Kulzer)	One-step self-etch	1.6
Non-Rinse Conditioner (Dentsply)	Two-step self-etch	1.0
AdheSE primer (Vivadent)	Two-step self-etch	1.4
OptiBond Solo Plus SE primer (Kerr)	Two-step self-etch	1.5
Clearfil SE Bond primer(Kuraray)	Two-step self-etch	1.9
Clearfil SE Bond Plus primer (Kuraray)	Two-step self-etch	2.0
Unifil Bond primer(GC)	Two-step self-etch	2.2
Panavia ED primer mixed (Kuraray)	Two-step self-etch	2.6
OptiBond Solo Plus primer/adhesive (Kerr)	Two-step etch&rinse	2.1
Prime&Bond NT primer/adhesive (Dentsply)	Two-step etch&rinse	2.2
Scotchbond 1 primer/adhesive(3M)	Two-step etch&rinse	4.7
OptiBond FL primer (Kerr)	Three-step etch-rinse	1.8

** Measured in-house using a digital pH meter (Inolab pH Level 2, WTW, Weilheim, Germany).*

number of pre-testing failures when tested following a microtensile bond strength approach (Inoue & others, 2001b, 2003; De Munck & others, 2003a). Besides the high initial acidity that appears to dramatically weaken the bonding performance, another concern is the effect of residual solvent (water) that remains within the adhesive interface, which can hardly can be completely removed. Further study needs to investigate the long-term stability of this strong self-etch approach.

"Mild" self-etch systems, in general, have a pH of around 2 (Table 1) and demineralize dentin only to a depth of 1 µm (Figures 15b and 17). This superficial demineralization occurs only partially, keeping residual hydroxyapatite still attached to collagen. Nevertheless, sufficient surface-porosity is created to obtain micromechanical interlocking through hybridization. The thickness of the hybrid layer is, however, much smaller than that produced by the strong self-etch or etch&rinse approach but has been proven to be minor in importance with regard to actual bonding effectiveness (Inoue & others, 2000, 2001b; De Munck & others, 2003a). The preservation of hydroxyapatite within the submicron hybrid layer may serve as a receptor for additional chemical bonding (Van Meerbeek & others, 2000; Yoshida & others, 2003). Carboxylic acid-based monomers like 4-MET (4-methacryloxyethyl trimellitic acid) and phosphate-based monomers, such as phenyl-P (2-methacryloxyethyl phenyl hydrogen phosphate), and 10-MDP (10-methacryloxydecyl dihydrogen phosphate) have a chemical bonding potential to calcium of residual hydroxyapatite (Yoshida & others, 2003). One may hypothesize that a weak self-etching effect is mandatory in order to (1) deal with the smear layer resulting from cavity preparation, (2) achieve micromechanical interlocking within etch pits at enamel and (3) achieve shallow micromechanical interlocking through hybridization at dentin. Micromechanical retention is thought to be necessary to resist acute de-bonding forces (such as those to which composite-tooth bonds are typically subjected during bond-strength testing). In addition, the exposed hydroxyapatite enamel surface and the hydroxyapatite crystals that remain around collagen (in the case of a "mild" self-etching or a glass-ionomer approach) are expected to be particularly advantageous. They enable more intimate chemical interaction with the functional monomers on a molecular level and may help prevent/retard marginal leakage. The challenge now is to have the functional monomers interact with hydroxyapatite so that the resulting calcium-carboxylate or calcium-phosphate bonds are stable within a hydrophilic environment long-term. Keeping hydroxyapatite around collagen may also better protect the collagen against hydrolysis and, thus, early degradation of the bond (Sano & others, 1999; Hashimoto & others, 2000, 2002). The weakest property of mild self-etch adhesives is their bonding potential to enamel. Therefore, developing monomers with stronger chemical bonding potential to hydroxyapatite may also help to further improve their bonding performance to enamel.

Some new adhesives, AdheSE (Vivadent) and OptiBond Solo Plus Self-etch (Kerr, Orange, CA, USA), were recently marketed and cannot be classified as "mild" or "strong" two-step self-etching adhesives. The pH of their self-etching primers is about 1.5 (Table 1) and, based on their interaction with dentin, the authors refer to them as "intermediary strong" two-step self-etch adhesives. Most typical is the two-fold build-up of the dentinal hybrid layer with a completely demineralized top layer and a partially demineralized base (Figures 18 and 19). Following an "etch&rinse" or "strong" self-etch approach, the transition of the exposed collagen fibril network to the underlying unaffected dentin is quite abrupt (see Figures 4, 5 and 16, respectively). Following an "intermediary strong" self-etch approach, the deepest region of the hybrid layer up to a maximum of 1 µm still con-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

tains hydroxyapatite, by which the transition of the hybrid layer to the underlying unaffected dentin is more gradual (Figures 18 and 19). These adhesives are more acidic than the "mild" self-etch adhesives, by which better micromechanical interlocking is achieved at enamel and dentin. The residual hydroxyapatite at the hybrid layer base may still allow for chemical intermolecular interaction, as was shown before for the "mild" self-etch adhesives. Based on the acidity (Table 1), the one-step self-etch adhesives i-Bond (Kulzer) and Xeno III (Dentsply, Milford, DE, USA) must also categorized as "intermediary strong" self-etch adhesives. Their resultant interfacial interaction is consequently expected to be similar to that produced by the intermediary "strong" two-step self-etch adhesives documented above.

Unicem (3M ESPE, St Paul, MN, USA) was recently launched as a possible first step towards self-adhesive resin-based restorative materials. This luting material is designed to be applied without any pre-treatment. TEM of the resultant interface showed a very superficial interaction with dentin (Figure 20). When applied to bur-cut dentin, a layer about 0.5-1 µm deep appeared less mineralized and most likely represented infiltration of Unicem components with a partially dissolved bur smear layer. This layer did not appear when Unicem was applied to fractured dentin that was free of cutting smear. Then, the interaction of Unicem with dentin could barely be morphologically detected. The actual bonding mechanism of this self-adhesive cement should be investigated in depth.

MEASURING BONDING EFFECTIVENESS: LABORATORY VERSUS CLINICAL TESTING

Laboratory Testing of Adhesives: Can They Predict Clinical Effectiveness?

Clinical trials are the ultimate test for dental restorations, but they cannot differentiate the true reason for failure due to the simultaneous impact of diverse stresses on restorations within the aggressive oral cavity. Lab testing can evaluate the effect of a single variable, while keeping all other variables constant. Based on this type of research, clear recommendations can be formulated toward clinicians with regard to the appropriate use and selection of dental materials. In general, laboratory testing is easy, fast and relatively cheap to screen new materials/techniques. They are useful in determining the "effectiveness" of adhesive materials within the specific test set-up. Ideally, the final objective should always be predicting clinical behavior long-term, though direct translation to the clinical situation is often difficult or even impossible.

Bond Strength Testing

In the mouth, the interface between restoration and tooth is exposed to diverse forces that act simultaneously. Already during setting of composite, resin shrinkage puts stress on the bond, pulling it away from the cavity wall (Versluis, Tantbirojn & Douglas, 1998). During function, mechanical stress by chewing forces, thermal and chemical stress with changes in temperature and pH will have an effect on the bond integrity as part of bio-tribocorrosive effects. The rationale behind bond strength testing is that the higher the actual bonding capacity of an adhesive, the better it will withstand such stresses and the longer the restoration will survive *in vivo*. Bond strength testing is relatively easy and fast and, in fact, besides a material tester does not require special equipment. It, therefore, remains the most popular methodology for measuring bonding effectiveness in the laboratory. Van Noort & others (1989), however, emphasized that bond strength cannot be regarded as a material property. The data obtained from bond strength tests largely depend on the actual test set-ups that may differ between laboratories for parameters such as specimen geometry, size of surface area, the type of composite and more. It is, therefore, not surprising that bond strength data substantially vary among laboratories throughout the world. The many variables involved make standardization of test methodologies for bond-strength measurements hardly achievable.

Most commonly, bond strength is measured by subjecting composites bonded-to-enamel/dentin to tensile or shear stress. However, at bond strength values higher than 20 MPa in a shear test, cohesive failures of the substrate will more likely occur (Schreiner & others, 1998). Therefore, a new test needed to be developed that differentiates between adhesives that produce higher bond strengths. A microtensile bond strength (µTBS) methodology was introduced by Sano and others in 1994(a). These authors showed that microtensile bond strength was inversely related to the bonded surface area (Sano & others, 1994a; Shono & others, 1999; Phrukkanon, Burow & Tyas, 1998a,b; Pashley & others, 1999) and that although much higher bond strengths were measured, most failures still occurred at the interface between tooth substrate and adhesive. Other advantages of µTBS-testing are that regional bond strengths and bonding effectiveness to clinically relevant tooth substrates such as carious (Nakajima & others, 1995; Yoshiyama & others, 2000) and sclerotic dentin (Tay & others, 2000; Kwong & others, 2002) can be measured (Pashley & others, 1999). The major disadvantage of µTBS-testing is the rather labor-intensive, technically demanding and relatively fragile sample preparation technique. Special care should be taken to avoid/reduce the production of microfractures at the interface during specimen preparation. They may weaken the bond and, thus, reduce the actual bond strength (Ferrari & Cardoso, 2002). Otherwise, one could argue that clinical restoration margins are

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

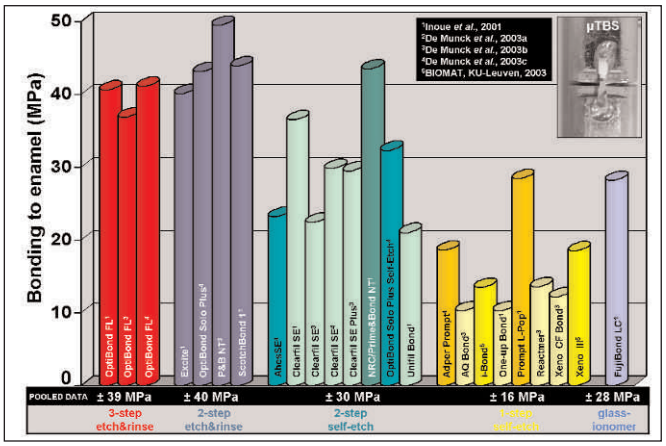


Figure 21. *Chart presenting the micro-tensile bond strength (µTBS) to enamel of diverse commercial adhesives. The data were gathered from diverse laboratory studies carried out at BIOMAT Leuven strictly following the same experimental protocol. The color code refers to the different kinds of adhesives following the classification presented in Figure 1. For the two-step self-etch adhesives, the light-green colored bars represent the data produced by "mild" self-etch adhesives, the intermediary-green colored bars those produced by "intermediary strong" self-etch adhesives, and the dark-green bars represent the µTBS-data produced by the "strong" self-etch adhesives. For the one-step self-etch adhesives, the light-yellow colored bars represent the data produced by "mild" self-etch adhesives, the intermediary-yellow colored bars those produced by "intermediary strong" self-etch adhesives, and the dark-yellow bars represent the µTBS-data produced by the "strong" self-etch adhesives. All data are pooled per group of adhesives underneath the chart.*

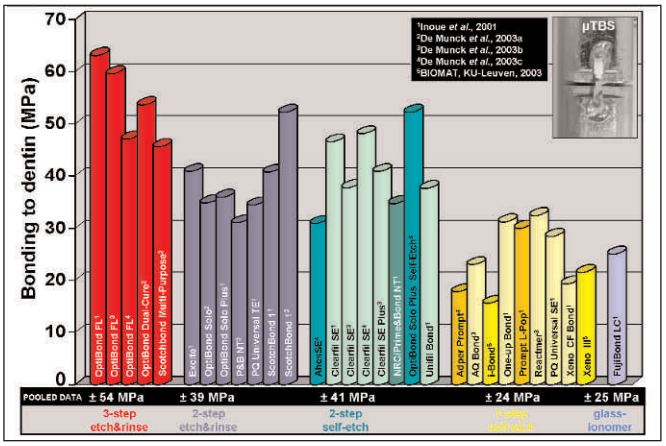


Figure 23. *Chart presenting the microtensile bond strength (µTBS) to dentin of diverse commercial adhesives. The data were gathered from diverse laboratory studies carried out at BIOMAT Leuven strictly following the same experimental protocol. The color code refers to the different kinds of adhesives following the classification presented in Figure 1. For the two-step self-etch adhesives, the light-green colored bars represent the data produced by "mild" self-etch adhesives, the intermediary-green colored bars those produced by "intermediary strong" self-etch adhesives, and the dark-green bars represent the µTBS-data produced by the "strong" self-etch adhesives. For the one-step self-etch adhesives, the light-yellow colored bars represent the data produced by "mild" self-etch adhesives, the intermediary-yellow colored bars those produced by "intermediary strong" self-etch adhesives, and the dark-yellow bars represent the µTBS-data produced by the "strong" self-etch adhesives. All data are pooled per group of adhesives underneath the chart.*

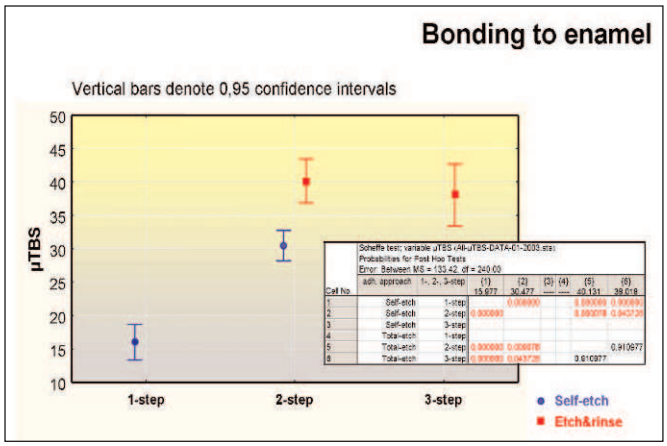


Figure 22. *Statistical analysis of the pooled enamel µTBS-data demonstrate that three-step etch&rinse adhesives bond equally well to enamel as two-step etch&rinse adhesives. Etch&rinse adhesives bond slightly, but statistically significantly better to enamel than two-step self-etch adhesives, that on their turn bond significantly much better than one-step self-etch adhesives. The actual p-values are mentioned in the table insert. All red-colored figures indicate statistical significant difference; black-colored figures indicate absence of statistical difference.*

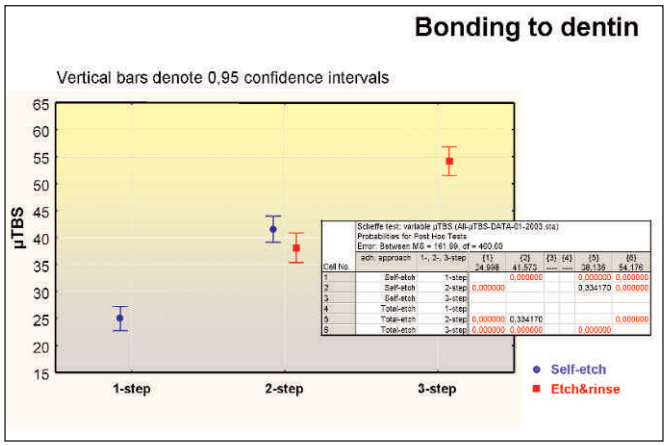


Figure 24. *Statistical analysis of the pooled dentin µTBS-data demonstrated that three-step etch&rinse adhesives bonded significantly better to dentin than two-step etch&rinse adhesives and two-step self-etch adhesives. Both two-step etch&rinse and self-etch bonded significantly much better than one-step self-etch adhesives. The actual p-values are mentioned in the table insert. All red-colored figures indicate statistical significant difference; black-colored figures indicate absence of statistical difference.*

subjected to similar stresses during finishing of composite restorations with diamonds. They also induce microfractures at the restoration-tooth transition. In this way, µTBS-sample preparation may actually better simulate clinical circumstances. Eventually, if all specimens are prepared in the same manner, no additional variable is introduced. In order to standardize sample preparation, at BIOMAT Leuven, the Iowa MicroSpecimen Former (Armstrong, Keller & Boyer, 2001; De Munck & others, 2003a,c) is used, which

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

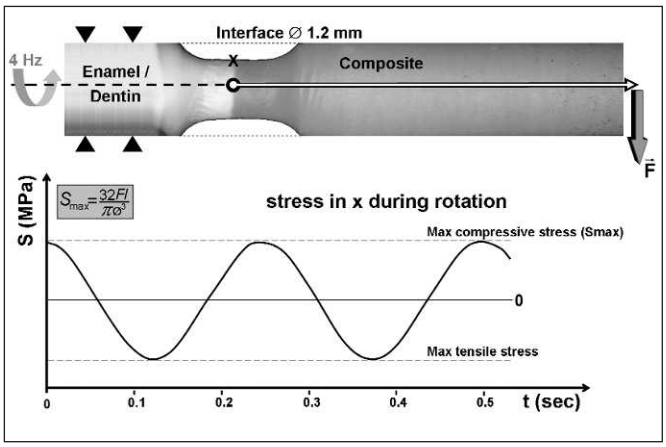


Figure 25. *Schematic explaining the principle of micro-rotary fatigue testing (μRF) and illustrating a specimen prepared for it. The sample is clamped at one end in the specimen grip and at the other end loaded with an adjustable weight. When the specimen is rotated, each spot "x" will be stressed cyclically in tensile and compression. The stress applied diminishes towards the center of the specimen.*

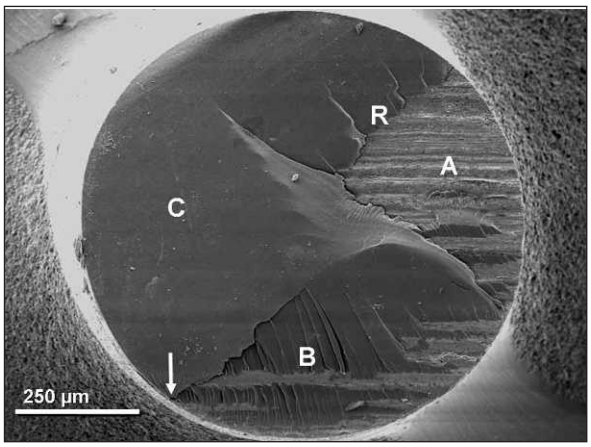


Figure 26. *Fe-SEM photomicrograph illustrating a resin-enamel μRF-specimen prepared using Clearfil SE (Kuraray) that failed after 25680 cycles at 19 MPa. The failure pattern was clearly mixed with "A" representing adhesive failure and "C" cohesive failure in the adhesive. The arrow probably represents the area where the crack was initiated. Typical beach marks are indicated by "B," where "R" represents the rest fracture.*

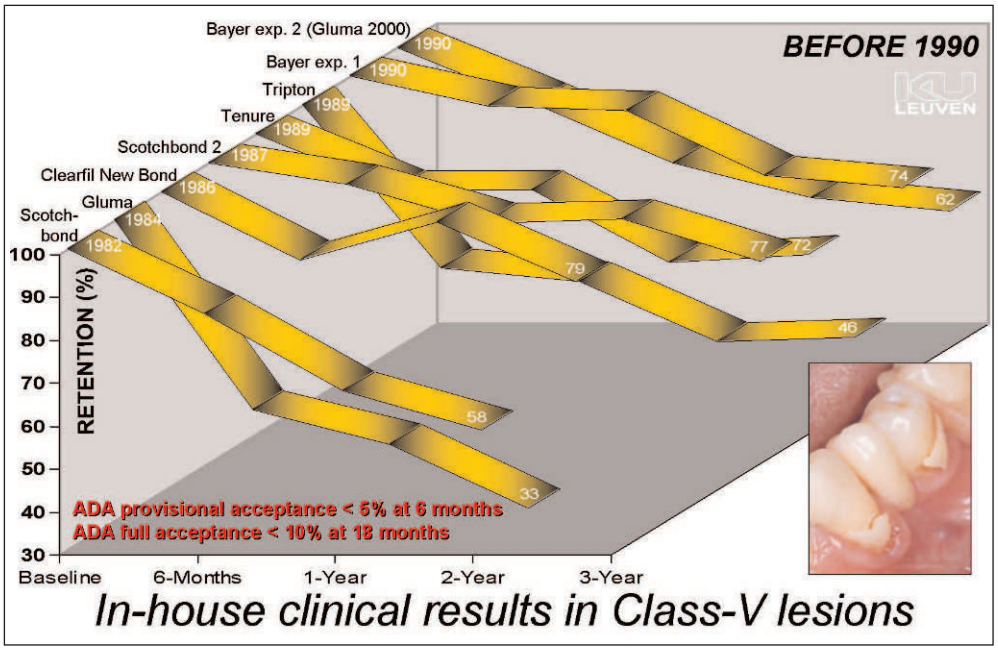


Figure 27. *Chart presenting the retention percentage of Class-V restorations in function of time when diverse adhesive/composite combinations were used to restore non-carious cervical lesions as part of in-house clinical trials, which were started before 1990.*

enables the production of stick-type specimens that are cylindrically constricted at the interface to ensure that the maximum tensile stress is concentrated at the actual interface. Hence, this μTBS-testing protocol must become the new standard for measure bonding effectiveness in the laboratory.

A modification of this test is the "micro-shear" test, which makes it more difficult to standardize the location of the force (Shimada & others, 2000). Nevertheless, the results obtained did not differ substantially from those gathered following a μTBS-protocol (Phrukkanon & others, 1998a,b).

Another less common technique is the push-out test (Frankenberger, Krämer & Petschelt, 1999, 2000b; Frankenberger & others, 2000a). A small resin composite cylinder in the middle of a dentin disc is pushed out, resulting in a shear stress at the interface. The main advantage of this technique is that failure is forced to occur along the adhesive interface (Drummond & others, 1996). However, this test is more time-consuming and cannot be applied for evaluating enamel bond strength. Also, push-out data are very comparable to traditional shear-bond strength testing (Drummond & others, 1996).

μTBS to Enamel—At Leuven, the μTBS of a large group of commercial and experimental adhesives to bur-cut enamel and dentin has been determined (Inoue & others, 2001a,b, 2003; De Munck & others, 2003a,c), always following the same experimental protocol, using one particular composite material (Z100, 3M ESPE). When bonding to enamel, an etch&rinse approach still results in the highest bonding effective-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

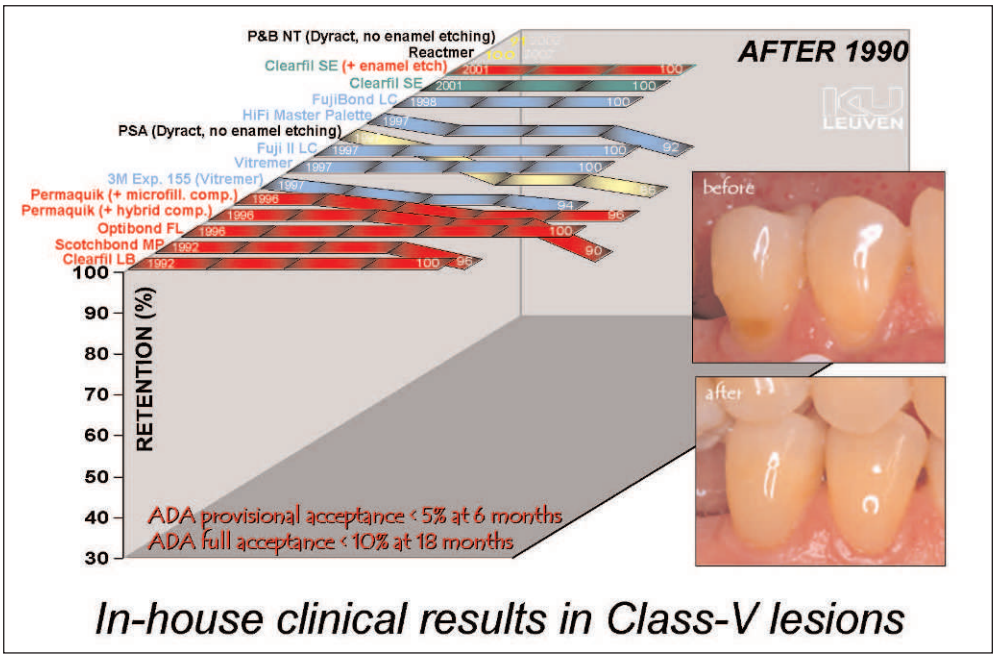


Figure 28. *Chart presenting the retention percentage of Class-V restorations in function of time when diverse adhesive/composite combinations were used to restore non-carious cervical lesions as part of in-house clinical trials, which were started after 1990. The color code refers to the different kinds of adhesives following the classification presented in Figure 1.*

ness irrespective of a two- or three-step procedure and the product tested (Figure 21). When pooling the μTBSs of all etch&rinse adhesives tested (or of which the μTBS was repeatedly measured as part of different studies), a μTBS of 39 and 40 MPa was achieved, respectively, for the three-step and two-step etch&rinse adhesives. As has been known from Buonocore (1955), bonding to enamel only requires an acid-etch step followed by the application of a fluid resin without the need for an intermediary primer step. The latter, on the other hand, does not negatively influence bonding effectiveness and is even mandatory when a "wet-bonding" procedure is carried out.

A self-etch procedure, in general, has resulted in a lower bonding effectiveness, though some adhesives approached the bonding effectiveness of etch&rinse adhesives (Figure 21). A pooled μTBS of about 30 MPa was obtained for two-step self-etch adhesives. The μTBS of the "strong" two-step self-etch adhesive NRC/Prime&Bond NT (Dentsply) was not significantly lower than that of the etch&rinse adhesives. Although Clearfil SE (Kuraray) and the experimental successor Clearfil SE Plus (Kuraray; containing the anti-microbial monomer methacryloyloxydodecylpyridinium bromide or MDPB; Imazato & others, 1997) belong to the group of "mild" two-step self-etch adhesives, their μTBS is not that much lower than etch&rinse adhesives. This may indicate that although they only interact superficially with enamel and, thus, their potential for micromechanical interlocking is much less than a phosphoric-acid treatment, the additional chemical bonding capacity to hydroxyapatite may have contributed to the actual favorable bonding effectiveness. Likewise, the bonding effectiveness of the "intermediary strong" two-step self-etch adhesive Optibond Solo Plus Self-etch (Kerr, Orange, CA, USA) approached that of etch&rinse adhesives.

One-step self-etch adhesives produced significantly lower μTBSs than etch&rinse and two-step self-etch adhesives (Figure 21). The pooled μTBS was about 16 MPa for the one-step self-etch adhesives. The "strong" one-step adhesives, Prompt L-Pop (3M ESPE), and its successor Adper Prompt (3M ESPE), presented with μTBSs in the same range as that recorded for the two-step self-etch adhesives. This most likely can be attributed to their higher acidity and, consequently, higher potential to achieve micromechanical interlocking at enamel.

The glass-ionomer adhesive Fuji Bond LC (GC) performed equally well as the two-step self-etch adhesives (Figure 21). However, during bond-strength testing, the glass-ionomer adhesive tended to fail more frequently in the glass-ionomer material itself than at the actual interface, where their actual bonding effectiveness to enamel was never measured and should at least be higher than the cohesive strength of the glass-ionomer adhesive (Inoue & others, 2000, 2001a).

Statistical analysis of the pooled enamel μTBS data (Figure 22) showed that etch&rinse adhesives, irrespective of a two- or three-step application procedure, bonded slightly but significantly stronger to enamel than two-step self-etch adhesives and significantly more strong than one-step self-etch adhesives.

μTBS to Dentin—At dentin, three-step etch&rinse adhesives still surpassed all other adhesives that use simplified application procedures (Figure 23). No significant difference could be recorded between the bonding effectiveness to dentin of two-step etch&rinse and two-step self-etch adhesives. Again, the "mild" two-step self-etch adhesive Clearfil SE (Kuraray) and the "intermediary strong" two-step self-etch adhesive Optibond Solo Plus Self-etch (Kerr) most closely approached the bonding effectiveness of the conventional three-step adhesives. The lowest μTBS was again

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

recorded for the one-step self-etch adhesives that performed similarly to the glass-ionomer adhesive Fuji Bond LC (GC).

Statistical analysis of the pooled dentin μTBS-data (Figure 24) showed that three-step etch&rinse adhesives bonded significantly more strongly to dentin than two-step etch&rinse and two-step self-etch adhesives. Both latter systems did not perform significantly different from each other. Again, the significantly least favorable μTBS-results were recorded for one-step self-etch adhesives.

μTBS to Hydroxyapatite—As mentioned above, the actual bonding effectiveness of glass ionomers and "mild" self-etch adhesives may result from combined micromechanical and chemical interaction with the tooth substrate. It is, however, currently not known how much chemical interaction contributes to the actual bonding effectiveness. Therefore, the authors determined the μTBS of a various group of adhesive materials to synthetic hydroxyapatite that, besides not having organic collagen, was highly polished and, thus, devoid of mechanical interlocking sites (Van Meerbeek & others, 2003). Among the adhesives tested, all specimens prepared with three-step etch&rinse adhesive Optibond FL (Kerr) failed prior to μTBS-testing (pre-testing failures), proving that any micromechanical retention was excluded. The two-step self-etch adhesive Clearfil SE (Kuraray) presented with a rather low μTBS, along with a high number of pre-testing failures, indicating that there is some chemical interaction that, however slightly (for about 7% as compared to its μTBS to dentin), contributed to the actual bond strength achieved at dentin. Clearly, much less pre-testing failure and a significantly higher μTBS were recorded for the resin-modified glass-ionomer adhesive Fuji Bond LC (GC) and the conventional glass-ionomer restorative material Fuji IX (GC). For Fuji Bond LC, the chemical interaction accounted for about 40% of the actual bond strength achieved at dentin. Chemical bonding of glass ionomer to hydroxyapatite depended greatly on the use of a separate polyalkenoic acid conditioner (Cavity Conditioner (GC); see also Figure 8). Without this pre-treatment, all specimens failed prior to testing. Equally effective as glass-ionomer materials with regard to chemical bonding potential, the resin-based luting material Panavia F (Kuraray) presented with a μTBS that accounted for about 67% of its actual bond strength to dentin. No pre-testing failures were recorded for Panavia F, indicating its relatively strong chemical bonding potential. Although Panavia F was applied following a self-etch approach using a 10-MDP-based primer solution, as in Clearfil SE, its chemical bonding effectiveness is much higher than the two-step self-etch adhesive Clearfil SE. Further in-depth analysis of the actual differences in composition and application procedures should help explain this difference in chemical bonding potential. Finally, the self-adhesive luting material Unicem (3M ESPE) presented with a relatively negligible chemical bonding potential in the same range recorded for the two-step self-etch adhesive (Clearfil SE). Note that the ratio of chemical bonding to eventual "total" bonding effectiveness must be regarded as arbitrary, since differences in substrate properties such as roughness, stiffness and so on between the hydroxyapatite and dentin specimens were ignored.

Effect of Aging—Most current adhesives perform well in bond-strength tests, at least when tested shortly after application and under controlled *in vitro* conditions (Inoue & others, 2001b, 2003; De Munck & others, 2003a,c). However, the oral cavity with temperature changes, chewing loads and chemical attacks by acids and enzymes forms a severe challenge for tooth-composite bonds to survive for a long time. Clinically, marginal deterioration of composite restorations remains problematic and forms the major reason that dramatically shortens the lifetime of adhesive restorations (Van Meerbeek & others, 1998a). A factor known to degrade tooth-composite bonds is exposure to water (Gwinnett & Yu, 1995; Sano & others, 1999, Armstrong & others, 2001). Among different forms of marginal leakage, nano-leakage, or the ingress of oral fluids through nanometer-sized channels along collagen fibrils within the hybrid layer, is considered detrimental to the bond integrity (Sano & others, 1995; Hashimoto & others, 2000, 2002).

In a recent paper (De Munck & others, 2003c), the authors studied the long-term degradation of resin-dentin bonds using a μTBS-testing methodology through exposure to water for four years, either directly or indirectly, when the resin-dentin interface was surrounded by resin bonded to enamel. The microtensile bond strength (μTBS) to dentin of two three-step etch&rinse adhesives (Optibond Dual-Cure, Kerr; Scotchbond Multi-Purpose, 3M ESPE) was compared to two two-step etch&rinse adhesives (Optibond Solo, Kerr; Scotchbond 1, 3M ESPE) after four years of storage in water. Direct exposure to water resulted in a significant decrease in the μTBS of the two-step but not of the three-step etch&rinse adhesives. Indirect exposure to water did not significantly reduce the μTBS of any adhesive, indicating that resin bonded to enamel protected the resin-dentin bond against degradation. This means that, in the clinical situation, one can rely on durable dentin bonding using three- or two-step etch&rinse adhesives if all cavity margins are located in enamel. For cavities with margins ending in dentin, three-step total-etch adhesives are preferred.

Marginal Sealing Effectiveness

Clinically, early loss of restoration is no longer a clinical problem when reliable (mostly conventional three-step etch&rinse) adhesives are used, even long-term (Van Meerbeek & others, 1994, 1998a; van Dijken,

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

2000a,b, 2001, 2002; De Munck & others, 2003b). However, marginal leakage and consequent marginal discoloration remains clinically the most frequent reason to replace/repair an adhesive restoration. Therefore, besides bond-strength testing, testing the marginal sealing effectiveness of adhesives is needed.

Marginal leakage has been defined as the clinically undetectable passage of bacteria, fluids, molecules or ions between a cavity wall and the restorative material applied to it (Kidd, 1976). All resin-based restorative materials shrink, which introduces shrinkage stress, pulling the adhesive away from the cavity wall, which may eventually form a gap. Today's adhesives are incapable of completely sealing restoration margins and, thus, preventing microleakage long-term (Pilo & Ben-Amar, 1999; Bouillaguet & others, 2000; Ceballos & others, 2001). Many methodologies have been introduced to assess microleakage and can be further subdivided in qualitative, semi-quantitative or true quantitative measurements of sealing effectiveness.

Qualitative Measurement of Sealing Effectiveness—The use of organic dyes as tracers is one of the oldest, most common methods of detecting leakage *in vitro*. A number of dyes varying in particle size and affinity to substrates have been used and are known to significantly influence microleakage results (Alani & Toh, 1997). In general, this method involves immersion of a restored tooth into a dye solution after having coated the unrestored tooth parts covered with a waterproof varnish until close to the restoration margin. After a certain time interval, the specimens are washed and sectioned into two or more slices to visually determine the extent of dye penetration along the restoration margin (Alani & Toh, 1997). The main problem is that this methodology basically is a qualitative evaluation method. It can be made semi-quantitative by applying a non-parametric scale (Castelnuovo, Tjan & Liu, 1996).

Semi-Quantitative Measurement of Sealing Effectiveness or Marginal Analysis—A number of *in vitro* studies have tested the performance of adhesives by semi-quantitatively evaluating by using SEM the marginal gap formation around restorations placed in extracted teeth (Roulet & others, 1989; Krejci, Kuster & Lutz, 1993; Roulet, 1994; Gladys & others, 1995; Blunck, Neumann & Roulet, 2000; Blunck & Roulet, 1999, 2002). This method assumes that if the forces generated during shrinkage or thermo-mechanical strains exceed the bond strength to enamel/dentin, an observable gap will form at the margin of the restoration. Although the literature also lacks clear evidence of any correlation of gap formation *in vitro* with the interfacial failures observed *in vivo*, it is reasonable to assume that this semi-quantitative mar-ginal gap analysis is clinically relevant (Roulet, 1994), certainly, when measurements are repeated after thermocycling (Krejci & others, 1993, Schuckar & Geurtsen, 1997).

Blunck and Roulet (2002) have semi-quantitatively analyzed the marginal adaptation of cervical restorations for a diverse group of adhesives, consistently following the same experimental protocol. Basically, their results correlated well with the μTBS-data recorded by the authors of this study at BIOMAT Leuven. After one-year water storage and two thermocycling sessions, still on average, 93% of the restoration margin length was gap-free for the three-step etch&rinse adhesive Optibond FL (Kerr) and 91% for the "mild" two-step self-etch adhesive Clearfil SE (Kuraray) (Blunck & Roulet, 2002). Two-step etch&rinse adhesives such as Excite (Vivadent), Optibond Solo Plus (Kerr) and Scotchbond 1 (3M ESPE) revealed significantly lower percentages of gap-free margin lengths of 80%, 82% and 63%, respectively. Less than half (48%) of the margin length was gap-free for the "strong" one-step self-etch adhesive Prompt-L Pop (3M ESPE).

Quantitative Measurement of Sealing Effectiveness or Flow Measurement—A quantitative method to assess microleakage is to measure the flow along the interface (Pagliarini & others, 1996) or from the pulp to a sealed dentin surface (Derkson, Pashley & Derkson, 1986; Del-Nero, Escribano & de la Macorra, 2000; Bouillaguet & others, 2000). The marginal sealing effectiveness is quantified using a "Flodec device" (De Marco Engineering, Geneva, Switzerland). The adhesively-restored tooth is brought under pressure with water from inside the dental pulp. The permeability of the tooth-restoration interface is then quantitatively determined through accurate measurement of the displacement of an air bubble within a water-filled micro-pipet (∅=0.7 mm) using a computer-driven optical system (Flodec device). The main advantages of this method are that it is fully quantitative and that the specimens can be longitudinally followed since it is a non-destructive method. However, one major problem using this technique is that leakage may also occur through the dental substrate itself and, thus, falsely increase the leakage values.

Nanoleakage—Sano and others (1994b, 1995) revealed that leakage can occur between the hybrid layer and intact dentin, even in the absence of a marginal gap. This leakage was assessed using Ag-ions that are extremely small (0.059 nm). It is hypothesized that it represents permeation through demineralized submicron spaces that have not been filled with adhesive resin (Sano & others, 1995). These voids are so small that bacteria may not be able to pass through, but these spaces may be more susceptible to degrada-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

tion by water and bacterial side products such as acids and enzymes (Paul & others, 1999). This phenomenon can be quantitatively assessed by measuring the dye penetration depth using, for instance, confocal laser scanning microscopy (Dörfer & others, 2000; Pioch & others, 2001, 2002) or TEM (Tay & others, 2002b).

Dynamic Fatigue Testing

Bonding effectiveness to tooth tissue is typically measured statically, for example, by shear bond or microtensile bond strength (μTBS) testing (see above). In the clinical situation, however, tooth-composite bonds are seldom imposed to such acute tensile/shear stresses. During its lifetime, a restoration is subjected to cyclic loading, each load is insufficient to provoke failure, but in the long-term, can possibly lead to marginal deterioration and loss of the restoration. Therefore, fatigue testing of dental adhesives is expected to better predict their *in vivo* performance.

There is, however, no standard fatigue test for dental adhesives. Possible methods are a cyclic shear test (Ruse, Shew & Feduik, 1995; Drummond & others, 1996; Dewji & others, 1998), a cyclic tensile test (Aquilino, Diaz-Arnold & Piotrowski, 1991; Givan & others, 1995), a cyclic fracture toughness test (Destoop, 2002) or a cyclic push-out test (Frankenberger & others, 1999). Another possibility is loading not only the interface but the whole tooth until the tooth-restoration complex fails (Fissore, Nicholls & Yuodelis, 1991).

At BIOMAT Leuven, the authors have developed a micro-rotary fatigue device that dynamically tests tooth-composite interfaces (De Munck & others, 2002). A macro-version was used prior to determine the fatigue resistance of soldered joints (Wiskott, Nicholls & Belser, 1994). In our test set-up, standard microtensile bond strength (μTBS) bar-type samples prepared with a rounded, constricted interface (Figure 25 and 26) were clamped in a pin-chuck and connected to a stepping motor with the free end loaded with a certain weight. By rotating the specimen, each spot at the outer surface of the interface underwent successively compressive and tensile loading following a sinusoidal function (Wiskott & others, 1994). Depending on the survival/failure of each sample after 10^5 cycles, the load imposed to the next sample was increased/decreased with ± 5%. The results of the fatigue test were analyzed using a logistic regression to determine the load at which 50% of the samples failed and was called the median micro-Rotary Fatigue Resistance (μRFR). In a pilot study, the μRFR of the three-step etch&rinse adhesive Optibond FL (Kerr) and the two-step self-etch adhesive Clearfil SE (Kuraray) to enamel and dentin was determined. The ranking of median μRFRs was in accordance with the ranking of the respective μTBSs obtained for the two adhesives bonded to enamel and dentin. They were about three-fourths of the respective μTBSs, except for Optibond FL bonded to dentin, which appeared to lose more of its static bond strength when tested dynamically. From this preliminary study, it could be concluded that fatiguing of tooth-composite interfaces is feasible, with consistent results provided. Because of the cyclic loading and high number of cycles (10^5), the resulting data might also be more clinically relevant, especially for assessing long-term bonding effectiveness, which is still a major shortcoming of contemporary adhesives.

Clinical Testing of Adhesives

New adhesives are continually being introduced to the dental profession, unfortunately, often without sufficient clinical validation (Van Meerbeek & others, 1998a, 2001a). In the mouth, multiple and mutually-interactive clinical variables related to the quality of tooth substrate and its immediate oral environment co-determine the eventual effectiveness of adhesives (Van Meerbeek & others, 1994). Adhesives have mainly been clinically tested in non-prepared cervical abrasions and erosions. Such "model" lesions are ideal test cavities, because they are located mainly in dentin and are widely available. They present no macro-mechanical undercuts, and they are usually found in anterior teeth or premolars with good access and in patients who have better than average oral hygiene. However, such clinical trials are limited in number and require several years with regular recalls in order to achieve sufficient clinical validation. Nevertheless, the more expensive and long-lasting clinical trials remain necessary to validate laboratory observations. Laboratory testing on near ideal substrates and under optimal *in vitro* conditions is valuable as a pre-clinical screening test of adhesive materials, at best, only a good prediction of clinical performance. Most Class-V clinical trials run for three years, although longer follow-up times may be desirable. However, after three years, most adhesives are outdated and are replaced by a successor that claims to be better.

At Leuven, the clinical effectiveness of adhesives has been routinely investigated in controlled follow-up studies using the same experimental protocol for almost 20 years. The clinical effectiveness of modern adhesives has significantly improved, allowing adhesive restorations to be placed with a high predictable level of clinical success. Most modern adhesive systems are superior to their predecessors, especially in terms of retention, making it no longer the main cause of premature clinical failure. This must, in part, be attributed to the introduction in the early 1990s of the *"total-etch"* (now referred to as "etch&rinse") technique, by which phosphoric acid is also applied to dentin. Earlier adhesives often showed many failures within the first six months when applied strictly to dentin without any selective phosphoric acid-etching of adjacent enamel (Figure 27). When following the same protocol in more

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

recent clinical trials (etch&rinse systems applied selectively to dentin), almost any early debonding failures were recorded (Figure 28). This must, to a great extent, be attributed to the enamel immediately adjacent to dentin always being (unintentionally) etched and the guarantee of a durable bond to the enamel margin. Adequate bonding to enamel, alone, may also keep such restorations in place. Nevertheless, bonding to dentin has improved substantially. However, in order to be considered clinically effective, adhesive systems should not only keep the restoration in place for a significant period of time, but also, and what clinically may even be more important, completely seal the restoration margins against the ingress of oral fluids and microorganisms. However, none of today's systems yet appears able to guarantee leakage-free margins for a significant amount of time, especially at the dentin site (Van Meerbeek & others, 1998a; De Munck & others, 2003b).

At Leuven, the excellent clinical performance of the three-step etch&rinse adhesive Optibond FL (Kerr), with a 100% retention rate at five years, is noteworthy (De Munck & others, 2003b). Likewise, 96% of the restorations were still in place at five years when the three-step etch&rinse adhesive Permaquick (Ultradent, South Jordan, UT, USA) was used (De Munck & others, 2003b). Besides the favorable clinical performance of etch&rinse adhesives, glass ionomers commonly present with high retention results, even up to three years of clinical service (Figure 28). In a recent double-blind, split-mouth, randomized controlled clinical trial, the clinical effectiveness of the "mild" two-step self-etch adhesive Clearfil SE (Kuraray) was evaluated following two experimental protocols (Peumans & others, 2003). Clearfil SE was applied either following the manufacturer's instructions or including prior selective acid etching of the enamel cavity margins with 40% phosphoric acid. At two years, no restoration losses were recorded for either experimental group (Figure 28). Besides a higher tendency toward small (but of clinically negligible relevance) marginal defects at the enamel side (when enamel was not etched beforehand with phosphoric acid), the "mild" self-etch approach of Clearfil SE still appears to be a clinically reliable, predictable and simplified adhesive technique.

In general, two-step etch&rinse adhesives perform clinically less favorably than conventional three-step adhesives (Sunnegardh & van Dijken, 2000; van Dijken, 2000a). For instance, still favorable seven-year retention rates of 84% and 79%, respectively, were recorded for the three-step etch&rinse adhesives Clearfil Liner Bond (Kuraray) and Optibond Dual-Cure (Kerr) (van Dijken, 2001). Two-step etch&rinse adhesives generally perform clinically less favorably in Class-V lesions. The results reported for this group vary more among the different research centers, which is probably indicative of their higher technique sensitivity. For instance, only 45% of the acetone-based adhesive One-Step (BISCO, Schaumberg, IL, USA) were retained at five years (van Dijken, 2001), and only 52% of Gluma 2000 (Kulzer) at five years (van Dijken, 2000a). Also, 25% of the Scotchbond 1 restorations were already lost at the three-year recall in a study by van Dijken (2001), while only 3% were lost at three years in a study by Ripps, Burgess and Rappold (2000). A loss rate of only 7% was recorded for Optibond Solo at three years (Swift & others, 2001). At three years, excellent and reasonably good clinical effectiveness was reported for Prime&Bond 2.1 (Dentsply), with a retention rate of 100% at three years by Martin, Jedynakiewicz and Fletcher (2002), and 89% at three years by Swift and others (2001).

Regarding the clinical effectiveness of two-step self-etch adhesives, less data is available in the literature. Latta and others (2000) reported a still favorable 92% retention rate at three years for Clearfil Liner Bond 2 and van Dijken (2002) reported a 91% retention rate at two years.

Finally, regarding one-step self-etch adhesives, strongly varying results were recorded for PSA (applied along with Dyract, Dentsply). Only a 5% loss rate at five years was reported by Folwaczny and others (2001), whereas, even 41% of the restorations placed using PSA (Dentsply) de-bonded within a four-year observation period, as reported by Unlu, Belli and Ozer (2001). A rather favorable retention rate of 84% at five years was reported by van Dijken (2000a). Several studies reported on the clinical performance of Prompt L-Pop (3M ESPE). Rather favorable short-term retention rates of 100% at six months and 96% at one year, respectively, were recorded by Munoz and others (2001) and by Boghosian (2002). However, relatively high loss rates of 21% at two years and 35% at one year were reported, respectively, by van Dijken (2002) and Brackett, Covey and St Germain (2002).

CONCLUSIONS

A great diversity in laboratory testing of adhesives exists. Modern determination of bonding effectiveness in the laboratory should involve (1) microtensile bond strength testing, (2) sealing effectiveness testing using semi-quantitative marginal analysis or fully quantitative margin permeability measurement and possibly (3) dynamic fatigue testing. There is a lack of standardization of testing methodologies. Nevertheless, good correlation exists between laboratory and clinical effectiveness, by which it can be concluded that laboratory testing CAN predict clinical effectiveness.

Diverse types of adhesives exist which can be classified following their bonding mechanism and clinical

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

application approach into etch&rinse, glass-ionomer and self-etch adhesives. Although there is a tendency toward adhesives with simplified application procedures, simplification does not guarantee equal or improved bonding effectiveness. Three-step etch&rinse adhesives still perform best in laboratory and clinical research. Because of an additional chemical bonding potential to hydroxyapatite, the mild self-etch approach may be most promising in terms of durable bonding to dental hard tissue using a simple, low, technique-sensitive application technique.

Acknowledgements

The Buonocore Memorial lecture is supported by a grant from Caulk Dental Manufacturing Co to the Academy of Operative Dentistry.

(Presented 27 February 2003)

References

Alani AH & Toh CG (1997) Detection of microleakage around dental restorations: A review *Operative Dentistry* **22(4)** 173-185.

Armstrong SR, Keller JC & Boyer DB (2001) The influence of water storage and C-factor on the dentin-resin composite microtensile bond strength and debond pathway utilizing a filled and unfilled adhesive resin *Dental Materials* **17(3)** 268-276.

Aquilino SA, Diaz-Arnold AM & Piotrowski TJ (1991) Tensile fatigue limits of prosthodontic adhesives *Journal of Dental Research* **70(3)** 208-210.

Blunck U, Neumann O & Roulet J-F (2000) Marginal adaptation of four one-bottle-adhesives depending on the application technique *Journal of Dental Research* **79(Special Issue)** Abstract #41 p 149.

Blunck U & Roulet JF (1999) Marginal adaptation of compomer Class V restorations *in vitro Journal of Adhesive Dentistry* **1(2)** 143-151.

Blunck U & Roulet JF (2002) Effect of one-year water storage on the effectiveness of dentin adhesives in Class V composite resin restorations *Journal of Dental Research* **81(Special Issue A)** Abstract #0946 p A-139.

Boghosian A (2002) Clinical evaluation of a self-etching adhesive: 1 year results *Journal of Dental Research* **81(Special Issue A)** Abstract #0192 p A-52.

Bouillaguet S, Duroux B, Ciucchi B & Sano H (2000) Ability of adhesive systems to seal dentin surfaces: An *in vitro* study *Journal of Adhesive Dentistry* **2(3)** 201-208.

Bouschlicher MR, Reinhardt JW & Vargas MA (1997) Surface treatment techniques for resin composite repair *American Journal of Dentistry* **10(6)** 279-283.

Brackett WW, Covey DA & St Germain HA Jr (2002) One-year clinical performance of a self-etching adhesive in Class V resin composites cured by two methods *Operative Dentistry* **27(3)** 218-222.

Buonocore DH (1955) A simple method of increasing the adhesion of acryl filling materials to enamel surfaces *Journal of Dental Research* **34** 849-853.

Castelnuovo J, Tjan AHL & Liu P (1996) Microleakage of multistep and simplified-step bonding systems *American Journal of Dentistry* **9(6)** 245-248.

Ceballos L, Osorio R, Toledano M & Marshall GW (2001) Microleakage of composite restorations after acid or Er:YAG laser cavity treatments *Dental Materials* **17(4)** 340-346.

Degrange H & Roulet JF (1997) Minimally invasive dentistry with bonding Chicago Quintessence Publishing.

Del-Nero MO, Escribano N & de la Macorra JC (2000) Analysis of sealing vs tensile bond strength of eight adhesive restorative material systems *Journal of Adhesive Dentistry* **2(2)** 117-127.

De Munck J, Van Meerbeek B, Inoue S, Vargas M, Yoshida Y, Armstrong S, Lambrechts P & Vanherle G (2003a) Micro-tensile bond strength of one- and two-step self-etch adhesives to bur-cut enamel and dentin *American Journal of Dentistry* (in press).

De Munck J, Van Meerbeek B, Peumans M & Lambrechts P (2003b) Five-year clinical effectiveness of two three-step total-etch adhesives and two composites in cervical lesions *Journal of Dental Research* **82(Special Issue)** Abstract 0907 (in press).

De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, Lambrechts P & Vanherle G (2003c) Four-year water degradation of total-etch adhesives bonded to dentin *Journal of Dental Research* **82(2)** 136-140.

De Munck J, Van Meerbeek B, Lambrechts P & Braem M (2002) Micro-rotary fatigue of tooth-biomaterial interfaces in Programme and abstracts 2002, *European Festival of Oral Science*, Cardiff, Wales, September 25-28, 2002 Abstract #406.

Denehy G, Bouschlicher M & Vargas M (1998) Intraoral repair of cosmetic restorations *Dental Clinics of North America* **42(4)** 719-737.

Derkson GD, Pashley DH & Derkson ME (1986) Microleakage measurement of selected restorative materials: A new *in vitro* method *Journal of Prosthetic Dentistry* **56(4)** 435-440.

Destoop V (2002) The cracking resistance and adhesion to dentine of dental restorative materials: A fracture mechanics approach Université Catholique de Louvain Doctoral dissertation.

Dewji HR, Drummond JL, Fadavi S & Punwani I (1998) Bond strength of BIS-GMA and glass ionomer pit and fissure sealants using cyclic fatigue *European Journal of Oral Sciences* **106(1)** 594-599.

Dörfer CE, Staehle HJ, Wurst MW, Duschner H & Pioch T (2000) The nanoleakage phenomenon: Influence of different dentin bonding agents, thermocycling and etching time *European Journal of Oral Sciences* **108(4)** 346-351.

Drummond JL, Sakaguchi RL, Racean DC, Wozny J & Steinberg AD (1996) Testing mode and surface treatment effects on dentin bonding *Journal of Biomedical Materials Research* **32(4)** 533-541.

Ferrari M & Cardoso PEC (2002) SEM evaluation of microtensile sample integrity before being tested *Journal of Dental Research* **81(Special Issue A)** Abstract #0951 p A-139.

Fissore B, Nicholls JI & Yuodelis RA (1991) Load fatigue of teeth restored by a dentin bonding agent and a posterior composite resin *Journal of Prosthetic Dentistry* **65(1)** 80-85.

Folwaczny M, Mehl A, Kunzelmann KH & Hickel R (2001) Clinical performance of a resin-modified glass-ionomer and a compomer in restoring non-carious cervical lesions. 5-year results *American Journal of Dentistry* **14(3)** 153-156.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Frankenberger R, Krämer N & Petschelt A (1999) Fatigue behaviour of different dentin adhesives *Clinical Oral Investigation* **3(1)** 11-17.

Frankenberger R, Kramer N, Oberschachtsiek H & Petschelt A (2000a) Dentin bond strength and marginal adaption after NaOCl pre-treatment *Operative Dentistry* **25(1)** 40-45.

Frankenberger R, Kramer N & Petschelt A (2000b) Technique sensitivity of dentin bonding: Effect of application mistakes on bond strength and marginal adaptation *Operative Dentistry* **25(4)** 324-330.

Fukuda R, Yoshida Y, Nakayama Y, Okazaki M, Inoue S, Sano H, Shintani H, Snauwaert J & Van Meerbeek B (2003) Bonding efficacy of polyalkenoic acids to hydroxyapatite, enamel and dentin *Biomaterials* (in press).

Givan DA, Fitchie JG, Anderson L & Zardiackas LD (1995) Tensile fatigue of a 4-META cement bonding three base metal alloys to enamel and comparison to other resin cements *Journal of Prosthetic Dentistry* **73(4)** 377-385.

Gladys S, Van Meerbeek B, Inokoshi S, Willems G, Braem M, Lambrechts P & Vanherle G (1995) Clinical and semi-quantitative marginal analysis of four tooth-coloured inlay systems at three years *Journal of Dentistry* **23(6)** 329-338.

Gwinnett AJ & Yu S (1995) Effect of long-term water storage on dentin bonding *American Journal of Dentistry* **8(2)** 109-111.

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, Kudoi Y, Oguchi H, Araki Y & Kuboto M (2002) Micromorphological changes in resin-dentin bonds after 1 year of water storage *Journal of Biomedical Materials Research* **63(3)** 306-311.

Imazato S, Kinomoto Y, Tarumi H, Torii M, Russell RR & McCabe JF (1997) Incorporation of antibacterial monomer MDPB into dentin primer *Journal of Dental Research* **76(3)** 768-772.

Inoue S, Van Meerbeek B, Abe Y, Yoshida Y, Lambrechts P, Vanherle G & Sano H (2001a) Effect of remaining dentin thickness and the use of conditioner on micro-tensile bond strength of a glass-ionomer adhesive *Dental Materials* **17(5)** 445-455.

Inoue S, Van Meerbeek B, Vargas M, Lambrechts P & Vanherle G (2000) Adhesion mechanism of self-etching adhesives in Tagami J, Toledano M, Prati C (eds) *Advanced Adhesive Dentistry Proceedings* book of the Granada 3rd International Kuraray Symposium: December 3-4, 1999, Cirimido: Grafiche Erredue p 131-148.

Inoue S, Vargas MA, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, Sano H & Van Meerbeek B (2001b) Micro-tensile bond strength of eleven contemporary modern adhesives to dentin *Journal of Adhesive Dentistry* **3(3)** 237-245.

Inoue S, Vargas MA, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, Sano H & Van Meerbeek B (2003) Micro-tensile bond strength of eleven modern adhesives to enamel *American Journal of Dentistry* (in press).

Kidd EAM (1976) Microleakage: A review *Journal of Dentistry* **4(5)** 199-206.

Krejci I, Kuster M & Lutz F (1993) Influence of dentinal fluid and stress on marginal adaptation of resin composites *Journal of Dental Research* **72(2)** 490-494.

Kwong SM, Cheung GS, Kei LH, Itthagarun A, Smales RJ, Tay FR & Pashley DH (2002) Micro-tensile bond strengths to sclerotic dentin using a self-etching and a total-etching technique *Dental Materials* **18(5)** 359-369.

Latta MA, Barkmeier WW, Triolo PT & Cavel WT (2000) Three-year clinical evaluation of the Clearfil Liner Bond 2 system *Journal of Dental Research* **79(Special Issue)** Abstract #1030 p 272.

Martin N, Jedynakiewicz NM & Fletcher JM (2002) A clinical evaluation of a new compomer restorative at 3 years in the restoration of non-retentive cervical cavities *Journal of Dental Research* **81(Special Issue A)** Abstract #0195 p A-52.

Munoz CA, Dunn JR, Bernal G, Torres J & Wilson A (2001) Clinical evaluation of Prompt L-Pop at 6 months *Journal of Dental Research* **80(Special Issue)** Abstract #237 p 65.

Nakajima M, Sano H, Burrow MF, Tagami J, Yoshiyama M, Ebisu S, Ciucchi B, Russell CM & Pashley DH (1995) Tensile bond strength and SEM evaluation of caries-affected dentin using dentin adhesives *Journal of Dental Research* **74(10)** 1679-1688.

Pagliarini A, Rubini R, Rea M, Campese C & Grandini R (1996) Effectiveness of the current enamel-dentinal adhesives: A new methodology for its evaluation *Quintessence International* **27(4)** 265-270.

Pashley DH & Tay FR (2001) Aggressiveness of contemporary self-etching adhesives. Part II: Etching effects on unground enamel *Dental Materials* **17(5)** 430-444.

Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA & Tay F (1999) The microtensile bond test: A review *Journal of Adhesive Dentistry* **1(4)** 299-309.

Paul SJ, Welter DA, Ghazi M & Pashley D (1999) Nanoleakage at the dentin adhesive interface vs microtensile bond strength *Operative Dentistry* **24(3)** 181-188.

Peumans M, Van Meerbeek B, De Munck J & Lambrechts P (2003) Two-year clinical effectiveness of a self-etch adhesive in cervical lesions *Journal of Dental Research* **82(Special Issue)** Abstract #0911 (in press).

Phrukkanon S, Burrow MF & Tyas MJ (1998a) Effect of cross-sectional surface area on bond strengths between resin and dentin *Dental Materials* **14(2)** 120-128.

Phrukkanon S, Burrow MF & Tyas MJ (1998b) The influence of cross-sectional shape and surface area on the microtensile bond test *Dental Materials* **14(3)** 212-221.

Pilo R & Ben-Amar A (1999) Comparison of microleakage for three one-bottle and three multiple-step dentin bonding agents *Journal of Prosthetic Dentistry* **82(2)** 209-213.

Pioch T, Staehle HJ, Duschner H & García-Godoy F (2001) Nanoleakage at the composite-dentin interface: A review *American Journal of Dentistry* **14(4)** 252-258.

Pioch T, Staehle HJ, Wurst M, Duschner H & Dorfer C (2002) The nanoleakage phenomenon: Influence of moist vs dry bonding *Journal of Adhesive Dentistry* **4(1)** 23-30.

Ripps A, Burgess J & Rappold A (2000) Clinical evaluation of self etching primer with compomer restorative material *Journal of Dental Research* **79(Special Issue)** Abstract #1035 p 273.

Roeters JJ (2000) A simple method to protect patient and environment when using sandblasting for intraoral repair *Journal of Adhesive Dentistry* **2(3)** 235-238.

Roulet J-F (1994) Marginal integrity: Clinical significance *Journal of Dentistry* **22(Supplement 1)** 9-12.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Roulet J-F, Reich T, Blunk U & Noack M (1989) Quantitative margin analysis in the scanning electron microscope *Scanning Microscopy* **3(1)** 147-158.

Ruse ND, Shew R & Feduik D (1995) *In vitro* fatigue testing of a dental bonding system on enamel *Journal of Biomedical Materials Research* **29(3)** 411-415.

Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R & Pashley DH (1994a) Relationship between surface area for adhesion and tensile bond strength—Evaluation of a micro-tensile bond test *Dental Materials* **10(4)** 236-240.

Sano H, Shono T, Takatsu T & Hosoda H (1994b) Microporous dentin zone beneath resin-impregnated layer *Operative Dentistry* **19(2)** 59-64.

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.

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.

Schreiner RF, Chappell RP, Glaros AG & Eick JD (1998) Microtensile testing of dental adhesives *Dental Materials* **14(3)** 194-201.

Schuckar M & Geurtsen W (1997) Proximo-cervical adaptation of Class II-composite restorations after thermocycling: A quantitative and qualitative study *Journal of Oral Rehabilitation* **24(10)** 766-775.

Shimada Y, Antonucci JM, Schumacher GE, McDonough WG & Tagami J (2000) Effects of regional tooth structure and sectioning orientation on micro-shear bond strength in Tagami J, Toledano M & Prati C (eds) *Advanced Adhesive Dentistry Proceedings* book of the Granada 3rd International Kuraray Symposium: December 3-4, 1999, Cirimido: Grafiche Erredue p 91-103.

Shono Y, Terashita M, Shimada J, Kozono Y, Carvalho RM, Russell CM & Pashley DH (1999) Durability of resin-dentin bonds *Journal of Adhesive Dentistry* **1(3)** 211-218.

Sunnegardh K & van Dijken JWV (2000) Three-year evaluation of 3 dentin bonding systems *Journal of Dental Research* **79(Special Issue)** Abstract #1032 p 272.

Swift EJ Jr, Perdigão J, Wilder AD Jr, Heymann HO, Sturdevant JR & Bayne SC (2001) Clinical evaluation of two one-bottle dentin adhesives at three years *Journal of the American Dental Association* **132(8)** 1117-1123.

Tay FR, Smales RJ, Ngo H, Wei SH & Pashley DH (2001) Effect of different conditioning protocols on adhesion of a GIC to dentin *Journal of Adhesive Dentistry* **3(2)** 153-167.

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

Tay FR, Pashley DH & Yoshiyama M (2002b) Two modes of nanoleakage expression in single-step adhesives *Journal of Dental Research* **81(7)** 472-476.

Tay FR, Kwong SM, Itthagarun A, King NM, Yip HK, Moulding KM & Pashley DH (2000) Bonding of a self-etching primer to non-carious cervical sclerotic dentin: Interfacial ultrastructure and microtensile bond strength evaluation *Journal of Adhesive Dentistry* **2(1)** 9-28.

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.

Tyas MJ, Anusavice KJ & Frencken JE (2000) Minimal intervention dentistry—a review *International Dental Journal* **50(1)** 1-12.

Unlu N, Belli S & Ozer F (2001) Clinical evaluation of a compomer resin in Class V abrasion lesions: Four year results *Journal of Dental Research* **81(Special Issue B)** Abstract #27 p B-236.

van Dijken JW (2000a) Clinical effectiveness of 12 adhesive systems in Class-V non-retentive lesions. A 5-year evaluation in Programme and abstracts of the 4th joint meeting of CED and NOF of IADR Warsaw, Poland, August 24-27, 2000 Abstract #42.

van Dijken JW (2000b) Clinical evaluation of three adhesive systems in Class V non-carious lesions *Dental Materials* **16(4)** 285-291.

van Dijken JW (2001) Data presented at symposium held during the CED-IADR meeting in Rome, Italy, September 2001.

van Dijken JWV (2002) Simplified adhesive systems in Class V non-carious cervical dentin lesions in Programme and abstracts of the European Festival of Oral Science, Cardiff, Wales, September 25-28, 2002 Abstract #8.

Van Meerbeek B, Conn L Jr, Duke ES, Robinson SJ, Eick JD & Guerrero D (1996) Correlative transmission electron microscopy examination of non-demineralized and demineralised resin-dentin interface formed by two dentin adhesive systems *Journal of Dental Research* **75(3)** 879-888.

Van Meerbeek B, De Munck J, Yoshida Y, Shirai K, Inoue S, Shintani H & Lambrechts P (2003) Chemical bonding potential of adhesive materials to hydroxyapatite *Journal of Dental Research* **82(Special Issue)** (abstract in press).

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

Van Meerbeek B, Peumans M, Verschueren M, Gladys S, Braem M, Lambrechts P & Vanherle G (1994) Clinical status of ten dentin adhesive systems *Journal of Dental Research* **73(11)** 1690-1702.

Van Meerbeek B, Vargas S, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001a) Adhesives and cements to promote preservation dentistry *Operative Dentistry* **(Supplement 6)** 119-144.

Van Meerbeek B, Yoshida Y, Inoue S, Fukuda R, Okazaki M, Lambrechts P & Vanherle G (2001b) Interfacial characterization of resin-modified glass-ionomers to dentin *Journal of Dental Research* **80(Special Issue)** Abstract #1701 p 739.

Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Abe Y, Fukuda R, Okazaki M, Lambrechts P & Vanherle G (2000) Bonding mechanism and micro-tensile bond strength of a 4-MET-based self-etching adhesive *Journal of Dental Research* **79(Special Issue)** Abstract #845 p 249.

Van Meerbeek B, Yoshida Y, Lambrechts P & Vanherle G (1998b) Mechanisms of bonding of a resin-modified glass-ionomer adhesive to dentin *Journal of Dental Research* **77(Special Issue)** Abstract #2236 p 911.

Van Meerbeek B, Yoshida Y, Lambrechts P, Vanherle G, Duke ES, Eick JD & Robinson SJ (1998c) A TEM study of two water-based adhesive systems bonded to dry and wet dentin *Journal of Dental Research* **77(1)** 50-59.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Van Noort R, Noroozi S, Howard IC & Cardew G (1989) A critique of bond strength measurements *Journal of Dentistry* **17(2)** 61-67.

Versluis A, Tantbirojn D & Douglas WH (1998) Do dental composites always shrink toward the light? *Journal of Dental Research* **77(6)** 1435-1445.

Wilson NHF, Setcos JC & Brunton P (2001) Replacement or repair of dental restorations in Wilson NHF, Roulet JF, Fuzzi M (eds) Advances in *Operative Dentistry*. Challenges of the future Chicago Quintessence Publishing p 105-115.

Wiskott AHW, Nicholls JI & Belser UC (1994) Fatigue resistance of soldered joints: A methodological study *Dental Materials* **10(3)** 215-220.

Yip HK, Tay FR, Ngo HC, Smales RJ & Pashley DH (2001) Bonding of contemporary glass ionomer cements to dentin *Dental Materials* **17(5)** 456-470.

Yoshida Y & Van Meerbeek B (2002) Unpublished observations.

Yoshida Y, Van Meerbeek B, Nakayama Y, Snauwaert J, Hellemans L, Lambrechts P, Vanherle G & Wakasa K (2000) Evidence of chemical bonding at biomaterial-hard tissue interfaces *Journal of Dental Research* **79(2)** 709-714.

Yoshida Y, Van Meerbeek B, Nakayama Y, Yoshioka M, Snauwaert J, Lambrechts P, Vanherle G, Wakasa K & Okazaki M (2001) Adhesion to and decalcification of hydroxyapatite by carboxylic acids *Journal of Dental Research* **80(6)** 1565-1569.

Yoshida Y, Van Meerbeek B, Snauwaert J, Hellemans L, Lambrechts P, Vanherle G, Wakasa K & Pashley DH (1999) A novel approach to AFM characterization of adhesive tooth-biomaterial interfaces *Journal of Biomedical Materials Research* **47(1)** 85-90.

Yoshida Y, Van Meerbeek B, Okazaki M, Shintani H & Suzuki K (2003) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **82(Special Issue)** abstract (in press).

Yoshioka M, Yoshida Y, Inoue S, Lambrechts P, Vanherle G, Nomura Y, Okazaki M, Shintani H & Van Meerbeek B (2002) Adhesion/decalcification mechanisms of acid interactions with human hard tissues *Journal of Biomedical Materials Research* **59(1)** 56-62.

Yoshiyama M, Urayama A, Kimochi T & Matsuo T (2000) Practical application of sealed restoration to dentin caries—Resin adhesion to caries-affected dentin in Tagami J, Toledano M, Prati C (eds) *Advanced Adhesive Dentistry Proceedings* book of the Granada 3rd International Kuraray Symposium: December 3-4, 1999, Cirimido: Grafiche Erredue p 177-194.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3**, 236-241

Clinical Research

In Vivo Kinetics of Bleaching Gel with Three-Percent Hydrogen Peroxide Within the First Hour

TA Al-Qunaian • BA Matis • MA Cochran

Clinical Relevance

The results of this study showed that hydrogen peroxide has an overall similar kinetics curve as carbamide peroxide, yet, it has a faster degradation rate. Therefore, hydrogen peroxide is indicated for daytime application; however, since hydrogen peroxide is used for shorter time periods, the results are still not conclusive since the duration of this study is only one hour.

SUMMARY

This *in vivo* study determined the kinetics of 3% hydrogen peroxide in a bleaching gel within the first hour. The material used in this study was 3% hydrogen peroxide gel (Perfecta 3/15, Premier Dental Products Co) and the study involved 10 subjects who met the inclusion and exclusion criteria. Each subject wore the tray with gel six different times on separate days. Evaluation of remaining amount of hydrogen peroxide was calculated by the method stated in US Pharmacopoeia. The study results indicate that the mean percentage of hydrogen peroxide recovered for 5, 10, 20, 30, 45 and 60 minutes was 61, 56, 49, 44, 38 and 32, respectively. The amount of hydrogen peroxide in the saliva sample after one hour was 0.42 mg. Excluding the first 10 minutes, the kinetics of hydrogen peroxide in the tray and teeth sample was exponential.

*Talal A Al-Qunaian, BDS, MSD, lecturer, Department of Restorative Dental Science, King Saud University, College of Dentistry, Saudi Arabia

Bruce A Matis, DDS, MSD, professor, director of Clinical Research Section, Indiana University School of Dentistry, Indianapolis, IN

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

*Reprint request: PO Box 60169, Riyadh 11545; e-mail: tqunaian@msn.com

INTRODUCTION

The appearance of an individual's teeth is a prime concern. Therefore, it is not surprising that tooth discoloration has been the subject of studies for many years. A healthy smile improves self-image and confidence and projects an aura of health to others.

During the past decade, the demand for cosmetics dentistry has grown dramatically, which has been fueled, in part, by different bleaching techniques. Based on current clinical studies, at-home bleaching is a safe, effective technique for whitening teeth. The kinetics cycle, according to the ADA (American Dental Association, 1998), is an important issue that determines the safety and effectiveness of at-home bleaching materials that are used during the day or at night. Nighttime bleaching agents use carbamide peroxide as the active agent, whereas, daytime bleaching agents use hydrogen peroxide (HP). There are studies that report the kinetics of carbamide peroxide (Gaiao, 1997; Wattanapayungkul & others, 1999; Matis & others, 2002); however, information regarding the kinetics of

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

hydrogen peroxide in bleaching agents is limited. The only published data is for the degradation of 10% hydrogen peroxide, which showed that only 50% of the hydrogen peroxide was recovered after 30 minutes (Clinical Research Associates, 1997), with a very small amount present in the tray after two hours. This *in vivo* study determined the kinetics of 3% hydrogen peroxide in a bleaching gel within the first hour.

METHODS AND MATERIALS

Patient Selection

This study was presented to the Institutional Review Board of Indiana University Purdue University Indianapolis. Upon approval, 10 human subjects were enrolled in the study. Tables 1 and 2 list the inclusion and exclusion criteria.

Table 1: *Inclusion Factors*

1. Presence of six caries-free maxillary anterior teeth that have not been restored facially.
2. No significant periodontal disease Gingival Index <1.0 (Loe and Silness, 1963)
3. Crown of the right central incisor at least 9 mm in length and 8 mm in width.
4. Patient agrees not to smoke during the study period.
5. Patient has the ability to return for periodic examinations.
6. Patient is willing to sign consent form.

Table 2: *Exclusion Factors*

1. History of any medical disease that may interfere with the study or require special consideration such as prescribing premedication before dental procedures.
2. Pregnant or lactating women.
3. Younger than 18 years of age.
4. Patient has smoked 15 days prior to being evaluated for acceptance into the study.

Methodology

Qualified subjects received a dental prophylaxis treatment with a rubber cup and pumice at least one week but no more than two months prior to the study. During the same visit, an alginate impression (Jeltrate Plus, Caulk Division Dentsply International Inc, Milford, DE, USA) of the upper arch was taken for each subject. The impression was then poured with Super Die fast-setting stone (Whip Mix Corp, Louisville, KY, USA). From this cast, a customized vacuum-formed tray was made with 0.035-inch soft vinyl sheets (Premier Dental Products Company, King of Prussia, PA, USA). The margins of the tray were trimmed with the Tray Magic Trimmer (Premier Dental Products Company) 1 mm short of the gingival margins. There were no reservoirs in the fabrication of the trays, according to the manufacturer's instructions.

The clinical procedure required a strip of 3% hydrogen peroxide (HP) bleaching gel (Premier Dental Products Company) be placed on the facial surfaces of the six maxillary anterior teeth spaces in the vacuum-formed tray. The filled tray was weighed on an analytical balance with +/- 0.1 mg sensitivity (Mettler AE100, Mettler Instrument Corp, Hightstown, NJ, USA). The tray was then placed into the subject's mouth and seated. Each subject wore the tray with the 3% hydrogen peroxide (HP) bleaching gel on separate days for 5, 10, 20, 30, 45 and 60 minutes. During tray use, the participants were asked to refrain from eating, drinking and speaking. They were provided a beaker for collecting their salivary secretions (saliva sample). The saliva was analyzed to evaluate the amount of hydrogen peroxide each subject would probably have swallowed. At the end of each evaluation period, the bleaching agent was retrieved from: 1) the tray (tray sample), 2) the teeth, by scraping the gel off with a spatula (tooth sample) and 3) the mouth, by having the subject rinse four times with deionized water and collecting the expectorant in a beaker (rinse sample).

Chemical Analysis

The method stated in United States Pharmacopeia (2000) and used in previous studies (Gaiao, 1997; Wattanapayungkul & others, 1999; Matis & others, 2002) was used to determine the amount of peroxide in each sample.

A solution of 0.025N sodium thiosulfate was used to determine the amount of hydrogen peroxide in each sample. Each ml of 0.025N sodium thiosulfate is equivalent to 1.7 mg of hydrogen peroxide. Therefore, the amount of recovered hydrogen peroxide could be identified by determining the volume of sodium thiosulfate used in triturating.

The concentration of hydrogen peroxide (wt %) for tray and teeth samples was determined by the following formula:

$$\text{Concentration of hydrogen peroxide} = \frac{V\,(0.025)\,(1.7)}{W}$$

V = volume of sodium thiosulfate (ml)

W= weight of the sample (milligrams of a recovered sample that incorporates gel and saliva into it)

The amount of hydrogen peroxide recovered in milligrams of HP present in the recovered sample was calculated using the following formula:

Amount of HP = V (0.025) (1.7)

V = the volume of sodium thiosulfate (ml). This formula was applied to the three samples.

The concentration of recovered gel was determined by calculating the percentage of the total physical amount of gel recovered from the tray, teeth and rinse samples compared to the initial amount of gel delivered:

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

$$\frac{\text{Concentration of HP at time}}{\text{initial gel delivered}} = \frac{\text{total gel recovered} \times 100}{}$$

The kinetics of hydrogen peroxide with each time was calculated from the ratio:

$$\frac{\text{Concentration of Hydrogen peroxide (t)}}{\text{Initial concentration of hydrogen peroxide}} \times 100$$

The initial concentration of bleaching gels was analyzed in triplicate by testing both pretreatment and post-treatment to determine the amount of hydrogen peroxide percentage at those times.

Statistical Analysis

A previous study (Gaiao, 1997) showed excellent within-subject repeatability. As a result, each subject wore the tray with bleaching agent only once for each specified time. The mean, standard deviation, standard error, range, 95% confidence interval for the mean concentration of hydrogen peroxide and percentage of hydrogen peroxide recovered were reported for each wearing time both for the total sample and the component tray, teeth and rinse samples. Regression analyses with a means to correlate the multiple measurements from each subject was used to model the degradation curves. A 5% significance level was used for all statistical comparisons.

RESULTS

The weight of gel delivered averaged 194.7 mg and ranged from 132.1 mg to 321.8 mg.

The amount of HP delivered averaged 6.04 mg and ranged from 4.10 mg to 9.98 mg.

The change in HP concentration in the tray and teeth samples (Figure 1) was 8.5 (p=0.0001) and 4.5 (p=0.0002) times greater, respectively, from five to 10 minutes compared to 10 to 60 minutes. The change in the amount of HP in the rinse and tray samples (Figure 2) was 28 (p=0.0004) and 4.0 (p=0.0012) times greater, respectively, from five to 10 minutes compared to 10 to 60 minutes. The amount of HP in the teeth sample (Figure 2) was relatively constant over time (p=0.2169). The change in the total amount of HP (Figure 2) was 5.9 times greater between five and 10 minutes compared to 10 and 60 minutes (p=0.0001). The amount of HP in the saliva sample increased over time (p=0.0212) but did not increase at a consistent rate (Figure 2).

The total percentage of HP recovered (Table 3, Figure 3) at 5, 10, 20, 30, 45 and 60 minutes was 61, 56, 49, 44, 38 and 32, respectively. All samples showed an exponential degradation of hydrogen peroxide after 10 minutes.

DISCUSSION

Tooth discoloration can be emotionally traumatic for people in a society that places a high value on appearance. Therefore, mastering using current at-home vital bleaching products allows the dentist to offer a reasonable treatment option along with other esthetic restorative options.

The term "kinetic cycle" means the degradation rate of bleaching-active agent at a specific time. The ADA has established the document *Acceptance Program Guidelines for Home-Use Tooth Whitening* (American

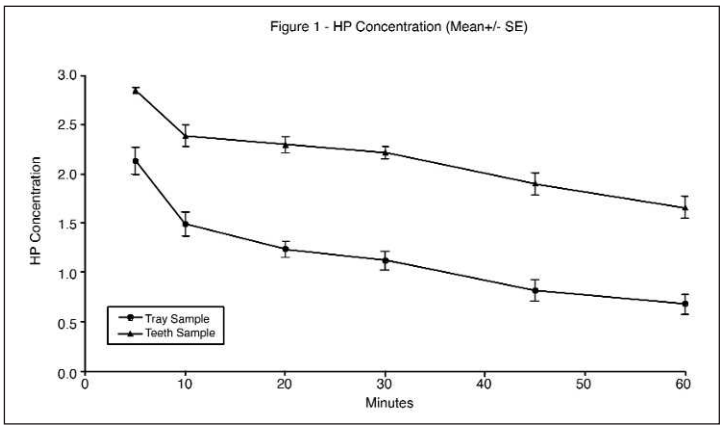


Figure 1. *HP Concentration (Mean +/- SE).*

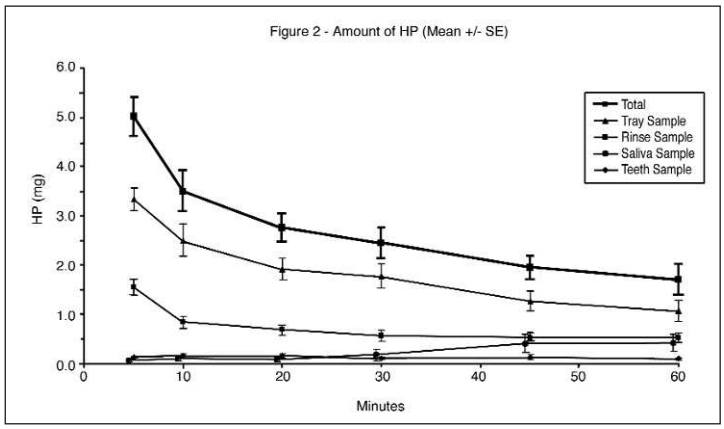


Figure 2. *Amount of HP Concentration (Mean +/- SE).*

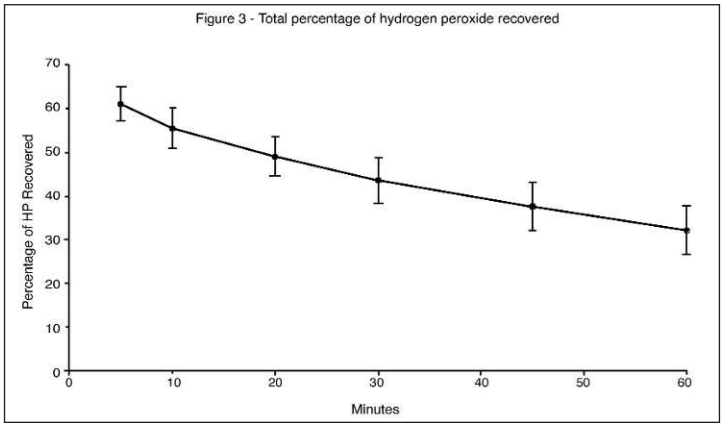


Figure 3. *Total percentage of hydrogen peroxide removed.*

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 3: *Total Percentage of Hydrogen Peroxide Recovered*

Minutes	N	Mean	SD	SE	Min	Max	95% CI for MEAN	
5	10	61.12	12.23	3.87	44.52	80.11	52.37	69.87
10	10	55.62	14.62	4.62	32.62	79.97	45.17	66.07
20	10	49.01	14.39	4.55	30.58	75.77	38.72	59.30
30	10	43.55	16.34	5.17	20.24	72.38	31.86	55.24
45	10	37.64	17.19	5.44	15.78	69.18	25.33	49.95
60	10	32.23	17.53	5.54	7.43	66.85	19.70	44.76

Dental Association, 1998). Section "8D" of that document states that manufacturers must supply the ADA with the "Kinetics of active ingredient release." The word "kinetics" was chosen instead of "degradation," because the rate of physical loss of peroxide from the tray and the rate of peroxide lost from the chemical breakdown are combined to determine the amount of active agent remaining at different time intervals. Therefore, "kinetics" appears to be a better term for this study than "degradation."

To grant their seal of approval, the ADA requires information about the kinetics of an at-home bleaching product. This information is essential to understanding the pattern of degradation and achieving ultimate results with limited side effects and recommended application time.

The methodology used in this study simulates the clinical situation for at-home vital bleaching. To facilitate the collection of the remaining gel, only six upper anterior teeth were selected for gel retrieval. For this study, subjects with large, caries-free teeth with no restoration on the facial surfaces were selected. Subjects with periodontal diseases were excluded to avoid any variable that might affect the kinetics of the bleaching gel by potential reaction with large numbers of microorganisms (Gurgan, Bolay & Alacam, 1996).

Trays were filled with a strip of bleaching gel, Perfecta 3/15 (Premier Dental Products Company), which was placed on the facial surfaces of the six anterior teeth as recommended by the manufacturer. A pilot study was conducted in an attempt to prevent under- or overfilling of the tray with bleaching gel. Underfilling could produce voids that might reduce the reaction of bleaching gel with the tooth surface, while overfilling could cause physical loss of the material.

The rapid kinetics in the first 10 minutes might be due to the saturation or diffusion of hydrogen peroxide into the tooth (Wattanapayungkul & others, 1999). Once hydrogen peroxide contacts the tooth, it breaks down to oxygen and water.

There is a significant difference between the kinetics of the tray and teeth samples. The kinetics of the teeth sample is significantly slower than the tray sample. Two factors could contribute to this result. First, the teeth sample was relatively non-contaminated since the gel was retrieved by scraping off just the incisal half of the anterior teeth where there would be a minimum of saliva. Second, saliva was present on the outside of the tray sample, with some occasionally found inside the tray. When the tray was removed from the subject's mouth and weighed, the weight included the gel recovered from the tray and the weight of the saliva contaminant. Therefore, a lower concentration would be obtained when the weight is greater. The concentration of hydrogen peroxide in the tray and the teeth samples did not reveal the exact kinetics process but gave a general predication for it. On the other hand, the total percentage obtained from the total amount of hydrogen peroxide retrieved from the tray, teeth and rinse samples provided precise kinetics information.

The results of this study agree with the hypothesis that the kinetics of hydrogen peroxide and carbamide peroxide are similar in the first hour in that, after a fast degradation, they decayed in an exponential manner.

In this study, the mean percentage of hydrogen peroxide recovered at one hour was 32.23 (Table 2, Figure 3). Fifty percent of the active agent was recovered in this study after 20 minutes in the mouth. Gaiao (1997) found that the kinetics of carbamide peroxide in trays with reservoirs after five minutes is exponential and the mean percentage of carbamide peroxide recovered at one hour is 63%; approximately half (52%) of the active agent was recovered after two hours in the mouth. Wattanapayungkul and others (1999) found that the kinetics of carbamide peroxide after five minutes is exponential and the mean percentage of carbamide peroxide recovered at one hour is 54%. Matis and others (2002) found that the kinetics of carbamide peroxide after two hours of bleaching using gel in trays with facial reservoirs was 52%; while the percentage of bleaching gel in trays without facial reservoirs was 23%. However, products tested with and without reservoirs had the same kinetics rate. These differences relate to tray design and possibly system components. In some studies using carbamide peroxide, trays with facial reservoirs were used. By using reservoirs, the amount of bleaching gel physically lost during active treatment is decreased by approximately 50% after two hours. In this study, trays with no reservoirs were used.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Carbamide peroxide is hydrogen peroxide coupled with urea. Urea and its byproducts appear to slow the kinetics of carbamide peroxide.

A study by Mokhlis and others (2000) found that teeth lightened with 20% carbamide peroxide were significantly lighter than teeth lightened with 7.5% hydrogen peroxide at one and two weeks but no significant difference was found at the end of 12 weeks. A study by Panich (1999) compared 15% carbamide peroxide with 5.5% hydrogen peroxide used in trays with reservoirs for 30 minutes twice a day. No significant difference in tooth whitening was found between them. However, Mokhlis and others (2000) noted that tooth lightening did not occur in the Panich study to the degree that it had in other overnight studies using 10% carbamide peroxide. On the other hand, the reversal was also much less. Kowitz and others (1991) compared 3% hydrogen peroxide to 10% carbamide peroxide *in vivo*. After four weeks, there was no significant difference between the whitening effects of the two products tested.

Saliva was collected from a patient's expectorate during the clinical procedure. This saliva was analyzed to determine the approximate amount of hydrogen peroxide that the subject might have swallowed during the bleaching treatment. In this study, the amount of hydrogen peroxide in the saliva sample after one hour was 0.42 mg (Figure 2). This is a significantly lower amount than the maximum amount ingested that could result in serious side effects. In addition, if a patient would wipe off the excess gel, expectorate and saliva and brush after the procedure, the amount of gel swallowed would be reduced even more.

Matis (1999) studied the degradation of whitening gel in saliva. He found that each patient's saliva degraded the whitening gel at a specific rate. A similar result was found in this study. Therefore, there are variants that affect the individual's kinetics rate. A possible factor that could be responsible for this individual variation is salivary antioxidants. Salivary antioxidants are essential for peroxide decomposition and are present intra- and extra-cellularly. There are factors that might affect the secretion level of salivary antioxidants and their ability to decompose peroxide. Attin & Hellwig (1996) found that dentifrices caused an elevated level of fluoride ions in saliva after tooth brushing. Larsen & others (1999) found that intraoral pH varies among individuals. Hannuksela & others (1994) observed that decreased pH and elevated fluoride ion concentration in saliva inhibit the activity of salivary antioxidants. This inhibition was observed only at a pH of less than 6.5 and with fluoride concentrations equal to or greater than 20 mM. Abbeele, Pourtois & Courtois (1992) found that salivary antioxidant activity was reduced to less than 40% of its initial activity value when fluoride concentration was equal to or greater than 0.1 mm. A study evaluating the effects of using different fluoride-containing products on the kinetics of peroxide gel would serve as a valuable contribution to the scientific literature.

CONCLUSIONS

This study concluded that:

1. Half or 50% of the active agent was recoverable after 20 minutes of tray use.
2. At one hour, the average of the total remaining hydrogen peroxide was 32.23%.
3. For one hour of bleaching treatment, the average concentration of hydrogen peroxide in collected saliva was 0.42 mg/100ml.
4. The kinetics rate was higher in the first 10 minutes for tray, teeth and rinse samples.

(Received 7 May 2002)

References

Abbeele A, Pourtois M & Courtois P (1992) Fluoride inhibition of SCN- and Cl- peroxidase activities in whole saliva and recombinant myeloperoxidase: Influence of pH and hydrogen peroxide concentration *Journal of De Biologie Buccale* **20(4)** 219-224.

American Dental Association (1998) Acceptance program guidelines for home-use tooth whitening products Council on Scientific Affairs Chicago, IL.

Attin T &Hellwig E (1996) Salivary fluoride content after tooth brushing with a sodium fluoride and an amine fluoride dentifrice followed by different mouthrinsing procedures *Journal Clinical Dentistry* **7(1)** 6-8.

Clinical Research Associates (1997) Tooth bleaching, state-of-art '97 *Clinical Research Associates Newsletter* **21(4)** 3.

Gaiao U (1997) A Clinical study of 10-percent carbamide peroxide degradation in bleaching trays [Thesis] Indianapolis: Indiana University School of Dentistry.

Gurgan S, Bolay S & Alacam R (1996) Antibacterial activity of 10 percent carbamide peroxide bleaching agent *Journal of Endodontics* **22(7)** 356-357.

Hannuksela S, Tenovuo J, Roger V, Lenader-Lumikari M & Ekstrand J (1994) Fluoride inhibits the antimicrobial peroxidase system; in human whole saliva *Caries Research* **28(6)** 429-434.

Kowitz GM, Rustogi KN, Wong R, Curtis JP &Wieckowski SE (1991) *In vivo* effect of peroxide on tooth coloration *Journal of Dental Research* Abstract #424, p 70.

Larsen MJ, Jensen AF, Madsen DM & Pearce EI (1999) Individual variation of pH, buffer capacity, and concentrations of calcium and phosphate in unstimulated whole saliva *Archives of Oral Biology* **44** 111-117.

Loe H & Silness J (1963) Periodontal disease in pregnancy: Prevalence and severity *Acta Odontologica Scandinavica* **21** 533-551.

Matis BA (1999) Degradation of whitening gel in saliva Personal Communication.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Matis BA, Yousef M, Cochran MA & Eckert GJ (2002) Degradations of nine bleaching gels after two hours *in vivo* *Operative Dentistry* **27(1)** 12-18.

Mokhlis GA, Matis BA, Cochran MA & Eckert GJ (2000) A three-month clinical evaluation of 20% carbamide peroxide and 7.5% hydrogen peroxide whitening agents during daytime use *Journal of the American Dental Association* **131** 1269-1277.

Panich M (1999) *In vivo* evaluation of 15% carbamide peroxide and 5.5% hydrogen peroxide whitening during daytime use [Thesis] Indianapolis: Indiana University School of Dentistry.

United States Pharmacopeia (2000) The National Formulary United States Pharmacopeial Convention, Inc, Rockville, MD.

Wattanapayungkul P, Matis BA, Cochran BA & Moore BK (1999) A clinical study of the effect of pellicle on the degradation of 10% carbamide peroxide within the first hour *Quintessence International* **30(11)** 737-741.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3,** 242-250

Comparison of Pulp Responses to Resin Composites

PE Murray • LJ Windsor • AA Hafez
RG Stevenson • CF Cox

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Clinical Relevance

Five commonly used adhesive systems for bonding resin composites have been compared in terms of bacterial microleakage, pulp inflammation, reactionary dentin formation and preserving the underlying odontoblast survival. These data clarify differences between the adhesive systems and the interactions between pulp injury and repair responses.

SUMMARY

Diverse reports have described how various types of adhesive systems cause disastrous pulp necrosis, chronic severe inflammation or failure to stimulate any pulp reactions. This article reports on the effects of five common adhesive systems and how they compare in terms of pulp injury as measured by odontoblast survival or dentin regeneration and reactionary dentin formation. One hundred and thirty Class V pulp, non-exposed cavities were prepared in non-human primate teeth and were restored with five different adhesive systems. After a period of time between 3 and 172 days, the teeth were extracted, fixed, processed and examined histomorphometrically. Bacterial microleakage was detected with McKays stain and inflammation was categorized according to the International Organization for Standardization (ISO) criteria. The number of odontoblasts and the area of reactionary dentin were measured. Pulp reactions of all adhesive systems were generally minimal, although some systems permitted bacterial microleakage in 33% of restorations, and some other systems were associated with pulp inflammation in 22% of restorations. These observations suggest that adhesive systems provide acceptable biocompatibility, however, there is strong potential for improvement.

*PE Murray BSc, PhD, assistant professor, Endodontics, College of Dental Medicine, Nova Southeastern University, Fort Lauderdale, FL

LJ Windsor, BSc, PhD, assistant professor, Oral Biology, Indiana University School of Dentistry, Indianapolis, IN

AA Hafez, BDS, MSc, postgraduate student, University of Southern California School of Dentistry, Los Angeles, CA

RG Stevenson, DDS, chairman of Restorative Dentistry, UCLA School of Dentistry, Los Angeles, CA

CF Cox, DMD, PhD, MSc, vice-chairman of Restorative Dentistry, UCLA School of Dentistry, Los Angeles, CA

*Reprint request: 3200 S University Dr, Fort Lauderdale, FL33328-2018; e-mail: pemurray@nova.edu

INTRODUCTION

The placement of resin composite (CR) systems is increasingly more popular due to reportedly improved longevity, biocompatibility, wear resistance and ability to compare favorably with traditional amalgam restorations (Vann, Barkmeier & Mahler, 1988; Berry & others, 1998; Kitasako, Inokoshi & Tagami, 1999). Since resin composites can match tooth color, they have greatly expanded the range of possibilities for restorative dentistry, thus, allowing deteriorated or debonded restorations to be repaired or replaced with minimal loss of tooth structure (Duke, 1993). Large undercuts of vital tooth structure that need to retain amalgam

restorations are generally not necessary (Shavell, 1980). This explains why placing these adhesive systems is increasing done at the expense of more traditional restorative materials (Clinical Research Associates, 1995).

The adhesive bonding systems used to bond resin composites to tooth structure have developed rapidly over the past 30 years, beginning in the 1960s with the development of the first commercial products, followed by the introduction of the acid-etch technique in clinical practice in the 1970s (Fusayama & others, 1979). Since then, bonding systems have become more steadily refined and diversified. Early single-step bonding systems became multi-step systems with complicated, time-consuming and operator-sensitive application procedures. The current trend is to reduce the multi-step adhesive process so as to lessen the sensitivity to errors of inaccurate or incorrect operator handling (Inoue & others, 2001). Dental companies usually classify bonding systems into six generations based mainly on the chronology of their release into the dental market (Kugel & Ferrari, 2000). However, when classified in terms of the number of steps, there are only three categories of systems that achieve bonding to tooth substrate. Depending on the clinical approach, Van Meerbeek and others (1998; 2000) have divided the modern systems into three categories based on the number of steps required to achieve bonding. Currently, many clinicians still use fourth-generation systems that involve three steps: etch & rinse, prime, bond or fifth-generation or two steps: etch and prime or prime and bond (Pilo & Ben-Amar, 1999). Each type of adhesive bonding system has its advantages and limitations, but it remains uncertain as to how these materials compare to each other in terms of pulp reactions. Investigating pulp reactions is important, especially since some reports of severe chronic inflammation (de Souza Costa & others, 2001) or even disastrous pulp necrosis following capping of the exposed pulp with certain adhesives (Pameijer & Stanley, 1998) are reported. These investigations contrast with reports of minimal or few pulp reactions, positive healing and reparative dentin-bridge formation (Tarim & others, 1998; Akimoto & others, 1998; Cox & others, 1998). Because of these diverse reports, controversy remains regarding the precise nature and severity of pulp reactions to using different adhesives.

Pulp injury and regeneration appear to be influenced by the summation of the effects of the initial injury, such as the initial carious lesion (Mjör, 2002), cavity preparation trauma (Swerdlow & Stanley, 1958), chemical activity of restorative materials (Murray & others, 2000), etching treatment (Murray & others, 2002a) and patient factors, including aging and treatment history, followed by postoperative complications such as bacterial microleakage (Murray & others, 2001; 2002b). In terms of restorative materials, the most important factors appear to be the ability of the restorative materials to avoid creating pulp injuries and preventing subsequent bacterial microleakage (Cox, 1994; Cox & Suzuki, 1994). Bacterial microleakage complications include postoperative sensitivity, inflammation, marginal discoloration, recurrent caries and the eventual need for endodontic therapy (Bergenholtz & others, 1982; Bergenholtz, 1990). Inflammation appears to be stimulated by bacterial microleakage more than material cytotoxicity (Watts & Paterson, 1987). Long-term pulp inflammation in the absence of bacterial microleakage is minimal in patients with mostly all common types of restorative materials (About & others, 2001). However, unresolved severe inflammation can progress to pulp necrosis and periapical lesion development with localized bone destruction (Bergenholtz, 1990; Bergenholtz, 2000). Therefore, it is important to ensure that bacterial microleakage and unresolved pulp inflammation are always prevented. Often, individual adhesives are compared with calcium hydroxide (Dycal) or a resin-modified glass ionomer as reference materials. Comparisons among these different types of adhesives are scarce. Moreover, how they compare with each other in terms of bacterial microleakage, inflammation, odontoblast survival and reactionary dentin repair is not known. This type of investigation is necessary to understand the biological processes that occur in the dentin-pulp complex during injury and repair aswell as to understand how restorative events can modify them, because these interactions underpin the success of much of restorative dentistry.

It is estimated that out of the 290-million cavity preparations restored each year in the United States, 200 million are replacements for failed restorations (Arnst & Carey, 1998). Surveys indicate that secondary (recurrent) caries is the most frequent reason for replacing amalgam and resin composite restorations, followed by mechanical failure and complications such as hypersensitivity (Mjör, Jokstad & Qvist, 1990; Maupome & Sheiham, 1998; Burke & others, 1999). These surveys continue to provide evidence that a high proportion of restored teeth show symptoms that require pulpectomy or extraction (Zöllner & Gaengler, 2000), suggesting that restorative treatments are not fully optimized. Consequently, this study investigated the effects of cavity restoration with five different types of adhesives and the effects each had on underlying pulp tissue. Bacterial microleakage, pulp inflammation, odontoblast survival and the reactionary dentin area were quantified to compare pulp injury and repair activities.

METHODS AND MATERIALS

One hundred and thirty-four sound teeth were used from eight adult *Rhesus macaca* monkeys. The animals were cared for according to the ethical and animal use

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 1: *Pulp Capping Materials*

Restorative Material	Adhesive System	Resin Composite	Testing Interval (Days)		Total Teeth
			3-79	80-172	
OB	Optibond[a]	Herculite XR[a]	12	7	19
AB	All Bond[b]	Aelitefil[b]	32	19	51
SB	Scotchbond 2[c]	Silux[c]	6	6	12
LB	Linerbond[d]	Clearfil AP-X[d]	6	6	12
AM	Amalgambond[e]	EPIC[e]	27	9	36

[a]Kerr, Orange, CA
[b]BISCO, Schaumburg, IL
[c]3M ESPE Dental Products, St Paul, MN
[d]Kurary Co, Osaka, Japan.
[e]Parkell, Farmingdale, NY

Table 2: *Histologic Criteria for Assessment of Pulpal Reactions*

Inflammatory Cell Response

None	The pulp contained few inflammatory cells, or an absence of inflammatory cells associated with the pulp beneath the cut tubules of the cavity floor.
Slight	The pulp had localized inflammatory cell lesions predominated by polymorphonuclear leukocytes or mononuclear lymphocytes.
Moderate	The pulp had polymorphonuclear leukocyte lesions involving more than one-third of the coronal pulp.
Severe	The pulp tissue is largely necrotic, following chronic inflammatory cell injury.

Reactionary Dentin

The area of dentin secretion by primary odontoblasts beneath the cut dentinal tubules of the cavity preparation.

Odontoblast Numbers

The number of intact palisade columnar cells with an eosinophilic cytoplasm and a nucleus located in a basal polarized position, adjacent to the pre-dentin.

Bacterial Microleakage

Absent	Absence of bacterial staining in any section.
Present	A positive bacterial staining reaction seen along the cavity walls, within the cut dentinal tubules of the cavity preparation, or within the dental pulp.

guidelines of the University Animal review procedures. Prior to the operative procedures, each animal was tranquilized and sedated. The teeth were scaled and polished with a rubber cup and pumice paste to remove plaque and calculus. Saliva was controlled by high-speed evacuation and quadrants of teeth were isolated with sterile cotton rolls. Non-pulp exposed standardized Class V cavities were prepared with a #330 carbide bur at ultra-high speed under water spray coolant. One new bur was used per cavity preparation. The cavities were cut into labial enamel with the distal margin no closer than 1 mm above the gingival margin. Accidental pulp exposure was avoided by checking the thickness of the remaining dentin using the UAB-modified Endocater Apex locator (Hygienic Corp, Akron, OH, USA). The cavities were prepared in dentin, leaving a range of remaining dentin thickness (RDT) between 1.70 and 0.011 mm, with a mean of 0.517 mm. The RDT was precisely measured from histological sections. Each cavity was immediately restored with the selected material. The adhesive systems and materials used to restore the cavity preparations were randomly distributed among the teeth and animals. The standardized methods and procedures used in this study are congruent with those described elsewhere (Cox, 1994; Cox & others, 1987; 1992; 1998). The entire cavity was acid-etched with 35% phosphoric acid for 15 seconds and washed with sterile saline solution for 15 seconds, leaving the surface slightly moist. The primer was evenly applied by gentle air dispersion for five seconds. The adhesive was then evenly applied and photopolymerized for 10 seconds using a visible light curing unit (Optilux 150, Demetron, Danbury, CT, USA). The resin composite materials filled the cavity preparations in three increments; each application was photopolymerized for 20 seconds using the same light curing unit. The irradiance of the light curing unit was not measured during the study. The materials were placed according to the manufacturers' instructions following International Organization for Standardization 7405 Usage Guidelines (1997). Following these guidelines, no positive or negative controls were employed; however, the evaluation guidelines of 70+/- five days were expanded to cover short intervals from 3-to-79 days to long 80-to-172 day terms. In total, five different adhesive systems were used to restore 130 teeth (Table 1). The animals were killed by left ventricular flushing with a 0.9% physiologic pH saline solution followed by a fast-penetrating gluteraldehyde-phosphate-buffered

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 3: *Statistical Differences Between Restorative Variables*

	Inflammation	Materials	Reactionary Dentin	Bacterial Microleakage	Odontoblast Numbers
Inflammation		p=0.0263 X^2=11.024	p=0.5725 X^2=0.319	p=0.0001 X^2=14.42	p=0.3104 F=1.037
Materials	p=0.0263 X^2 = 11.024		p=0.0063 X^2 = 14.35	p=0.1655 X^2 = 6.488	p=0.5180 F = 0.815
Reactionary Dentin	p=0.5725 X^2=0.319	p=0.0063 X^2=14.35		p=0.2910 X^2=1.115	p=0.3959 F=0.726
Bacterial Microleakage	p=0.0001 X^2=14.42	p=0.1655 X^2=6.488	p=0.2910 X^2=1.115		p=0.1455 F=2.145
Odontoblast Numbers	p=0.3104 F=1.037	p=0.5180 F=0.815	p=0.3959 F=0.726	p=0.1455 F=2.145	

X^2 = Chi square test
F=Analysis of variance test

Table 4: *Restorative Materials,Bacterial Microleakage and Pulp Inflammation*

Restorative Material	Pulpal Inflammation			
	Absent		Present	
	- bacteria	+ bacteria	- bacteria	+ bacteria
OB	100%	0%	0%	0%
	(19)	(0)	(0)	(0)
AB	72%	6%	12%	10%
	(37)	(3)	(6)	(5)
SB	75%	17%	8%	0%
	(9)	(2)	(1)	(0)
LB	67%	33%	0	0
	(8)	(4)	(0)	(0)
AM	83%	11%	0%	6%
	(30)	(4)	(0)	(2)

para-formaldehyde fixative (Cox, Heys & Heys, 1977). The restored teeth were dehydrated in a series of alcohols, demineralized in EDTA and processed for light microscope analysis. Serial sections of 7 µm thickness stained with hematoxylin and eosin were examined by light microscopy, and histomorphometric analysis was conducted on the lingual-labial longitudinal serial sections of each specimen that exhibited the least dentin thickness and greatest pulp area, as previously described by Murray & others, (2000; 2001; 2002a). Masson trichrome staining was used to assess soft tissue organization and reactionary (or reparative) dentin formation. The area of reactionary dentin was measured at 100x magnification in each histological section using an eyepiece graticule superimposed over the reactionary dentin. The area of reactionary dentin was used to provide an assessment of pulp repair activity (Table 2). Odontoblast cells were categorized on the basis of their morphology and position within the tooth pulp. Only intact cells that displayed a clearly identifiable nucleus were included. Odontoblasts were identified as being the outermost stratum of palisaded, columnar cells that are immediately adjacent to the predentin that displayed eosinophilic cytoplasm and a nucleus located in a basal polarized position. Odontblast cell numbers were counted per mm of pulp dentin border beneath the cut dentinal tubules of cavity preparations and immediately opposite and independent of the site of cavity preparation as a control measure for comparison.

The inflammatory response of each pulp was categorized from "none," "slight" and "moderate" to "severe," according to ISO guidelines and published criteria (Mjör, 1983; Mjör & Ferrari, 2002; Mjör, 2002). The categories of pulp inflammation were ranked in order of severity from none, slight, moderate or severe (Table 2): None means that the pulp contained few inflammatory cells or an absence of inflammatory cells associated with cut tubules of the cavity floor. Slight signifies that the pulp had localized inflammatory cell lesions predominated by polymorphonuclear leukocytes or mononuclear lymphocytes. Moderate indicates that the pulp had polymorphonuclear leukocyte lesions involving more than one-third of the coronal pulp. Severe signifies that the pulp tissue was largely necrotic, following chronic inflammatory cell injury. Bacterial contamination of each restoration was assessed using McKay's (1970) stain to detect the presence of gram positive and negative microorganisms (Table 2). Data were analyzed using multivariate analysis of variance (ANOVA) and Chi-square (X^2) statistical tests at a confidence level of 95% (STATview software, SAS Inc, Cary, NC, USA). The raw numerical data on the numbers of odontoblasts per unit area were analyzed statistically by using a single ANOVA test to compare the interactions among the categories of inflammation, materials, bacterial microleakage and the area of reactionary dentin. The raw categorical data on inflammation, materials and bacterial microleakage were compared with each other using separate Chi-square tests for each interaction of the complete raw data for each vari-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

able. These statistical procedures are reportedly among the most versatile and conservative of the multiple comparison tests (Dawson-Saunders & Trapp, 1994).

RESULTS

Cavity Restoration Materials

The major differences between individual adhesive systems were noted in the categories of pulp inflammation and reactionary dentin area (Table 3). Non-statistically significant differences ($p>0.05$) were observed between certain adhesive systems and bacterial microleakage, and also odontoblast cell numbers (Table 3). No pulp inflammation was observed following restoration with OB or LB (Table 4). This lack of inflammation compares with 6% of AM, 8% of SB and 22% of AB restorations (Table 4). However, when the incidence of pulp inflammation is expressed as a mean category, it is clear that the average category of inflammation is between absent and slight (Figure 1); therefore, these adhesive systems provide an acceptable biocompatible response according to ISO guidelines (1997). There appeared to be some differences between adhesive systems and the stimulation of reactionary dentin, because over the long-term (80-172 days), the mean area of reactionary dentin beneath the site of AM cavity preparations was approximately 57% less than that observed beneath SB cavity preparations (Figure 3).

Pulp Inflammation

Pulp inflammation appeared to be primarily influenced by bacterial microleakage, followed by differences between restorative materials (Table 3). Inflammation appeared to have little effect on the reactionary dentin area and odontoblast numbers (Table 3). Whenever bacterial microleakage was detected, the category of pulp inflammation was increased (Figure 1). Following restoration with OB and LB, no pulp inflammation was observed, which compares to some inflammation in the absence of bacterial microleakage with AB and SB, and some inflammation with AB, SB and AM in the presence of bacterial microleakage (Figure 1). Overall, pulp inflammation was detected in 14 of the 130 cavity restorations, seven in the absence of bacteria and seven in the presence of bacteria (Table 4). This compares with 13 cavity restorations with bacterial microleakage but without any pulpal inflammation, indicating that pulpal inflammation is not always observed when bacterial microleakage is present (Table 4).

Reactionary Dentin

In this investigation, the reactionary dentin area was used as a measure of pulp regeneration. Reactionary dentin was identified as tertiary dentin with a tubular continuity with the primary odontoblasts (Figure 4). No reparative dentin was observed during this study. The only variable influencing the reactionary dentin area was the difference between adhesive systems; all the other variables had little effect (Table 3). However, the reactionary dentin area was a very time-dependent repair activity. Considerably greater areas of reactionary dentin were observed over longer periods (80-to-172 days), compared with shorter periods (3-to-79 days). Using SB as a baseline measure, the long-term reactionary dentin area of other restorative materials as a fraction of SB was OB (92%), AB (63%), LB (47%) and AM (43%) (Figure 2). However, the large standard deviation of the mean area indicates that the area of reactionary dentin was highly variable within each type of adhesive system.

Bacterial Microleakage

The microleakage of bacteria into cavity restorations appeared to have some effect on pulp inflammation but not any of the other factors, such as the type of adhesive system. This suggests that bacterial microleakage stimulated pulp inflammation (Figure 1), but the ability of the systems to prevent bacterial microleakage were similar to each other (Table 3). OB was the only system to totally prevent bacterial microleakage (Table 4), while 16% of AB, 16% of SB, 17% of AM and 33% of LB showed some infiltration of bacterial microleakage into cavity margins.

Odontoblast Numbers

The number of odontoblasts was used as a measure of pulp injury. Odontoblast numbers beneath the cavity preparation were reduced by an average of -22% compared with odontoblast numbers that were independent of the site of cavity preparation (Figure 3). Some differences were observed between systems as follows: OB (-13%), LB (-21%), AM (-21%), SB (-23%) and AB (-26%) (Figure 3). However, none of these differences were statistically significantly different (Table 3). Inflammation, bacterial microleakage and reactionary dentin had little effect on odontoblast numbers (Table 3).

Cavity Remaining Dentin Thickness

No statistical differences at the $p=0.05$ significance were found between adhesive systems and resin composites and the RDT of cavity preparations.

DISCUSSION

All of the adhesive systems evaluated in this study were of the third- or fourth-generation, which are the most commonly used by clinicians (Pilo & Ben-Amar, 1999). Investigations of pulp injury, repair, inflammation and bacterial microleakage are necessary to improve the success of restorative dental treatment. Previous reports of pulp reactions highlighted pulp injury and inflammation problems with earlier types of restorative materials, especially calcium hydroxide (Cox, 1994; Cox & Suzuki, 1994) and early adhesive systems (Fukushi & Fusayama, 1980). These reports have led to the development of systems with improved sealing properties, thus, explaining the reduced inci-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

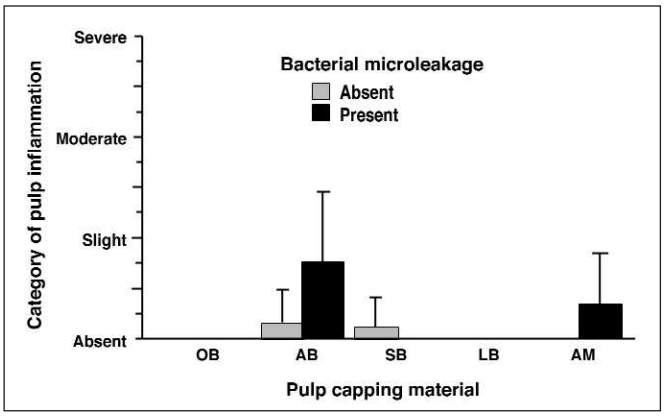


Figure 1. *Resin composite materials and inflammation.*

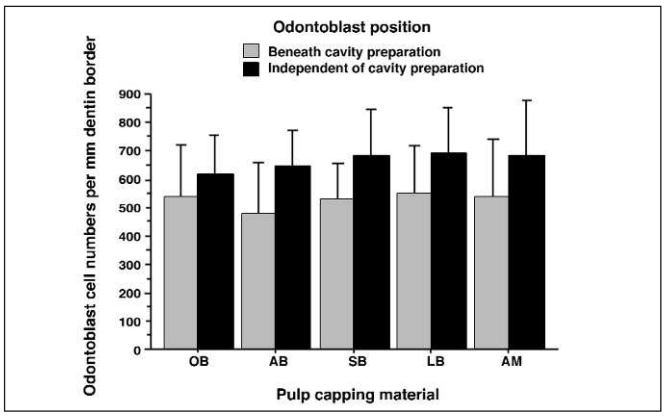


Figure 2. *Resin composite materials and odontoblast cell numbers.*

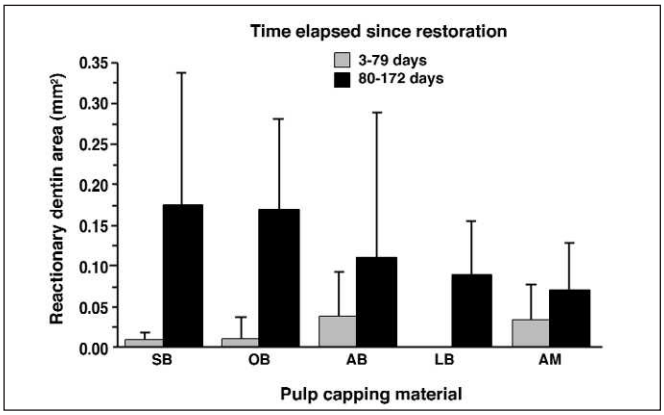


Figure 3. *Resin composite materials and reactionary dentin area.*

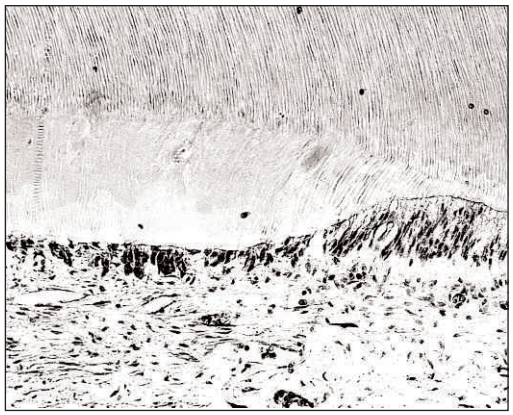


Figure 4. *Histology of reactionary dentin.*

dence of bacterial microleakage observable with currently available products. Nevertheless, despite this improvement, the findings of this investigation demonstrate that current adhesive systems are not yet fully optimized. This is because up to 33% of teeth restored with some type of adhesive permit bacterial microleakage, and 22% of the teeth restored with certain types of adhesives showed some inflammation, sometimes, even in the absence of bacterial microleakage. This study did not examine the chemical components of the adhesive products, but chemical differences in their formulation may explain some differences in pulpal reactions.

Previous investigations of cavity restoration have been criticized for being short-term, with some lasting between three and 60-days (Kitasako & others, 1999), while long-term ISO guidelines (1997) specify 70 +/- 5 days. While the accuracy of short-term investigations appears questionable to determine long-term responses, the authors' observations of the reactionary dentin area illustrate this point. Consider that it was not possible to predict the long-term area of reactionary dentin (80-172 days) from the short-term area of reactionary dentin (3-79 days). It is felt that this dilemma, created by the time-line of observation for measuring pulp responses, explains many differences as reported between investigations. A prime example is pulp inflammation that has often been associated with applying certain adhesives, particularly the self-etching types, onto exposed pulp tissue (Pameijer & Stanley, 1998) and deep cavity preparations (Hebling, Giro & Costa, 1999). While these studies have shown persistent pulpitis or chronic necrosis, long-term studies of up to 381 days in human teeth have shown that all pulp inflammation will resolve within 30 weeks, following non-pulp exposed restoration in the absence of bacterial microleakage (About & others, 2001). These examples indicate that the capacity of pulp regeneration and the resolution of inflammation may not be fully realized when investigations are conducted only to the ISO timeline guidelines. Consequently, the authors suggest that future investigations of pulp reactions to restorative treatment should always consider the effects of time and, if possible, the testing interval should be extended to provide for a more accurate long-term assessment of biocompatability.

The methods used in this study to examine pulp injury and healing have shown that using ANOVA and X^2 statistical tests allow for investigation of the inter-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

action of pulp injury and healing variables. Using these statistical methods has avoided any problems that would be created by the disparity in tooth numbers between groups. Future investigations should advance these statistical methods using post hoc pairwise comparisons of the data; this more in-depth analysis was not done in this study. While many statistical methods have been used to determine correlations between variables in previous investigations (Warfvinge, 1987; Cox & others, 1992; Murray & others, 2000; 2001), this is the first study to demonstrate P-value correlations for interactions between all the variables examined. While it may never be possible to fully correlate all pulp injury and repair reactions with aspects of cavity preparation, restorative treatment, patient factors and postoperative complications, it must be accepted that a failure to consider the effects of some variables has led to misinterpretations of results in the past. A notable example is the pulp injuries that were attributed to the chemical activity of restorative materials when, in fact, the source of the pulp injury was bacterial microleakage (Watts & Paterson, 1987). It is apparent that more complete statistical evaluations would help to improve the accuracy of histological investigations; in the future, this should allow for factors that contribute to clinical success to be more clearly identified.

Unresolved pulp inflammation is associated with postoperative sensitivity, discoloration, failure of pulp healing, reduced reactionary dentin formation and the eventual need for endodontic treatment (Bergenholtz & others, 1982). For these reasons the inflammation must always be avoided. In many observations, the majority of pulp inflammation was associated with bacterial microleakage (Cox, 1994; Bergenholtz, 1990), leading to the recommendation that the most important factor for achieving clinical success is the prevention of bacterial microleakage (Cox & Suzuki, 1994; Murray & others, 2002b). A total of between seven and 130 cavity preparations were associated with pulpal inflammation in the absence of bacterial microleakage, suggesting that some pulpal irritation from the chemical activity of the adhesive systems has occurred. There are many reports of adverse pulp reactions to adhesive systems, especially the self-etching types investigated in this study (Hebling & others, 1999; de Souza Costa & others, 2001). However, these observations must be put into perspective. Only absent or slight categories of pulp inflammation were observed, and the mean level of pulp inflammation in the absence of bacterial microleakage was less than when bacterial microleakage was present. Furthermore, these seven cavity preparations were not only compared with the 13 cavity preparations associated with bacterial microleakage, but also an absence of pulpal inflammation. This data suggests that adhesive systems are more likely to prevent pulpal inflammatory activity in the presence of bacterial microleakage, rather than stimulate inflammatory activity due to chemical irritation. Clearly, there is a need to avoid all sources of pulp inflammation, and the 22% of AB, 8% of SB and 6% of AM-restored teeth exhibiting inflammation suggest that there is potential for improvement, overall, because each of these materials passed the inflammation requirements according to ISO guidelines (1997).

Reactionary dentin, sometimes referred to as reparative dentin secretion by odontoblasts, appears to be primarily stimulated by a loss of dentin or the degree of injury of the physiological dentin matrix (Cox & others, 1992), although the restorative material also appears to have some influence (Mjör, 1983; Mjör & Ferrari, 2002). While calcium hydroxide is often associated with comparatively large areas of reactionary dentin, and zinc oxide eugenol is normally associated with the smallest area of reactionary dentin (Stanley, 1968), adhesive systems are associated with intermediate areas of reactionary dentin (Murray & others, 2000; 2001). The ability of pulpal odontoblasts to secrete reactionary dentin has recently come under renewed focus, following the realization that the regeneration of dentin can be used to accomplish successful restorative treatment (Mjör, 2002). In addition to reactionary dentin, odontoblasts can also increase the secretion of peritubular dentin beneath caries lesions and dentin damage or loss. This reduces dentin permeability and the penetration of caries lesions (Mjör, 2002). The slow, stepwise removal of deeply penetrating caries lesions, can stimulate reparative dentin secretion and avoid pulp exposure (Ricketts, 2001). Avoidance of pulp exposure can provide successful restorative treatment in 86% of cases, lasting more than 10 years (Mertz-Fairhurst & others, 1998); this compares with 13% of successful direct pulp capping treatments over the same time period (Barthel & others, 2000). This investigation has shown that SB, OB and AB are associated with greater reactionary dentin secretion than LB or AM, but all of these adhesive systems provide a suitable environment for pulp regeneration to proceed normally. RDT is extremely important in stimulating pulp reactions; therefore, great care has been taken to ensure that RDT was randomly distributed among restorative materials to minimize any possible differences in RDT. ANOVA statistics found no statistical differences between the RDT of cavity preparations restored with the different restorative materials. Therefore, the effects of RDT were not examined in this study. To accomplish successful treatment, the use of restorative materials must be congruent with the natural healing activity of teeth (Murray & others, 2000). In this study, none of the tested adhesive systems appeared to impair the healing of teeth following cavity restoration. Although increasing the number of histological investigations and reviews supports adhesive systems, particularly for restoring posterior, non-exposed preparations in the teeth of healthy young adults (Ranly & García-Godoy, 2000; Schuurs,

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Gruythuysen & Wesselink, 2000), reports of certain adhesives being associated with persistent pulpitis and delayed pulp regeneration and dentin secretion (Costa, Mesas & Hebling 2000; de Souza Costa & others, 2001) remains an issue. An important factor linking most of the negative reports of certain adhesives is that they are frequently used in the most severe circumstances when the pulp has already been mechanically injured. If the tooth pulp is injured to the extent that restorative treatment is prone to fail, there are probably no types of restorative materials available that will enable the pulp capping treatment to be successful. Nevertheless, it must be accepted that operator sensitivity of adhesive systems (Lutz, Krejci & Besek, 1997) and careful selection of the types of injured teeth to be restored might have dramatic effects on the clinical success of these systems. Consequently, careful selection of the use of systems is recommended, together with the continued use of conventional materials, particularly, amalgam and resin-modified glass ionomers. In some circumstances, these materials may provide a better clinical performance for restoring teeth, particularly when the benefits of using adhesive systems are not required.

CONCLUSIONS

This study has shown that all tested adhesives provide for successful pulp healing following non-exposed pulp cavity restoration. The preservation of odontoblast numbers beneath cavity preparations demonstrates the lack of pulp injury associated with adhesives, while the secretion of reactionary dentin showed that the natural regenerative activity of pulp proceeds normally. Pulp inflammation remained at very low levels, passing the ISO guidelines for biocompatibility. However, 33% of pulp with bacterial microleakage and 22% of pulp with inflammation remain a matter of concern. Consequently, improvements in adhesives are still necessary. These improvements should include: antibacterial activity (Imazato & others, 2001), improved sealing and bond strengths (Pereira, Segala & Souza Costa, 2000), caries prevention (Christensen, 2000), improved biocompatibility and improved ease of handling.

(Received 7 June 2002)

References

About I, Murray PE, Franquin J-C, Remusat M & Smith AJ (2001) Pulpal inflammatory responses following non-carious Class V restorations *Operative Dentistry* **26(4)** 336-342.

Akimoto N, Momoi Y, Kohno A, Suzuki S, Otsuki M, Suzuki S & Cox CF (1998) Biocompatibility of Clearfil Liner Bond 2 and Clearfil AP-X system on non-exposed and exposed primate teeth *Quintessence International* **29(3)** 177-188.

Arnst C & Carey J (1998) Biotech bodies *Business Week* **July** 42-49.

Barthel CR, Rosenkranz B, Leuenberg A & Roulet JF (2000) Pulp capping of carious exposures: Treatment outcome after 5 and 10 years: A retrospective study *Journal of Endodontics* **26(9)** 525-528.

Bergenholtz G, Cox CF, Loesche WJ & Syed SA (1982) Bacterial leakage around dental restorations: Its effect on the dental pulp *Journal of Oral Pathology* **11(6)** 439-450.

Bergenholtz G (1990) Pathogenic mechanisms in pulpal disease *Journal of Endodontics* **16(2)** 98-101.

Bergenholtz G (2000) Evidence for bacterial causation of adverse pulpal responses in resin-based dental restorations *Critical Reviews in Oral Biology & Medicine* **11(4)** 467-480.

Berry TG, Summit JB, Chung AK & Osborne JW (1998) Amalgam at the new millennium *Journal of the American Dental Association* **129(11)** 1547-1556.

Burke FJ, Cheung SW, Mjör IA & Wilson NH (1999) Reasons for the placement and replacement of restorations in vocational training practices *Primary Dental Care* **6(1)** 17-20.

Christensen GJ (2000) The need for caries-preventive restorative materials *Journal of the American Dental Association* **131(9)** 1347-1349.

Clinical Research Associates Newsletter (1995) Use survey **19** 4

Costa CA, Mesas AN & Hebling J (2000) Pulp response to direct pulp capping with an adhesive *American Journal of Dentistry* **13(2)** 81-87.

Cox CF, Heys DR & Heys RJ (1977) A gravity perfusion technique for lab animals *Laboratory Animal* **6(4)** 18-22.

Cox CF, Keall CL, Keall HJ, Ostro E & Bergenholtz G (1987) Biocompatibility of surface-sealed dental materials against exposed pulps *Journal of Prosthetic Dentistry* **57(1)** 1-8.

Cox CF, White KC, Ramus DL, Farmer JB & Snuggs HM (1992) Reparative dentin: Factors affecting its deposition *Quintessence International* **23(4)** 257-270.

Cox CF (1994) Evaluation and treatment of bacterial microleakage *American Journal of Dentistry* **7(5)** 293-295.

Cox CF & Suzuki S (1994) Re-evaluating pulp protection: Calcium hydroxide liners vs cohesive hybridization *Journal of the American Dental Association* **125(7)** 823-831.

Cox CF, Hafez AA, Akimoto N, Otsuki N, Suzuki S & Tarim B (1998) Biocompatibility of primer, adhesive and resin composite systems on non-exposed and exposed pulps of non-human primate teeth *American Journal of Dentistry* **11(Special Issue)** S55-S63.

Dawson-Saunders B & Trapp RG (1994) *Basic and Clinical Biostatistics* 2nd ed Norwalk, CT, Appleton & Lange, p 210-231.

de Souza Costa CA, Lopes DO, Nascimento AB, Teixeira HM & Fontana UF (2001) Response of human pulps capped with a self-etching adhesive system *Dental Materials* **17(3)** 230-240.

Duke ES (1993) Adhesion and its application with restorative materials *Dental Clinics of North America* **37(3)** 329-340.

Fukushi Y & Fusayama T (1980) Effect of cyanoacrylate treatment of cavity walls *Journal of Dental Research* **59(4)** 662-669.

Fusayama T, Nakamura M, Kurosaki N & Iwaku M (1979) Non-pressure adhesion of a new adhesive restorative resin *Journal of Dental Research* **58(4)** 1364-1370.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Hebling J, Giro EMA & Costa CAS (1999) Human pulp response after an adhesive system application in deep cavities *Journal of Dentistry* **27(8)** 557-564.

Inoue S, Vargas MA, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, Sano H & Van Meerbeek B (2001) Microtensile bond strength of 11 contemporary adhesives to dentin *Journal of Adhesive Dentistry* **3(3)** 237-245.

ISO 7405 (1997) Pre-clinical evaluation of biocompatibility of medical devices used in dentistry: Test methods for dental materials Geneva, Switzerland: *International Organization for Standardization*.

Imazato S, Torii Y, Takatsuka T, Inoue K, Ebi N & Ebisu S (2001) Bactericidal-effect of dentin primer containing antibacterial monomer methacryloyloxydodecylpyridinium bromide (MDPB) against bacteria in human carious dentin *Journal of Oral Rehabilitation* **28(4)** 314-319.

Kugel G & Ferrari, M (2000) The science of bonding: From first- to sixth-generation *Journal of the American Dental Association* **131(Supplement)** 20S-25S.

Kitasako Y, Inokoshi S & Tagami J (1999) Effects of direct pulp capping techniques on short term response of the mechanically exposed pulps *Journal of Dentistry* **27(4)** 257-263.

Lutz FU, Krejci I & Besek M (1997) Operative dentistry: The missing clinical standards *Practical Periodontal Aesthetic Dentistry* **9(5)** 541-548.

Maupome G & Sheiham A (1998) Criteria for restoration replacement and restoration life-span estimates in an educational environment *Journal of Oral Rehabilitation* **25(12)** 896-901.

McKay GS (1970) Gram stain modified to improve color contrast *Journal of Clinical Pathology* **23(2)** 191.

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(1)** 55-66.

Mjör IA (1983) Biological and clinical properties in Mjör IA, editor, *Dental Materials, Biological Properties and Clinical Evaluation*, Boca Raton; CRC Press Boca Raton, FL 91-121.

Mjör IA, Jokstad A & Qvist V (1990) Longevity of posterior restorations *International Dental Journal* **40(1)** 11-17.

Mjör IA & Ferrari M (2002) Pulp-dentin biology in restorative dentistry Part 6: Reactions to restorative materials, tooth restoration interfaces, and adhesive techniques *Quintessence International* **33(1)** 35-63.

Mjör IA (2002) Pulp-dentin biology in restorative dentistry Quintessence Publishing, London.

Murray PE, About I, Lumley PJ, Smith G, Franquin J-C & Smith AJ (2000) Post operative pulpal and repair responses *Journal of the American Dental Association* **131(3)** 321-329.

Murray PE, About I, Franquin J-C Remusat M & Smith AJ (2001) Restorative pulpal and repair responses *Journal of the American Dental Association* **132(4)** 482-491.

Murray PE, Smyth TW, About I, Remusat R, Franquin J-C & Smith AJ (2002a) The effect of etching on bacterial microleakage of an adhesive composite restoration *Journal of Dentistry* **30(1)** 29-36.

Murray PE, Hafez AA, Smith AJ & Cox CF (2002b) Bacterial microleakage and pulp inflammation associated with various restorative materials *Dental Materials* **18(6)** 470-478.

Pameijer CH & Stanley HR (1998) The disastrous effects of the "Total Etch" technique in vital pulp capping in primates *American Journal of Dentistry* **11(Special Issue**) S45-S59.

Pereira JC, Segala AD & Souza Costa CA (2000) Human pulpal response to direct pulp capping with an adhesive system *American Journal of Dentistry* **13(3)** 139-147.

Pilo R & Ben-Amar A (1999) Comparison of microleakage for three one-bottle and three multiple-step dentin bonding agents *Journal of Prosthetic Dentistry* **82(2)** 209-213.

Ranly DM & García-Godoy F (2000) Current and potential pulp therapies for primary and young permanent teeth *Journal of Dentistry* **28(3)** 153-161.

Ricketts D (2001) Management of the deep carious lesion and the vital pulp dentine complex *British Dental Journal* **191(11)** 606-610.

Schuurs AH, Gruythuysen RJ & Wesselink PR (2000) Pulp capping with adhesive resin-based composite vs calcium hydroxide: A review *Endodontics & Dental Traumatology* **16(6)** 240-250.

Shavell HM (1980) The amalgapin technique for complex amalgam restorations *Journal of the California Dental Association* **8(4)** 48-55.

Stanley HR (1968) Design for a human pulp study I *Oral Surgery Oral Medicine Oral Pathology* **25(4)** 633-647.

Swerdlow H & Stanley HR (1958) Reaction of the human dental pulp to cavity preparation I Effect of water spray at 20,000 rpm *Journal of the American Dental Association* **56(3)** 317-329.

Tarim B, Hafez AA, Suzuki SH, Suzuki S & Cox CF (1998) Biocompatibility of Optibond and XR-Bond adhesive systems in non-human primate teeth *International Journal of Periodontics & Restorative Dentistry* **18(1)** 86-99.

Vann WF Jr, Barkmeier WW & Mahler DB (1988) Assessing composite resin wear in primary molars: Four-year findings *Journal of Dental Research* **67(5)** 876-879.

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, Perdigão J, Lambrechts P & Vanherle G (2000) Enamel and dentin adhesion in RS Schwartz, JB Summit & JW Robbins (eds) *Fundamentals of Operative Dentistry: A Contemporary Approach*, Chicago, Quintessence Publishing 2nd edition.

Warfvinge J (1987) Morphometric analysis of teeth with inflamed pulp *Journal of Dental Research* **66(1)** 78-83.

Watts A & Paterson RC (1987) Bacterial contamination as a factor influencing the toxicity of materials to the exposed dental pulp *Oral Surgery, Oral Medicine, Oral Pathology* **64(4)** 466-474.

Zöllner A & Gaengler P (2000) Pulp reactions to different preparation techniques on teeth exhibiting periodontal disease *Journal of Oral Rehabilitation* **27(2)** 93-102.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3**, 251-259

Laboratory Research

Cytotoxicity of Conventional and Modified Glass Ionomer Cements

WH Lan • WC Lan • TM Wang • YL.Lee
WY Tseng • CP Lin • MC Chang • JH Jeng

Clinical Relevance

Since resin-modified glass ionomer cements such as Compoglass, ProTec CEM, Fuji II LC and GC Lining cement show strong toxicities to pulp cells, it is not recommended that resin-modified GICs be directly applied onto dental pulp cells.

SUMMARY

Various glass ionomer cements (GICs) and resin-modified GICs are widely used as tooth-colored restorative materials. However, their potential effects on pulp tissues are not fully understood. In this study, the authors compared the toxicity of nine types of GICs on cultured human dental pulp cells. Exposure of pulp cells to GICs for five days led to differential growth inhibition as analyzed by 3-(4,5-dimethyl-thiazol-2-yl)-2,5-diphenyl-tetrazolium bromide (MTT) assay. Exposure of pulp cells to ProTec CEM, Fuji II LC, Compoglass and GC Lining cement for five days decreased the cell numbers to 11%, 12%, 19% and 25%, respectively, of the control. Exposure of pulp cells to Fuji IX, GIC FX and Fuji II SC also decreased cell numbers by 62%, 33% and 24%, respectively. By contrast, Hy-Bond and Fuji I showed only mild suppression on the growth of pulp cells, with 12% and 16% decreased cell numbers. Morphologically, marked retraction and rounding of pulp cells were noted following exposure to GC Lining cement; in addition, cell surface blebbing was noted following exposure to Compoglass, Fuji II LC and ProTec CEM. Exposure of the pulp cells to Fuji II SC and Fuji IX, however, led to decreases in the cell density, with no obvious morphological changes. These results indicate that resin-modified GICs, such as Compoglass, Fuji II LC, ProTec CEM and GC Lining cements, are more toxic to pulp cells than conventional GICs. It is not recommended that resin-modified GICs be directly applied onto dental pulp cells. However, additional *in vivo* studies

Wan-Hong Lan, BDS, DMSc, professor and dean, National Taiwan University, Taipei, Taiwan

Wen-Chien Lan, MS, National Taiwan University, Taipei, Taiwan

Tong-Mei Wang, BDS, MS, National Taiwan University, Taipei, Taiwan

Yuan-Ling Lee, BDS, MS, PhD student, National Taiwan University, Taipei, Taiwan

Wan-Yu Tseng, BDS, MS, teaching assistant, National Taiwan University, Taipei, Taiwan

Chun-Pin Lin, BDS, MS, PhD, professor and head, National Taiwan University, Taipei, Taiwan

Mei-Chi Chang, MS, PhD, associate professor, Chang-Gung Institute of Nursing

*Jiiang-Huei Jeng, BDS, PhD, associate professor, Department of Dentistry, National Taiwan University Hospital and Laboratory of Dental Pharmacology, Toxicology and Pulp Biology, National Taiwan University College of Medicine

*Reprint request: No 1, Chang-Te Street, Taipei, Taiwan; e-mail: huei@ha.mc.ntu.edu.tw

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

are needed to evaluate the potential toxicities of these resin-modified GICs during clinical operative procedures.

INTRODUCTION

Glass ionomer cements (GICs) are widely used as restorative materials, due to fluoride release, dentin adhesion and their esthetic color. GICs are also used as root canal sealers (Saunders & others, 1992), retrofilling material (Peltola, Salo & Oikarinen, 1992), bone substitutes (Brook & others, 1992) and to bond fractured roots (Firedman, Moshonov & Trope, 1993). However, GIC lacks sufficient mechanical properties and is brittle in nature. Resin-modified GICs were, therefore, developed to provide improved mechanical properties while retaining the advantages of conventional GICs (Mathis & Ferracane, 1989).

After operative procedures, the release of components from various dental materials may diffuse through dentinal tubules and potentially attack the target tissues, causing an adverse pulpal reaction (al-Fawaz, Gerzina & Hume, 1993; Ratanasathien & others, 1995; Geurtsen, 1998; Schmalz, 1998). Generally, the biocompatibility of conventional GICs is considered to be good, with minimal release of organic components (Kuhn, Lesan & Painter, 1983; Schmalz & others, 1994). Mjör, Nordahl & Tronstad (1991) filled Class V cavities of dog teeth with GIC and found that only slight pulpal reactions occurred. Nevertheless, if GIC was placed in direct contact with the pulp tissue, abscess formation was observed (Paterson & Watts, 1987; Schmalz & others, 1994; Schmalz, 1998). This reaction may result from the release of toxic ingredients from GIC, because GIC (Ketac-Fil, ESPE, BRD) eluents may inhibit the proliferation of gingival fibroblasts and rat osteosarcoma cells (Peltola & others, 1992). In addition, few studies have investigated the pulpal effects of resin-modified GICs. Felton and others (1991) found that light-cured GICs induce few toxic effects on the pulp, whereas, various GIC preparations have recently been reported to exhibit differential toxic effects on tissues. Sasanaluckit and others (1993) tested nine different GICs (including resin-modified GIC) and found that Vitrebond (3M, St Paul, MN, USA, a resin-modified GIC) is an extremely toxic GIC, whereas, Chem Fil, Ketac Fil and Ketac Silver exhibited few toxic effects on cultured cells. These differential toxic effects may be due to disparities in the compositions of GICs, such as polyacrylic acid, itaconic acid, tartaric acid, resin monomers and more (Stanley, 1992). Generally, conventional GICs are suggested to have minimal toxicity, whereas, resin-modified GICs are shown to exert cytotoxicity and genotoxicity (Sidhu & Schmalz, 2001).

In this study, the authors investigated the toxic effects of nine popularly used GICs on cultured human dental pulp cells to further evaluate whether conventional GICs are biocompatible to the pulp cells and whether resin-modified GICs are more toxic than conventional GICs.

METHODS AND MATERIALS

Chemicals and Materials

The nine materials used in this study were GC Lining cement, Fuji I, Fuji II SC, Fuji II LC, Fuji IX, ProTec CEM, Compoglass, GIC FX and Hy-Bond. Table 1 describes their primary use, curing method and whether or not the material is resin-modified. Compoglass, ProTec CEM and Fuji II LC are resin-modified glass ionomer cements.

Cultures of Human Dental Pulp Cells

Human dental pulp cells were cultured using an explant technique as described previously (Chang & others, 1998a,b; Chen & others, 2001). Briefly, healthy human teeth were obtained during third-molar extraction after proper informed written consent was obtained. For culture of dental pulp cells, freshly extracted human third molars were immediately split with a hammer. The pulp tissues were removed with a curette. Pulp tissues were minced into small pieces and cultured in Dulbecco's modified Eagle's medium (DMEM) containing 10% fetal calf serum (FCS) with penicillin/streptomycin. Cell passages from numbers three to eight were used for this study.

Preparation of GICs and Resin-Modified GICs

The ratio of powder and liquid of the various GICs and resin-modified GICs were used according to the instructions supplied by the respective manufacturers. The materials were dispensed, mixed and applied onto the base of the transwells (Costar Transwell-Clear, 6.5 mm diameter, with a pore size of 0.4 μm) to create a 2-mm material thickness. Resin-modified GICs were cured using a halogen lamp (Optilux, Demetron Research Corporation, CT, USA), whereas, self-curing GICs were allowed a one-hour setting period prior to being transferred into 24-well culture plates in order to compare the relative toxicities of the GICs. When the materials are not transferred to the culture wells, the components in the GICs will not diffuse into the culture medium and make contact with pulp cells. In this experimental condition, the relative toxicity of different GICs with pulp cells can be compared in equal condition.

Effect of GICs on Pulp Cell Growth

For comparing the effects of these GICs on the growth of pulp cells, the transwells containing various GICs and resin-modified GICs were transferred onto 24-well culture wells that were seeded with pulp cells (5×10^3 cells/well) 24 hours prior. Cells were further incubated for five days. Morphological changes in pulp cells were photographed under a phase-contrast

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 1: *Primary Uses, Curing Condition, Source, Resin Contents and Chemical Compositions of the Nine Tested GICs*

Material	Primary Uses	Curing Condition	Resin-Modified or Not	Components	Source
GIC FX	Class III and Class V cavity restoration	Self-curing	No	Powder: 34% silica, 26% aluminum oxide, 6.5% phosphoric anhydride, 10% fluorine, 5% strontium oxide, 6.5% calcium oxide, 12% lanthanum oxide and pigment; Liquid: 95% copolymer of acrylic acid and tricarboxylic acid, 5% tartaric acid.	Shofu Inc, Kyoto, Japan
Hy-Bond, GIC CX	Restoration of Class V Cavities	Self-curing	No	Powder: 97.85% Frit (SiO_2, $Al(OH)_3$, $AlPO_4$, CaF_2, SrF_2, LaF_3), 2.4% SiO_2, 0.25% additions ($C_{16}H_{10}O_9$, SnF_2, SrF_2, ZnO) and pigment; Liquid: 87.4% copolymer of acrylic acid and tri-carboxylic acid, and 6.5% tartaric acid.	Shofu Inc, Kyoto, Japan
ProTec CEM	Cementation of inlays, crowns and bridges	Self-curing	Yes	Powder: 2% silicon dioxide, 72% Ba-Al-fluorosilicate, 25% ytterbium trifluoride, initiator, particle size 7.5 µm; Liquid: 25% methacrylate-modified polyacrylic acid, 28% dimethacrylate, 28% HEMA, water	Vivadent Dental GmbH, Jagst, Germany
Compoglass	Class I, II, III and V cavity restoration	Light-curing	Yes	Powder (77%): ytterbium trifluoride, Ba-Al-fluorosilicate, catalyst, mixed oxide, particle size 0.2-3 µm; Liquid (22.75%): urethane dimethyacrylate (UDMA), tetraethylene glycol dimethacrylate (TEGDMA), cycloaliphatic dicarboxylic acid dimethacrylate	Vivadent Dental GmbH, Jagst, Germany
Fuji I	Base and liner, or for cementation of crowns, bridges and banding	Self-curing	No	Powder: 95% fluoro-alumino silicate glass, 5% polyacrylic acid powder; Liquid: 40% polyacrylic acid, 10% polybasic carboxylic acid.	GC Corp, Tokyo, Japan
GC Lining cement	Liner	Self-curing	No	Powder: 100% fluoro-alumino silicate glass, Liquid: 40% polyacrylic acid.	GC Corp, Tokyo, Japan
Self-curing Fuji II	Class III and V cavity restoration and primary teeth	Self-curing	No	Powder: 95% fluoro-alumino silicate glass, 5% polyacrylic acid powder; Liquid: 40% polyacrylic acid, 10% polybasic carboxylic acid.	GC Corp, Tokyo, Japan
Fuji II LC	Class III and V operative restoration	Light curing	Yes	Powder: 100% fluoro-alumino silicate glass; Liquid: 50% polyacrylic acid, 15% 2-hydroxyethyl methacrylat (HEMA) and initiator.	GC Corp, Tokyo, Japan
Fuji IX	Class I, II, III, and V cavity restoration (non-load bearing area), core build-up	Self-curing	No	Powder: 95% fluoro-alumino silicate glass, 5% polyacrylic acid powder; Liquid: 40% polyacrylic acid, 10% polybasic carboxylic acid.	GC Corp, Tokyo, Japan

microscope using a Nikon camera. Cell numbers were measured with a 3-(4,5-dimethyl- thiazol-2-yl)-2,5-diphenyl-tetrazolium bromide (MTT) assay as described previously (Jeng & others, 1999; Jeng & others, 2000; Chen & others, 2001). Briefly, transwells with or without tested materials (negative control) were removed, and cell layers were incubated with fresh culture medium containing 0.5 mg/ml of MTT for two hours. The produced formazan was dissolved in 0.5 ml of dimethylsulfoxide (DMSO) and read with a Dynatech Microwell plate reader at an optical density of 540 nm. The original optical density values of test

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

wells at 540 nm were expressed as a percentage of the optical density of the untreated control after subtraction of the blank value. A blank well was regularly used for data substraction by placing culture medium with MTT into culture wells that contained a transwell but no materials. Five separate experiments were performed for comparison. Results are expressed as absorbance (Abs) value at OD_{540} (MTT reduction) and as percentages of the control (as 100%) (mean ± SE) (n=5). Regression analysis was used to correlate cell numbers and OD_{540} values. One-way ANOVA was used to compare the differences between test materials and the untreated control

RESULTS

Untreated fibroblasts proliferated to a nearly confluent state after five days of incubation. These pulp cells generally appeared spindle-shaped with extended cellular processes (Figure 1a). Following exposure to GC Lining cement, the density of pulp cells decreased with the presence of marked intercellular spaces. Some of the pulp cells became retracted or rounded, with a loss of functional organization (Figure 1b). Exposure of pulp cells to GIC FX, Fuji I and Hy-Bond for five days produced few effects on cell density and morphology (data not shown). Most of the pulp cells became rounded with marked surface blebbing (arrowheads) after a five-day exposure to Compoglass (Figure 1c). Self-curing Fuji II (Fuji II SC) induced no marked morphological changes in pulp cells under the phase contrast microscope. Pulp cells are generally spindle-shaped in appearance. However, the density of cells within a defined area is below the control well (Figure 1d). Fuji II LC and ProTec CEM led to cell rounding and surface blebbing of pulp cells, similar to the pulp cells treated with Compoglass (data not shown). Pulp cells appeared more slender in appearance with more extended cellular processes following exposure to Fuji IX, even though a decrease in cell density was noted (Figure 1e).

Mitochondrial dehydrogenase activity has been widely used for measuring chemical cytotoxicity. Viable cells can reduce MTT to insoluble formozan by mitochondrial dehydrogenase. The authors, therefore, used the 3-(4,5-dimethyl-thiazol-2-yl)-2,5-diphenyl-tetrazolium bromide (MTT) assay to elucidate changes in pulp cell numbers following exposure to various GICs for five days. Generally, the optical density of MTT reduction produced a highly significant (r^2=0.99) linear correlation with cell numbers for cell densities ranging from 1.25 x 10^4 cells/well to 10 x 10^4 cells/well (data not shown).

Consistently, exposure to GICs and resin-modified GICs for five days showed differential effects on the growth of pulp cells. As shown in Figure 2a, the control pulp cells were viable and able to reduce MTT to insoluble formozan following culturing for five days, as indicated by an optical density (OD_{540}) value of 0.371. This OD_{540} value of 0.371 is comparable to about 3 x 104 cells, indicating proliferation of pulp cells in this culture condition. Exposure of pulp cells to GIC FX, Fuji I, Hy-Bond and Fuji II SC decreased the OD_{540} value to 0.256, 0.315 (p>0.05), 0.289 and 0.283 (p<0.05), which showed no or a marginally significant difference. On the contrary, ProTec CEM, Compoglass, GC Lining cement, Fuji II LC and Fuji IX markedly decreased the OD_{540} value (p<0.01), indicating the induction of moderate to severe cytotoxicity. In order to easily evaluate the extent of cytotoxicity following exposure to GICs, viable cell numbers are also presented as a percentage of the control (as 100%). As shown in Figure 2b, ProTec CEM, Fuji II LC, Compoglass and GC Lining cement showed moderate to severe toxicity as revealed by decreases in cell numbers to 11%, 12%, 19% and 25% of the control, respectively. Exposure to Fuji IX, GIC FX and Fuji II SC also decreased cell numbers to 38%, 67% and 76%, respectively, relative to the untreated control. Hy-Bond and Fuji I slightly suppressed the growth of pulp cells with 12% and 16% decreases in cell numbers.

DISCUSSION

Although a number of *in vivo* and *in vitro* studies have revealed little pulpal adverse effects of GIC (Kuhn & others, 1983; Mjör & others, 1991; Schmalz & others, 1994), direct contact of pulp tissue with GIC may exert potential pulpal damage (Sasanaluckit & others, 1993; Schmalz & others, 1994; Schmalz, 1998) especially when resin-modified GICs are used. Disparities in the cytotoxicities of different GICs on cultured mammalian cells have also been reported (Sasanaluckit & others, 1993). The authors found that pulp cells showed significantly different responses to the nine different kinds of popularly used GICs.

Fuji I luting cement was developed for optimal film thickness to ensure complete seating of cemented restorations (Ewoldsen, 2000). It contains 95% fluoroalumino silicate and 5% polyacrylic acid powder, whereas, its liquid contains 40% polyacrylic acid and 10% polybasic carboxylic acid (Table 1). Cell culture media that contains eluents of Fuji I cement has been shown to exert only mild inhibitory effects on the growth of hamster oral epithelial cells with 12% inhibition (Lewis & others, 1996). This is generally consistent with previous reports that have found that conventional GICs show little detrimental effect to the pulp (Kuhn & others, 1983; Mjör & others, 1991; Schmalz & others, 1994). Consistently, Fuji I has also been shown to exert few toxic effects on pulp cells in this study. Hy-Bond, the other conventional GIC that contains mainly acrylic acid, tricarboxylic acid and tartaric acid (Table 1), also showed no marked effect on the growth of pulp cells. A previous report suggests that type II GIC (Fuji II) is more biocompatible than the Type I luting GIC

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Figure 1: *Morphological alterations of pulp cells following exposure to various GICs: (a) Untreated pulp cells and pulp cells exposed to (b) GC Lining cement, (c) Compoglass, (d) self-curing Fuji II (Fuji II SC) and (e) Fuji IX. Arrowhead indicated cell surface blebbing. (100x, original magnification). (bar=100 µm.*

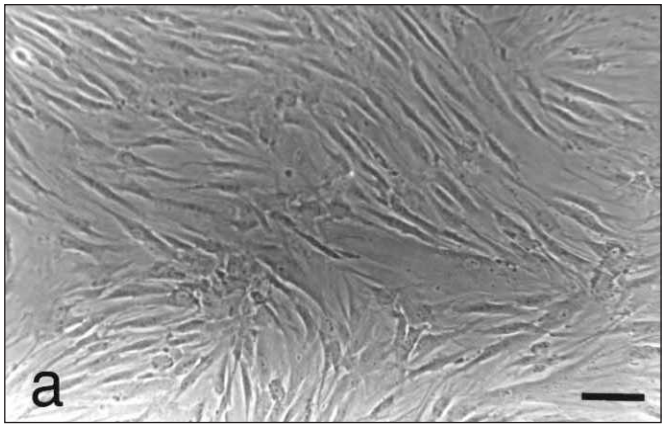


Figure 1A. *Morphology of cultured dental pulp cells.*

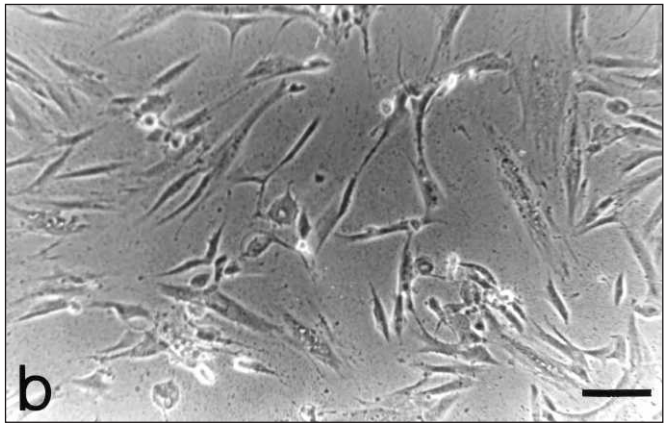


Figure 1B. *Morphology of pulp cells treated wtih GC Lining cement.*

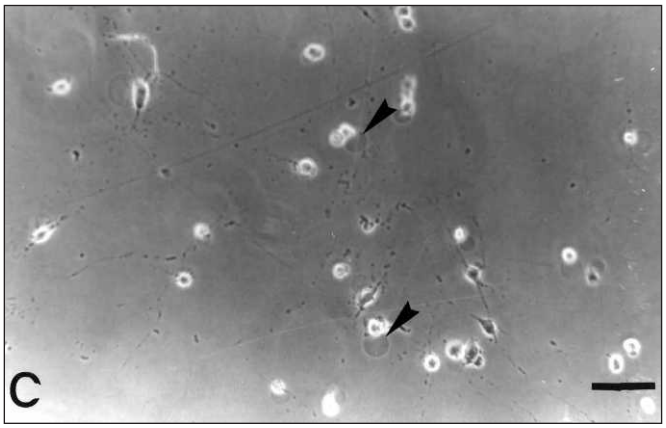


Figure 1C. *Morphology of pulp cells treated with Compoglass.*

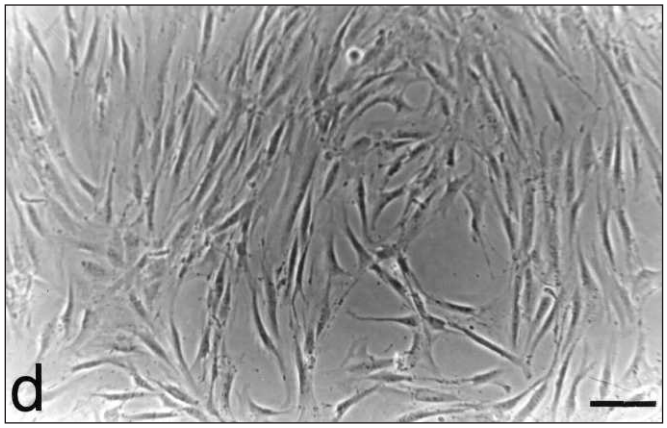


Figure 1D. *Morphology of pulp cells treated with Fuji SC.*

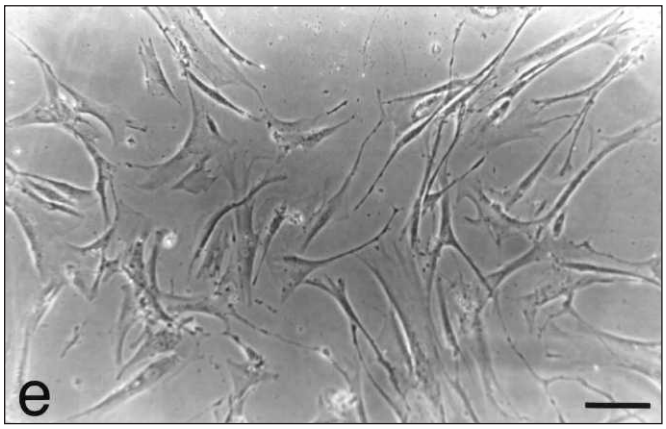


Figure 1E. *Morphology of pulp cells treated with Fuji IX.*

(Fuji I) (Mjör & others, 1991). However, the authors have found that self-curing Fuji II (Fuji II SC) was more toxic to pulp cells than Fuji I. Since Fuji I and Fuji II SC contain similar powder and liquid compositions (Table 1), differential toxicities may be due to disparities in the particle size of the powder or setting properties that affect the elution of chemicals from GIC. However, the clinical relevance of the differential toxicity is not clear. Mjör and others (1991) use GICs (Fuji I and Fuji II GIC) to restore the Class V cavities of dog teeth and found that only mild inflammatory reaction and mineralization disturbance occurred *in vivo*. The presence of the dentin barrier (>0.6 mm) that limits the direct contact of pulp cells with GIC may partially explain the results of this *in vivo* study. In addition to Fuji II SC, Fuji IX and GIC FX have evidently suppressed the growth of pulp cells in this study. This indicates that, in addition to differences in chemical composition of these GICs, additional factors such as curing condition and particle size may affect the toxicity of GICs. Recent *in vivo* study has noted that filling of rat upper molar Class V cavities with Fuji IX GIC induced disruption of the odontoblast layer, dilatation of blood vessels and transient inflammatory reactions after eight days. After 30-days of Fuji IX filling, the pulp tissue recovered and dis-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Figure 2: *Effect of nine kinds of GICs on the growth of pulp cells. Pulp cells ($5x10^5$) were incubated in DMEM supplemented with 10% FCS in transwells containing various GICs. Cells were further cultured for five days. Cell number was measured using a MTT assay. (a) Extent of MTT reduction as indicated by the absorbance (Abs) value at a wavelength of OD_{540}. *Denotes a significant difference (p<0.05). (b) Results are expressed as percentage of the untreated control (as 100%).*

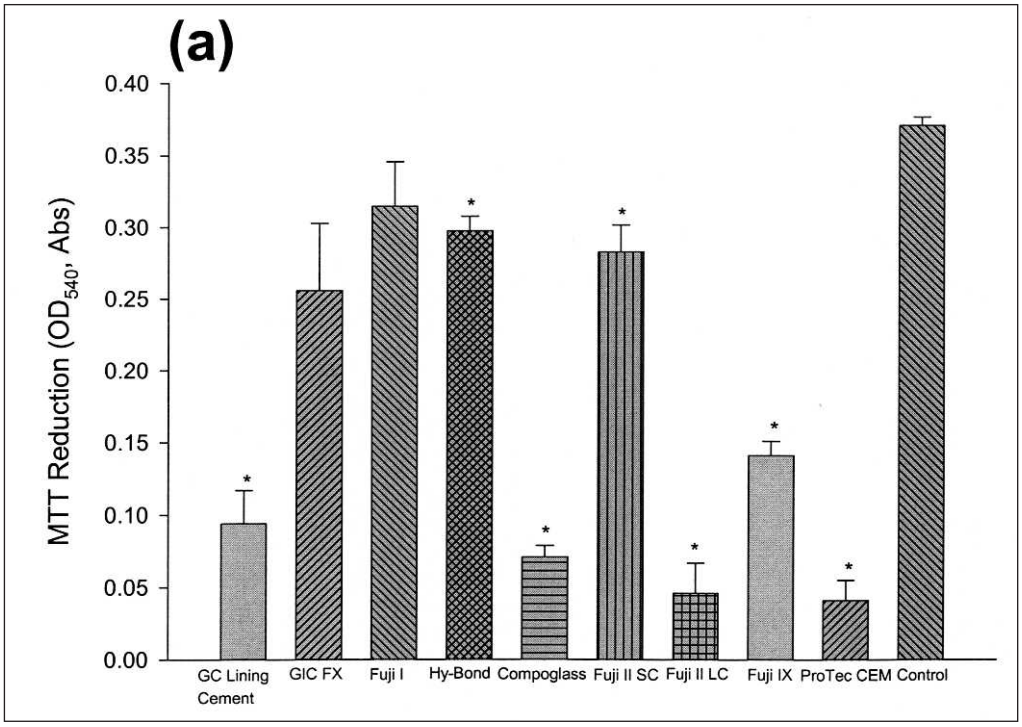


Figure 2A: *Effects of nine kinds of GICs on MIT reduction of pulp cells.*

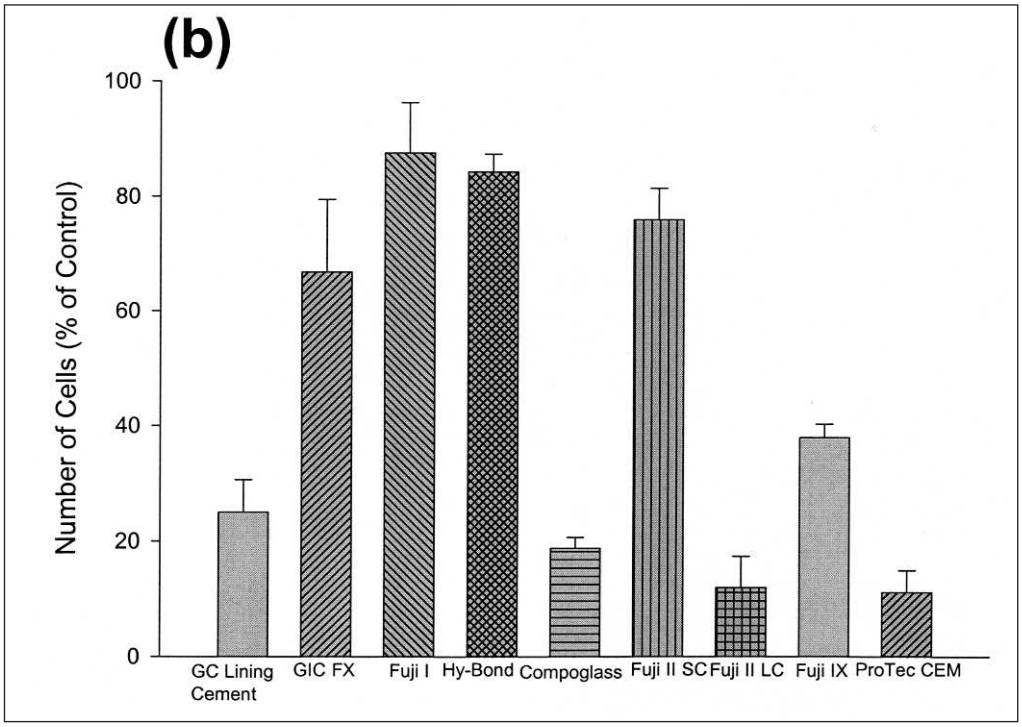


Figure 2B: *Effects of nine kinds of GICs on viable cell number.*

played a normal appearance (Six, Lasfargues & Goldberg, 2000). The presence of moderate toxicity of Fuji IX and GIC FX suggests that clinical application of these GICs must take into consideration the potential reversible or irreversible damage to dental pulp.

Few studies have addressed the toxicity of GIC lining cement on pulp cells. Recently, GC lining cement has been shown to induce morphological changes in pulp cells and inhibit the adhesion of pulp cells to substrate (Müller & others, 1990). Direct exposure of mouse L929 fibroblasts to medium eluents of Fuji II GIC elicited a strong cytotoxic response (Hume & Mount, 1988), which was more toxic than GC Lining cement. Similarly, the authors also noted that GC lining cement induced retraction and disruption of the functional cellular organization of dental pulp cells. Self-curing Fuji II (Fuji II SC) showed lower cytotoxicity to pulp cells than GC lining cement. Differences in the powder and liquid compositions, curing extent or particle size (Table 1) may partially explain these disparities. Using a transwell culture system for our tests, the exposure area of materials to the culture medium was consistently set at a 6.5-mm diameter transwell for all tested GICs. This is generally a more-simulating clinical exposure of pulp tissue to restorative materials, compared to most previous studies that used medium eluents of various GICs.

Fuji II SC and Fuji II LC were shown to exert little cytotoxicity on human osteoblasts. Vitremer, which

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

releases 2-hydroxyethyl-methacrylate (HEMA), however, showed strong cytotoxicity (Oliva & others, 1996). Stanislawski and others (2000) elucidated that Fuji II SC eluent is the most toxic agent to gingival fibroblasts (GF), followed sequentially by Fuji II LC and Compoglass. Toxicity of these biomaterials is related to their potency to reduce intracellular glutathione levels. However, Fuji II LC, which contains 15% HEMA (Table 1), showed marked cytotoxicity to pulp cells in this study. Moreover, it showed stronger toxic effects to pulp cells than conventional self-curing Fuji GICs. These findings generally agree with a previous report that resin-modified GICs (Vitrebond and Vitremer) show more-evident toxicity than conventional GICs (Chem Fil and Ketac Fil) on GF (Consiglio & others, 1998). Consiglio and others (1998) compared the cytotoxicity of four conventional GICs and two resin-modified GICs on GF. Results indicated that both conventional and resin-modified GICs may suppress the protein synthetic activity of GF. Several studies have suggested that toxicity of GIC may be related to changes in pH (Hume & Mount, 1988; Consiglio & others, 1998), and the possible release of fluoride (Sasanaluckit & others, 1993; Consiglio & others, 1998), HEMA (Ratanasathien & others, 1995; Consiglio & others, 1998) and aluminum ion (Forss, 1993). In addition to the contents of common ingredients of conventional GICs such as aluminum silicate glass, calcium, fluoride and polyacidic liquid (Smith, 1990; Oliva & others, 1996), HEMA has been found to release from Fuji II LC and light-curing GC Lining cement (Palmer, Anstice & Pearson, 1999). Thus, HEMA may be the major contributing factor to pulp toxicity. The lower powder/liquid ratio of GC Lining cement relative to Fuji II LC is another possible causative factor (Palmer & others, 1999), resulting in lower toxicity of GC Lining cement relative to Fuji II LC. The prolonged setting and hardening times of GICs prior to exposure of cells may possibly diminish the toxicity of GICs as revealed by decreasing cytotoxicity of Fuji II LC after a 24-hour setting period (Oliva & others, 1996). A more complete setting may decrease the amount of components released and allow for the evaporation of some of the toxic organic components from GICs leading to a rapid decrease in GIC cytotoxicity after setting (Hanks, Anderson & Craig, 1981; Meryon & Browne, 1984).

Filling human Class V cavities with Vitrebond resin-modified GIC induced marked inflammatory pulpal reactions and a large necrotic zone (do Nascimento & others, 2000). Compoglass and ProTec CEM are the other two widely used resin-modified GICs. Using Macaca monkeys as experimental animals, Compoglass was tested as a restorative material for Class V caries cavities and was shown to induce more evident pulpal inflammatory responses than IRM, whereas, bacterial ingrowth was suggested to mediate this inflammatory reaction (Tarim & others, 1997). However, Compoglass, which contains UDMA and TEGDMA (Table 1), was a strong cytotoxic agent to pulp cells in this study. Stanislawski and others (1999) suggested the leaching of toxic components such as HEMA and TEGDMA. Fuji II LC, being slightly more toxic than Compoglass, a finding consistent with that reported by Stanislawski and others (1999), suggested that HEMA may be a more toxic agent than other resin monomers in Compoglass. The authors of this research, however, have found that few studies have addressed the toxicity of ProTec CEM on cultured cells. This study has noted the potent cytotoxicity of ProTec CEM may be due to its contents of HEMA, dimethylacrylate and methacrylate-modified polyacrylic acid (Table 1). Moreover, these resin monomers (HEMA, TEGDMA and GMA) are also shown to impair the growth and functions of macrophages and may induce micronuclei formation in V79 cells (Bouillaguet & others, 2000; Schweikl, Schmalz & Spruss, 2001). Taken together, these results indicate that resin-modified GICs show markedly greater toxicity to pulp cells compared to conventional GICs. Differential cytotoxicities of GICs and resin-modified GICs may be due to disparities in the composition of powder and liquid and possibly particle size and setting properties. The clinical relevance of the observed toxicity of GICs on pulp cells is not fully clear. It is well known that calcium hydroxide also produces certain levels of necrosis (Yamamura, 1985); however, it is not considered a detrimental treatment by most clinicians. In fact, some investigators agree that it maybe a necessary pulpal response to produce a dentinal bridge (Yamamura, 1985). Thus, more *in vivo* studies are needed to confirm whether resin-modified GICs are more toxic to pulp tissues when applied as restorative materials and their clinical relevance.

CONCLUSIONS

1. Resin-modified GICs, such as Compoglass, ProTec CEM, Fuji II LC and GC Lining cement, were the most toxic to dental pulp cells, followed by conventional GICs such as Fuji IX, GIC FX and Fuji II SC. Hy-Bond and Fuji I showed little pulpal cytotoxicity.
2. It is not recommended that resin-modified GICs be directly applied onto dental pulp cells.

Acknowledgements

The authors thank Ms HF Jeng for her technical assistance. This study was supported by a grant from the National Science Council, Taiwan, ROC (NSC-89-2314-B002-187).

(Received 9 May 2002)

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

References

al-Fawaz A, Gerzina TM & Hume WR (1993) Movement of resin cement components through acid-treated dentin during crown cementation *in vitro Journal of Endodontics* **19(5)** 219-223.

Bouillaguet S, Wataha JC, Virgillito M, Gonzalez L, Rakich DR & Meyer JM (2000) Effect of sub-lethal concentrations of HEMA (2-hydroxyethyl methacrylate) on THP-1 human monocyte-macrophages, *in vitro Dental Materials* **16(3)** 213-217.

Brook IM, Craig GT, Hatton PV & Jonck LM (1992) Bone cell interactions with a granular glass-ionomer bone substitute material: *In vivo* and *in vitro* culture models *Biomaterials* **13 (10)** 721-725.

Chang MC, Lin CP, Haung TF, Lan WH, Lin YL, Hsieh CC & Jeng JH (1998a) Thrombin-induced DNA synthesis of cultured human dental pulp cells is dependent on its proteolytic activity and modulated by prostaglandin E2 *Journal of Endodontics* **24(11)** 709-713.

Chang MC, Lan WH, Chan CP, Lin CP, Hsieh CC & Jeng JH (1998b) Serine protease activity is essential for thrombin-induced protein synthesis in cultured human dental pulp cells: Modulation role of prostaglandin E2 *Journal of Oral Pathology & Medicine* **27(1)** 23-29.

Chen RS, Liuiw CC, Tseng WY, Hong CY & Jeng JH (2001) The effect of curing light intensity on the cytotoxicity of a dentin-bonding agent *Operative Dentistry* **26(5)** 505-510.

Consiglio R, Rengo S, Liguoro D, Riccitiello F, Formisano S, Palumbo G & Di Jeso B (1998) Inhibition by glass-ionomer cements of protein synthesis by human gingival fibroblasts in continuous culture *Archives of Oral Biology* **43(1)** 65-71.

do Nascimento AB, Fontana UF, Teixeira HM & Costa CA (2000) Biocompatibility of a resin-modified glass-ionomer cement applied as pulp capping in human teeth *American Journal of Dentistry* **13(1)** 28-34.

Ewoldsen N (2000) Fugi I luting cement *Journal of the American Dental Association* **131(2)** 148-150.

Felton DA, Cox CF, Odom M & Kanoy BE (1991) Pulpal response to chemically cured and experimental light-cured glass ionomer cavity liners *Journal of Prosthetic Dentistry* **65 (5)** 704-712.

Firedman S, Moshonov J & Trope M (1993) Resistance to vertical fracture of roots, previously fractured and bonded with glass ionomer cement, composite resin and cyanoacrylate cement *Endodontics & Dental Traumatology* **9(3)** 101-105.

Forss H (1993) Release of fluoride and other elements from light-cured glass ionomers in neutral and acidic conditions *Journal of Dental Research* **72(8)** 1257-1262.

Geurtsen W (1998) Substances released from dental resin composites and glass ionomer cements *European Journal of Oral Science* **106(2 Pt 2)** 687-695.

Hanks CT, Anderson M & Craig RG (1981) Cytotoxic effects of dental cements on two cell culture systems *Journal of Oral Pathology* **10(2)** 101-112.

Hume WR & Mount GJ (1988) *In vitro* studies on the potential for pulpal cytotoxicity of glass ionomer cements *Journal of Dental Research* **67(6)** 915-918.

Jeng JH, Chan CP, Ho YS, Lan WH, Hsieh CC & Chang MC (1999) Effects of butyrate and propionate on the adhesion, growth, cell cycle kinetics and protein synthesis of cultured human gingival fibroblasts *Journal of Periodontology* **70(12)** 1434-1442.

Jeng JH, Ho YS, Chan CP, Wang YJ, Hahn LJ, Lei D, Hsu CC & Chang MC (2000) Areca nut extract upregulates prostaglandin production, cyclooxygenase-2 mRNA and protein expression of human oral keratinocytes *Carcinogenesis* **21(7)** 1365-1370.

Kuhn AT, Lesan WA & Painter HA (1983) Release of organic species from glass ionomer cements *Journal Material Science Letter* **2** 224.

Lewis J, Nix L, Schuster G, Lefebvre C, Knoernschild K & Caughman G (1996) Response of oral mucosal cells to glass ionomer cements *Biomaterials* **17(11)** 1115-1120.

Mathis RS & Ferracane JL (1989) Properties of a glass ionomer resin composite hydrid material *Dental Materials* **5(5)** 355-358.

Meryon SD & Browne RM (1984) *In vitro* cytotoxicity of a glass ionomer cement of a new generation *Cell Biochemistry & Function* **2(1)** 43-48.

Mjör IA, Nordahl I & Tronstad L (1991) Glass ionomer cements and dental pulp *Endodontics & Dental Traumatology* **7(2)** 59-64.

Müller J, Horz W, Bruckner G & Kraft E (1990) An experimental study on the biocompatibility of lining cements based on glass ionomer as compared with calcium hydroxide *Dental Materials* **6(1)** 35-40.

Oliva A, Dellaragione F, Salerno A, Riccio V, Tartaro G, Cozzolino A, D'amati S, Pontoni G & Zappia V (1996) Biocompatibility studies on glass ionomer cements by primary cultures of human osteoblasts *Biomaterials* **17(13)** 1351-1356.

Palmer G, Anstice HM & Pearson GJ (1999) The effect of curing regime on the release of hydroxyethyl methacrylate (HEMA) from resin modified glass-ionomer cements *Journal of Dentistry* **27(4)** 303-311.

Paterson RC & Watts A (1987) Toxicity to the pulp of a glass-ionomer cement *British Dental Journal* **162(3)** 110-112.

Peltola M, Salo T & Oikarinen K (1992) Toxic effects of various retrograde root-filling materials on gingival fibroblasts and rat sarcoma cells *Endodontics & Dental Traumatology* **8(3)** 120-124.

Ratanasathien S, Wataha JC, Hanks CT & Dennison JB (1995) Cytotoxic interactive effects of dentin bonding components on mouse fibroblasts *Journal of Dental Research* **74(9)** 1602-1606.

Sasanaluckit P, Albustany KR, Doherty PJ & Williams DF (1993) Biocompatibility of glass ionomer cements *Biomaterials* **14(12)** 906-916.

Saunders WP, Saunders EM, Herd D & Stephens E (1992) The use of glass ionomer as a root canal sealer—A pilot study *International Endodontic Journal* **25(5)** 238-244.

Schmalz G, Thonemann B, Riedel M & Elderton RJ (1994) Biological and clinical investigations of a glass ionomer base material *Dental Materials* **10(5)** 304-313.

Schmalz G (1998) The biocompatibility of non-amalgam dental filling materials *European Journal of Oral Science* **106(2 Pt 2)** 696-706.

Schweikl H, Schmalz G & Spruss T (2001) The induction of micronuclei *in vitro* by unpolymerized resin monomers *Journal of Dental Research* **80(7)** 1615-1620.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Sidhu SK & Schmalz G (2001) The biocompatibility of glass-ionomer cement materials. A status report for the American Journal of Dentistry *American Journal of Dentistry* **14(6)** 387-396.

Six N, Lasfargues JJ & Goldberg M (2000) *In vivo* study of the pulp reaction to Fuji IX, a glass ionomer cement *Journal of Dentistry* **28(6)** 413-422.

Smith DC (1990) Composition and characteristics of glass ionomer cements *Journal of the American Dental Association* **120(1)** 20-22.

Stanley HR (1992) Local and systemic response to dental composites and glass ionomers *Advances in Dental Research* **6** 55-64.

Stanislawski L, Daniau X, Lauti A & Goldberg M (1999) Factors responsible for pulp cell cytotoxicity induced by resin-modified glass ionomer cements *Journal of Biomedical Material Research* **48(3)** 277-288.

Stanislawski L, Soheili-Majd E, Perianin A & Goldberg M (2000) Dental restorative biomaterials induce glutathione depletion in cultured human gingival fibroblast: Protective effect of N-acetyl cysteine *Journal of Biomedical Material Research* **51(3)** 469-474.

Tarim B, Hafez AA, Suzuki SH, Suzuki S & Cox CF (1997) Biocompatibility of compomer restorative systems on non-exposed dental pulps of primate teeth *Operative Dentistry* **22(4)** 149-158.

Yamamura T (1985) Differentiation of pulpal cells and inductive influences of various matrices with reference to pulpal wound healing *Journal of Dental Research* **64** 530-540.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3,** 260-266

Thermal Emission by Different Light-Curing Units

AUJ Yap • MS Soh

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Clinical Relevance

LED (Light-Emitting Diodes) light curing units produce significantly less heat than halogen lights. High-intensity halogen lights are potentially hazardous to pulp.

SUMMARY

This study quantified and compared the thermal emission of different light curing units (LCU). Three LED (Elipar Freelight [3M]; GC e-light [GC]; Coolblu [Dentalsystems.com]) and three halogen (Max [Dentsply-Caulk]; Elipar Trilight [3M]; Astralis 10 [Ivoclar-Vivadent]) lights were selected for the study. Thermal emission of the LCUs, when used in various curing modes, was assessed using a K-type thermocouple and a digital thermometer at distances of 3 mm and 6 mm compared to the conventional halogen LCU (Max). The temperature profiles and mean maximum temperature change (n=7) generated by each LCU were obtained. Data was subjected to ANOVA/Scheffe's post-hoc test and Independent Samples *t*-test at significance level 0.05. At 3 mm, temperature rise observed with LED lights ranged from 4.1°C to 12.9°C, while halogen lights ranged from 17.4°C to 46.4°C. At 6 mm, temperature rise ranged from 2.4°C to 7.5°C and 12.7°C to 25.5°C for LED and halogen lights, respectively. Thermal emission of LED lights was significantly lower than halogen lights. Significant differences in temperature rise were observed between different curing modes for the same light and between different LED/halogen lights.

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

MS Soh, BSc (Hons), Research Scholar, Department of Restorative Dentistry, National University of Singapore

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

INTRODUCTION

The potential damaging effects of temperature increase on pulp tissue during restorative treatment has been a matter of concern to dentistry for many years. Light curing units (LCUs) can cause a temperature increase that could damage the pulp (Hussey, Biagioni & Lamey, 1995; Hannig & Bott, 1999). Thermal transfer to pulp is affected by material shade, thickness, composition, porosity, curing time and residual dentin thickness (McCabe, 1985; Goodis & others, 1989; Shortall & Harrington, 1998). It also varies with the type of curing unit, quality of light filter, output intensity and irradiation time (Goodis & others, 1997; Shortall & Harrington, 1998; Hannig & Bott, 1999). Temperature rise during the curing of restorative materials is, however, mainly contributed by the light source (Lloyd, Joshi & McGlynn, 1986).

LED (Light-Emitting Diodes) LCUs were recently introduced to the dental profession. They are solid-

Table 1: *Details of the Light Curing Units (LCU) and the Various Curing Modes Evaluated*

LCU	Curing Modes	Curing Profiles		
Elipar Freelight (LED) 3M-ESPE, St Paul, MN, USA	Standard (FL1)	400 mW/cm² (20 seconds)		
	Standard (FL2)	400 mW/cm² (40 seconds)		
	Exponential (FL3)	0-400 mW/cm² (12 seconds)	→	400 mW/cm² (28 seconds)
GC e-Light (LED) GC Europe, Leuven, Belgium	Pulse Curing (EL1)	750 mW/cm² (10 pulses x 2 seconds)		
	Standard (EL2) (40 seconds)	350 mW/cm²		
	Turbo (EL3)	600 mW/cm² (20 seconds)		
	Soft-Start Curing A (EL4)	0-600 mW/cm² (20 seconds)	→	0-600 mW/cm² (20 seconds)
	Soft-Start Curing B (EL5)	0-300 mW/cm² (20 seconds)	→	0-300 mW/cm² (20 seconds)
CoolBlu (LED) Dental Systems.Com, Tokyo, Japan	Mode 3 (CB1)	350 mW/cm² (20 seconds)		
	Mode 6 (CB2)	140 mW/cm² (6 seconds)	→	350 mW/cm² (15 seconds)
Max (Halogen) Dentsply-Caulk, Milford, DE, USA	Standard (MX)	400 mW/cm² (20 seconds)		
Elipar Trilight (Halogen) 3M-ESPE, St Paul, MN, USA	Standard (TL1)	800 mW/cm² (40 seconds)		
	Exponential (TL2)	100-800 mW/cm² (40 seconds)		
Astralis 10 (Halogen) Ivoclar-Vivadent, Liechtenstein, Austria	High Power (AS1)	1200 mW/cm² (10 seconds)		
	Adhesive Program (AS2)	650 mW/cm² (20 seconds)		
	Pulse Program (AS3)	150 to 650mW/cm² (10 seconds)	→	Pulsating between 650 and 1200 mW/cm²
	ECS-Program (AS4)	1200mW/cm² (30 seconds)		

Curing profiles are based on manufacturers' information.

state semiconductor devices that convert electrical energy directly to heat. Thermal light sources (halogen and plasma lights) emit light by electrical heating. The generation of light produced by LEDs results in high efficacy as most of the energy radiated falls within the absorption spectrum of camphorquinone photoinitiators (Mills, Jandt & Ashworth, 1999). Cited advantages of LED lights include: (a) cost-efficiency with a longer-lasting light source compared to halogen or other devices, (b) clinician friendliness with cordless features and a more slim-line construction, (c) equivalent or improved physical properties of polymerized composite resin, (d) battery operation with no bulb aging, (e) no decrease in output as bulb ages, (f) less heat production during routine and extended use of the polymerization device and (g) no fan necessary (Duke, 2001). Although some research has been conducted on the use of LED lights on composite hardness, modulus, depth of cure, compressive and flexural strengths (Mills & others, 1999; Stahl & others, 2000; Jandt & others, 2000; Kurachi & others, 2001), the thermal emission of LED lights has not been investigated.

This study quantified the thermal emission of three LED and halogen lights. Temperatures changes associated with various curing modes of each LCU were also compared.

METHODS AND MATERIALS

The light curing units selected for this study included three LED lights (Elipar Freelight [3M-ESPE, St Paul, MN, USA]; GC e-light [GC]; Coolblu [Dental Systems.com]) and three halogen lights (Max [Dentsply-Caulk, Milford, DE, USA]; Elipar Trilight [3M-ESPE]; Astralis 10 [Ivoclar-Vivadent]). Details of the LCUs and curing modes evaluated are shown in Table 1. Thermal emission of the LCUs was measured

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

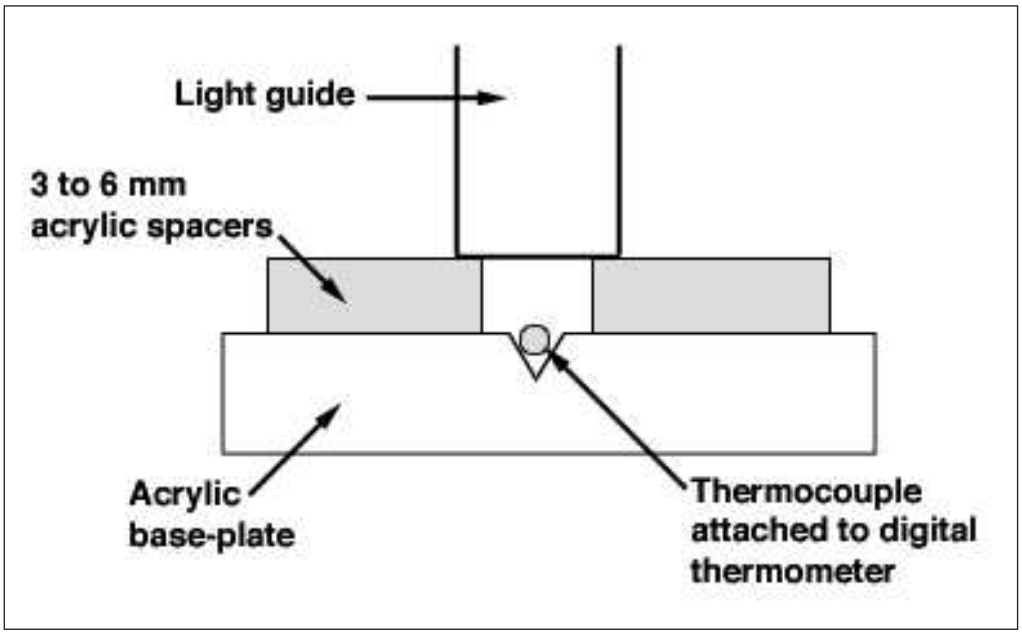


Figure 1: *Diagrammatic representation of the experimental set-up.*

Table 2: *Mean Maximum Temperature Rise Observed with the Various LCUs/Curing Modes*

Light-Curing Units	Light-Curing Modes	At 3 mm [°C]	At 6 mm [°C]
Elipar Freelight	FL1	11.0 (0.22)	7.5 (0.21)
	FL2	12.9 (0.17)	6.6 (0.18)
	FL3	10.9 (0.31)	7.2 (0.24)
GC e-Light	EL1	8.1 (0.16)	4.9 (0.20)
	EL2	5.5 (0.10)	3.4 (0.23)
	EL3	7.5 (0.20)	4.1 (0.24)
	EL4	8.4 (0.16)	4.5 (0.13)
	EL5	4.1 (0.29)	2.4 (0.11)
CoolBlu	CB1	5.8 (0.21)	3.9 (0.11)
	CB2	5.5 (0.23)	3.9 (0.17)
Max	MX	17.4 (0.70)	12.7 (0.28)
Elipar Trilight	TL1	26.7 (0.39)	19.8 (0.32)
	TL2	22.6 (0.16)	18.3 (0.41)
Astralis 10	AS1	36.0 (0.88)	20.2 (0.20)
	AS2	24.4 (0.42)	14.6 (0.23)
	AS3	34.6 (0.31)	17.9 (0.15)
	AS4	46.4 (0.55)	25.5 (0.22)

Standard deviations in parentheses.
See Table 1 for times and intensities of light-curing modes.

by a K-type thermocouple and a digital thermometer (305, Peacock Precision Instruments, Singapore). The thermocouple was secured onto a groove in an acrylic base-plate so that the surface of the thermocouple was flushed against the top surface of the base-plate (Figure 1). Two clear acrylic plates 3 mm and 6 mm in thickness with a 7 mm diameter hole served as spacers to control the thermocouple-light guide exit window distance. The experimental set-up allowed the thermocouple to be positioned at the center of the 7-mm hole. The light guide exit windows of the LCUs were placed over the 7-mm hole of the upper acrylic plates and activated. Temperature rise during irradiation can, therefore, be measured at distances of 3 mm and 6 mm away from the thermocouple.

A pilot study was first conducted to determine the effects of environmental temperature on temperature rise during light irradiation using the Max polymerization unit. The experiment was conducted in a controlled and enclosed environment (Concept 300 Workstation; Ruskin Technology Limited, Yorkshire, UK) at preset temperatures of 25°C and 37°C. Temperature rise associated with the Max polymerization unit at both preset environmental temperatures was measured at distances of 3 mm and 6 mm. Five readings were taken at five-minute intervals for each preset temperature and distance. Results were analyzed with paired sample *t*-test at significance level 0.05. At 3 mm, temperature rise was 15.2 ± 0.1 and 15.3 ± 0.2°C for environmental temperatures of 37°C and 25°C, respectively. At 6 mm temperature rise was 10.8 ± 0.2 and 10.8 ± 0.3°C for environmental temperatures of 37°C and 25°C, respectively. As no significant difference in temperature rise was observed between the two environmental temperatures at both distances, the main experiment was conducted under ambient room temperature.

The ambient room temperature was recorded and maximum temperature rise during light activation was obtained for the different LCUs and curing modes. Seven readings were obtained for each light-curing mode combination. To minimize the effects of heating, a five-minute hiatus was implemented between each

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 3: *Comparison of Mean Maximum Temperature Rise of the Various Curing Modes for the Same LCU*

Light Guide Exit Window Distance	Light Curing Unit	Differences
3 mm	Elipar Freelight	FL2>FL1, FL3
	GC e-light	EL1, EL4>EL3>EL2>EL5
	CoolBlu	NS
	Elipar Trilight	TL1>TL2
	Astralis 10	AS4>AS1>AS3>AS2
6 mm	Elipar Freelight	FL1, FL3>FL2
	GC e-light	EL1>EL4>EL3>EL2>EL5
	CoolBlu	NS
	Elipar Trilight	TL1>TL2
	Astralis 10	AS4>AS1>AS3>AS2

Results of one-way ANOVA/Scheffe's post-hoc test or independent sample t-test ($p<0.05$). > indicates statistical significance while NS denotes no statistical significance.

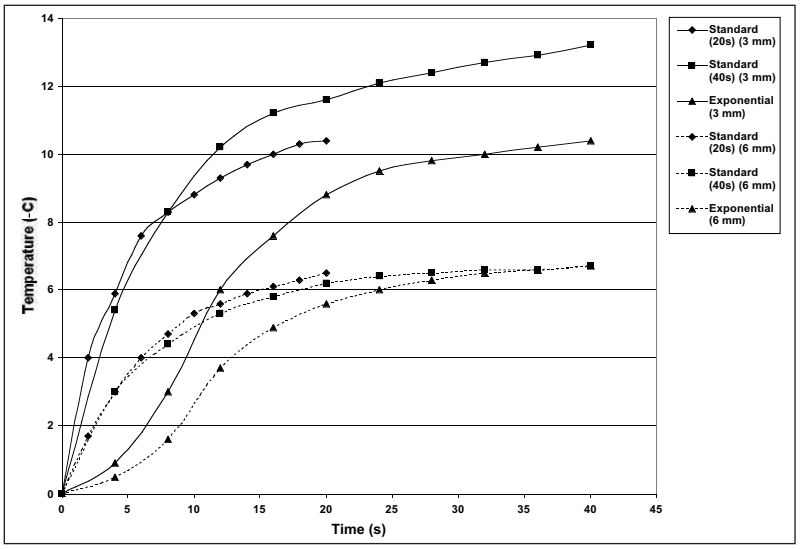


Figure 2: *Temperature rise profile of Elipar Freelight.*

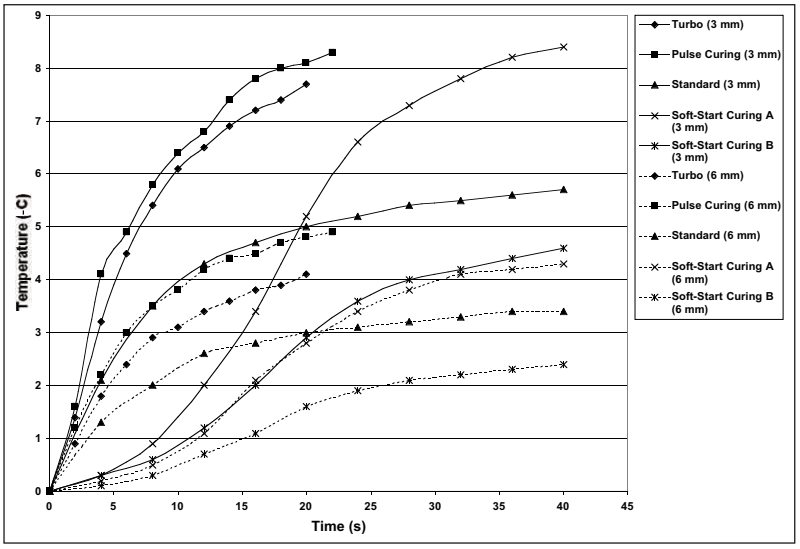


Figure 3: *Temperature rise profile of GC e-light.*

curing cycle. The temperature rise profiles of the various lights and their different curing modes were also determined by obtaining 10 temperature readings at equal time intervals over the light curing period. Data was subjected to one-way ANOVA/ Scheffe's post-hoc test and Independent Samples *t*-test at significance level 0.05. The mean maximum temperature rise of the different LCUs/curing modes was compared to the conventional halogen LCU (Max). In addition, differences between the curing modes for the same light and different LED/halogen lights were also compared. Temperature changes at 3 mm and 6 mm were also contrasted.

RESULTS

Table 2 shows the mean maximum temperature rise observed with the various LCUs/curing modes. The temperature rise profiles of the various LCUs/curing modes are reflected in Figures 2 through 7.

The temperature rise observed at 3 mm was significantly higher than at 6 mm. At 3 mm the temperature rise observed with LED lights ranged from 4.1°C to 12.9°C, while the halogen lights showed a range of 17.4°C to 46.4°C. At 6 mm, temperature rise ranged from 2.4°C to 7.5°C and 12.7°C to 25.5°C for LED and halogen lights, respectively. Thermal emission of LED lights was significantly lower than halogen lights at both distances. Table 3 reflects the significant differences in temperature rise among different curing modes of the same curing light. For Freelight and e-light, minor variations in significant differences between curing modes were observed between 3 mm and 6 mm. No significant difference in temperature rise was observed between the two curing modes for Coolblu. At both distances, the thermal emission of Freelight, with its various curing modes, was significantly higher than the other LED lights. Among the halogen lights, curing with the ECS mode (designed for curing of resin cements through ceramic restorations) for 30 seconds resulted in the most heat generation. Maximum or peak temperatures were consistently observed toward the end of the curing cycles and duration lasted no more than 15 seconds (Figure 2 through 7).

DISCUSSION

Light guide exit window distances of 3 mm and 6 mm were used to mimic distances encoun-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

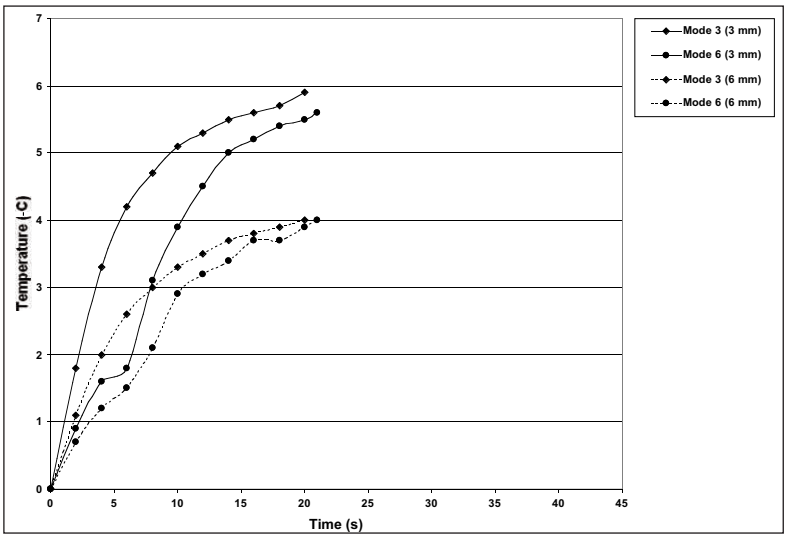


Figure 4: *Temperature rise profile of CoolBlu.*

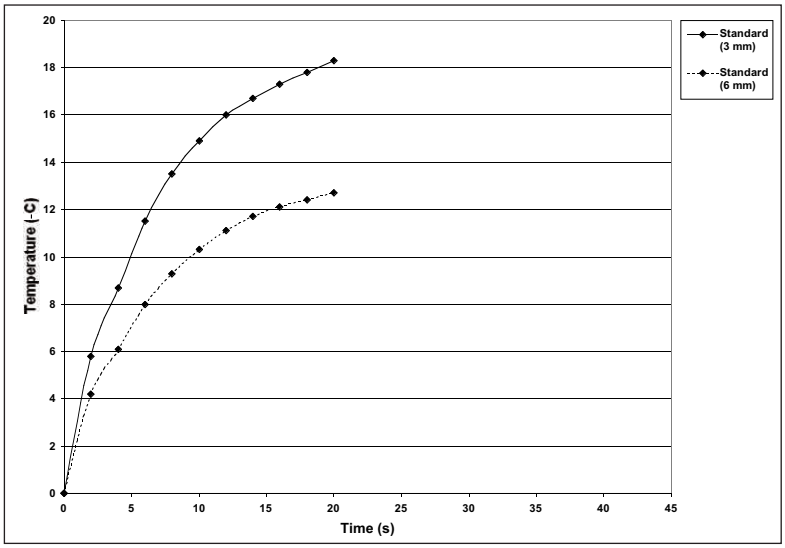


Figure 5: *Temperature rise profile of Max.*

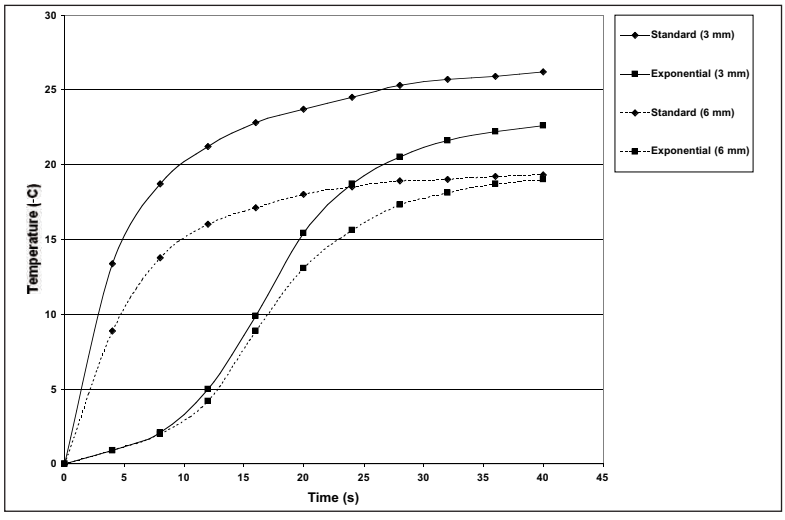


Figure 6: *Temperature rise profile of Elipar Trilight.*

tered when curing deep Class I and Class II cavities. The experimental set-up also allowed for the simulation of a confined cavity as in the case of a Class II cavity with matrix and rubber dam placement. In addition, the 3 mm distance approximates the proximity of the light guide exit window to the top layer of restorative materials during clinical restorative procedures. Since the acrylic spacers used have a low thermal conductivity, the maximum temperature rise observed represents the worst case scenario. Restorative materials and teeth were excluded from the experiment design to minimize the number of variables involved. By doing so, the data obtained can also be applied to light-(heat) enhanced bleaching procedures and thermal expansion of composites during curing.

In this study, temperature rise decreased significantly with increased light guide exit window distance. Results concur with those of Shortall and Harrington (1998), who investigated temperature rise due to radiation energy at various cavity depths. Although the light output of LCUs (350 and 710 mW/cm^2) used by the latter group were similar to that of Max and Trilight (400 and 800 mW/cm^2, respectively) in this study, maximum temperature rise observed at 6 mm distance was considerably lower (2.0°C and 3.7°C, compared to 12.7°C and 19.8°C). This may be partially attributed to using black nylon spacers that may absorb part of the heat emitted, instead of clear acrylic ones.

The thermal emission of LED lights was significantly lower than halogen lights at both distances. Rather than a hot filament (as used in halogen bulbs), LEDs use junctions of doped semiconductors (p-n junctions) for the generation of light (Nakamura, Mukai & Senoh, 1994). Under proper forward biased conditions, electrons and holes recombine at the LED's p-n junctions, leading to the emission of blue light in the case of gallium nitride LEDs. As the spectral output of gallium nitride blue LEDs falls within the absorption spectrum of the camphoroquinone photoinitiators, no light filters are required. The latter (light filters), however, serve as partial thermal buffers in curing lights (Shortall & Harrington, 1998). From Table 2, it is apparent that LED LCUs still emit heat and the thermal emission from different LED lights varies significantly. The temperature rise observed with Freelight was significantly higher than e-light and Coolblu despite the same or a lesser number of LEDs used (Freelight and

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Coolblu 19 LEDs; e-light 64 LEDs). The maximum temperature observed with Freelight is expected to be even higher if not for the aluminum casing cum handle used. This serves to conduct heat and cool the unit. Reasons for the higher thermal emission of Freelight are not known. Possible hypotheses include LED size and inter-LED spacing.

Among the halogen lights, curing modes utilizing high light outputs generally resulted in significantly greater thermal emission. The lowest temperature rise was observed with the Max polymerization unit that had the lowest light output among the three halogen lights evaluated. The clinical experience with conventional halogen LCUs (<500 mW/cm^2) indicates that pulp appears able to recover from transient heating from light-curing. Zach and Cohen (1965) reported that 15% of teeth in rhesus monkeys developed necrosis when healthy pulps were exposed to a temperature increase of only 5.5°C. These findings and those of Pohto and Scheinin (1958) suggest that the critical temperature for irreversible damage to the pulp begins at between 42°C and 42.5°C. Hannig and Bott (1999) measured the pulp chamber temperature increase induced during resin composite polymerization with various LCUs using a tooth model (Class II cavity with a 1 mm dentin layer between the pulp chamber and proximal cavity wall), K-type thermocouple positioned at the pulp-dentin junction and 2 mm composite layers. They found that LCUs with outputs greater than 670 mW/cm^2 generated temperature increases of more than 5.5°C when used for 40 seconds. Taking this into consideration, the maximum temperature rise detected in Trilight (800 mW/cm^2 for 40 seconds) should be viewed as critical, especially where residual dentin thickness is limited. In spite of the very high value observed with Astralis High Power mode (AS1—designed for curing composite restorations), the very short-term temperature peak may not be relevant to pulpal damage (Figure 7). The Astralis ECS mode should, however, never be used for curing composites and bonding agents.

For an individual tooth, it is nearly impossible for a clinician to predict the temperature rise that may occur when curing a restoration. In general, the thicker the dentin and the shorter the curing time, the smaller the temperature increase (Loney & Price, 2001). Clinicians should be aware of the potential thermal hazard associated with using high intensity lights when curing composites in deep cavities. Minimum irradiation times should also be used when curing bonding agents with these lights in view of the absence of a composite thermal buffer. A simple and effective way to protect the pulp is to apply a cement base or lining material to the cavity floor (Hansen & Asmussen, 1993).

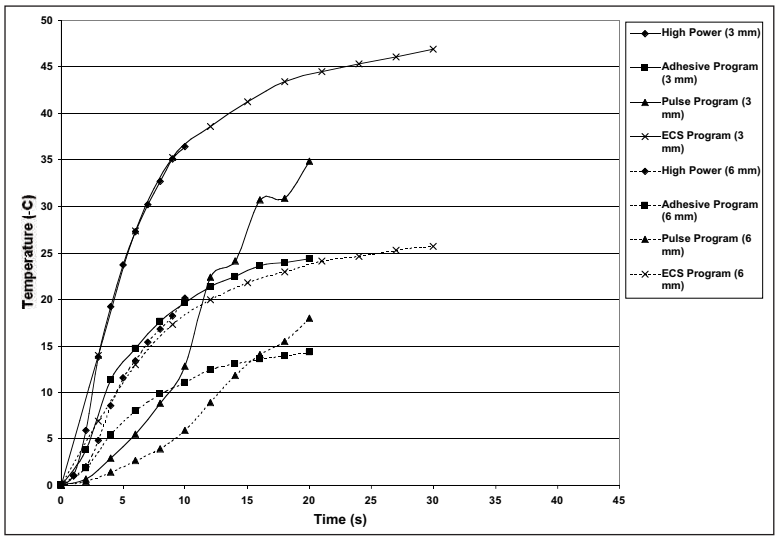


Figure 7: *Temperature rise profile of Astralis 10.*

CONCLUSIONS

Under the conditions of this *in-vitro* study:

1. LED lights emit significantly less heat than halogen lights.
2. The heat emitted by individual curing lights depends on the curing mode used.
3. The heat emitted by different LED/halogen lights varies significantly.

(Received 17 May 2002)

References

Duke ES (2001) Light-emitting diodes in composite resin photopolymerization *Compendium* **22(9)** 722-725.

Goodis HE, White JM, Marshall GW, Yee K. Fuller N, Gee L & Marshall SJ (1997) Effects of Nd: and Ho :yttrium-aluminum-garnet lasers on human dentin fluid flow and dental pulp-chamber *in vitro Archives of Oral Biology* **42(12)** 845-854.

Goodis HE, White JM, Andrews J & Watanabe LG (1989) Measurement of temperature generated by visible-light-cure lamps in an *in-vitro* model *Dental Materials* **5(4)** 230-234.

Hannig M & Bott B (1999) *In-vitro* pulp chamber temperature rise during composite resin polymerization with various light-curing sources *Dental Materials* **15(4)** 275-281.

Hansen EK & Asmussen E (1993) Correlation between depth of cure and temperature rise of a light-activated resin *Scandinavian Journal of Dental Research* **101(3)** 176-179.

Hussey DL, Biagioni PA & Lamey PJ (1995) Thermographic measurement of temperature change during resin composite polymerization *in vivo Journal of Dentistry* **23(5)** 267-271.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Jandt KD, Mills RW, Blackwell GB & Ashworthv STT (2000) Depth of cure and compressive strength of dental composites cured with blue light emitting diodes (LEDs) *Dental Materials* **16(1)** 41-47.

Kurachi C, Tuboy AM, Magalhaes DV & Bagnato VS (2001) Hardness evaluation of a dental composite polymerized with experimental LED-based devices *Dental Materials* **17(4)** 309-315.

Lloyd CH, Joshi A & McGlynn E (1986) Temperature rises produced by light sources and composites during curing *Dental Materials* **2(4)** 170-174.

Loney RW & Price RB (2001) Temperature transmission of high-output light-curing units through dentin *Operative Dentistry* **26(5)** 516-520.

McCabe JF (1985) Cure performance of light-activated composites by differential thermal analysis (DTA) *Dental Materials* **1(6)** 231-234.

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.

Nakamura S, Mukai T & Senoh M (1994) Candela-class high-brightness InGaN/AlGaM double-heterostructure blue-light-emitting diodes *Applied Physics Letters* **64(13)** 1687-1689.

Pohto M & Scheinin A (1958) Microscopic observations on living dental pulp *Acta Odontologica Scandinavia* **16** 303-327.

Shortall AC & Harrington E (1998) Temperature rise during polymerization of light-activated resin composites *Journal of Oral Rehabilitation* **25(12)** 908-913.

Stahl F, Ashworth SH, Jandt KD & Mills RW (2000) Light-emitting diode (LED) polymerisation of dental composites: Flexural properties and polymerization potential *Biomaterials* **21(13)** 1379-1385.

Zach L & Cohen G (1965) Pulp response to externally applied heat *Oral Surgery, Oral Medicine, Oral Pathology* **19** 515-530.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3,** 267-273

Microleakage Evaluation of a Flowable Polyacid-Modified Resin Composite Used as Fissure Sealant on Air-Abraded Permanent Teeth

HC Güngör • MD Turgut
N Attar • N Altay

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Clinical Relevance

On air-abraded permanent teeth, using a flowable polyacid-modified resin composite as fissure sealant following acid etching and/or bonding agent application provided comparable microleakage results to that of a resin-based fluoride sealant.

SUMMARY

This study evaluated the microleakage of a flowable polyacid-modified resin composite used as a fissure sealant on air-abraded teeth in comparison to a resin-based fluoride sealant. Sixty extracted human third molars were divided into six subgroups (n=10) under two main groups (fissure sealant materials). The occlusal surfaces of the teeth were air-abraded at 80 psi using 50 µm particles of aluminum oxide for 30 seconds, followed by rinsing and drying. Delton FS+ (Dentsply International) and Dyract Flow (Dentsply DeTrey) were applied to the teeth in subgroups following application of: a) an acid etching and bonding agent, b) bonding agent alone or c) air-abrasion alone, respectively. Acid etching (Delton EZ Etch, Dentsply International) and bonding agent application (Prime & Bond NT, Dentsply DeTrey) were done according to the manufacturers' instructions. The teeth were thermocycled for 500 cycles between 5°C and 55°C with dwell time of 15 seconds. Basic fuchsin (0.5%) staining followed by buccolingual sectioning was performed. Microleakage was evaluated at 20x optical magnification. Kruskal–Wallis test was used to make comparisons among six subgroups. Pairwise comparisons were done with the Mann–Whitney U test with the level of significance set as α=0.05.

Dyract Flow application as a fissure sealant on air-abraded permanent teeth in combination with acid etching and/or bonding agent provided microleakage results comparable to Delton FS+. Results also showed that the use of air abrasion, alone, resulted in significantly higher microleakage scores.

*H Cem Güngör, DDS, PhD, research assistant, Department of Pedodontics, Hacettepe University, Faculty of Dentistry, Ankara, Turkey

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

Nuray Attar, DDS, PhD, research assistant, Department of Conservative Dentistry, Hacettepe University Faculty of Dentistry, Ankara, Turkey

Nil Altay, DDS, PhD, associate professor, Department of Pedodontics, Hacettepe University, Faculty of Dentistry, Ankara, Turkey

*Reprint request: 06100 Ankara, Turkey; e-mail: cemgungor@hotmail.com

INTRODUCTION

Epidemiological data shows that the prevalence of dental caries in most developed countries has declined in recent decades (Brunelle & Carlos, 1982; Kalsbeek &

Verrips, 1990; Brown & Selwitz, 1995). However, as stated by Ripa (1985; 1993), the caries decline is not uniform for all tooth surfaces, and the reduction of smooth surface caries has been more pronounced than for pit and fissure lesions. The high caries susceptibility of the pit and fissure surfaces of posterior teeth has been recognized for many years (Simonsen, 1987). These sites have accounted for more than 85% of the total caries experienced in the 1986-87 NIDR survey of US schoolchildren (Hicks & Flaitz, 1993). Pit and fissure sealants are the most effective means of reducing the risk of caries that arises from these sites, and their success largely depends on the retention of sealant (Waggoner & Siegal, 1996).

With microleakage receiving much attention in the literature, this is another important factor that has contributed to success with dental sealants (Barnes & others, 2000). Microleakage has been defined as the clinically undetectable passage of bacteria, fluids, molecules or ions between the cavity wall and the applied restorative material (Kidd, 1976). The success of a pit and fissure sealant can be hindered if the applied material cannot resist microleakage. In this case, microleakage results in the initiation and/or progression of caries under sealed surfaces, which lowers its cost–effectiveness (Jensen & Handelman, 1980; Jeronimus, Till & Sveen, 1975). Since its introduction, acid etching has become a crucial and indispensable step in sealant applications. Etching produces microscopic porosities in the enamel surface into which the unpolymerized sealant flows and hardens in tag-like projections that attach the material to the tooth surface. Satisfactory acid etching has been reported to be a primary determinant for success (Simonsen, 1987).

Air-abrasive technology, introduced in the 1950s, has been reported to prepare enamel and dentin for bonding in a similar way to acid etching (Goldstein & Parkins, 1994). Air abrasion uses a high speed stream of purified aluminum oxide particles delivered by air pressure, which creates a roughened enamel surface that may make it more conducive to bonding (Goldstein & Parkins, 1994; Wright & others, 1999). Clinicians have shown considerable interest in using air abrasion to treat incipient pit and fissure carious lesions (Hamilton & others, 2001). Some studies have suggested that this may eliminate the need for acid etching when applying pit and fissure sealants (Goldstein & Parkins, 1995).

Polyacid-modified resin composite introduced in the early 1990s has been developed as a direct esthetic restorative material that combines the desirable properties of light-curing composites with those of fluoride-releasing glass-ionomer cements (Duke, 1999; Luo & others, 2000). It has become an important part of many practices throughout the world and has also enjoyed a growing interest as a result of clinical studies (Luo & others, 2000; Çehreli & Altay, 2000). Newer brands of these restorative materials with flow characteristics have been marketed recently. This *in vitro* study evaluated the microleakage of a flowable polyacid-modified resin composite used as fissure sealant on air-abraded permanent teeth when compared to a resin-based fluoride fissure sealant.

METHODS AND MATERIALS

Sixty extracted human teeth (30 lower and 30 upper third molars) were used in the study. Organic remnants on teeth were removed with a surgical blade and the occlusal surfaces of the teeth were cleaned with a bristle brush rotating on a slow-speed handpiece with irrigation. After drying, the teeth were macroscopically evaluated under a light source by the naked eye and a dental probe. Any teeth with a visible carious lesion, hypoplasia and extraction damage were discarded. Teeth were stored at room temperature in distilled water that was changed daily.

The occlusal surfaces of teeth were prepared with the Microetcher (Danville Engineering Inc, San Ramon, CA, USA), which was set at 80 psi for air abrasion using 50 µm particles of aluminum oxide. Air abrasion was applied for 30 seconds in five continuous, overlapping sweeps across each occlusal surface. During air abrasion, care was taken to hold the nozzle perpendicular to the occlusal surface at a maximum distance of 5 mm as instructed by the manufacturer. In order to keep this distance, the operator placed his hands on a rest pad and avoided lifting his arms during operation. Following rinsing with air/water spray for 15 seconds and thoroughly drying with compressed air, the teeth were divided into six subgroups (n=10) under two main groups: Group A teeth—a resin-based fluoride fissure sealant, Delton FS+ (Dentsply International York, PA, USA) was used; Group B teeth—a flowable polyacid-modified resin composite, Dyract Flow (Dentsply DeTrey, Konstanz, Germany) was applied. In this study, Delton EZ Etch (Dentsply International, York, PA, USA) and Prime & Bond NT (Dentsply DeTrey, Konstanz, Germany) were also used in their respective subgroups. All procedures that related to sealant applications were performed by one operator. The detailed procedures in subgroups are as follows:

A1: Air-abrasion + Delton EZ Etch + Prime & Bond NT + Delton FS+

A2: Air-abrasion + Prime & Bond NT + Delton FS+

A3: Air-abrasion + Delton FS+

B1: Air-abrasion + Delton EZ Etch + Prime & Bond NT + Dyract Flow

B2: Air-abrasion + Prime & Bond NT + Dyract Flow

B3: Air-abrasion + Dyract Flow

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

In subgroups where Delton EZ Etch and Prime & Bond NT were applied, manufacturers' instructions were followed. Subgroup A1 and B1 teeth were etched for 30 seconds, rinsed with air/water spray for 15 seconds and air dried for 10 seconds. Prime & Bond NT was applied for 20 seconds on the occlusal surfaces, and the solvent was removed by blowing gently with air for five seconds, followed by 10 seconds of light-curing. Fissure sealant materials were applied on the occlusal surfaces and light cured for 40 seconds. In subgroups A2 and B2, the same procedures were followed for Prime & Bond NT and respective fissure sealants. In subgroups A3 and B3, teeth received their respective fissure sealant materials that were directly applied on the air-abraded occlusal surfaces and light cured for 40 seconds. Light-curing procedures were done with the Hilux 200 Curing Light (Benlioglu Dental Inc, Ankara, Turkey). The tip of the light source was placed on the occlusal cusps, thereby, making it possible to keep the minimum distance from the occlusal surface (De Craene & others, 1989). The curing unit had a light output of 500 mW/cm^2 and the curing efficiency was assessed before use in each subgroup.

Specimens were then stored in distilled water for 24 hours and thermocycled for 500 cycles between 5°C and 55°C with a dwell time of 15 seconds. The roots were sealed with modeling wax and the teeth were completely covered with three coats of nail varnish except for the area covered with sealant and 1 mm of the surrounding enamel. The specimens were immersed in 0.5% basic fuchsin dye for 24 hours. The teeth were sectioned longitudinally in a buccolingual direction in a dry environment with diamond disc separators (Diamond Disc Superflex, 910S/220, North Bel, Italy) in order to obtain three sections of 1 mm-thickness from each specimen. The dust from sectioning was removed with compressed air and the sections were kept in dry conditions. Examinations related to the sealants' degree of microleakage and photographing were carried out under a stereomicroscope at 20x magnification (Wild Type 308700, Heerbruug, Switzerland).

The following criterion were used to evaluate microleakage (Ovrebo & Raadal, 1990; Park & others, 1993): 0=no dye penetration; 1=dye penetration restricted to outer half of the sealant; 2=dye penetration restricted to the inner half of the sealant and 3=extensive dye penetration to the bottom of the sealant. The mean score for each specimen was obtained. In order to obtain mean scores for the subgroups, the mean of means for specimens in each subgroup was calculated.

The study was originally designed as two–way (2x3) ANOVA. However, statistical analysis was performed by Kruskal–Wallis test to make comparisons among six subgroups since the parametric test assumptions (normality of distributions and homogeneity of variances) were not satisfied. Pairwise comparisons were done with the Mann–Whitney U test with Bonferroni correction. The level of significance was set at α=0.05.

RESULTS

The total number of sections examined for dye penetration was 180. Table 1 shows the dye penetration for all sections. When all sections were considered, regardless of the method of application and sealant type used, 101 sections (56%) had no microleakage. However, extensive dye penetration (score 3) was observed in 48 sections (27%) (Table 2). Extensive dye penetration was found mainly in subgroups where the sealant materials were directly applied on the air abraded occlusal surfaces (subgroups A3 and B3). Stereomicroscopic photographs of sections at 20x magnification are presented in Figures 1–3.

Statistically significant differences were found among six subgroups (KW=47.277, p<0.001). Range (minimum, maximum values) and medians for each subgroup are presented in Table 3. Looking at the combinations of sealants and the application methods, differences between subgroup A1 and subgroups A2, A3, B2 and B3 were found to be statistically significant

Table 1: *Microleakage Presence Under All Sealant Sections*

	Sealant Types			
	Delton FS+		Dyract Flow	
Method of Application	**Absent**	**Present**	**Absent**	**Present**
Acid etch+bonding agent	29	1	28	2
Bonding agent only	21	9	22	8
Air-abrasion only	1	29	0	30
TOTAL	**51**	**39**	**50**	**40**

Table 2: *Dye Penetration Scores Obtained in Subgroups (DEZ=Delton EZ Etch; PBNT=Prime & Bond NT; DFS=Delton FS+; DFW=Dyract Flow)*

		Dye Penetration Scores			
Subgroup	**Procedure**	**0**	**1**	**2**	**3**
A1	Air-abrasion + DEZ + PBNT + DFS	29	1	0	0
A2	Air-abrasion + PBNT + DFS	21	8	1	0
A3	Air-abrasion + DFS	1	2	3	24
B1	Air-abrasion + DEZ + PBNT + DFW	28	2	0	0
B2	Air-abrasion + PBNT + DFW	22	6	2	0
B3	Air-abrasion + DFW	0	3	3	24
TOTAL		**101**	**22**	**9**	**48**

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 3: *Summary of Results. DEZ=Delton EZ Etch; PBNT=Prime & Bond NT; DFS=Delton FS+ and DFW=Dyract Flow*

Subgroup	Procedure	Median	Min	Max
A1	Air-abrasion + DEZ + PBNT + DFS[a]	0.0000	0.0000	0.3300
A2	Air-abrasion + PBNT + DFS[b]	0.3333	0.0000	1.0000
A3	Air-abrasion + DFS[c]	3.0000	1.6700	3.0000
B1	Air-abrasion + DEZ + PBNT + DFW[a,d]	0.0000	0.0000	0.3300
B2	Air-abrasion + PBNT + DFW[b,d]	0.1667	0.0000	1.0000
B3	Air-abrasion + DFW[c]	3.0000	1.6700	3.0000

Superscripts ([a, b, c, d]) represent statistically insignificant difference for subgroups with same letters (p>0.05).

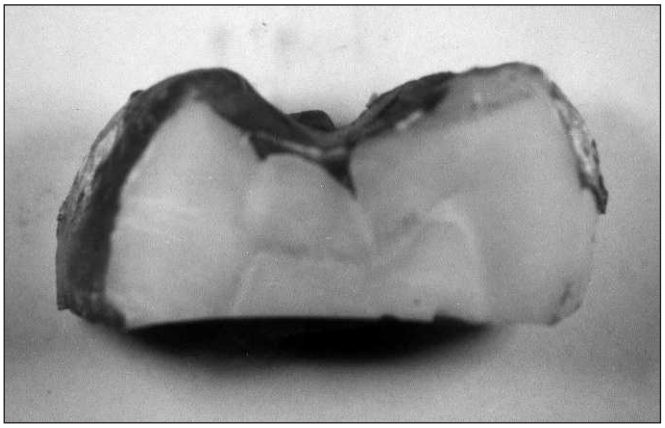


Figure 1: *Microleakage (score=3) represented by a specimen in subgroup A3 (Delton FS+ was directly applied on air-abraded occlusal surface).*

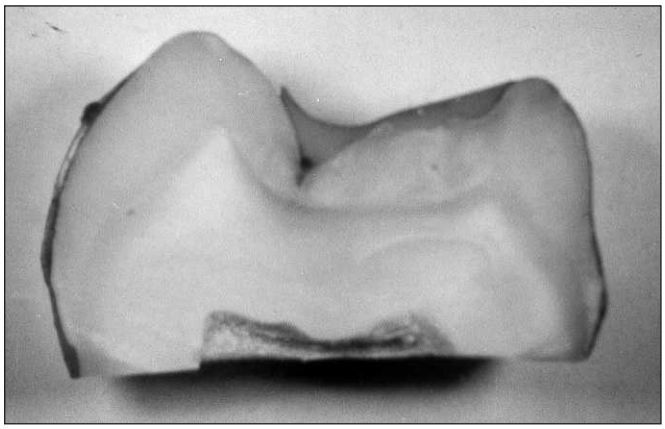


Figure 2: *Section showing severe microleakage (score=3), where Dyract Flow was applied to occlusal surfaces following air-abrasion only (subgroup B3).*

($p<0.05$). The differences between subgroup A2 and subgroups A3, B1 and B3 were statistically significant ($p<0.05$). Statistically significant differences were also found between subgroup A3 and subgroups B1, B2; subgroup B1 and subgroup B3; subgroup B2 and subgroup B3 ($p<0.05$). However, the differences between subgroups A1 and B1; A2 and B2; A3 and B3; B1 and B2 were statistically insignificant ($p>0.05$).

DISCUSSION

Microleakage of sealants has been investigated by numerous *in vitro* studies where air abrasion was used to condition the tooth surfaces prior to fissure sealing. Results of these studies have shown that sealant applications had microleakage scores in varying degrees (Hatibovic-Kofman, Wright & Braverman, 1998; Wright & others, 1999; Hatibovic-Kofman, Butler & Sadek, 2001). In this study, the use of air abrasion alone in subgroups A3 and B3 resulted in significantly higher microleakage scores. In these subgroups, Delton FS+ and Dyract Flow were directly applied to air-abraded occlusal surfaces. This result is consistent with the findings of studies that have reported air abrasion, alone, without acid etching, produces significantly more microleakage (Hatibovic-Kofman & others, 1998; Sams & others, 1995; Haws & others, 1996). In a clinical study by Hamilton and others (2001), air-abrasion, alone was used to treat questionable carious lesions. After 12-month follow-up, the authors reported that of the 113 teeth with questionable incipient caries that were air abraded and restored in the treatment group, 50 (44%) had caries extending into dentin and required re-treatment. They concluded that "the merit of treating questionable incipient pit-and-fissure carious lesions with air-abrasion has not been demonstrated."

In this study, applying acid etching and bonding agent prior to applying fissure sealant was found to have a significant effect on the results obtained. Subgroups where sealant materials were applied on air-abraded occlusal surfaces followed by acid etching and bonding agent application (subgroups A1 and B1) have shown significantly less microleakage. Furthermore, the difference between subgroup A1 and A2 was also significant. These results indicate the importance of acid etching in fissure sealant applications on air-abraded enamel (Hatibovic-Kofman & others, 2001; Sams & others, 1995; Haws & others 1996). The results of an *in vitro* study by Hatibovic-Kofman and others (2001) has emphasized that microleakage could most effectively be prevented with a combination of air abrasion and acid etching. They have reported that air abrasion has provided a level of surface roughening that was not as effective as acid etching. According to the authors, adding acid etching to the air-abrasion procedure would result in a further reduction in microleakage.

Acid etching was omitted in subgroups A2 and B2, and the use of the bonding agent Prime & Bond NT promoted the resins to attain favorable marginal sealing when compared to subgroups A3 and B3. The

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

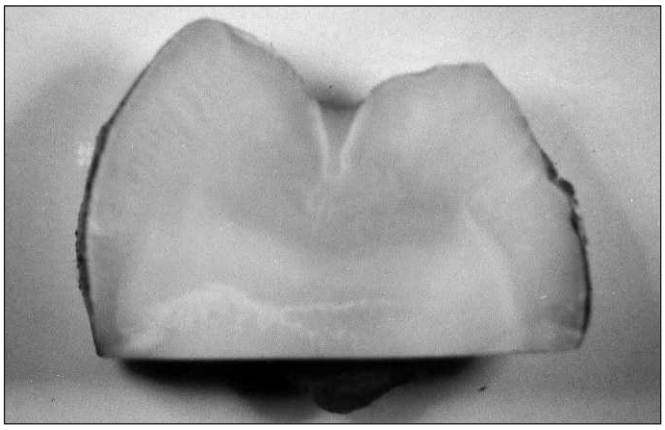


Figure 3: *Photograph of a section in subgroup B2 showing no microleakage. Dyract Flow was applied to air-abraded occlusal surfaces in this subgroup following application of bonding agent Prime & Bond NT.*

potential advantages of using hydrophilic dentin bonding agents as an additional adhesive layer between etched enamel and sealant have been investigated by several studies (Symons, Chu & Meyers, 1996; Tulunoglu & others, 1999; Hitt & Feigal, 1992; Boreem & Feigal, 1994). These studies have reported a marked advantage to this added layer by increasing bond strength and decreasing microleakage, particularly on teeth that had etched enamel contaminated with saliva prior to sealant placement.

The most significant finding of this study was obtained when comparing subgroups B1 and B2. The difference between these subgroups was statistically insignificant. In subgroup B2, using the bonding agent, Prime & Bond NT alone resulted in a comparable finding to that of subgroup B1 (acid etching and bonding agent). This finding may be explained by the components of materials used and their functions. Dyract Flow is a polyacid-modified resin composite with flow characteristics. It contains ammonium salt of PENTA (dipentaerythritol penta acrylate monophosphate) and N, N-dimethyl aminoethyl methacrylate and carboxylic acid modified macromonomers. It also contains strontium-alumino-fluoro-silicate glass particles as the fluoride source. Every molecule of aminopenta contains five polymerizable methacrylate groups and one phosphate group. Nakabayashi, Kojima and Masuhara (1982) reported that methacrylates with the hydrophilic and hydrophobic groups promote the diffusion of monomers due to their affinity for tissues. The hydrophilic carboxylate groups and the phosphate groups of the monomers are responsible for the wetting ability of Dyract Flow. PENTA is also available in Prime & Bond NT and functions as an adhesion promoter, wetting aid and cross-linker. Acetone is another component in Prime & Bond NT and serves as solvent, carrier for the resins and water displacer. Therefore, just using a bonding agent might have promoted Dyract Flow penetrating into the microporosities created by air abrasion in subgroup B2. However, this finding should be investigated further in clinical and laboratory studies.

A contradiction may exist between the results of subgroups A1 and A2 and subgroups B1 and B2. Although subgroups A1 and A2 bear a resemblance to subgroups B1 and B2 with regard to the study design except for the sealant materials, a statistically significant result was obtained for subgroups A1 and A2 only. This condition can be explained by the sealants used. Dyract Flow has enhanced flowing properties when compared to Delton FS+, which is a 55% filled resin sealant. Delton FS+ contains two sources of releasable fluoride, barium alumino fluroboro silicate glass and sodium fluoride. With the use of filled sealants, it is anticipated that poorer sealing, greater void formation and greater microleakage might occur due to their higher viscosity and lower flow properties (Barnes & others, 2000; Hatibovic-Kofman & others, 2000; Flanagan & Pearson, 1988; Percinoto & others, 1995; Irinoda & others, 2000). In a recent *in vitro* study, the effect of viscosity on the penetration of resin sealants into etched human enamel was investigated (Irinoda & others, 2000). The authors reported that the presence of filler particles and tinting agents in the tested sealants tested increased their viscosity and lowered their penetration into etched enamel to reach the depth of acid etching. They concluded that the higher viscosity of sealants might have affected their ability to maintain good marginal seals. Ten Cate, Keizer and Arends (1975) have also observed an increase in tag distribution and length as the viscosity of the resin decreased. However, Barnes and others (2000) reported that the viscosity and flow properties of filled and unfilled fissure sealants did not affect their sealing ability. Another point that may help to explain the results is the use of Prime & Bond NT. Due to its components and hydrophilic characteristics, it might also have contributed better flow of Dyract Flow into the microporosities created by air abrasion than for Delton FS+.

CONCLUSIONS

In this *in vitro* study, the use of air abrasion, alone, in Delton FS+ and Dyract Flow sealed teeth has resulted in extensive dye penetration. Keeping in mind that the flowable polyacid-modified resin composites were introduced to the dental profession only a few years ago, the importance of clinical studies related to them can be better recognized. However, within the limitations of this study, it can be concluded that using Dyract Flow as a fissure sealant on air abraded permanent teeth in combination with acid etching and/or bonding agent has provided comparable microleakage results to that of Delton FS+.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Acknowledgements

The authors express their gratitude to Professor Dr Senay Canay from the Department of Prosthetic Dentistry at Hacettepe University, Faculty of Dentistry for her generous help in obtaining Microetcher. Erdem Karabulut MSc from the Department of Biostatistics Hacettepe University is gratefully acknowledged for performing the statistical analysis.

(Received 20 May 2002)

References

Barnes DM, Kihn P, von Fraunhofer JA & Elsabach A (2000) Flow characteristics and sealing ability of fissure sealants *Operative Dentistry* **25(4)** 306-310.

Boreem LM & Feigal RJ (1994) Reducing microleakage of sealants under salivary contamination: Digital image analysis evaluation *Quintessence International* **25(4)** 283-289.

Brown LJ & Selwitz RH (1995) The impact of recent changes in the epidemiology of dental caries on guidelines for the use of dental sealants *Journal of Public Health Dentistry* **55(5 Special No)** 274-291.

Brunelle JA & Carlos JP (1982) Changes in the prevalence of dental caries in US schoolchildren, 1961-1980 *Journal of Dental Research* **61(Special Issue)** 1346-1351.

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.

De Craene GP, Martens LC, Dermaut LR & Surmont PA (1989) A clinical evaluation of a light-cured fissure sealant (Helioseal) *Journal of Dentistry for Children* **56(2)** 97-102.

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

Flanagan RA & Pearson GJ (1988) Evaluation of dye penetrations around two resin based materials used as fissure sealants *Biomaterials* **9(5)** 460-462.

Goldstein RE & Parkins FM (1994) Air-abrasive technology: Its new role in restorative dentistry *Journal of the American Dental Association* **125(5)** 551-557 Review.

Goldstein RE & Parkins FM (1995) Using air-abrasive technology to diagnose and restore pit and fissure caries *Journal of the American Dental Association* **126(6)** 761-766.

Hamilton JC, Dennison JB, Stoffers KW & Welch KB (2001) A clinical evaluation of air-abrasion treatment of questionable carious lesions. A 12-month report *Journal of the American Dental Association* **132(6)** 762-769.

Hatibovic-Kofman S, Wright GZ & Braverman I (1998) Microleakage of sealants after conventional, bur and air abrasion preparation of pits and fissures *Pediatric Dentistry* **20(3)** 173-176.

Hatibovic-Kofman S, Butler SA & Sadek H (2001) Microleakage of three sealants following conventional, bur and air-abrasion preparation of pits and fissures *International Journal of Paediatric Dentistry* **11(6)** 409-416.

Haws SM, Oliveira ML, Vargas MA & Kanellis MJ (1996) Air abrasion and microleakage of pit and fissure sealants *Journal of Dental Research* **75** Abstract #1301 p 180.

Hicks MJ & Flaitz CM (1993) Epidemiology of dental caries in the pediatric and adolescent population: A review of past and current trends *Journal of Clinical Pediatric Dentistry* **18(1)** 43-49.

Hitt JC & Feigal RJ (1992) Use of a bonding agent to reduce sealant sensitivity to moisture contamination: An *in vitro* study *Pediatric Dentistry* **14(1)** 41-46.

Irinoda Y, Matsumura Y, Kito H, Nakano T, Toyama T, Nakagaki H & Tsuchiya T (2000) Effect of sealant viscosity on the penetration of resin into etched human enamel *Operative Dentistry* **25(4)** 274-282.

Jensen OE & Handelman SL (1980) Effect of an autopolymerizing sealant on viability of microflora in occlusal dental caries *Scandinavian Journal of Dental Research* **88(5)** 382-388.

Jeronimus DJ Jr, Till MJ & Sveen OB (1975) Reduced viability of microorganisms under dental sealants *Journal of Dentistry for Children* **42(4)** 275-280.

Kalsbeek H & Verrips GH (1990) Dental caries prevalence and the use of fluorides in different European countries *Journal of Dental Research* **69(Spec No)** 728-732, discussion 820-823.

Kidd EA (1976) Microleakage in relation to amalgam and composite restorations: A laboratory study *British Dental Journal* **141(10)** 305-310.

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

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.

Ovrebo RC & Raadal M (1990) Microleakage in fissures sealed with resin or glass ionomer cement *Scandinavian Journal of Dental Research* **98(1)** 66-69.

Park K, Georgescu M, Scherer W & Schulman A (1993) Comparison of shear strength, fracture patterns, and microleakage among unfilled, filled, and fluoride–releasing sealants *Pediatric Dentistry* **15(6)** 418-421.

Percinoto C, Cunha RF, Delbem AC & Aragones A (1995) Penetration of a light- cured glass ionomer and a resin sealant into occlusal fissures and etched enamel *American Journal of Dentistry* **8(1)** 20-22.

Ripa LW (1985) The current status of pit and fissure sealants: A review *Journal of the Canadian Dental Association* **51(5)** 367-380 Review.

Ripa LW (1993) Sealants revisited: An update of the effectiveness of pit-and-fissure sealants *Caries Research* **27(Supplement 1)** 77-82 Review.

Sams DR, Dickinson GL, Russell CM & Dadian T (1995) Prophylaxis with microprophy or micro etcher for pit and fissure sealants *Journal of Dental Research* **74** Abstract #490 p 73.

Simonsen RJ (1987) Retention and effectiveness of a single application of white sealant after 10 years *Journal of the American Dental Association* **115(1)** 31-36.

Symons AL, Chu CY & Meyers IA (1996) The effect of fissure morphology and pretreatment of the enamel surfaces on penetration and adhesion of fissure sealants *Journal of Oral Rehabilitation* **23(2)** 791-798.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

ten Cate JM, Keizer S & Arends J (1975) Polymer adhesion to enamel: The influence of viscosity and penetration *Journal of Oral Rehabilitation* **4** 149-156.

Tulunoglu Ö, Bodur H, Üçtasli M & Alaçam A (1999) The effect of bonding agents on the microleakage and bond strength of sealant in primary teeth *Journal of Oral Rehabilitation* **26(5)** 436-441.

Waggoner WF & Siegal M (1996) Pit and fissure sealant application: Updating the technique *Quintessence International* **127(3)** 351–361, quiz 391-392 Review.

Wright GZ, Hatibovic-Kofman S, Millenaar DW & Braverman I (1999) The safety and efficacy of treatment with air abrasion technology *International Journal of Paediatric Dentistry* **9(2)** 133-140.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3,** 274-280

Physiological Remineralization of Artificially Demineralized Dentin Beneath Glass Ionomer Cements With and Without Bacterial Contamination *In Vivo*

Y Kitasako • M Nakajima • RM Foxton
K Aoki • PNR Pereira • J Tagami

Clinical Relevance

Artificially demineralized dentin was shown to be physiologically remineralized beneath glass ionomer restorations.

SUMMARY

This study evaluated the physiological remineralization of artificially demineralized dentin beneath glass ionomer cements with and without bacterial contamination. The artificially demineralized dentin was produced on 84 monkey teeth using a decalcifying solution. Half the samples were left open to the oral cavity for one week, then, all cavities were restored with two glass-ionomer cements: Fuji IX or Fuji II LC improved (n=7). The nanohardness of the artificially demineralized dentin at 3, 90 and 360 days was measured using a nanoindentation tester (ENT-1100, Elionix) and compared statistically by two-way ANOVA and Fisher's PLSD test ($p<0.05$). Each mineral (Ca, Mg, P, F) within the demineralized dentin was also analyzed using Electron Probe Microanalysis. For the samples, the mean nanohardness of the three-day samples was significantly lower than the 360-day samples ($p<0.05$). Although there was no significant difference in the mean nanohardness within all the bacterially-contaminated groups through the experimental periods ($p>0.05$), the mean nanohardness of the bacterial-contaminated samples were significantly lower than the non-bacteria-contaminated samples ($p<0.05$). From the EPMA results, fluoride release from both cements to the bottom of the artificially demineralized dentin was detected within three days. Although Ca density was sparse within this demineralized dentin lesion, for the Fuji IX sample, a high Mg density within this lesion was detected at 360 days.

*Yuichi Kitasako, DDS, PhD, instructor, Tokyo Medical and Dental University, Graduate School, Department of Restorative Sciences, Cariology and Operative Dentistry, Tokyo, Japan

Masatoshi Nakajima, DDS, PhD, lecturer, Tokyo Medical and Dental University, Tokyo, Japan

Richard M Foxton, DDS, graduate student, Tokyo Medical and Dental University, Fixed Prosthodontics, Tokyo Japan

Kazuhiro Aoki, DDS, PhD, instructor, Tokyo Medical and Dental University, Department of Hard Tissue Engineering, Pharmacology, Tokyo Japan

Patricia NR Pereira, DDS, PhD, assistant professor, University of North Carolina at Chapel Hill, School of Dentistry

Junji Tagami, DDS, PhD, professor and chairman, Tokyo Medical and Dental University

*Reprint request: 5-45 Yushima 1-chome, Bunkyo-ku, Tokyo 113–8549, Japan; e-mail: oztasnurhan@hotmail.com

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

INTRODUCTION

Glass ionomer cements (GIC) are potentially caries-inhibitory because of their fluoride release and uptake ability (Forsten, 1998; Mount, 1999). Previous fluoride release studies reported the resistance imparted by restorative materials to secondary caries (Mjör, 1996; Pereira, Inokoshi & Tagami, 1998; Randall & Wilson, 1999). Fluoride and other ions have been detected in high concentrations in the dentin adjacent to GIC restorations (Skartveit & others, 1990; Mukai & others, 1993; Nagamine & others, 1997), and by placing GIC onto demineralized dentin, it resulted in hypermineralization of the dentin (ten Cate & Van Duinen, 1995). Moreover, GICs have been shown to be more effective than non-fluoride containing materials in preventing *in vitro* recurrent root caries (Hsu & others, 1988). Root surface caries has become a significant clinical problem in the dentate elderly (Espelid & Tveit, 1991). Adhesive fluoride-releasing restorative materials such as resin-modified glass ionomer cements are suitable for restoring root caries lesions, because they possess an anticariogenic effect (Pereira & others, 1998; Hsu & others, 1988; Dionysopoulos & others, 1998).

There have been several reports of a substantial reduction in the number of cariogenic microorganisms remaining in soft carious dentin following cavity sealing with GIC (Weerheijm & others, 1993; Weerheijm & others, 1999; Seppä, Torppa-Saarinen & Luoma, 1992). GIC restorations may prevent caries progression by favoring remineralization and/or by interfering with the growth and metabolism of the remaining cariogenic bacteria in the dentin, presumably by the release of various ions (Fischman & Tinanoff, 1994). A packable glass ionomer cement, Fuji IX (GC Corp Tokyo, Japan), with a high flexural strength, lower solubility and increased wear resistance compared to conventional glass ionomer cements, has been introduced for use in the Atraumatic Restorative Technique (ART) approach. This approach has been developed to provide urgently needed treatment of dental caries in populations where conventional methods of dental care are not available or too costly. Because of the increase in life expectancy and retained teeth in the elderly population, this approach for treating root caries lesions would also be beneficial for the underlying vital pulp. However, the mechanism of *in vivo* remineralization of demineralized dentin beneath fluoride-releasing materials is not fully understood.

Artificial caries media have been used to evaluate the remineralization of dentin (ten Cate & Van Duinen, 1995; Arends & others, 1989; Tatsumi & others, 1992; Creanor & others, 1998). Tatsumi and others (1992) demonstrated that artificially demineralized dentin could be produced in monkey teeth to simulate a clinical carious cavity. Physiological remineralization of artificially demineralized monkey teeth, accompanied by an increased hardness and calcium content recovery, occurs only in the underlying dentin by normal biologic processes (Tatsumi & others, 1992). However, physiological remineralization of artificially demineralized dentin beneath glass ionomer cement is not fully understood, particularly in relation to non-bacteria-contaminated or bacteria-contaminated environments *in vivo*. Using the artificial caries model, this *in vivo* study evaluated the physiological remineralization of artificially demineralized dentin beneath glass ionomer cements with and without bacterial contamination.

METHODS AND MATERIALS

Three monkeys (Macaca fuscata) were housed in facilities approved by the Tokyo Medical and Dental University. The principles of laboratory animal care were followed as well as specific national laws. The Ethics Committee for Animal Care at Tokyo Medical and Dental University approved the animal use protocol. Because two experimental groups (non-bacteria contaminated or the bacteria-contaminated group), two glass ionomer cements and three different observation periods (3, 90 and 360 days) were used in this study (Figure 1), samples (n=7) of one experimental group were obtained from 28 cavities per animal (total 84 samples).

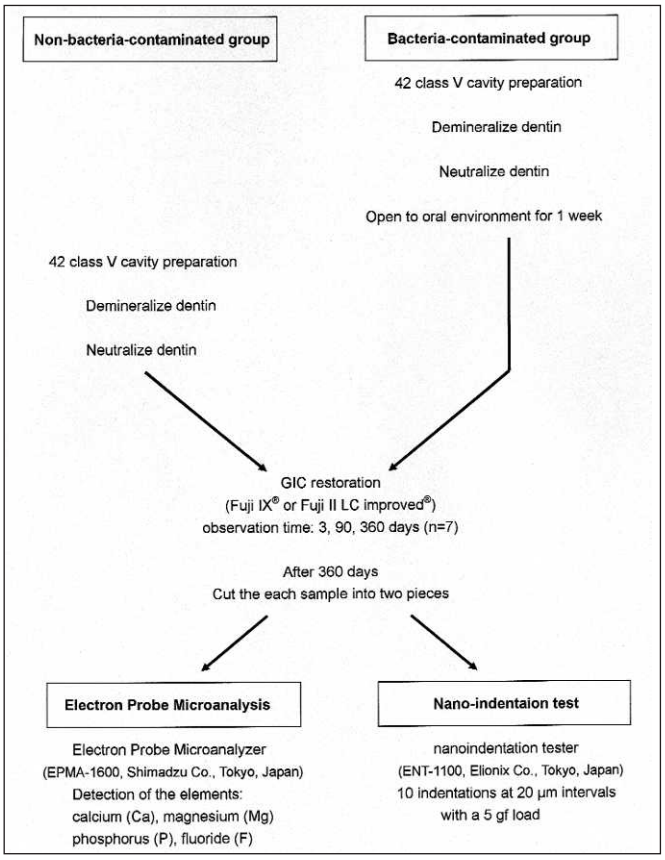


Figure 1: *Flow chart of the experimental procedure.*

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Materials and Specimen Preparation

The animals were placed under general anesthesia by intramuscular injection of 20 mg/kg ketamine (Ketaral, Sankyo Co, Tokyo, Japan) and intravenous injection of 10 mg/kg pentobarbital sodium (Nembutal Sodium Solution, Abbott Laboratories, Abbott Park, IL, USA). Class V cavities were prepared on the facial surface of 84 intact teeth using a high-speed tapered diamond bur (ISO #170, GC Corp, Tokyo, Japan) under water-spray. To simulate a carious cavity, artificially demineralized dentin was produced in the axial walls by means of a decalcifying solution (Tatsumi & others, 1992). After protecting the enamel surface with a varnish, a quick decalcifying solution (7 gm $AlCl_3$, 8.5 cm^3 37% HCl and 5 cm^3 95% formic acid in distilled water, 100 cm^3 in total) was applied to the cavity at a rate of one drop/second for five minutes and neutralized by applying 5% sodium sulfate at a rate of two drops/second for one minute. This technique produced artificially demineralized dentin that was detectable with a caries detector dye (Fusayama, Okuse & Hosoda, 1966). This cavity was, therefore, considered representative of a clinical carious cavity. In this study, physiological remineralization of the artificially demineralized dentin layer beneath the glass ionomer cement was evaluated with and without bacterial contamination.

Forty-two prepared cavities were left open to the oral environment for one week to obtain the bacteria contaminated sample (Sasafuchi & others, 1999). After one week, another 42 cavities were prepared. All cavities were restored with one of two glass ionomer cements: a packable glass ionomer cement (Fuji IX, GC Corp Tokyo, Japan) or a resin-modified glass ionomer cement (Fuji II LC improved, GC Corp Tokyo, Japan). For the bacteria-contaminated group, water spray cleaning with a five-second blast of air was performed to remove food debris before placing the restoration (Brännström & Nyborg, 1973). After the cement had set, varnish for Fuji IX (Fuji VARNISH, GC Corp, Tokyo, Japan) and varnish for Fuji II LC (Fuji II COAT LC, GC Corp) were applied to the cement surface, respectively. Two non-bacteria-contaminated cavities for the non-bacteria-contaminated group were prepared as a control sample to evaluate the baseline for the nanohardness.

At 360 days after preparation, the monkeys were sacrificed by intravenous injection of 250 mg/kg thiopental sodium (Ravonal, Tanabe Pharmaceutical Co, Osaka, Japan). The teeth were extracted and immersed in a 10% neutral buffered formalin solution for two weeks. Two 200 µm-thick longitudinal sections through the center of the restoration were cut with a diamond disk under running water. One section was polished with waterproof silicon carbide papers (grit #600, 800, 1000, 1200 and 1500) to a thickness of 100 µm under running water. After polishing with diamond pastes (particle size 6, 3, 1, 0.25 µm) under running water, the artificial demineralized dentin layer was observed under light microscopy (PM-10AK, Olympus Optical Corp, Tokyo, Japan) at a magnification of 40x (Figure 2).

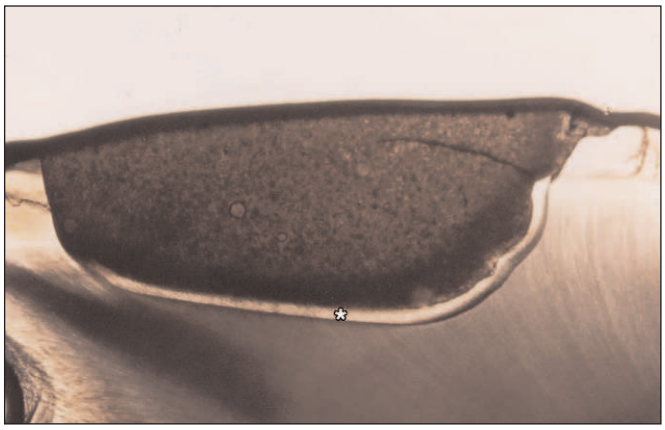


Figure 2: *Light microscopic photograph of an experimental sample (Fuji IX, non-bacteria-contaminated group, three day, 40x). The Artificially demineralized dentin layer (asterisk) was observed beneath the glass ionomer cement. This layer was completely demineralized by using Plank Rychlo's decalcifying solution.*

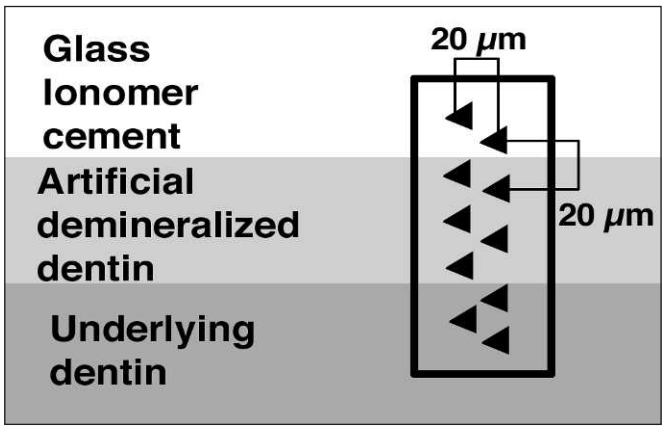


Figure 3: *Nano-indentation test. Ten indentations were made at 20 µm intervals across the demineralized layer from the glass ionomer cement to the undemineralized dentin.*

Electron Probe Microanalysis

For Electron Probe Microanalysis, the light microscopic section was successively dehydrated in ethanol of increasing concentrations (50%, 70%, 80%, 90%, 95% and 100%), immersed in a stylene monomer (Niishin EM Co, Tokyo, Japan) for three days and embedded in a benzoyl peroxide-added polyester resin (Oken Trade Co, Tokyo, Japan). After setting, the surface was polished with waterproof silicon carbide papers (grit #600, 800, 1000, 1200 and 1500) and diamond pastes (particle size 6, 3, 1, 0.25 µm) under running water. They were then vacuum-coated with carbon. The carbon-coated samples were used to detect the elements calcium (Ca), magnesium (Mg), phosphorus (P) and fluoride (F) in demineralized dentin using an Electron Probe

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Microanalyzer (EPMA-1600, Shimadzu Co, Tokyo, Japan). During the EPMA analysis, Backscatter electronic images (BEI) were also photographed at a magnification of 1000x. The Electron microscope settings were 15 kV (accelerating voltage) and 100 nA (probe current) with a probe diameter of 1 µm on setting. A scanning spot with a spatial resolution of 400 X 400 pixels and a dwell time of 50m seconds per pixel was used for the elemental mapping. With this instrument, the distribution of each element is shown in 16 pseudo colors, depending on the X-ray intensity. Mapping images of each element could be obtained by EPMA.

Nanohardness Test

The final section was embedded in epoxy resin (Epon 815, Niishin EM Co, Tokyo, Japan). After resin polymerization for 24 hours, the surfaces were polished with waterproof silicon carbide papers (grit #600, 800, 1000, 1200, 1500) and diamond pastes (particle size 6, 3, 1, 0.25 µm) under running water. The sample molds were positioned on a heated modeling compound that served to stabilize the specimen surface and orient it parallel to the stage of the nanoindentation tester (ENT-1100, Elionix Co, Tokyo, Japan) with a 5 gf load. The 10% formalin solution was intermittently applied in drops to prevent drying. The instrument used for this nanohardness testing was a depth sensing computer-controlled instrument with a three-sided pyramidal diamond probe (Urabe & others, 2000). After setting a sample on the stage of the testing machine, the instrument was programmed to create the position of indentations. Ten indentations were made at 20-µm intervals across the demineralized dentin layer from the glass ionomer cement to the undemineralized dentin (Figure 3). The load on the indenter was 5 gf (Urabe & others, 2000). After making the indentation, nanohardness was calculated by an attached computer. Mean nanohardness was compared statistically by two-way ANOVA and Fisher's PLSD test ($p<0.05$).

RESULTS

Width of Demineralized Dentin Layer (Table 1)

For the non-bacteria-contaminated groups, the width of the demineralized dentin layer ranged from 86 µm to 128 µm, with the means for the groups ranging from 99 µm to 116 µm. For the bacteria contaminated groups, the width of the demineralized dentin layer ranged from 47 µm to 75 µm, with the means for the groups ranging from 59 µm to 67 µm. Although no significant differences were found among the widths of the demineralized dentin layers at each time interval ($p=0.99$) for both uninfected and infected experimental groups, there were significant differences between the non-bacteria-contaminated and bacteria-contaminated groups ($p<0.05$).

EPMA Findings

For both the bacteria-free and contaminated groups at three days after operation, fluoride (F) release to the bottom of the artificially demineralized dentin occurred and calcium (Ca), magnesium (Mg) and phosphorus (P) loss were clearly detected within this demineralized dentin lesion. At 90 and 360 days after operation, in the case of the non-bacteria-contaminated groups, a high density of F within the bottom of this demineralized dentin lesion was still detected. For the bacteria-contaminated groups, each mineral density at 90 and 360 days after operation was not clear under EPMA. Although Ca was sparsely detected within this demineralized dentin lesion at 360 days after operation, in the non-bacteria-contaminated groups, a high density of Mg and a moderate density of P was also detectable within this lesion (Figure 4).

Nanohardness Test Findings (Table 1)

There were no significant differences in mean nanohardness among the three-day control, non-bacteria-contaminated and bacteria-contaminated samples ($p>0.05$). The mean nanohardness (Kg/mm^3±SD) for the non-bacteria-contaminated groups at the different

Table 1: *Nano-Hardness (Kg/mm^3) and Width of Demineralized Dentin (µm)*

Experimental Groups		Non-Bacteria-Contaminated Group								Bacteria-Contaminated Group					
Time Intervals		0 day	3 day		90 day		360 day			3 day		90 day		360 day	
Glass ionomer		Non-Restored Control	Fuji IX	Fuji II LC	Fuji IX	Fuji II LC	Fuji IX	Fuji II LC		Fuji IX	Fuji II LC	Fuji IX	Fuji II LC	Fuji IX	Fuji II LC
Nano-hardness	mean	23.3	22.1	20.6	25.8	29.7	41.8	30.8		21.2	22.2	16.7	18.6	17.2	16.4
	SD	5.9	9.1	7.7	9.3	9.0	18.3	8.5		6.9	6.1	7.4	5.0	8.2	8.9
Width of Demineralized Dentin	mean	128	116	104	108	99	105	108		67	64	63	62	60	59
	min	122	98	95	91	86	88	96		52	55	59	54	49	47
	max	138	128	110	122	106	121	119		74	73	75	71	68	64

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

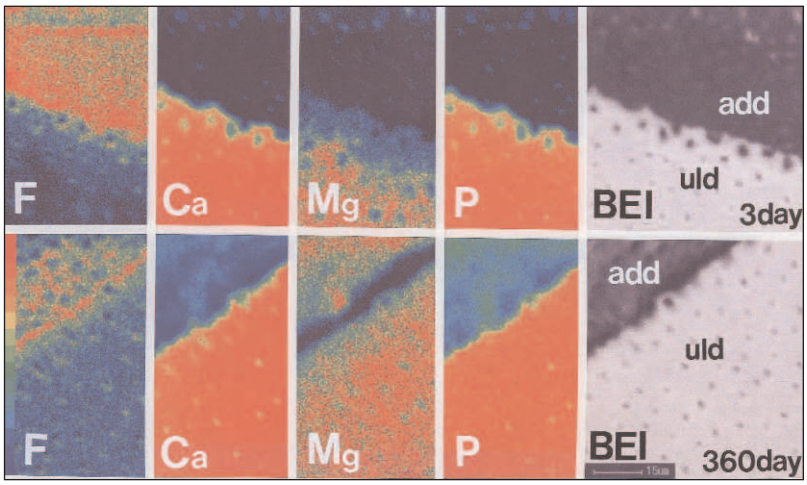


Figure 4: *EPMA and Backscatter electronic image (BEI) photographs of the non-bacteria contaminated Fuji IX samples at 3 and 360 days after operation (X1000). At three days after operation, fluoride (F) release to the bottom of the artificially demineralized samples occurred completely, and calcium (Ca), magnesium (Mg) and phosphorus (P) loss was obviously detected within this demineralized dentin lesion. Although Ca density was sparse within this demineralized lesion at 360 days after operation, for the non-bacteria-contaminated groups, high density of Mg content and moderate density of P content were also detected within this lesion.*

times (3, 90 and 360 days) for Fuji II LC were 20.6±7.7, 29.7±9.0, 30.8±8.5, while the means for Fuji IX were 25.8±9.3, 22.2±9.1 and 41.8±18.3, respectively. Although there was no significant difference in mean nanohardness between the 3- and 90-day samples ($p>0.05$), the mean nanohardness of the three-day samples were significantly lower than the 360-day samples ($p<0.05$). There was a significant difference in mean nanohardness between Fuji IX and Fuji II LC that improved at 360 days after operation ($p>0.05$). On the other hand, for the bacteria-contaminated groups, the mean nanohardness (Kg/mm^3±SD) at the different times (3, 90 and 360 days) for Fuji II LC were 22.2±6.1, 29.7±9.0 and 16.4±8.9, and for Fuji IX, they were 21.2±6.9, 14.7±8.4 and 16.4±8.9, respectively. Although there was no significant difference in mean nanohardness within all the bacteria-contaminated groups through the experimental periods ($p>0.05$), the mean nanohardness of the bacteria-contaminated samples was significantly lower than the non-bacteria-contaminated samples ($p<0.05$).

DISCUSSION

In vitro and *in vivo* researchers examining the demineralization and remineralization of teeth have used several tools and approaches, including change in hardness of the lesion, contact-microradiography and various analytical techniques such as x-ray diffraction or EDAX (Cox & others, 1980; Akimoto & others, 2001). In this study, the nano indentation tester provided useful data on the artificial demineralized dentin that could not be evaluated using the classical type of hardness tester (Tatsumi & others, 1992). On the other hand, electron-probe microanalysis (EPMA) enables for the identification and quantification of elements present on de- and remineralized dentin (Frank, Capitant & Goni, 1966; Yamamoto & others, 2001). It is expected that from the EPMA results, fluoride release to the bottom of the artificially demineralized dentin might occur within three days of the operation. The clinical significance of the EPMA findings is that stability of the incorporated fluoride within the demineralized dentin lesions would promote a dentin remineralization cycle, contributing to the control of caries disease. However, in the case of the bacteria-contaminated groups, each mineral density at 90 and 360 days after preparation could not be clearly detected using EPMA. Even after water spray cleaning of the bacteria-contaminated group, bacteria might exist within the artificial demineralized dentin (Brännström & Nyborg, 1973). However, for bacterial contamination groups, there was no significant difference in the mean microhardness through the experimental periods ($p>0.05$). Further bacterial studies are needed to evaluate the changing environment within the bacteria-contaminated layer beneath glass ionomer restorations.

The dentin matrix consists of mineral and organic components similar to those of other mineralized tissues, such as bone and cementum. The prototype for the mineral is hydroxyapatite, which contains calcium, phosphorus, oxygen, magnesium and hydrogen as essential elements in its structure. The EPMA characteristic areas with a high density of magnesium content suggest that magnesium might act as an important cofactor for enzyme activation (Althoff & others, 1982; Wiesmann & others, 1997). Because magnesium in hydroxyl apatite crystals is among the first elements to be dissolved in the demineralization process (Tjäderhane, Hietala & Larmas, 1995), magnesium-elemental loss and gain might be effective in promoting the condition of dentin remineralization. Magnesium affects mineral metabolism (Wallach, 1991) and the pattern of mineral formation (Boskey & others, 1992) or the control apatite crystal size (Bigi & others, 1992; Tsuboi & others, 1994; Aoba, Moreno & Shimoda, 1992). Bigi and others (1992) reported that apatite destabilization in the presence of magnesium is associated with a reduction in apatite crystal size and the Ca/P molar ratio but not with a change in lattice dimensions. The high density of magnesium content in the demineralized dentin areas appears to be unstable, and during physiological remineralization, it could be released from its original location, resulting in a more homogenous magnesium distribution in physiological remineralized dentin.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Caries resistance and the formation of the inhibition zone appear to be associated with the level of fluoride release from glass ionomer restorations (ten Cate & Van Duinen, 1995; Okuda & others, 2002). Diaz-Arnold and others (1995) observed that conventional glass ionomer cement released greater amounts of fluoride than a resin-modified glass ionomer cement. Although the mean nanohardness of the bacteria-free Fuji IX and Fuji II LC samples was significantly lower than the 360-day samples ($p<0.05$), there was a significant difference in mean nanohardness between the two cements at 360 days after operation. Not only did the differences in material composition and fluoride releaseinfluence the nanohardness data, but other factors such as type of cure of the cements, underlying vital pulp or other elements from the ionomer materials may have influenced this data. The results of this *in vivo* study might provide new and important information related to how the glass ionomer-demineralized dentin interface responds to dentin remineralization under a dynamic environment. It should lead to an improvement in the formulations of resin-modified and packable glass ionomers.

Because physiological remineralization of artificial demineralized monkey teeth occurs only in inner demineralized dentin (Tatsumi & others, 1992), the outer demineralized dentin with bacterial infection should be removed during caries treatment (Fusayama & others, 1966). In this study on outer demineralized dentin, the mean nanohardness of the bacteria-contaminated samples was significantly lower than the non-bacteria-contaminated samples at 90 and 360 days after preparation ($p<0.05$). Moreover, in the case of the bacteria-contaminated groups, there were some areas that had the same nanohardness as the non-bacteria-contaminated samples. In the clinical situation, dentists, to date, have been guided regarding the extent of carious dentin by referring to its softness, detection with hand instruments and discoloration observed by the naked eye (Gao, Smales & Yip, 2000). However, the hardness and color of carious dentin changes gradually and continuously from the cavity floor to unaffected normal dentin without any clinically recognizable boundaries (Ogawa & others, 1983). Consequently, carious dentin is frequently left at the enamel-dentin junction after conventional caries removal (Anderson & Charbeneau, 1985), and excessive remineralizable dentin such as outer demineralized dentin may be removed when using rotary instruments (Smales & Fang, 1999). With the increased life expectancy and number of retained teeth in the elderly, root surfaces become exposed to the oral environment, increasing the risk of root surface caries. Unfortunately, there is no scientific basis to guide dentists in how much caries-affected dentin should be removed according to the minimum intervention concept. For the physiological remineralization of demineralized dentin, glass ionomer cements would be suitable for promoting the environment and effectively changing the remineralization cycle.

CONCLUSIONS

Artificially demineralized dentin was proven to physiologically remineralize beneath well-sealed glass ionomer restorations.

(Received 20 May 2002)

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

References

Akimoto N, Yokoyama G, Ohmori K, Suzuki S, Kohno A & Cox CF (2001) Remineralization across the resin-dentin interface: *In vivo* evaluation with nanoindentation measurements, EDS, and SEM *Quintessence International* **32(7)** 561-570.

Althoff J, Quint P, Krefting & Hohling HJ (1982) Morphological studies on the epiphyseal growth plate combined with biochemical and X-ray microprobe analyses *Histochemistry* **74(4)** 541-552.

Anderson MH & Charbeneau GT (1985) A comparison of digital and optical criteria for detecting carious dentin *Journal of Prosthetic Dentistry* **53(5)** 643-646.

Aoba T, Moreno EC & Shimoda S (1992) Competitive adsorption of magnesium and calcium ions onto synthetic and biological apatites *Calcified Tissue International* **51(2)** 143-150.

Arends J, Christoffersen J, Ruben J & Jongebloed WL (1989) Remineralization of bovine dentine *in vitro.* The influence of the F content in solution on mineral distribution *Caries Research* **23(5)** 309-314.

Bigi A, Foresti E, Gregorini R, Ripamonti A, Roveri N & Shah JS (1992) The role of magnesium on the structure of biological apatites *Calcified Tissue International* **50(5)** 439-444.

Boskey AL, Rimnac CM, Bansal M, Federmann M, Lian J & Boyan BD (1992) Effect of short-term hypomagsemia on the chemical and mechanical properties of rat bone *Journal of Orthopaedic Research* **10(6)** 774-783.

Brännström M & Nyborg H (1973) Cavity treatment with a microbicidal fluoride solution: Growth of bacteria and effect on the pulp *Journal of Prosthetic Dentistry* **30(3)** 303-310.

Cox CF, Heys DR, Gibbons PK, Avery JK & Heys RJ (1980) The effect of various restorative materials on the microhardness of reparative dentin *Journal of Dental Research* **59(2)** 109-115.

Creanor SL, Awawdeh LA, Saunders WP, Foye RH & Gilmour WH (1998) The effect of a resin-modified glass ionomers restorative material on artificially demineralized dentine caries *in vitro Journal of Dentistry* **26(5-6)** 527-531.

Diaz-Arnold AM, Holmes DC, Wistrom DW & Swift EJ Jr (1995) Short-term fluoride release/uptake of glass ionomer restoratives *Dental Materials* **11(2)** 96-101.

Dionysopoulos P, Kotsanos N, Papadogiannis Y & Konstantinidis A (1998) Artificial caries formation around fluoride-releasing restorations in roots *Journal of Oral Rehabilitation* **25(11)** 814-820.

Espelid I & Tveit AB (1991) Diagnosis of secondary caries and crevices adjacent to amalgam *International Dental Journal* **41(6)** 359-364.

Fischman SA & Tinanoff N (1994) The effect of acid and fluoride release on the antimicrobial properties of four glass ionomers cements *Pediatric Dentistry* **16(5)** 368-370.

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

Frank RM, Capitant M & Goni J (1966) Electron probe studies of human enamel *Journal of Dental Research* **(Supplement 3)** 672-682.

Fusayama T, Okuse K & Hosoda H (1966) Relationship between hardness, discoloration and microbial invasion in carious dentin *Journal of Dental Research* **45(4)** 1033-1046.

Gao W, Smales RJ & Yip HK (2000) Demineralization and remineralization of dentine caries, and the role of glass-ionomer cements *International Dental Journal* **50(1)** 51-56 Review.

Hsu CYS, Donly KJ, Drake DR & Wefel JS (1988) Effect of aged fluoride-containing restorative materials on recurrent root caries *Journal of Dental Research* **77(2)** 418-425.

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

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

Mukai M, Ikeda M, Yanagihara T, Hara G, Kato K, Nakagaki H & Robinson C (1993) Fluoride uptake in human dentine from glass-ionomer cement *in vivo Archives of Oral Biology* **38(12)** 1093-1098.

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

Ogawa K, Yamashita Y, Ichijo T & Fusayama T (1983) The ultrastructure and hardness of the transparent layer of human carious dentin *Journal of Dental Research* **62(1)** 7-10.

Okuda M, Pereira PN, Nikaido T & Tagami J (2002) Evaluation of *in vitro* secondary caries using confocal laser scanning microscope and X-ray analytical microscope *American Journal of Dentistry* (in press).

Pereira PNR, Inokoshi S & Tagami J (1998) *In vitro* secondary caries inhibition around fluoride releasing materials *Journal of Dentistry* **26(5-6)** 479-485.

Randall RC & Wilson NH (1999) Glass-ionomer restoratives: A systematic review of a secondary caries treatment effect *Journal of Dental Research* **78(2)** 628-637.

Sasafuchi Y, Otsuki M, Inokoshi S & Tagami J (1999) The effects on pulp tissue of microleakage in resin composite restorations *Journal of Medical and Dental Sciences* **46(4)** 155-164.

Seppä L, Torppa-Saarinen E & Luoma H (1992) Effect of different glass ionomers on the acid production and electrolyte metabolism of Streptococcus mutans Ingbritt *Caries Research* **26(6)** 434-438.

Skartveit L, Tveit AB, Tøtdal B, Øvrebo R & Raadal M (1990) *In vivo* fluoride uptake in enamel and dentin from fluoride-containing materials *Journal of Dentistry for Children* **57(2)** 97-100.

Smales RJ & Fang DT (1999) *In vitro* effectiveness of hard excavation of caries with the ART technique. A traumatic restorative treatment *Caries Research* **33(6)** 437-440.

Tatsumi T, Inokoshi S, Yamada T & Hosoda H (1992) Remineralization of etched dentin *Journal of Prosthetic Dentistry* **67(5)** 617-620.

ten Cate JM & Van Duinen RN (1995) Hypermineralization of dentinal lesions adjacent to glass-ionomer cement restorations *Journal of Dental Research* **74(6)** 1266-1271.

Tjäderhane L, Hietala EL & Larmas M (1995) Mineral element analysis of carious and sound rat dentin by electron probe microanalyzer combined with back-scattered electron image *Journal of Dental Research* **74(11)** 1770-1774.

Tsuboi S, Nakagaki H, Ishiguro K, Kondo K, Mukai M, Robinson C & Weatherell JA (1994) Magnesium distribution in human bone *Calcified Tissue International* **54(1)** 34-37.

Urabe I, Nakajima M, Sano H & Tagami J (2000) Physical properties of the dentin-enamel junction region *American Journal of Dentistry* **13(3)** 129-135.

Wallach S (1991) Relation of magnesium to osteoporosis and calcium urolithiasis *Magnesium & Trace Elements* **10(2-4)** 281-286 Review.

Weerheijm KL, de Soet JJ, van Amerongen WE & de Graaff J (1993) The effect of glass-ionomer cement on carious dentine. An *in vivo* study *Caries Research* **27(5)** 417-423.

Weerheijm KL, Kreulen CM, de Soet JJ, Groen HJ & van Amerongen WE (1999) Bacterial counts in carious dentine under restorations: 2-year *in vivo* effects *Caries Research* **33(2)** 130-134.

Wiesmann HP, Tkotz T, Joos U, Zierold K, Stratmann U, Szuwart T, Plate U & Hohling HJ (1997) Magnesium in newly formed dentin mineral of rat incisor *Journal of Bone & Mineral Research* **12(3)** 380-383.

Yamamoto H, Iwami Y, Unezaki T, Tomii Y & Ebisu S (2001) Fluoride uptake in human teeth from fluoride-releasing restorative material *in vivo* and *in vitro*: Two-dimensional mapping by EPMA-WDX *Caries Research* **35(2)** 111-115.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3,** 281-286

Assessing the Surface Roughness of a Posterior Resin Composite: Effect of Surface Sealing

CYG Takeuchi • VH Orbegoso Flores • RG Palma Dibb
H Panzeri • EHG Lara • W Dinelli

Clinical Relevance

Although the surface sealing of polymerized composites has been widely suggested, the use of surface sealants has not reduced the surface roughness of a posterior resin composite after simulated toothbrushing.

SUMMARY

This study assessed the *in vitro* influence of surface sealing on the surface roughness of a posterior resin composite before and after toothbrushing. Thirty specimens (13 mm diameter x 1 mm high) were fabricated from Filtek-P60 resin composite and randomly assigned to three groups (n=10): a non-sealed control and two groups sealed with one of the tested materials—a surface-penetrating sealant (Protect-it!-PI) and a one bottle adhesive system (Single Bond-SB). The samples were subjected to a surface roughness reading to determine the initial roughness, then submitted to simulated toothbrushing with 35,600 cycles for 100 minutes. Specimens were then cleaned and a post-abrasion surface roughness reading accomplished. Means (µm), recorded before (B) and after (A) toothbrushing, and standard deviations were: Control—(B): 0.032 (±0.005), (A): 0.054 (±0.005); PI—(B): 0.034 (±0.005), (A): 0.060 (±0.034); SB (B): 0.031 (±0.004), (A): 0.047 (±0.007). Data were tabulated and submitted to two-way ANOVA. No statistically significant difference was observed when the control and experimental groups were compared. However, a significant difference ($p<0.05$) was found between the measurements performed before and after toothbrushing. Based on these results, it may be concluded that using either a surface penetrating sealant or a one bottle adhesive system did not provide the optimization of superficial integrity. The use of a dentifrice and toothbrush resulted in significant alterations to the surface smoothness of the resin composite.

*Cristina Yoshie Garcia Takeuchi, DDS, post-graduate student, Department of Restorative Dentistry, Araraquara Dental School, São Paulo State University (UNESP), Brazil

Victor Humberto Orbegoso Flores, DDS, MS, PhD, research fellow, Araraquara Dental School, São Paulo State University (UNESP), Brazil

Regina Guenka Palma Dibb, DDS, MS, PhD, professor, Department of Operative Dentistry, Ribeirão Preto Dental School, University of São Paulo (USP), Brazil

Heitor Panzeri, DDS, MS, PhD, professor, Department of Dental Materials and Prosthodontics, Ribeirão Preto Dental School, University of São Paulo (USP), Brazil

Elza Helena Guimarães Lara, DDS, MS, PhD, professor, Department of Pharmaceutical Sciences, Pharmaceutical Sciences School, University of São Paulo (USP), Brazil

Welingtom Dinelli, DDS, MS, PhD, professor, Department of Restorative Dentistry, Araraquara Dental School, São Paulo State University (UNESP), Brazil

*Reprint request: R Humaitá, n° 1680 CEP: 14801-903, Araraquara-SP, Brasil; e-mail: cristakeuchi@yahoo.com.br

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

INTRODUCTION

The advent of the acid-etching technique (Buonocore, 1955) and the further introduction of BIS-GMA resin composites (Bowen, 1956) has undoubtedly represented the most remarkable approaches in the outstanding progress of adhesive dentistry, as it has widened the scope in oral health care and has decisively prompted the development and optimization of innumerable restorative systems and techniques. Therefore, the indications and applicability of adhesive materials have been increasingly extended and widespread.

For more than 30 years, resin composite has been employed in posterior teeth. Although the earliest formulations yielded a number of problems, such as high polymerization shrinkage, marginal microleakage that invariably led to secondary caries, low wear resistance and loss of anatomic form, (Phillips & others, 1971; Phillips & others, 1973; Leinfelder & others, 1975), composites have recently experienced a notable improvement with their physical properties (Ramos & others; Wang, 2002). However, despite advances, wear of composite materials still persists. Even after accomplishing appropriate finishing and polishing techniques, the surface exhibits microirregularities that inherently lead to material wear, deterioration and marginal infiltration, resulting mainly from the abrasive processes to which the restoration is subjected in the oral environment. Wear is, therefore, a complex process that involves several features—masticatory efforts, fatigue, friction, erosion and oral health care procedures that interact among themselves (Heath & Wilson, 1976; Araújo & Araújo, 1983; Knibbs & Pearson, 1984; Boghosian, Randolph & Jekkals, 1987; Pratten & Johnson, 1988; Dickinson & others, 1990; Leinfelder, 1991; Berastegui & others, 1992). Clinically, the wear of a restoration may result from centric and functional contacts, attrition of food bolus, interproximal contact areas, and toothbrushing (abrasion wear) through the mechanical action of the toothbrush and dentifrice (Kanter, Koski & Martin, 1982; Ehrnford, 1983; Goldstein & Lerner, 1991).

In an attempt to overcome this problem, using a thin layer of a low-viscosity resin over polymerized composite restoration has been investigated. The so-called surface-penetrating sealant or rebonding agent should be able to fill, by capillary action, the structural microdefects and microfissures that are formed during the insertion technique and finishing/polishing procedures. This approach is assumed to provide a more uniform, regular surface, thereby enhancing surface smoothness. In addition, the coating resin would be capable of penetrating deeply into the interfacial microgaps, thus providing improved marginal sealing, especially in the dentin/cementum margins.

Although the properties of the latest posterior resin composites have been optimized, there is still a lack of studies reporting whether the surface integrity of such materials may be enhanced by the use of low-viscosity surface sealant. Moreover, although some studies have supported the idea that using specific surface-penetrating sealants is essential for an optimal resealing of polymerized resin composite restorations, a sufficiently low-viscosity resin agent with proper characteristics and formulation, even though not specifically developed for such purpose, could be successfully used as a surface sealant.

Taking this into consideration, this research assessed the influence of surface sealing on surface roughness of posterior resin composite before and after toothbrushing.

METHODS AND MATERIALS

A total of 30 disks (13 mm diameter and 1 mm thick) were fabricated from Filtek P60 posterior resin composite (3M Dental Products, St Paul, MN, USA) using a *Teflon* matrix. The restorative material was inserted according to an incremental filling technique and light cured for 20 seconds, as recommended by manufacturer's instructions, using a visible light-curing unit with 500mW/cm^2 output (XL 3000, 3M Dental Products). To insure the fabrication of specimens with highly regular surfaces before photo-polymerizing the last increment, a microscopic slide with a 1,650g weight was placed over the resin/matrix block in order to compact the material and provide a smooth, standardized surface. After 30 seconds, the weight was removed and the resin composite was light cured through the slide.

Resin composite samples were randomly assigned to three groups of equal size (n=10) according to the surface treatment: a non-sealed control group and two experimental groups in which the surface sealing was accomplished with one of the tested agents—a surface-penetrating sealant (Protect-it!-PI) (Pentron Inc, Wallingford, CT, USA) and a one bottle adhesive system (Single Bond-SB) (3M-ESPE Dental Products, St Paul, MN, USA). Table 1 lists the tested materials with their compositions, specifications and manufacturers.

Table 1: *Materials Tested*

Material	Category	Composition	Batch #
Filtek P-60	Posterior resin composite	Zircon/Silica BIS-GMA, UDMA and BIS-EMA	1KF, 0BR
Single Bond	Ethanol-and water-based, one-bottle adhesive system	Water/ethanol/HEMA, BIS-GMA, Dimentacrylate, Copolymers, Polyacrylic acid and Itaconic acid	1FC
Protect-it!	Surface-penetrating sealant	BIS-GMA, UDMA, TEGDMA, THFMA, Photoinitiators, Accelerator, Stabilizer	41205

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

For the sealed groups, both materials were applied following the same guidelines that were established for the surface sealing technique: the resin composite surface was etched with a 37% phosphoric acid gel for 15 seconds, rinsed thoroughly for 15 seconds and the excess water removed with a mild oil-free air stream for 10 seconds. A uniform layer of the low-viscosity agent was applied over the etched area using a disposable microbrush with light scrubbing motion for 20 seconds, gently air-thinned for five seconds to insure an even distribution and light-cured for 20 seconds. It is important to emphasize that although Single Bond is an ethanol- and water-based one bottle adhesive system and works best with some water present, the goal of this study was to assess the viability of using the one bottle adhesive system as a surface sealant. Therefore, Single Bond was applied following the same directions recommended for a surface-penetrating sealant.

Following the surface treatment, the specimens were stored in distilled water at 37°C for seven days. The samples were then subjected to a one-minute cleansing in ultra-sound with distilled water and 1% detergent and carefully rinsed, blotted with absorbing paper and air dried for 15 seconds (Working Draft for Wear Test by Tooth Brushing, 1996). Samples were then subjected to a surface roughness reading to determine the initial roughness in a roughness meter (Prazis RUG 03 Digital Roughness Meter, ARO-Argentina). Each specimen was carefully fixed with wax on a metallic support, with the needle situated at the extremity of the equipment's arm positioned on the sample surface and programmed to trace a course of 4.8 mm, providing the first measure. Two additional measurements were accomplished by rotating the disk through at an angle of 90°, and the average (*Ra* initial-µm) was obtained from the three values. This measure was defined as an average of the pick-up arm's distances over and below the central line adopted for reference (Leitão & Hegdahl, 1981).

After the surface roughness reading, the samples were prepared for the wear test. For purposes of the study, each resin composite disk was individually fixed on the center of a *Plexglass* plate by adding a drop of acrylic resin monomer (JET, Clássico, 05458-001, Sao Paulo, SP, Brazil) on its surface and pressing the specimen against the plate for 10 seconds. The *Plexglass* plates were constructed with the dimensions required by the toothbrushing equipment: 50 mm large, 20 mm wide and 3 mm thick (Figure 1).

Plexglass patterns, designed with the same dimensions as the resin composite disks, were also prepared to act as a reference for the wear test. It has been advocated that, due

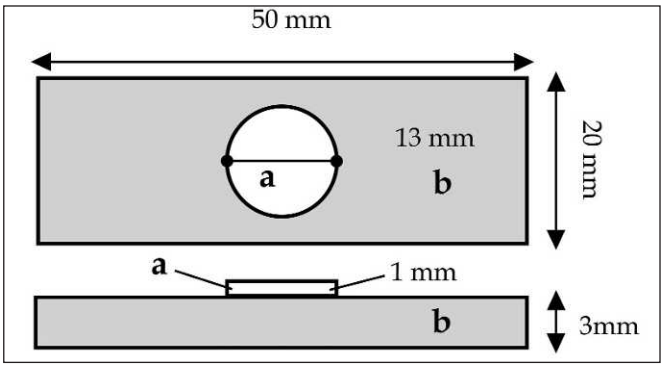


Figure 1.

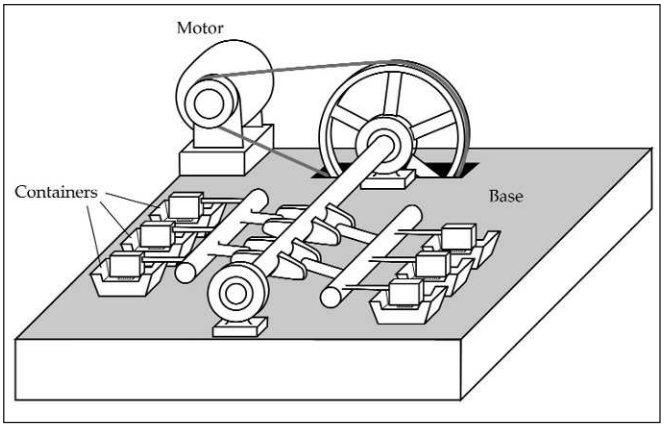


Figure 2.

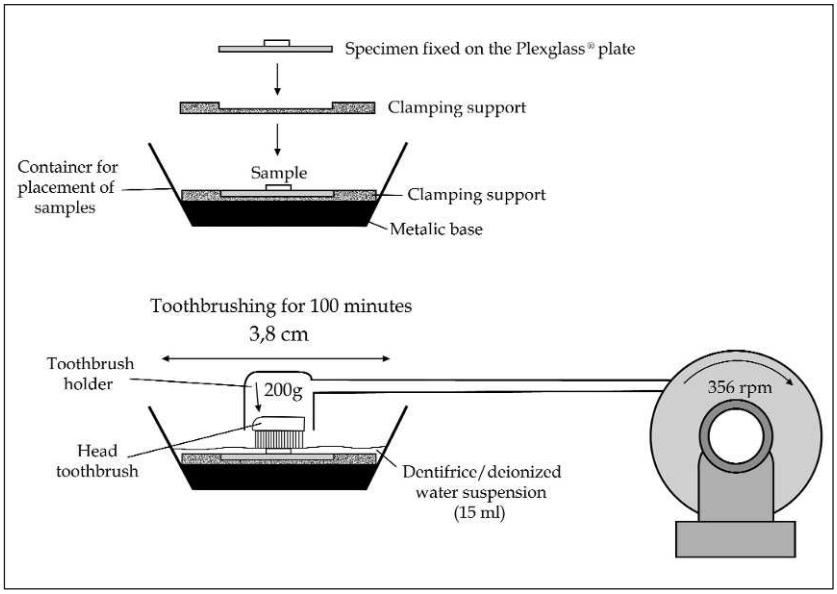


Figure 3.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

to its highly homogenous composition, *Plexglass* ensures increased reliability to the results as it enables wear to be evaluated whether or not the specimens were subjected to uniform wear during simulated toothbrushing (Ramos, Palma Dibb & Lara, 1999).

The simulated toothbrushing apparatus was the Pepsodent type (Figure 2) developed at the Precision Workshop at the University of Sao Paulo, Brazil. Earlier investigations have described this equipment as a valuable tool to provide precise, well-controlled toothbrushing (Vieira, 1960; Vieira & Phillips, 1960; Lara, 1988; Ramos & others, 1999; Chimello & others, 2001; Wang, 2001).

For each sample, a Kolynos toothbrush with soft bristles (Kolynos do Brasil Ltda, Osasco, SP 06020-170, Brazil) was used. As abrasive agent, a suspension of dentifrice Kolynos (Kolynos do Brasil Ltda, Osasco, SP, 06020-170) and deionized water was prepared in a 1:1 ratio at room temperature. Both slurry and toothbrush heads were replaced for every new sample.

The equipment allowed simultaneous toothbrushing of six samples: five resin composite disks and one Plexiglass pattern. The composite specimens were randomly assigned to groups of equal size and individually fixed with wax in a clamping support positioned inside the containers and fully covered with 15 ml of the dentifrice/water suspension (Figure 3). Before the wear test began, the equipment was calibrated to level the toothbrush bristles parallel to the sample surface, thereby, providing uniform toothbrushing (Harrington & others, 1982). Afterwards, toothbrushing was accomplished at a frequency of 356 rpm with horizontal movements of the toothbrush heads under a 200g weight and a traveled course of 3.8cm. Each group of specimens was subjected to a 100-minute simulated toothbrushing that performed a total of 35,600 brushings per test. This interval is reported to correspond to a two-year toothbrushing exposure (Phillips & Van Huysen, 1948) provided by an individual with good oral health care (Vieira, 1960).

Following the wear test, samples were carefully cleaned, as previously described, and submitted to a post-abrasion surface roughness reading. Each specimen was tested for the first reading: three measurements were provided and the average (*Ra* final-µm) was calculated. However, at this time, the equipment's arm was positioned in such a way that the tracing direction was perpendicular to the direction of the toothbrushing, with the aim of registering the undulations caused by the abrasion procedure. The roughness meter detected the surface irregularities transverse to matrix direction that correspond to peaks, valleys and waves produced by toothbrushing.

Furthermore, the dentifrice was assessed for its abrading degree at the laboratory of Pharmaceutical Sciences Department at the Pharmaceutical Sciences School, University of Sao Paulo. The analysis of the wear suffered by the material during toothbrushing consisted of weighing the specimens in an analytic electronic balance (Ohaus Corporation, Union, NJ, USA) before and after the toothbrushing test to calculate the mass difference in grams. The average mass loss was 14.52 mg, which corresponds to a low abrading level (Working Draft for Wear Test by Tooth Brushing, 1996).

The collected data were submitted to two-way ANOVA at 5% significance level.

RESULTS

The analysis of data revealed a statistically significant difference ($p<0.05$) between the measurements performed before and after the wear test, with increased roughness recorded after toothbrushing.

Means and standard deviations are shown in Table 2.

As the tested conditions were compared, no statistical difference was noticed among the groups.

DISCUSSION

The findings of the conducted research disclosed that toothbrushing provided a significant increase in surface roughness of the resin composite. These results are similar to those reported by Goldstein and Lerner (1991) and Chimello and others (2001), who noticed surface alteration on the specimens after toothbrushing. Roughness may be influenced by different features, such as the amplitude of the traveled course of the toothbrush, the weight applied on the toothbrush head and the dentifrice abrading degree and temperature (Heath & Wilson, 1976). Moreover, it has been demonstrated that the dentifrice rate in the suspension and toothbrushing speed may lead to surface alteration (Ehrnford, 1983). Taking this into consideration in the present study, the same brand of dentifrice and toothbrush were employed in all tests and the above factors were assessed in order to standardize the experiment and evaluate solely the influence of the surface sealants on surface texture.

A rough surface may decrease the wear resistance of the restorative material and significantly increase the number of sites on the restoration's surface that are prone to accumulating bacterial biofilm, which inher-

Table 2: *Initial and Post-Abrasion Surface Roughness Means (µm) and Standard Deviation for the Tested Conditions*

Group	Before	After
Control	0.032 (±0.005)[a]	0.054 (±0.005)[b]
Single Bond	0.031 (±0.004)[a]	0.047 (±0.007)[b]
Protect-it!	0.034 (±0.005)[a]	0.060 (±0.034)[b]

Same letters indicate statistical similarity

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

ently leads to a greater incidence of oral diseases apart from making the restorations more susceptible to staining and loss of brilliance (Lutz, Setcos & Phillips, 1983; Pratten & Johnson, 1988).

The surface sealing technique was proposed in an attempt to eliminate the microfissures and irregularities that resulted from removing several surface particles or microfractures on the enamel/adhesive interface caused by polymerization shrinkage or finishing and polishing. Surface sealants were specifically developed to provide an increase in wear resistance and optimize the marginal sealing of resin composites applied to posterior teeth (Dickinson & Leinfelder, 1993). Clinical trials have reported a significant increase in marginal integrity and wear resistance (Dickinson & others, 1990; Dickinson & Leinfelder, 1993). In addition, *in vitro* investigations have concluded that resealing restoration margins may optimize restorations' marginal sealing (Ramos & others, 2000).

Studies (Torstenson, Brännstrom & Mattson, 1985; Reid, Saunders & Chen, 1991) have suggested coating polymerized resin composite with different materials, such as adhesive system or fissure sealants. However, studies have also postulated (Tjan & Tan, 1991) that using any low-viscosity resin system as a surface sealant would provide an incomplete wetting and may not yield the required effectiveness in enhancing surface integrity by sealing structural microdefects due to improper fluidity (poor wettability) disparities in formulation, diluents (ethanol/water or acetone), viscosity modifiers (materials with or without filler particles) and curing mechanisms. Since the degree of penetration of surface sealants relies upon their viscosity and the ability to wet and spread over an etched surface, the efficiency of these materials depends on how deep they penetrate into microcracks before polymerization is completed.

Accordingly, some authors (Dickinson & others, 1990; Dickinson & Leinfelder, 1993) have suggested that a specially formulated surface-penetrating sealant might be desirable in order to optimize the depth of penetration into the debonded surface and ensure complete wetting and the consequent sealing of microdefects. The first surface–penetrating sealants (SPS) were developed and made commercially available in the early 1990s. The use of these agents provided optimal performance, mainly in enhancing marginal integrity (Ramos & others, 2000). However, their efficiency in improving the wear resistance of resin composites is still controversial (Ramos & others, 1999). Apart from this, the use of a specific surface sealant inevitably results in additional cost for both the clinician and patient, which needs to be considered. As a result, this investigation assessed whether using an adhesive system would lead to results similar or comparable to those reached by the surface-penetrating sealants with regard to surface roughness.

In this study, the surface sealants yielded a performance similar to the surface roughness of the resin composite assessed before and after surface sealing. Likewise, no difference was observed when the experimental groups were compared to the non-sealed control. These findings corroborate those by Ramos and others (1999) that reported tested surface sealants did not interfere with the degree of wear and surface roughness of resin composite. Such results could possibly be ascribed to the extremely thin layer of surface sealant that solely fills the most superficial defects of resin composite without providing an adequate overall surface covering (Bertrand & others, 2000). Besides, polymerization failure of the sealant may occur in the presence of oxygen (Rueggeberg & Margeson, 1990). As a result, it can be easily removed by the action of saliva, food, brushing, antagonistic surfaces or other agents (Bertrand & others, 2000).

The constant improvement of dental materials may result in the development of resin systems with appropriate flow rate and properties that perform the role of the surface-penetrating sealants with the same efficiency at no additional cost to clinician or patient. Nevertheless, further research is necessary to confirm this assumption. Moreover, studies must also be focused on the long-term evaluation of sealed posterior composite restorations in order to provide the clinician with guidelines for a proper indication and correct handling of such restorations.

CONCLUSIONS

The results of this study may conclude that:

1. Using surface penetrating sealant and one bottle adhesive system did not provide optimal superficial integrity;
2. Using dentifrice and a toothbrush resulted in considerable alterations on the surface smoothness of the resin composite.

Acknowledgements

The authors thank pharmacist José Orestes Del Ciampo for his valuable contribution, accomplishing the toothbrushing tests for this study.

(Received 28 May 2002)

References

Araújo MAM & Araújo MAJ (1983) [Estudo rugosimétrico da superfície de uma resina microparticulada com carga mista, tratada com diferentes agentes de acabamento] *Odontologo Moderno* **10(7)** 24-30.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Berastegui E, Canalda C, Brau E & Miguel C (1992) Surface roughness of finished composite resins *Journal of Prosthetic Dentistry* **68(5)** 742-750.

Bertrand MF, Leforestier E, Muller M, Lupi-Pégurier L & Bolla M (2000) Effect of surface penetrating sealant on surface texture and microhardness of composite resins *Journal Biomedical Materials Research* **53(6)** 658-663.

Boghosian AA, Randolph RG & Jekkals VJ (1987) Rotary instrument finishing of microfilled and small particle hybrid composite resins *Journal of the American Dental Association* **115(2)** 299-301.

Bowen RL (1956) Use of epoxy resins in restorative materials *Journal of Dental Research* **35(3)** 360-369.

Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surface *Journal of Dental Research* **34(6)** 849-853.

Chimello DT, Palma Dibb RG, Corona SAM & Lara EHG (2001) Assessing wear and surface roughness of different composite resins after toothbrushing *Materials Research* **4(4)** 285-289.

Dickinson GL, Leinfelder KF, Mazer RB & Russel CM (1990) Effect of surface penetrating sealant on wear rate of posterior composite resins *Journal of the American Dental Association* **121(2)** 251-255.

Dickinson GL & Leinfelder KF (1993) Assessing the long-term effect of a surface penetrating sealant *Journal of the American Dental Association* **124(7)** 68-72.

Ehrnford L (1983) Surface microstructure of composite resins after toothbrush-dentifrice abrasion *Acta Odontologica Scandinavica* **41(4)** 241-245.

Goldstein GR & Lerner T (1991) The effect of tooth brushing on a hybrid composite resin *Journal of Prosthetic Dentistry* **66(4)** 498-500.

Harrington E, Jones PA, Fisher SE & Wilson HJ (1982) Toothbrush-dentifrice abrasion: A suggested standard method *British Dental Journal* **153(4)** 135-138.

Heath JR & Wilson HJ (1976) Abrasion of restorative materials by toothpaste *Journal of Oral Rehabilitation* **3(2)** 121-138.

Kanter J, Koski RE & Martin D (1982) The relationship of weight loss to surface roughness of composite resins from simulated toothbrushing *Journal of Prosthetic Dentistry* **47(5)** 505-513.

Knibbs PJ & Pearson GJ (1984) Finishing glass ionomer cement. A scanning electron microscope examination of the surface on an anhydrous glass-ionomer cement *in vivo* *Brazilian Dental Journal* **157(11)** 398-400.

Lara EHG (1988) [*Estudo do comportamento de algumas preparações básicas de dentifrícios na forma de pasta. I-Tecnologia. II-Propriedades reológicas. III-Índice de abrasividade*] Tese (Livre Docência), Faculdade de Ciências Farmacêuticas de Ribeirão Preto, Universidade de Ribeirão Preto.

Leinfelder KF, Sluder TB, Sockwell CL, Strickland WD & Wall JT (1975) Clinical evaluation of composite resins as anterior and posterior restorative materials *Journal of Prosthetic Dentistry* **33(4)** 407-416.

Leinfelder KF (1991) Using composite resin as a posterior restorative material *Journal of the American Dental Association* **122(4)** 65-70.

Leitão J & Hegdahl T (1981) On the measuring of roughness *Acta Odontológica Scandinavica* **39(6)** 379-84.

Lutz F, Setcos JC & Phillips RW (1983) New finishing instruments for composite resins *Journal of the American Dental Association* **107(4)** 575-580.

Phillips RW & Van Huysen G (1948) Dentifrices and tooth surface *American Perfumer and Essential Oil Review* **50(1)** 33-37.

Phillips RW, Avery DR, Mehra R, Swartz ML & Mc Cune RJ (1971) One-year observations on a composite resin for Class II restorations *Journal of Prosthetic Dentistry* **26(1)** 68-77.

Phillips RW, Avery DR, Mehra R, Swartz ML & Mc Cune RJ (1973) Observations on a composite resin for Class II restorations: Three-year report *Journal of Prosthetic Dentistry* **30(6)** 891-897.

Pratten DH & Johnson GH (1988) An evaluation of finishing instruments for an anterior and posterior composite *Journal of Prosthetic Dentistry* **60(2)** 154-158.

Ramos RP, Palma Dibb RG & Lara EHG (1999) [Influência de diferentes agentes de cobertura na integridade superficial de uma resina composta] *Revista Odontológica UNESP* **28(2)** 451-464.

Ramos RP, Chimello DT, Chinelatti MA, Palma Dibb RGP & Mondelli J (2000) Effect of three surface sealants on marginal sealing of Class V composite resin restorations *Operative Dentistry* **25(5)** 448-453.

Reid JS, Saunders WP & Chen YY (1991) The effect of bonding agent and fissure sealant on microleakage of composite resin restorations *Quintessence International* **22(4)** 295-298.

Rueggeberg FA & Margeson DH (1990) The effect of oxygen inhibition on an unfilled/filled composite system *Journal of Dental Research* **69(10)** 1652-1658.

Tjan AHL & Tan DE (1991) Microleakage at gingival margins of Class V composite resin restorations rebonded with various low-viscosity resin systems *Quintessence International* **22(7)** 565-573.

Torstenson B, Brännstrom M & Mattson B (1985) A new method of sealing composite resin contraction gaps in lined cavities *Journal of Dental Research* **64(3)** 450-453.

Vieira DF (1960) Studies on hardness and abrasion resistance of acrylic resins (Thesis-Master degree), Indiana University School of Dentistry p 102.

Vieira DF & Phillips RW (1960) Influence of certain variables on the abrasion of acrylic resin veneering materials *Journal Prosthetic Dentistry* **12(4)** 720-731.

Wang L (2001) *In vitro* evaluation of abrasion resistance of "condensable" composite resins after toothbrushing. Wear and superficial roughness analysis (Thesis) Bauru School of Dentistry, University of São Paulo p 133.

Working draft for wear test by tooth brushing. Part 1. International Organization for Standard Technical Specification 14569-1 Switzerland, ISO, 1996.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3**, 287-296

The Effect on Shear Bond Strength of Rewetting Dry Dentin with Two Desensitizers

MQ Al Qahtani • JA Platt
BK Moore • MA Cochran

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Clinical Relevance

HurriSeal desensitizer has been effective as a dentin rewetting agent and can render the bonding procedure less technique sensitive; however, Protect desensitizer reduced the shear bond strength of dentin bonding agents to the dentin surface.

SUMMARY

The difficulty related to achieving a balance between wet and dry dentin makes the dentin bonding technique extremely sensitive. This study evaluated the effect of rewetting dried dentin with two commercial desensitizing agents (Protect and HurriSeal) on the dentin shear bond strength of three total-etch dentin bonding agents (Syntac Single-Component, OptiBond Solo Plus and Prime & Bond NT) and compared both to applying these same bonding agents to moist dentin and dry dentin. Each bonding agent was paired with an appropriate resin composite from the same manufacturer (Table 1). Recently extracted, formalin-treated caries-free human molars were used. The occlusal surface of each tooth was ground to create a flat dentin surface. Then, each tooth was mounted in acrylic. Twelve groups (n=15) were prepared: 1) Syntac Single-Component with Heliomolar resin composite (SSC/H) to moist dentin; 2) SSC/H to dry dentin; 3) SSC/H to dried dentin rewet with Protect; 4) SSC/H to dried dentin rewet with HurriSeal; 5) OptiBond Solo Plus with Point 4 resin composite (OBS+/P4) to moist dentin; 6) OBS+/P4 to dry dentin; 7) OBS+/P4 to dried dentin rewet with Protect; 8) OBS+/P4 to dried dentin rewet with HurriSeal; 9) Prime & Bond NT with TPH Spectrum resin composite (PBNT/TPH) to moist dentin; 10) PBNT/TPH to dry dentin; 11) PBNT/THP to dried dentin rewet with Protect and 12) PBNT/TPH to dried dentin rewet with HurriSeal. Groups 1, 5 and 9 were placed according to manufacturers' instructions (moist dentin) as control groups. All the other groups received a 15-second air blast after etching and prior to applying the one bottle adhesive or desensitizer and one bottle adhesive. Resin composite cylinders [4 mm in diameter and 2 mm in height] were then placed. The specimens were stored in distilled water at 37°C for 24 hours prior to thermocycling 2,500 times (at 8°C and 48°C). Shear bond strengths (SBSs) were measured one week after

*Mohammed Q Al Qahtani, BDS, MSD, lecturer, Department of Restorative Dental Science, King Saud University, College of Dentistry, Saudi Arabia

Jeffrey A Platt, DDS, MSD, assistant professor, director, Division of Dental Materials, Indiana University School of Dentistry, Indianapolis, IN

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

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

*Reprint request: PO Box 60169, Riyadh 11545, Saudi Arabia; e-mail: mqahtani@mail.com

fabrication by using a circular knife-edge and crosshead speeds of 0.5 mm/minute. Failure modes of debonded specimens were determined under a stereomicroscope (30x). Failed specimens with the low and high shear bond strengths in each group were evaluated under a low vacuum Scanning Electron Microscope (SEM-LV). One-way ANOVA and Tukey's tests were used to compare the different conditions for each bonding system. In the Syntac Single-Component bonding agent groups, there was no significant difference in shear bond strength between the control (15.73 MPa), dry (18.11 MPa) and HurriSeal (16.18 MPa) specimens. Protect specimens showed significantly lower shear bond strength (6.39 MPa). In the OptiBond Solo Plus bonding agent groups, there was no significant difference between the control (20.79 MPa) and the HurriSeal (21.29 MPa) groups, and both groups had significantly greater bonds than the dry (14.13MPa) and Protect (9.57 MPa) groups. In the Prime & Bond NT bonding agent groups, the shear bond strength of the HurriSeal group (20.73 MPa) was significantly higher than the other groups: control (8.05 MPa), dry (5.73 MPa) and Protect (5.45 MPa).

INTRODUCTION

Between 1982 and 1985, Nakabayashi described the micromechanical bonding mechanism of dentin bonding agents. The most widely accepted theory today is that acid conditioners partially demineralize the dentin surface, leaving an exposed collagen network that can be penetrated by resin, creating an intricate micromechanical attachment called the "hybrid layer" (Nakabayashi, Kojima & Masuhara, 1982; Nakabayashi, 1985). The demineralized dentin matrix has been described as shrinking and collapsing easily when it is dried with air after being rinsed with water (Perdigão & Lopes, 1999; Pashley, Horner & Brewer, 1992; Titley & others, 1994; Kato & Nakabayashi, 1998). Any collapse of the collagen matrix as a result of over-drying may prevent monomers from penetrating into deeper areas and increase the risk of adhesive failures and nanoleakage (Nakabayashi, Ashizawa & Nakamura, 1992; Pioch & others, 1999). For that reason using a dry-bonding technique is perhaps not the best choice for resin composite restorations.

Introducing the moist or wet bonding technique is one way to preserve the micromorphological integrity of the collagen (Tay & others, 1995). Kanka (1992b) found that moist dentin resulted in an increase in shear bond strength compared to dry dentin. He also found that moisture plays a significant role in facilitating resin penetration of acid-conditioned dentin of appropriately formulated and constituted resins. The benefit of the "wet-bonding technique" is derived from the ability of water to maintain the collagen framework and intertubular porosity patent for monomer infiltration (Tay, Gwinnett & Wei, 1996). Most studies have reported that with a moist surface, optimum infiltration of adhesive resin into the demineralized layer occurs and higher bond strength values are achieved (Kanka, 1996; 1997; Swift & Triolo, 1992; Perdigão, Swift & Cloe, 1993; Han & others, 2000). The risk with moist dentin is an overwet condition that results in excessive water, which appears to cause phase separation of the hydrophobic and hydrophilic monomer components, resulting in blister and globule formation spaces at the resin-dentin interface (Tay & others, 1996).

Therefore, it appears that a difficulty exists in achieving a balance between overdrying or overwetting the conditioned dentin, both which may have undesirable effects clinically on bonding performance. Perdigão and others (1999) have shown that re-wetting the etched dentin with an aqueous HEMA solution re-established the level of bond strength obtained to moist dentin and served as an effective means to reopen the interfibrillar spaces for penetration of adhesive primer and/or priming-bonding resin. In another study by Perdigão and others (1998), the application of Aqua-Prep to dried dentin surfaces was found to restore or increase the bond strengths of acetone-based water-free dentin adhesives (One-Step, Prime & Bond 2.1) to the same level as bonding to moist substrates. Pilo and others (2001) concluded that pretreatment of etched dentin with either a disinfectant (Consepsis 2% chlorhexidine digluconate, Tubulicid 2% EDTA and 1% bezyalkonium chloride) or rewetting (Aqua prep 35% HEMA) agents may have a positive effect on the shear bond strength of resin to dentin, but an aqueous HEMA solution was the most effective material and could render the bonding procedure less technique sensitive.

Table 1: *Materials Used in this Study*

Materials	Manufacturer	Batch #
Syntac Single-Component	Ivoclar/Vivadent, Amherst, NY, USA	D56201
OptiBond Solo Plus	Kerr Corp, Orange, CA, USA	009565
Prime & Bond NT	Dentsply/Caulk, Milford, DE, USA	010609
Heliomolar-A2	Ivoclar/Vivadent, Amherst, NY, USA	D59377
Point 4-A2	Kerr Corp, Orange, CA, USA	29877
TPH spectrum-A2	Dentsply/Caulk, Milford, DE, USA	0107033
Protect Desensitizer	Butler Company, Chicago, IL, USA	0A021
HurriSeal Desensitizer	Beutlich, Waukegan, IL, USA	5693315
Elipar Highlight Light Cure Unit	ESPE, Seefeld, Germany	3804934
Model 100 Curing Radiometer	Demetron Research Corp, Danbury, CT, USA	106646

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Platt, Duke and Rhodes (2001) evaluated the effect of desensitizers (Vivadent and Gluma) used as rewetting agents on dentin shear bond strength. Vivadent is a solution of 5% glutaraldehyde and 35% polyethylene glycol dimethacrylate, and Gluma is a solution of glutaraldehyde and HEMA. The results of that study indicated that Vivadent and Gluma Desensitizers did not interfere with the performance of the tested dentin bonding systems and may serve as effective dentin rewetting agents.

This *in vitro* study evaluated the effect of rewetting dried dentin with two desensitizers (Protect and HurriSeal) on the dentin shear bond strength of three total-etch dentin bonding agents (Syntac Single-Component, OptiBond Solo Plu and Prime & Bond NT) and compared them to applying these bonding agents to moist dentin (control) and dry dentin.

The null hypothesis for this study was that there would be no significant difference in dentin shear bond strengths under any of three conditions (moist dentin, dried dentin and dried dentin rewetted with different desensitizers).

METHODS AND MATERIALS

Table 1 lists the materials used in this study. One hundred and eighty recently extracted, caries-free human molars were used. After extraction, the teeth were placed in jars containing 10% buffered formalin for two-to-four weeks. They were then debrided with periodontal curettes and cleaned. After cleaning, the teeth were stored in distilled water at 3-4°C until used.

Specimen Preparation

The occlusal surfaces of the teeth were ground at slow speed with a 180-grit silicon carbide paper mounted on a water-cooled wheel to create a flat dentin surface. They were examined under a stereomicroscope to make sure that no enamel was left on the bonding area. Then, each tooth was mounted in a cylindrical Plexiglass mold and a chemically cured acrylic resin. The dentin was finished and polished in order on wet 240, 320, 400 and 600-grit silicon carbide papers. The specimens were placed in distilled water until ready for use.

Restorative Procedures

These specimens were divided into 12 groups. Each group contained 15 teeth. The groups were:

Group 1. Syntac Single—Component bonding agent with Heliomolar resin composite (SSC/H) placed on moist dentin as a control group.

Group 2. SSC/H placed on dry dentin.

Group 3. SSC/H placed on dried dentin rewet with Protect.

Group 4. SSC/H placed on dried dentin rewet with HurriSeal.

Group 5. OptiBond Solo Plus bonding agent with Point 4 resin composite. (OBS+/P4) placed on moist dentin as a control group.

Group 6. OBS+/P4 placed on dry dentin.

Group 7. OBS+/P4 placed on dried dentin rewet with Protect.

Group 8. OBS+/P4 placed on dried dentin rewet with HurriSeal.

Group 9. Prime & Bond NT bonding agent with TPH spectrum resin composite (PBNT/TPH) placed on moist dentin as a control group.

Group 10. PBNT/TPH placed on dry dentin.

Group 11. PBNT/TPH placed on dried dentin rewet with Protect.

Group 12. PBNT/TPH placed on dried dentin rewet with HurriSeal.

Table 2: *Dentin Bonding Agents and Bonding Procedures According to Manufacturer's Instructions*

Bonding System	Materials	Solvent Used	Directions
Syntac Single-Component	37% Phosphoric acid, (one bottle primer and adhesive)	Water (46% deionized water, 43.6% HEMA)	Etch for 15 seconds, rinse, lightly dry to a moist surface, apply and scrub adhesive on surface for 10 seconds, leave it undisturbed for 20 seconds, dry it lightly, when the material has visibly thickened, a strong blast of air direct onto surface to disperse the remaining adhesive, light cure for 20 seconds, apply a second coat of adhesive, dry immediately, light cure for 20 seconds, dentin after treatment with two coats of adhesive should appear shiny, if it appears dull apply another coat and do the same for a second coat, place resin composite and light cure for 40 seconds.
OptiBond Solo Plus	37% Phosphoric acid, (one bottle primer and adhesive)	Ethanol	Etch for 15 seconds, rinse, lightly dry to a moist surface, apply adhesive on surface for 15 seconds, air thin for three seconds, light cure for 20 seconds, place resin composite and light cure for 40 seconds.
Prime & Bond NT	34% Phosphoric acid, (One bottle primer and adhesive)	Acetone	Etch for 15 seconds, rinse, lightly dry to a moist surface, apply adhesive surface and leave it for 20 seconds, air dry for 10 seconds, light cure for 10 seconds, place resin composite and light cure for 40 seconds.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Groups 1, 5 and 9 were placed according to the manufacturer's instructions (moist dentin) by blotting with gauze to serve as the control groups (Table 2). Groups 2, 6 and 10 received a 15-second air blast (using oil-free compressed air from an air-syringe, keeping the syringe 2 cm from the surface) after etching, rinsing and before applying one bottle adhesive. Group 3, 7 and 11 received a 15-second air blast after etching and rinsing; the dry dentin was remoistened by using the desensitizer (Protect) prior to applying the one bottle adhesive. Groups 4, 8 and 12 received a 15-second air blast after etching and rinsing; the dry dentin was remoistened by using the desensitizer (HurriSeal) prior to applying the one bottle adhesive. After applying the bonding agent on each specimen, light activated polymerization was accomplished with an Elipar Highlight at 400 mW/cm^2-verified with a Model 100 Curing Radiometer. Resin composite cylinders 2 mm high and 4 mm in diameter were placed over the adhesives. The resin composites were Heliomolar for groups 1 to 4, Point 4 for groups 5 to 8 and TPH spectrum for groups 9 to 12. Then, the resin was polymerized for 40 seconds using the light-curing unit Elipar Highlight at 400 mW/cm^2-verified with a Model 100 Curing Radiometer.

Testing Procedure

The specimens were stored in distilled water at 37°C for 24 hours; the specimens were thermocycled between 8°C and 48°C water baths for 2,500 cycles with a 30-second dwell time and a 10-second transfer time. The specimens were then returned to storage until the one-week storage time was completed. Each specimen was loaded into a 1123 MTS Renew Testing Machine using Test Works 4 software for testing, with the long axis of the specimen being perpendicular to the direction of the applied force. The circular knife-edge was located at the interface between the composite post and the dentin surface, and the specimen was marked with pencil to identify orientation of the shear knife. Bond strength was measured in the shear mode at a crosshead speed of 0.5 mm/minute until fracture occurred.

Failure Mode Determination

After shear bond strength testing, the failure sites of the debonded specimens were examined under a stereomicroscope (30x) to determine failure mode. The failure mode of each specimen was recorded according to the type that was most representative of the failure in each specimen as follows:

A_T=Adhesive failure between bonding agent and tooth (dentin).

A_R=Adhesive failure between bonding agent and resin composite.

C_R=Cohesive failure within resin composite.

C_T=Cohesive failure within tooth (dentin).

Scanning Electron Microscope

The specimens were removed from water storage and examined without applying a metallic coating under a JEOL JSM-5310LV Scanning Microscope operated in a low vacuum mode (SEM-LV). Failed specimens with the lowest and highest shear bond strength in each group were examined throughout the debonded area with SEM-LV (X1,000). Cross-sectional evaluations of some of these failed specimens were done under SEM-LV (X1,000) to evaluate the difference among the three conditions (moist dentin, dry dentin, dry dentin rewetted with two different desensitizers).

Statistical Analysis

A one-way analysis of variance (ANOVA) was used to compare the four dentin treatments for differences in shear bond strength for each of three bonding agents. Dentin treatments were compared using Tukey's multiple comparison procedure to control the overall significance level of the tests. Kaplan-Meier estimates of the probability of not failing were also calculated in order to graph the probability of failure by strength for each type of treatment. Cox's regression was also used to determine the hazard ratios for each treatment compared to the control (Moist).

RESULTS

A one-way ANOVA test showed that dentin treatments had a significant effect on bond strength ($p<0.0001$) when using Syntac Single-Component, OptiBond Solo Plus or Prime & Bond NT bonding agents. Figure 1 presents the mean shear bond strengths of all groups of different bonding agents. Different statistical analysis tests are summarized in Table 3. The specimen numbers of different failure modes for each group are presented in Table 4.

Syntac Single-Component Bonding Agent

The treatments are listed from the highest mean shear bond strength to the lowest: dry dentin (18.11 ± 3.50

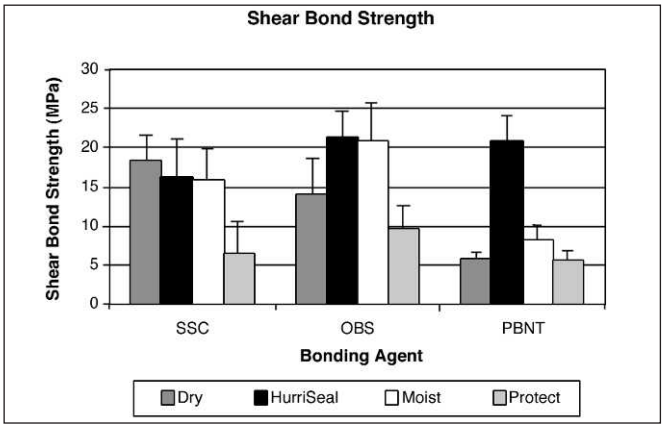


Figure 1.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 3: *Statistical Analysis Summary*

Material	Mean Shear Bond Strength ± SD In Mpa		Tukey's Test			Cox's Regression Test	
	Group	Value	Group	Group	*p*-value	Hazard Ratio of Failure	
Syntac Single-Component + Heliomolar	Moist **	15.73 ± 4.13	Dry	HurriSeal	0.5915	Moist	----
	Dry **	18.11 ± 3.50	Dry Dry	Moist Protect	0.4117 <0.0001	Dry	0.595
	Rewet with Protect	6.39 ± 4.17	HurriSeal HurriSeal	Moist Protect	0.9909 <0.0001	Protect	9.359
	Rewet with HurriSeal**	16.18 ± 4.89	Moist	Protect	<0.0001	HurriSeal	0.749
OptiBond Solo Plus + Point 4	Moist**	20.79 ± 4.91	Dry	HurriSeal	<0.0001	Moist	----
	Dry	14.13 ± 4.56	Dry Dry	Moist Protect	0.0002 0.0163	Dry	5.002
	Rewet with Protect	9.57 ± 3.08	HurriSeal HurriSeal	Moist Protect	0.9870 <0.0001	Protect	31.637
	Rewet with HurriSeal**	21.29 ± 3.33	Moist	Protect	<0.0001	Hurriseal	1.019
Prime & Bond NT + TPH Spectrum	Moist	8.05 ± 2.08	Dry	HurriSeal	<0.0001	Moist	----
	Dry**	5.73 ± 1.01	Dry Dry	Moist Protect	0.0287 0.9844	Dry	4.921
	Rewet with Protect**	5.45 ± 1.49	HurriSeal	Moist	<0.0001	Protect	4.788
	Rewet with HurriSeal	20.73 ± 3.45	HurriSeal Moist	Protect Protect	<0.0001 0.0110	HurriSeal	Less than 0.0005

*N= 15 for each group, Groups with ** had no significant difference at* p*<0.05.*

Table 4: *Specimen Numbers for the Different Failure Modes*

Material	Mean SBS ± SD In Mpa		Failure Modes			
	Groups	Value	A_R	A_R	C_R	C_R
Syntac Single-Component + Heliomolar	Moist **	15.73 ± 4.13	1 (6.66%)	9 (60.00%)	5 (33.33%)	-
	Dry **	18.11 ± 3.50	- -	7 (46.66%)	8 (53.33%)	-
	Rewet with Protect	6.39 ± 4.17	13 (86.66%)	2 (13.33%)	-	-
	Rewet with HurriSeal**	16.18 ± 4.89	1 (6.66%)	6 (40.00%)	4 (26.66%)	4 (26.66%)
OptiBond Solo Plus + Point 4 5.002	Moist**	20.79 ± 4.91	1 (6.66%)	4 (26.66%)	3 (20.00%)	7 (46.66%)
	Dry	14.13 ± 4.56	7 (46.66%)	7 (26.66%)	-	4 (26.66%)
	Rewet with Protect	9.57 ± 3.08	13 (86.66%)	2 (13.33%)	-	-
1.019	Rewet with HurriSeal**	21.29 ± 3.33	-	-	1 (6.66%)	14 (93.33%)
Prime & Bond NT + TPH Spectrum	Moist	8.05 ± 2.08	15 (100%)	-	-	-
	Dry**	5.73 ± 1.01	15 (100%)	- -	- -	- -
	Rewet with Protect**	5.45 ± 1.49	15 (100%)	-	-	-
	Rewet with HurriSeal	20.73 ± 3.45	1 (6.66%)	-	3 (20.00%)	11 (73.33%)

MPa), dry dentin rewetted with HurriSeal (16.18 ± 4.89 MPa), moist dentin (15.73 ± 4.13 MPa) and dry dentin rewetted with Protect (6.39 ± 4.17 MPa) (Table 3). The first three treatments were not significantly different at $p<0.05$; however, rewetting with Protect was significantly lower than all other methods of treatment ($p<0.0001$). From Figure 2, the probability of failure at low shear bond strengths for the treatment with Protect was high compared to the other treatments. From the results of Cox's regression, the odds of failure with the Protect treatment were 9.359 times the odds of failure with the moist treatment.

OptiBond Solo Plus Bonding Agent

The treatments are listed from the highest mean

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

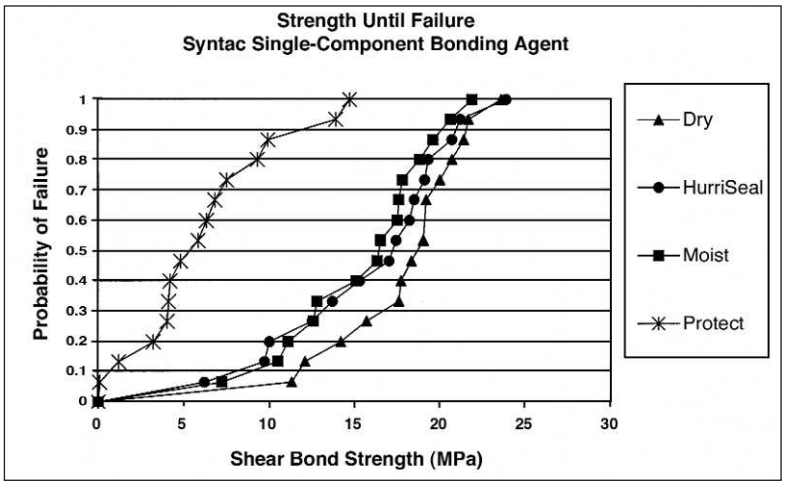


Figure 2.

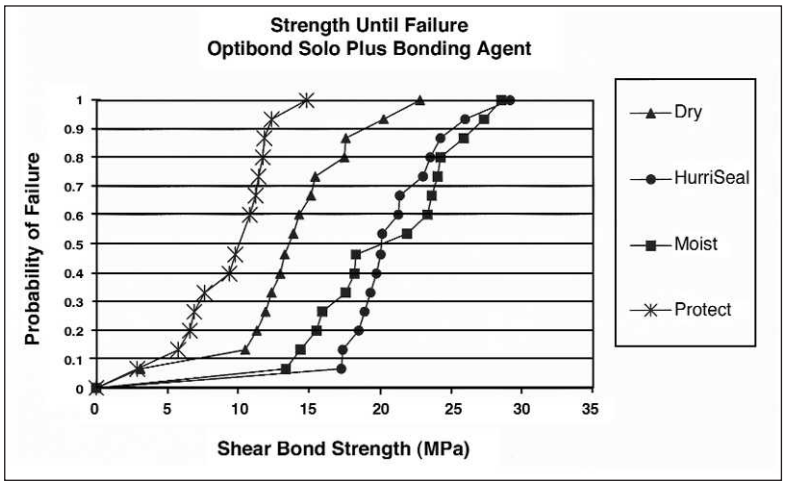


Figure 3.

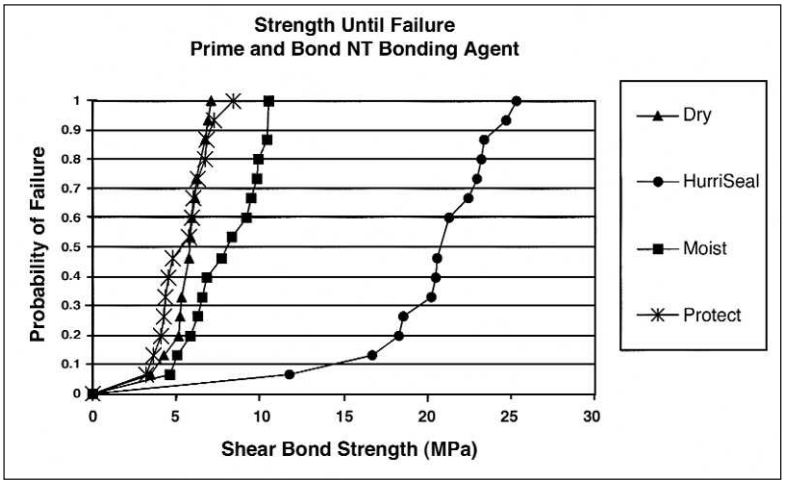


Figure 4.

shear bond strength to the lowest: dry dentin rewetted with HurriSeal (21.29 ± 3.33 MPa), moist dentin (20.79 ± 4.91 MPa), dry dentin (14.13 ± 4.56 MPa) and dry dentin rewetted with Protect (9.57 ± 3.08 MPa) (Table 3). The first two treatments were not significantly different at $p<0.05$. Treatment with Hurriseal was significantly higher than treatment with dry dentin ($p<0.0001$) and Protect ($p<0.0001$). Moist treatment was significantly higher than dry treatment ($p=0.0002$) and Protect ($p<0.0001$). Dry dentin treatment was also significantly higher than treatment with Protect ($p<0.0163$). From Figure 3, the probability of failure at low shear bond strengths for the Protect treatment and the dry treatment were high compared to the other two treatments. From the results of Cox's regression, the odds of failure with the Protect treatment were 31.637 times that of the moist treatment. The odds of failure with the dry treatment were 5.002 times the odds of failure with the moist treatment. The odds of failure of the Hurriseal treatment were the same as the moist treatment.

Prime & Bond NT Bonding Agent

The treatments are listed from the highest mean shear bond strength to the lowest: dry dentin rewetted with Hurriseal (20.73 ± 3.45 MPa), moist dentin (8.05 ± 2.08 MPa), dry dentin (5.73 ± 1.01 MPa) and dry dentin rewetted with Protect (5.45 ± 1.45 MPa) (Table 3). The last two treatments were not significantly different at $p<0.05$. Treatment with Hurriseal was significantly higher than all the other treatments ($p<0.0001$). Moist treatment was significantly higher than dry treatment ($p=0.0287$) and Protect treatment ($p=0.0110$). From Figure 4, the probability of failure at low shear bond strengths for the moist dentin, dry dentin and dry dentin rewetted with Protect treatments were higher than the dry dentin rewetted with Hurriseal treatment. From the results of Cox's regression, the odds of failure with the Protect treatment were 4.788 times the odds of failure with the moist treatment. The odds of failure with the dry treatment were 4.921 times the odds of failure with the moist treatment. The odds of failure of the Hurriseal treatment were less than 0.0005 times that of the moist treatment.

Scanning Electron Microscope Observations

The SEM-LV images (X1,000) in Figure 5 A, B, C and D show different types of failure modes in the debonded area of the failed specimens, and these were: A_T=Adhesive failure between bonding agent and tooth (dentin), A_R=Adhesive failure

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

between bonding agent and resin composite, C_R=Cohesive failure within resin composite and C_T=Cohesive failure within tooth (dentin).

The SEM-LV images (1,000x) in cross-section show that bonding to moist dentin produced a uniform hybrid layer (Figure 6A), bonding to dry dentin resulted in a non-uniform and thin hybrid layer (Figure 6B), bonding to dry dentin rewetted with Protect desensitizer produced no hybrid layer at all (Figure 6C) and bonding to dry dentin rewetted with Hurriseal desensitizer produced a uniform hybrid layer (Figure 6D).

DISCUSSION

For Syntac Single-Component there was no significant difference in mean shear bond strengths between moist dentin (15.73±4.13 MPa), dry dentin (18.11±3.50 MPa) and rewetting with HurriSeal (16.18±4.89 MPa). The higher bond strength to dried dentin may be due to the contents of this bonding agent (43.6% HEMA and 46% deionized water, Table 2), which could assist in rehydrating or rewetting air dried dentin and, thus, collapsed collagen, transforming it into a loosely arranged network that simultaneously allows the hydrophilic monomer to interdiffuse (VanMeerbeek & others, 1998). However, the mean shear bond strength to moist dentin was less than to dried dentin, which may be due to it being overwet. This could cause phase separation of the hydrophobic and hydrophilic monomer components and result in the blister and globule formation space at the resin-dentin interface (Tay & others, 1996).

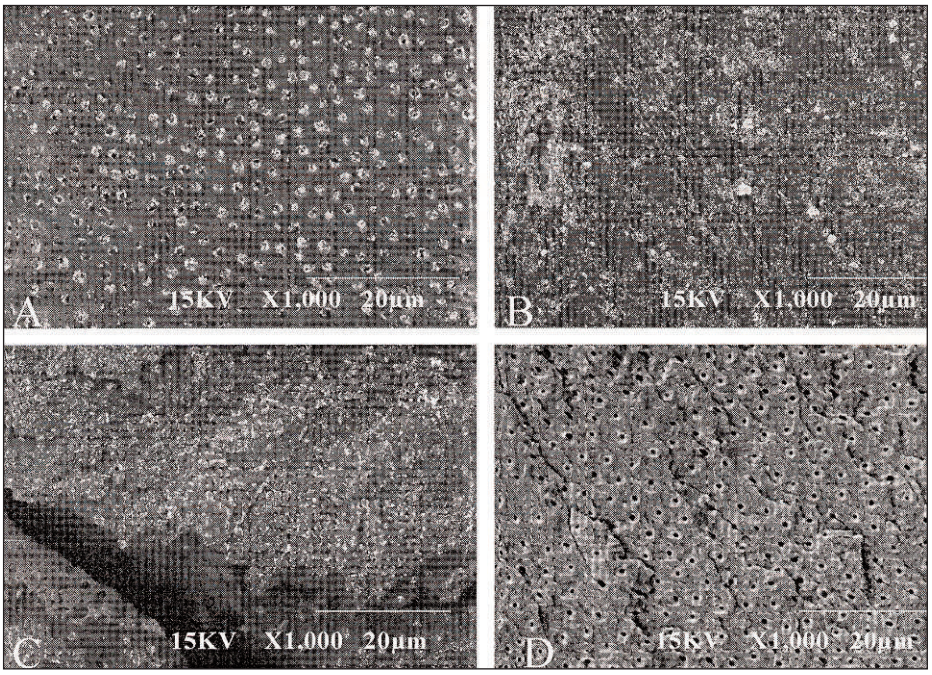


Figure 5.

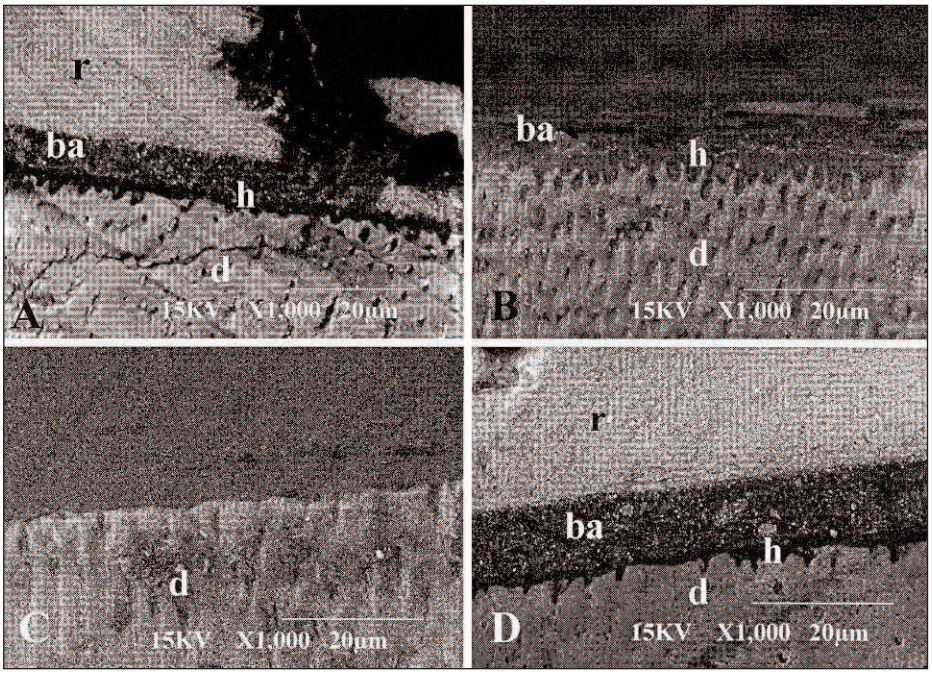


Figure 6: *ba= bonding agent, d = dentin, h = hybrid layer, r = resin composite.*

The presence of organic solvents, such as ethanol or acetone, in the composition of bonding agents can dislocate water from the moist collagen network, thus, promoting the infiltration of the resin monomers through the nano-spaces of the dense collagen. This is referred to as the wet-bonding technique that enhances bond strength (Jacobsen & Söderholm, 1995; Perdigão & others, 1999; Kanka, 1992a; Jacobsen & Söderholm, 1998).

For OptiBond Solo Plus there was a significant difference in mean shear bond strengths between moist dentin (20.79±4.91 MPa) and dry dentin (14.13±4.56 MPa). This may result from better wetting and infiltration of the monomer to moist dentin. Dried dentin can have the collagen collapsed, reducing the wetting and infiltration of the monomer, then, the shear bond

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

strength. These results agree with a study by (Perdigão & others (1999) that showed a significant reduction in bond strength of OptiBond Solo Plus to dried dentin and high bond strength to moist dentin.

For Prime & Bond NT there was a significant difference in mean shear bond strength to moist dentin (8.05±2.08 MPa) and dry dentin (5.73±1.01 MPa); however, both groups had low bond strengths. Other studies have reported that the mean shear bond strengths of Prime & Bond NT bonding agent to moist dentin were (8.1±3.8 and 5.9±2.6 MPa) (Rosa & Perdigão, 2000; Blomlof & others, 2001). Other studies have shown that air drying of etched dentin decreases the bond strength of acetone-based dentin bonding agents from one-third to one-half of the bond strength obtained to moist dentin (Perdigão & others, 1999; Halvorson, 1997; Barkmeier, Hammesfahr & Latta, 1999). Acetone in bonding agents is an excellent solvent and carrier for adhesive monomers. It requires water-filled channels or spaces located between the collagen fibrils. However, there is a problem regarding how much water remains after rinsing the etched dentin, then drying. Another factor that can affect the bond strength is air drying of the bonding agent. Caution should be taken to avoid excessive air drying, which may result in reducing the bond strength due to incorporating oxygen into the bonding agent that impairs monomer conversion (Glasspoole & Erickson, 1992).

Rewetting the etched dried dentin with Protect desensitizer resulted in low shear bond strengths for all three bonding agents used in this study: Syntac Single-Component (6.39±4.17 MPa), OptiBond Solo Plus (9.57±3.08 MPa) and Prime & Bond NT (5.45±1.49 MPa). This may result from the presence of monohydrogen-monopotassium oxalate in this desensitizer, which may precipitate and cover the surface, thus, minimizing the chance of forming a hybrid layer, resulting in a decreased bond strength. The results of this study are supported by a study by Pashley, Tao and Pashley (1993), where they found that monohydrogen-monopotassium oxalate solution adversely affected the shear bond strength of dentin bonding agents (Superbond, All Bond and Scotchbond 2). Results from this study are also supported by the SEM-LV image in (Figure 6C). There was no hybrid layer, which may result in low shear bond strength.

Rewetting of the etched dried dentin with HurriSeal desensitizer resulted in high shear bond strengths for all bonding agents used in this study: Syntac Single-Component (16.18±4.89 MPa), OptiBond Solo Plus (21.29±3.33 MPa) and Prime &Bond NT (20.73±3.45 MPa). This may result from the presence of 35% HEMA, benzalkonium chloride and 0.5% sodium fluoride and water in this desensitizer solution. The HEMA component is an excellent hydrophilic wetting agent that can reduce the surface tension and reopen the interfibrillar spaces. This allows for rapid transport of the desensitizing material into the open dentin tubules. A combination of benzalkonium chloride and sodium fluoride wraps itself around the collagen fibers and strengthens them to create a better fiber field to which the dentin bonding agents can hybridize, resulting in a strong hybrid layer (Hansen & Asmussen, 1997; Perdiãgo & others, 1998, 1999; Ross, 1998; Leinfelder, 1999). These results are also supported by the SEM-LV image in (Figure 6D). There was a uniform hybrid layer, which may result in high shear bond strength.

Bonding to dentin creates two interfaces: the dentin/adhesive interface and the adhesive/resin composite interface. Due to the similarity in the chemical composition of adhesive bonding agent and resin composite, the bond strength of the adhesive/resin composite interface should be higher than the dentin/adhesive interface.

Failure modes classification of the debonded specimens can provide important information in analyzing the obtained dentin shear bond strength. Olio (1993) stated that failure mode determination is an important observation in bond strength testing.

In this study, it can be seen generally, not statistically, that the number of the specimens exhibiting A_T failure mode were high with the low shear bond strengths; the number of specimens exhibiting A_R failure mode were high with intermediate to high shear bond strengths and the number of specimens exhibiting C_R and C_T failure modes were high with high shear bond strengths.

Adhesive failure between bonding agent and dentin (A_T) occurred in specimens with low shear bond strength. This might be due to incomplete wetting of the bonding agent to dentin (Figures 5A), non-uniform and thinner hybrid layer (Figure 6B) or the absence of a hybrid layer (Figure 6C).

Adhesive failure between the bonding agent and resin composite (A_R) occurred in the specimens with medium to high shear bond strengths (Figure 5B). This might be due to excessive water that causes phase separation of hydrophilic and hydrophobic monomers as in water-based bonding agents (Tay & others, 1996) or from an oxygen inhibition layer between the bonding agent and resin composite due to excessive air drying (Glasspoole & Erickson, 1992).

Cohesive failure within the resin composite (C_R) occurred in specimens with high shear bond strengths (Figure 5C). This might be due to cracks in the resin composite or errors such as bending moments and/or misalignment during the test.

Cohesive failure within dentin (C_T) occurred in specimens with the high shear bond strengths (Figure 5D). This might be due to good wetting and interaction

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

between the bonding agent and dentin, uniform hybrid layers in (Figure 6A and D), cracks in the dentin or errors such as bending moments and/or misalignment during the test.

From Table 4 we can see that the dry, rewet with Hurriseal and moist (control) groups for Syntac Single-Component bonding agent, rewet with HurriSeal and moist (control) groups for OptiBond Solo Plus bonding agent and rewet with HurriSeal group for Prime & Bond NT bonding agent exhibited high bond strengths. Each group had zero or one specimen with adhesive failure between the bonding agent and dentin (A_T). On the other hand, rewet with Protect group for Syntac Single-Component bonding agent; dry and rewet with Protect groups for OptiBond Solo Plus bonding agent; moist (control), dry and rewet with Protect groups for Prime & Bond NT bonding agent exhibited low shear bond strengths. These groups had most or all of their specimens showing adhesive failure between bonding agent and dentin (A_T).

CONCLUSIONS

The difficulty in achieving a balance between moist and dry dentin makes the dentin bonding technique extremely sensitive. The clinician should have a clear and thorough understanding of the chemical composition and adhesive mechanism of various dentin bonding systems.

HurriSeal was effective as a rewetting agent and can render the bonding procedure as less technique sensitive. Using Protect, on the other hand, reduced the shear bond strength of dentin bonding agents to the dentin surface.

(Received 29 May 2002)

References

Barkmeier WW, Hammesfahr PD & Latta MA (1999) Bond strength of composite to enamel and dentin using Prime & Bond 2.1 *Operative Dentistry* **24(1)** 51-56.

Blomlof J, Cederlund A, Jonsson B & Ohlson NG (2001) Acid conditioning combined with single-component and two-component dentin bonding agents *Quintessence International* **32(9)** 711-715.

Glasspoole EA & Erickson RL (1992) Effect of air thinning of adhesive resin on the bond strength measurements *Journal of Dental Research* **71** Abstract #257 p 10-12.

Halvorson R (1997) Effect of drying technique on bond strengths of one-bottle adhesives *Journal of Dental Research 76* Abstract #417.

Han L, Abu-Bakr N, Okamoto A & Iwaku M (2000) WDX study of resin-dentin interface on wet vs dry dentin *Dental Materials Journal* **19(3)** 317-325.

Hansen EK & Asmussen E (1997) Improved efficacy of dentin-bonding agents *European Journal of Oral Sciences* **105(5 Pt 1)** 434-439.

Jacobsen T & Söderholm KJ (1995) Some effects of water on dentin bonding *Dental Materials* **11(2)** 132-136.

Jacobsen T & Söderholm KJ (1998) Effect of primer solvent, primer agitation, and dentin dryness on shear bond strength to dentin *American Journal of Dentistry* **11(5)** 225-228.

Kanka J 3rd (1992a) Effect of resin primer solvents and surface wetness on resin composite bond strength to dentin *American Journal of Dentistry* **5(4)** 213-215.

Kanka J 3rd (1992b) Resin bonding to wet substrate: Bonding to dentin *Quintessence International* **23(1)** 39-41.

Kanka J 3rd (1996) Wet bonding: Effect of drying time and distance *American Journal of Dentistry* **9(6)** 273-276.

Kanka J 3rd (1997) One step bond strength to enamel and dentin *American Journal of Dentistry* **10(1)** 5-8.

Kato G & Nakabayashi N (1998) The durability of adhesion to phosphoric acid etched, wet dentin substrates *Dental Materials* **14(5)** 347-352.

Leinfelder KF (1999) Solutions for postoperative sensitivity *Compendium of Continuing Education in Dentistry* **20(5)** 488-490, 492, 494.

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

Nakabayashi N (1985) Bonding of restorative materials to dentin: The present status in Japan *International Dental Journal* **35(2)** 145-154.

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.

Olio G (1993) Bond strength testing. What does it mean? *International Dental Journal* **43(5)** 492-498.

Pashley DH, Horner JA & Brewer PD (1992) Interactions of conditioners on the dentin surface *Operative Dentistry* **(Supplement 5)** 137-150.

Pashley EL, Tao L & Pashley DH (1993) Effects of oxalate on dentin bonding *American Journal of Dentistry* **6(3)** 116-118.

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

Perdigão J, Swift EJ & Cloe BC (1993) Effects of etchants, surface moisture, and resin composite on dentin bonding strengths *American Journal of Dentistry* **6(2)** 61-64.

Perdigão J, Swift EJ, Heymann HO & Malek MA (1998) Effect of a re-wetting agent on the performance of acetone-based dentin adhesives *American Journal of Dentistry* **11(5)** 207-213.

Perdigão J, Van Meerbeek B, Lopes MM & Ambrose WW (1999) The effect of a rewetting agent on dentin bonding *Dental Materials* **15(4)** 282-295.

Pilo R, Cardash HS, Oz-Ari B & Ben-Amar A (2001) Effect of preliminary treatment of the dentin surface on the shear bond strength of resin composite to dentin *Operative Dentistry* **26(6)** 569-575.

Pioch T, Kobaslija S, Schagen B & Götz H (1999) Interfacial micromorphology and tensile bond strength of dentin bonding systems after NAOCl treatment *Journal of Adhesive Dentistry* **1(2)** 135-142.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Platt JA, Duke ES & Rhodes B (2001) The effect of desensitizers on dentin shear bond strength Indiana University School of Dentistry–Research Day Abstract #13.

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

Ross WN (1998) Quick tips-an effective dentin desensitizer at last *Contemporary of Esthetic in Restorative Practice* **July/August** 1-2 (Technical Report).

Swift EJ & Triolo PT (1992) Bond strengths of Scotchbond Multi-Purpose to moist dentin and enamel *American Journal of Dentistry* **5(6)** 318-320.

Tay FR, Gwinnett AJ, Pange KM & Wei SH (1995) The overwet phenomenon. A transmission electron microscopy study of possible mechanisms *Journal of Dental Research* **74** Abstract #15.

Tay FR, Gwinnett AJ & Wei SH (1996) The overwet phenomenon: An optical micromorphological study of surface moisture in the acid-conditioned resin-dentin interface *American Journal of Dentistry* **9(1)** 43-48.

Titley K, Chernecky R, Maric B & Smith D (1994) Penetration of a dentin bonding agent into dentin *American Journal of Dentistry* **7(4)** 190-194.

VanMeerbeek B, Yoshida Y, Lambrechts P, Vanherle G, Duke ES, Eick JD & Robinson SJ (1998) A TEM study of two water-based adhesive systems bonded to dry and wet dentin *Journal of Dental Research* **77(1)** 55-59.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3,** 297-302

Effect of Cooled Composite Inserts in the Sealing Ability of Resin Composite Restorations Placed at Intraoral Temperatures: An *In Vitro* Study

FJ de la Torre-Moreno • JI Rosales-Leal • M Bravo

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Clinical Relevance

Using cooled composite inserts improves the sealing ability of Class V resin composite restorations.

SUMMARY

Polymerization shrinkage causes microleakage of resin composite restorations. New materials and operative techniques should be developed in order to reduce polymerization shrinkage. This research studied the effects of cooled composite inserts and room-temperature composite inserts in the sealing ability of resin composite restorations placed at intraoral temperatures. Forty-eight extracted human molars (providing a total of 155 sections) were kept at intraoral temperatures, and Class V cavities were restored with an ormocer-based resin composite (Admira, Voco). Three restorative techniques were used: conventional bulk insertion (Group I or control group) (n=53 sections), room-temperature resin composite inserts (Group II) (n=52) and cooled resin composite inserts (Group III) (n=50). Microleakage and penetrating microleakage were studied under the microscope. Cooled composite inserts reduce microleakage at the gingival margins with respect to Groups I (p=0.002) and II (p=0.014). When small-size cooled composite inserts were used, the sealing ability at the gingival margins of Class V composite restorations was improved with respect to the bulk insertion technique and the room-temperature composite inserts technique.

*Francisco José de la Torre-Moreno, DDS, PhD, assistant professor, Universidad de Granada, Granada, Spain

Juan Ignacio Rosales-Leal, DDS, PhD, assistant professor, Universidad de Granada, Granada, Spain

Manuel Bravo, DDS, PhD, associate professor, Universidad de Granada, Granada, Spain

*Reprint request: , Gran Vía de Colón 46, 2E, E-18010 GRANADA (SPAIN); e-mail: fjtorre@ugr.es

INTRODUCTION

The marginal integrity of resin composite restorations has yet to be perfected. Reducing polymerization shrinkage seems to be the key to producing a leakage-free restoration. Therefore, new materials (with lower contraction) and improved operative techniques are needed. A glass-ceramic inserts technique was proposed to reduce microleakage and increase wear resistance (Bowen, Eichmiller & Marjenhoff, 1991; Bowen & others, 1993). These inserts, designed to fill as much of the cavity as possible, reduce the composite volume needed for a restoration and should decrease polymerization shrinkage stresses. Several *in vitro* studies found less microleakage when ceramic inserts were used (Godder & others 1994; George, Richards & Eichmiller, 1995; Ölmez, Öztas & Bilici, 1998), while

others did not (Applequist & Meiers, 1996; Coli, Derhami & Brännström, 1997). Regarding *in vivo* studies, although a two-year performance study (Kiremitçi, Bolay & Gürgan, 1998) reported glass-ceramic improvement of the overall properties of resin composite restorations, Sjögren and others (2000) found that failure of the restorations (loss and bulk fracture) increased with time (over two years). These results suggest that the weakest part of the restoration is the interface between the ceramic inserts and resin composite. Ceramic inserts can be contaminated during manipulation, which could affect the longevity of the restoration (Worm & Meiers, 1996). The use of prepolymerized resin composite inserts could improve some properties related to the insert-resin composite interface. Some studies (Godder & others, 1994; Applequist & Meiers, 1996) placed resin composite inserts (equal in size to ceramic ones) in light-cured resin composite restorations but did not find a reduction in microleakage when compared to conventional techniques; these results were attributed to the size of the composite inserts, which are less light-transmitting than ceramic ones, and so, the deeper areas of the restoration could not be cured properly. Moreover, these inlay-like restorations have an unfavorable C-factor (Feilzer, de Gee & Davidson, 1987; Davidson, Van Zeghbroeck & Feilzer, 1991), because the curing layer is reduced to a luting layer. Small composite inserts could help reduce leakage, unless they displace less curable material.

Light-cured composites have a very fast-setting reaction that limits flow (Feilzer, de Gee & Davidson, 1993). Some factors can decelerate the setting reaction of light-cured composites, thereby reducing the setting stress (Feilzer & others, 1993; Condon & Ferracane, 1998). Low contraction forces applied over a long period permit composite flow and allow for stress relaxation; examples of this effect are soft-start polymerization and pulse-delay curing devices (Uno & Asmussen, 1991; Goracci, Mori & Casa de Martinis, 1996; Sakaguchi & Berge 1998; Silikas, Eliades & Watts, 2000; Sahafi, Peutzfeldt & Asmussen, 2001; Yap & Seneviratne, 2001). Temperature could be another way to reduce setting stresses, as curing degree and curing rate of resin composites decrease with lower temperatures (Bandyopadhyay, 1982; Bennett & others, 1994; Vaidyanathan & Vaidyanathan, 1992; Lovell, Newman & Bowman, 1999). The use of cooled inserts could reduce the setting stress by changing the reaction kinetics. In addition, a thermal expansion of cooled inserts is expected (when they reach intraoral temperatures) to counteract part of the polymerization shrinkage.

In this study, small composite inserts (stored at room temperature and at –5°C) were placed in a bulk increment of the same material. The control group was restored in bulk, because the bulk technique is reproduced by placing inserts (inserts must be inserted in a bulk increment of resin composite); moreover, it has been questioned whether incremental techniques reduce shrinkage with respect to bulk insertion (Versluis & others, 1996). The thermal effect of composite inserts must be studied at intraoral temperatures. A new *in vitro* method has been developed to simulate intratooth temperature and pulp pressure during restorative procedures. This work tested whether the sealing ability of Class V composite restorations is affected when small composite inserts, stored at room temperature and cooled, are used.

METHODS AND MATERIALS

Forty-eight extracted, non-carious human molars were used within three months after extraction and stored in an aqueous 1% chloramin T solution at 4°C until prepared. The prepared teeth were randomly assigned to (and blocked [five sessions]) three experimental groups (16 teeth each) that corresponded to the different restorative techniques. The first three sessions (30 teeth) corresponded to the pilot study (see sample size calculation below). All the restorative procedures were conducted by the same operator at a room temperature of 22°C and 50% humidity.

In order to simulate intraoral tooth temperature and pulp pressure, teeth were prepared as follows (Figure 1). The apical third of each tooth was removed. The permeability of two root canals to the pulp chamber was verified by means of endodontic K-files (#20). Then, the

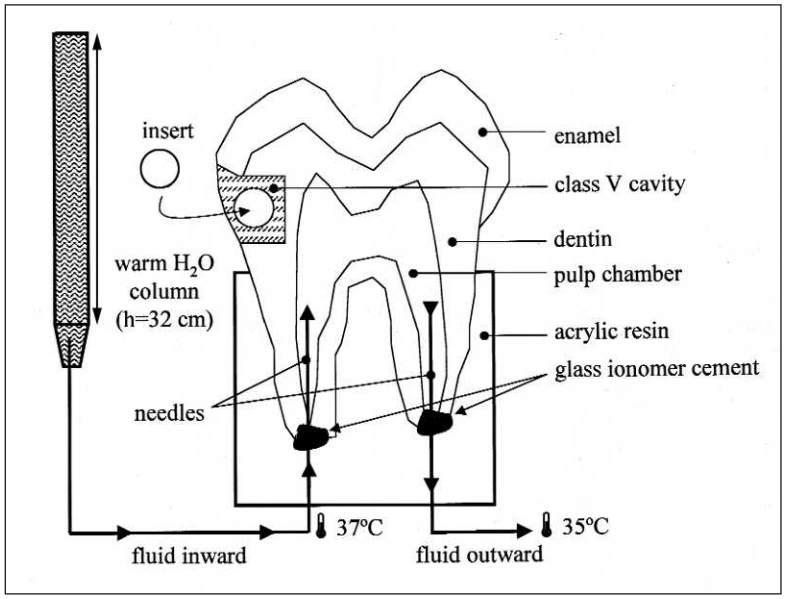


Figure 1: *Tooth preparation.*

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

teeth were apically penetrated from these two root canals to the pulpal chamber with two needles fixed to the root with Vitrebond (3M, St Paul, MN, USA). The needles were then connected to a simulated pulpal circuit of incoming and outgoing warm water under pressure (32 cm water column). The circuit was stabilized when the incoming water was at 37°C and the outgoing water was at 35°C.

Standardized buccal Class V cavities (5 mm long, 3 mm wide and 2.5 mm deep) were prepared in each specimen with a #330 diamond bur in a high-speed handpiece and air-water spray. A new bur was used for every three preparations. The gingival walls were extended to the cemento-enamel junction, and the enamel margins were beveled. For each Class V restoration, two spheres (1.5 mm. diameter) of an A3-shade ormocer-based resin composite (Admira, Voco, Cuxhaven, Germany) were made by light curing for 40 seconds in a silicone mold 24 hours before placing the restoration, then stored dry in hermetically-sealed containers (50% of the inserts were stored at room-temperature and 50% at –5°C).

The three experimental groups are: Group I (control group)—cavities were cleaned and air dried. Dentin and enamel were etched with 35% phosphoric acid (Vococid, Voco) for 15 seconds and rinsed with water for 10 seconds. Excess humidity was removed from the cavity, and one coat of adhesive (Admira Bond, Voco) was applied and light cured for 20 seconds. The teeth were then filled with one bulk increment of A3-shade resin composite (Admira, Voco) and light cured for 40 seconds with a Spectrum 800 (Dentsply de Trey, Konstanz, Germany). The intensity of the curing light was monitored at 500 mW/cm^2 for every restoration. The restorations were polished with Sof Lex discs (3M). Group II—the acid etching and application of adhesive followed the same steps as in Group I. After light curing the adhesive, a bulk increment of A3-shade resin composite was placed and two spheres of prepolymerized room-temperature composite were inserted in the center of the restoration, displacing the excess resin composite. After the excess material was removed, the restorations were light cured for 40 seconds and polished with discs. Group III—the teeth were restored as in Group II, but the pre-polymerized composite inserts were cooled to –5°C for 24 hours before placement.

The restored teeth were stored in water at 37°C for 24 hours. Needles placed on the apical zone of the teeth were then removed to prevent leakage from this area. This procedure left apical orifices that were subsequently filled with IRM (Dentsply de Trey). Two coats of nail varnish were applied to the root surface, except for 1 mm around the cavity margins. The teeth were then placed in a 0.5% fuchsin solution for 24 hours before being embedded in auto-polymerizable resin (Inplex, Remet sas, Bologna, Italy). Finally, the teeth were sectioned in a bucco-lingual direction with a cutting machine (Accutom 50, Struers, Copenhagen, Denmark). Sections 0.9-mm thick were obtained for each restoration (one restoration with two sections, 35 with three sections and 12 with four sections, totaling 155 sections). The sections were coded and randomly observed under the microscope in a blinded condition by an experienced observer. The depth of microleakage was recorded in milimeters both in the occlusal and gingival margins of the restorations. Dentinal penetration of the dye solution (penetrating microleakage) was also scored as absent or present. Penetration into the interface between the inserts and curable material was also scored as absent or present. To check the examiner's reliability, all observations were repeated by the same observer, thus, obtaining an ICC (intraclass correlation coefficient) of 0.97 for microleakage data and a 1.00 kappa for penetration data.

The sample size in each group was estimated based on the first 30 molars (giving 95 sections) using the formula n_c=Deff (z_α+ z_β)2 ($2\sigma^2/\Delta^2$), where n_c is the clustered-corrected sample size in each group, Deff is the design effect for the mean, z_α corresponds to a two-sided significance level (α), z_β corresponds to power (1-β), σ^2 is the applicable variance and Δ is the expected difference between two groups (Koch & Paquette, 1997). Considering microleakage in a gingival wall as the main variable in this study, a 1.46 Deff was found, a 1.7% type I error (α) (taking into account Bonferroni's correction for three comparisons; see below) (z_α=2.39), 20% type II error (β) (z_β=0.84), with the observer σ=0.51 mm and considering Δ=0.40 mm (approximate observed mean difference in gingival microleakage among the four gingival microleakage grades with clinical significance: No microleakage, less than half of the interface, more than half of the interface and extension to axial wall), the sample size necessary in each group was estimated as 49.5 and, consequently, the sample size was incremented up to approximately that figure.

The *t*-test (microleakage data) and chi-square test (penetration data) were adjusted for lack of independence of sections within the molars using SUDAAN v.7.0 (Shah, BarnWell & Bieler, 1996). The *p*-value to declare a difference as significant was 0.017 (after Bonferroni correction for three comparisons and an uncorrected 0.05 *p*-value).

RESULTS

Table 1 presents the microleakage and penetration data in the occlusal and gingival walls of this study. In the occlusal margins, both microleakage and penetration are very low, with non-significant differences between the groups. In gingival margins, the cooled composite inserts technique (Group III) produced significantly less microleakage in the gingival margins than in Group I (bulk technique) and Group II (room-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

temperature composite inserts), with non-significant differences between Groups I and II. Regarding penetrating microleakage, only Group I showed higher figures than Group III in gingival margins. Further results, not shown in Table 1, indicate that no leakage was found between inserts and the base material.

DISCUSSION

No microleakage was found between the composite inserts and polymerizable material. These results suggest that this interface can be less critical than that achieved between resin composite and glass-ceramic inserts (Worm & Meiers, 1996; Sjögren & others, 2000). Further research is needed to determine whether these insert-surrounding areas can be damaged by setting stresses, mechanical and physical properties of the material.

Gingival margins of Class V fillings are the weakest part of these restorations. The adhesion to enamel margins is much stronger than to dentin and cementum. As a result, the stress generated by polymerization shrinkage is released in gingival margins and microleakage appears in these areas.

Previous work has reported a reduction in microleakage using glass-ceramic inserts (Godder & others, 1994; George & others, 1995; Ölmez & others, 1998). However, some authors (Godder & others, 1994; Applequist & Meiers, 1996) have noted that composite inserts did not improve leakage of light-cured composites as expected. Nevertheless, composite inserts placed in self-curing material are known to improve marginal integrity (Dietschi & Herzfeld, 1998). This can be explained by the vast size of the composite inserts (same size as the ceramic ones) used by these studies. However, using small, room-temperature composite inserts did not significantly reduce the micro-leakage or penetrating micro-leakage in this study with respect to the conventional bulk technique. This suggests that with the curing phase reduction achieved with room temperature, small-composite inserts are not enough to significantly decrease microleakage or penetrating microleakage.

The use of small, cooled composite inserts (Group III) reduced microleakage significantly in gingival margins with respect to Groups I and II. This suggests that the thermal effect is responsible for this reduction. The reduction of penetrating microleakage in gingival margins with respect to bulk insertion suggests that the hybrid layer remained intact to protect dentin when cooled inserts were used.

Under the conditions of this study, the sealing ability of Group III restorations is improved as an effect of the thermal changes introduced by means of cooled inserts. Because temperature changes affect the properties of composite restorations, *in vitro* studies should be performed at intraoral temperatures.

As microleakage of resin composite restorations is mainly caused by polymerization shrinkage, a significant reduction in microleakage with the same materials (restorative + adhesive) may be produced by modifying the restorative procedures. In terms of future research, it is important to study the influence this study has in the following factors, which affect polymerization shrinkage:

Thermal Expansion of the Inserts: A thermal expansion of the cooled inserts and cooled surrounding areas should occur when they reach intraoral temperatures. This thermal expansion occurs in the early stages of the setting reaction (Bandyopadhyay, 1982) when the higher contraction stresses are produced. Therefore, this expansion can reduce leakage as a difference with hygroscopic expansion produced by water sorption (Donly & others, 1990; Hirasawa & others, 1983). Additional research is necessary to characterize the evolution of thermal changes and setting stresses throughout the reaction.

Reaction Kinetics: Insert surrounding areas are cooled by contact with the cooled inserts. The curing rate and degree of cure decrease when setting reaction occurs at lower temperatures (Bandyopadhyay, 1982;

Table 1: *Microleakage (mm) and Penetration (%) Data in Cavity Margins*

Group	n	Occlusal Wall	Gingival Wall
Microleakage (mm) ($\bar{x}\pm s$[a])			
Group I (bulk technique)	53	0.080 (0.299)	0.582 (0.605)
Group II (composite inserts)	52	0.058 (0.149)	0.463 (0.626)
Group III (cooled composite inserts)	50	0.015 (0.059)	0.159 (0.309)
Comparison by pairs[b]		I *vs* II: p=0.657	I *vs* II: p=0.470
		I *vs* III: p=0.177	I *vs* III: p=0.002[d]
		II *vs* III: p=0.024	II *vs* III: p=0.014[d]
Penetration (n (%))			
Group I (bulk technique)	53	1(1.9)	19(35.8)
Group II (composite inserts)	52	0(0)	9(17.3)
Group III (cooled composite inserts)	50	0(0)	2(4.0)
Comparison by pairs[c]		I *vs* II: p=0.317	I *vs* II: p=0.124
		I *vs* III: p=0.316	I *vs* III: p=0.003[d]
		II *vs* III: ---	II *vs* III: p=0.108

a: mean ± standard deviation; b: after t-test$_{adj}$ *(adjusted for multiple sections within molars); c: after* χ^2_{adj} *(adjusted for multiple sections within molars); d: statistically significant for an uncorrected* p*-value 0.05, after Bonferroni correction for three comparisons (see Methods).*

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Bennett & others, 1994; Vaidyanathan & Vaidyanathan, 1992; Lovell, & others, 1999). Thus, in cooled, insert-surrounding areas, the pre-gel period could be longer and, the setting stresses could be relieved more easily by flow. It has been reported that although glass-ceramic inserts reduce polymerization shrinkage, the contraction force is not always reduced during polymerization (Tani & others, 1993). The use of cooled inserts could help to reduce or modulate these forces. However, the kinetics of a diffusion-controlled reaction that involves auto-acceleration and auto-deceleration (Anseth & others, 1996; Lovell & others, 1999) is a complex phenomenon that needs further research, especially when there are different setting temperatures in the same restoration.

Shrinkage Vectors: Light-cured composites shrink toward the center of the material and shrinkage vectors are strongly influenced by the boundary conditions and slightly influenced by light activation (Versluis, Tantbirojn & Douglas, 1998). The effect of temperature should be considered in determining shrinkage vectors, because different temperatures within the same restoration could make the material shrink somewhat from the cooled areas toward the warmed (by contact with warm tooth) adhesive layer. The higher temperatures should increase the curing rate and degree of conversion and counteract the light attenuation that makes the material shrink slightly towards the curing light.

Curing Light Thermal Effect: The material can reach temperatures higher than intraoral temperatures by the thermal effect of the curing light (Loney & Price, 2001; Knezevic & others, 2001). Consequently, a thermal contraction of the restorative should be induced when it drops to intraoral temperatures. The thermal effect of the curing light has been determined to be about 30% of the final shrinkage force (Versluis, Sakaguchi & Douglas 1994). This effect is reduced when cooled inserts are used, because the overall temperature of the restoration should be lower during light curing.

CONCLUSIONS

This *in vitro* study concludes that when small-size cooled composite inserts are used, the sealing ability at the gingival margins of Class V composite restorations is improved with respect to the bulk insertion technique and the room-temperature composite inserts technique.

(Received 30 May 2002)

References

Anseth KS, Goodner MD, Reil MA, Kannurpatti SM, Newman SM & Bowman CN (1996) The influence of comonomer composition on dimethacrylate resin properties for dental composites *Journal of Dental Research* **75(8)** 1607-1612.

Applequist EA & Meiers JC (1996) Effect of bulk insertion, prepolymerized resin composite balls, and beta-quartz inserts on microleakage of Class V resin composite restorations *Quintessence International* **27(4)** 253-258.

Bandyopadhyay S (1982) A study of the volumetric setting shrinkage of some dental materials *Journal of Biomedical Materials Research* **16(2)** 135-144.

Bennett B, Puckett A, Pettey D & Roberts B (1994) Light source distance and temperature effects on composite polymerization [abstract] *Journal of Dental Research* **73(Special Issue)** p 1002.

Bowen RL, Eichmiller FC & Marjenhoff WA (1991) Glass-ceramic inserts anticipated for "megafilled" composite restorations *Journal of the American Dental Association* **122(3)** 71-75.

Bowen RL, George LA, Eichmiller FC & Misra DN (1993) An esthetic glass-ceramic for use in composite restoration inserts *Dental Materials* **9(5)** 290-294.

Coli P, Derhami K & Brännström M (1997) *In vitro* marginal leakage around Class II resin composite restorations with glass-ceramic inserts *Quintessence International* **28(11)** 755-760.

Condon JR & Ferracane JL (1998) Reduction of composite contraction stress through non-bonded microfiller particles *Dental Materials* **14(4)** 256-260.

Davidson CL, Van Zeghbroeck L & Feilzer AJ (1991) Destructive stresses in adhesive luting cements *Journal of Dental Research* **70(5)** 880-882.

Dietschi D & Herzfeld D (1998) *In vitro* evaluation of marginal and internal adaptation of Class II resin composite restorations after thermal and occlusal stressing *European Journal of Oral Sciences* **106(6)** 1033-1042.

Donly KJ, Dowell A, Anixiadas C & Croll TP (1990) Relationship among visible light source, composite resin polymerization shrinkage, and hygroscopic expansion *Quintessence International* **21(11)** 883-886.

Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.

Feilzer AJ, de Gee AJ & Davidson CL (1993) Setting stresses in composites for two different curing modes *Dental Materials* **9(1)** 2-5.

George LA, Richards ND & Eichmiller FC (1995) Reduction of marginal gaps in composite restorations by use of glass-ceramic inserts *Operative Dentistry* **20(4)** 151-154.

Godder B, Zhukovsky L, Trushkowsky R & Epelboym D (1994) Microleakage reduction using glass-ceramic inserts *American Journal of Dentistry* **7(2)** 74-76.

Goracci G, Mori G & Casa de Martinis L (1996) Curing light intensity and marginal leakage of resin composite restorations *Quintessence International* **27(5)** 355-362.

Hirasawa T, Hirano S, Hirabayashi S, Harashima I & Aizawa M (1983) Initial dimensional change of composites in dry and wet conditions *Journal of Dental Research* **62(1)** 28-31.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Kiremitçi A, Bolay S & Gürgan S (1998) Two-year performance of glass ceramic insert-resin composite restorations: Clinical and scanning electron microscopic evaluation *Quintessence International* **29(7)** 417-421.

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.

Koch GG & Paquette DW (1997) Design principles and statistical considerations in periodontal clinical trials *Annals of Periodontology* **2(1)** 42-63.

Loney RW & Price RB (2001) Temperature transmission of high-output light-curing units through dentin *Operative Dentistry* **26(5)** 516-520.

Lovell LG, Newman SM & Bowman CN (1999) The effects of light intensity, temperature, and comonomer composition on the polymerization behavior of dimethacrylate dental resins *Journal of Dental Research* **78(8)** 1469-1476.

Ölmez A, Öztas N & Bilici S (1998) Microleakage of resin composite restorations with glass-ceramic inserts *Quintessence International* **29(11)** 725-729.

Sahafi A, Peutzfeldt A & Asmussen E (2001) Effect of pulse-delay curing on *in vitro* wall-to-wall contraction of composite in dentin cavity preparations *American Journal of Dentistry* **14(5)** 295-296.

Sakaguchi RL & Berge HX (1998) Reduced light energy density decreases post-gel contraction while maintaining degree of conversion in composites *Journal of Dentistry* **26(8)** 695-700.

Shah BV, BarnWell BG & Bieler GS (1996) SUDAAN software for the statistical analysis of correlated data user's manual Release 7.0 Research Triangle Park, NC: Research Triangle Institute.

Silikas N, Eliades G & Watts DC (2000) Light intensity effects on resin composite degree of conversion and shrinkage strain *Dental Materials* **16(4)** 292-296.

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(1)** 25-31.

Tani Y, Nambu T, Ishikawa A & Katsuyama S (1993) Polymerization shrinkage and contraction force of composite resin restorative inserted with "Megafiller" *Dental Materials Journal* **12(2)** 182-189.

Uno S & Asmussen E (1991) Marginal adaptation of a restorative resin polymerized at reduced rate *Scandinavian Journal of Dental Research* **99(5)** 440-444.

Vaidyanathan J & Vaidyanathan TK (1992) Interactive effects of resin composition and ambient temperature of light curing on the percentage conversion, molar heat of cure and hardness of dental composite resins *Journal of Materials Science: Materials in Medicine* **3(1)** 19-27.

Versluis A, Sakaguchi RL & Douglas WH (1994) Stress development in composite resins during polymerization *Journal of Dental Research* **73(Special Issue)** Abstract #1000.

Versluis A, Douglas WH, Cross M & Sakaguchi RL (1996) Does an incremental filling technique reduce polymerization shrinkage stresses? *Journal of Dental Research* **75(3)** 871-878.

Versluis A, Tantbirojn D & Douglas WH (1998) Do dental composites always shrink toward the light? *Journal of Dental Research* **77(6)** 1435-1445.

Worm DA & Meiers JC (1996) Effect of various types of contamination on microleakage between beta-quartz inserts and resin composite *Quintessence International* **27(4)** 271-277.

Yap AUJ & Seneviratne C (2001) Influence of light energy density on effectiveness of composite cure *Operative Dentistry* **26(5)** 460-466.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3**, 303-306

The Effect of Elevated Temperatures on the Dentin Adhesion of Resin Composites

WW Brackett • DA Covey • LD Haisch

Clinical Relevance

Ingestion of hot food or beverages could compromise adhesive resin composite restorations.

SUMMARY

Although resin composite restorations may undergo relatively extreme temperature changes in the oral cavity, little is known about the effects of temperature on their adhesion to tooth structure. This study evaluated the effect of temperature on shear bond strength to dentin of three commercial resin dentin adhesives through testing of matured specimens over the 20° to 55°C temperature range. A significant difference ($p<0.05$) was observed between 20°C and 55°C for all the materials, and for one of the materials, a significant difference was also observed between 20°C and 37°C.

INTRODUCTION

Due to modern dietary practices, dental restorations are subjected to relatively extreme transient temperatures that range from 15°C to 68°C on tooth surfaces following ingestion of cold or hot beverages (Youngson & Barclay, 2000). Although the low thermal diffusivity of both tooth structure and resin restorative materials (Craig, 1997) would limit the depth of such temperature extremes, according to finite element analyses (Fenner, Robinson & Cheung, 1998; Hecox-Hohl & Beatty, 2001), they tend to affect the cavosurface margins of a restoration and indirectly induce significant stresses deeper into the tooth at the resin/tooth structure interface. Hecox-Hohl and Beatty (2001) modeled Class V resin composite restorations of abfraction lesions and predicted induced stresses from a short exposure to high temperatures greater than 40 MPa at the dentin/dentin adhesive interface.

Elevated temperatures have also been long recognized as being detrimental to the properties of resin composite restorative materials (Torgalkar, 1973; Draughn, 1981; Vijayaraghavan & Hsiao, 1994), and because resins and tooth structure differ greatly in coefficient of thermal expansion (Anusavice & Phillips, 1996), it has also long been accepted that specimens for *in-vitro* investigations of adhesion should be stressed by undergoing temperatures typical of the oral environment before testing (Guzman, Swartz & Phillips, 1969). Despite the temperature extremes of the oral environment and the prevalence of thermocycling and the accelerated thermal stressing of adhesive specimens in laboratory research, previous research has only focused on the effects of temperature and humidity at the time of placement on the adhesion of resin to dentin, which were minimal (Burrow & others, 1995; Burrow & others, 1996). No investigations have been published on

*William W Brackett, DDS, MSD, associate professor, University of Nebraska Medical Center, Lincoln, NE

David A Covey, DDS, MS, MS, associate professor, University of Nebraska Medical Center, Lincoln, NE

Larry D Haisch, DDS, associate professor, University of Nebraska Medical Center, Lincoln, NE

*Reprint request: Section of Operative Dentistry, Department of Adult Restorative Dentistry, College of Dentistry, PO Box 830740, Lincoln, NE 68583-0750; e-mail: wbracket@unmc.edu

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

the effect of elevated temperatures applied after placement of matured resins to tooth structure on the adhesion.

This study evaluated the changes in dentin bond strength of three commercial resin dentin adhesives over the 20° to 55°C temperature range common in both the oral environment and in laboratory research protocols.

METHODS AND MATERIALS

Freshly-extracted human third molars were debrided and stored in a 1% thymol solution at room temperature. Within two weeks of extraction, the teeth were invested in clear resin cylinders, with the occlusal surface of the tooth oriented perpendicular to the long axis of the cylinder and protruding approximately 3 mm out one end of the cylinder. During setting of the investing resin, the specimens were stored at 100% relative humidity. For each invested tooth, the occlusal surface and the end of the resin cylinder were ground until the occlusal enamel was removed and the cylinder end and resulting dentin surface were perpendicular to the long axis of the cylinder. This was accomplished by affixing the cylinders in the auto-polishing attachment of a precision grinding/polishing machine (Model VP160/AP60, Leco Corp, St Joseph, MI, USA) and grinding with wet SiC abrasive paper of 180, 240 and 320 grits. After grinding, the invested teeth were stored in 37°C water.

Within two days of grinding the dentinal surfaces of the teeth, cylinders of three representative hybrid resin composites, listed in Table 1 and randomly assigned to the invested teeth, were affixed at room temperature. The dentinal surfaces were initially etched for 15 seconds with the supplied phosphoric acid. After etching, the dentinal surfaces were rinsed and lightly air dried, then a mask of adhesive tape was placed to restrict the area of adhesion to a 4-mm diameter circle. The supplied single-bottle dentin adhesive for each resin was then applied onto the area defined by the mask according to manufacturer's instructions, air thinned and light cured for 10 seconds. This was immediately followed by attachment of the resin composite using a #5 gelatin capsule oriented perpendicular to the dentin surface. The resulting cylindrical button was 4 mm in diameter and 6 mm in length. The resin composite was light cured in bulk from all directions for two minutes. Intensity of the light-curing unit (Elipar TriLight, ESPE America Inc, Norristown, PA, USA) was monitored throughout the study with the unit's built-in radiometer and was found to be 800 mW/cm^2 on all occasions.

The completed specimens were stored in 37°C water for one week, after which the adhesive tape masks were removed. The resin cylinders were then fractured from the dentin substrate in shear, while immersed in a temperature-controlled water bath using a servohydraulic testing machine (Model 8500, Instron Corp, Canton, MA, USA) at a crosshead speed of 0.5 mm/minute. One shearing fixture was a custom-made brass device designed to secure the previously described invested teeth and to position the resin composite cylinders perpendicular to crosshead travel. This was opposed by a chisel-shaped steel blade that engaged the resin composite cylinder at its junction with the dentin surface.

Specimens of each material were tested at three temperatures, 20°, 37° and 55°C, with each placed in the water bath for 20 minutes prior to testing so that the temperature of the entire specimen could equilibrate to the desired temperature. The type of failure was visu-

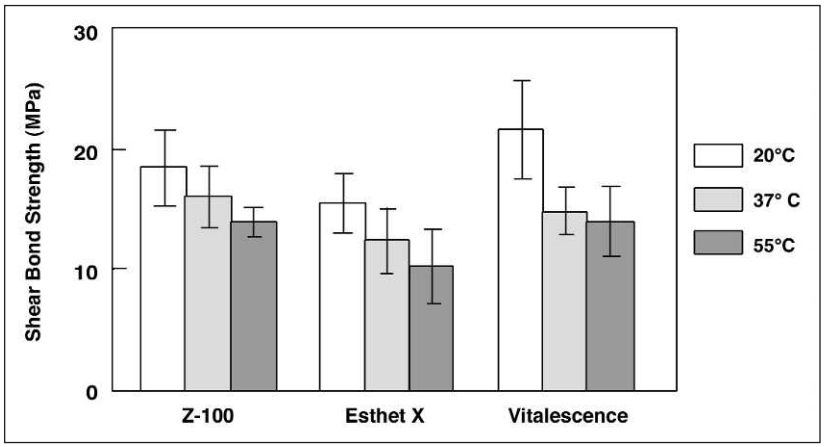


Figure 1: *Shear bond strength to dentin of three resin composites at three temperatures (MPa). Height of error bars equals one standard deviation.*

Table 1

Resin Composite	Adhesive	Manufacturer
Z 100	Single Bond	3M Dental Products Division St Paul, MN, USA
Esthet X	Prime & BondNT	DENTSPLY/Caulk Milford, DE, USA
Vitalescence	PQ1	Ultradent Products, Inc South Jordan, UT, USA

Table 2: *Shear Bond Strength to Dentin of Three Resin Composites at Three Temperatures MPa (SD)*

Resin Composite	20°C	37°C	55°C
Z 100/Single Bond	18.5 (3.2)	16.1 (2.6)	14.0 (1.3)*
Esthet X/Prime & Bond NT	15.6 (2.5)	12.5 (2.7)	10.3 (3.2)*
Vitalescence/PQ1	21.8 (4.1)	15.0 (2.0)*	14.1 (3.0)*

**significantly different than 20°C for the same material, one-way ANOVA; Neuman-Keuls multiple comparison test (p<0.05).*

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

ally observed and recorded as adhesive, cohesive or mixed.

The sample size for each combination of material and temperature was five. Because temperature, not material, was the independent variable of interest, average shear bond strength values produced between each resin composite and dentin at the various temperatures were statistically compared using three separate one-way analyses of variance. For each of the three, follow-up multiple comparisons of means were made using a Neuman-Keuls multiple comparison test. No comparison was made between resin composites.

RESULTS

Each of the resin composite/adhesive combinations tested showed decreasing bond strength to dentin with increasing temperatures. For each of the experimental groups, data were found to be distributed normally. A significant difference ($p<0.05$) was observed between 20°C and 55°C for all the materials, and for Vitalescence, a significant difference was also observed between 20°C and 37°C. All failures were of the adhesive type. Complete results are listed in Table 2 and displayed in Figure 1.

DISCUSSION

A large adhesive resin composite restoration is likely to undergo a variety of challenges, including abrasion, chemical degradation and mechanical stresses. This study's findings implied that the decrease in bond strength with the intake of a hot food or beverage would likely compound the already-hostile environment of such a restoration. The frequency with which a patient ingests very hot foods and beverages should probably be considered prior to electing a dentin-adhesive restoration, while patients with such restorations should avoid extremely hot foods and beverages.

The authors concede that a restored tooth does not reach the steady-state elevated temperature of the bond strength specimens in this study; however, they believe that no experimental model can effectively separate the effects of temperature from the effects of temperature-induced stresses due to the different thermal coefficients of resin and tooth structure. This study was undertaken in an attempt to define boundary conditions as a basis for further study. The diminished bond strength with temperature observed may be a compound effect of stresses on the adhesive bond produced by greater expansion of the resin than the tooth and the plasticizing effect of heat on resins. The authors acknowledge that a sample size of five is relatively small but wish to point out that sample size is factored into the statistical test employed, and that the greatest risk presented by use of such a small sample size is that of a real difference going unnoticed. This may have occurred in this study in terms of the comparison between room and body temperature.

The authors also believe, based on many previous microleakage studies of Class V restorations and the previously cited finite element analysis (Hecox-Hohl & Beatty, 2001), that the resin/dentin interface is more susceptible to disruption than the resin/enamel interface, and the resin/dentin interface was the better choice for testing in this initial study. It is expected that these results generalize to resin-enamel adhesion, however, further study is indicated to determine the extent.

In addition to the above-cited clinical implications, this study also raises questions about the appropriate temperature for *in-vitro* testing of bond strength. In support of the customary testing at room temperature is the lack of any significant difference between room and body temperature for two of the three materials, and that the rank order of bond strength did not change for the three materials at any temperature. Of concern is the significant difference between bond strengths obtained at room temperature and 55°C for all the materials, and the significant differences observed among all three temperatures for the Ultradent product. It is unclear why this product, which had the highest bond strength at room temperature, was more sensitive to temperature than the others, although its adhesive is a filled resin, unlike the other products and, consequently, forms a thicker layer.

These results lend support to the thermocycling of laboratory specimens for adhesion testing. The increased plasticity of resins produced by heating during the thermocycling of microleakage specimens might compound or reduce the deleterious effect on marginal adaptation of thermal stresses, depending on the cavity design tested.

CONCLUSIONS

Elevated temperatures degrade the *in-vitro* dentin adhesion of the products tested.

(Received 5 June 2002)

References

Anusavice KJ & Phillips RW (1996) *Phillips' Science of Dental Materials* Philadelphia WB Saunders, 10th ed p 286.

Burrow MF, Nikaido T, Satoh M & Tagami J (1996) Early bonding of resin cements to dentin-effect of bonding environment *Operative Dentistry* **21(5)** 196-202.

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.

Craig RG (1997) Restorative Dental Materials St Louis Mosby 10th ed p 44.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Draughn RA (1981) Effects of temperature on mechanical properties of composite dental restorative materials *Journal of Biomedical Material Restoration* **15(4)** 489-495.

Fenner DN, Robinson PB & Cheung PM (1998) Three-dimensional finite element analysis of thermal shock in a premolar with a composite resin MOD restoration *Med Eng Phys* **20(4)** 269-275.

Guzman HJ, Swartz ML & Phillips RW (1969) Marginal leakage of dental restorations subjected to thermal stress *Journal of Prosthetic Dentistry* **21(2)** 166-175.

Hecox-Hohl R & Beatty MW (2001) FEA stresses in restored cervical lesions under combined loading conditions *Journal of Dental Research* **80(Special Issue)** Abstract #1577 p 233.

Torgalkar AM (1973) Effect of temperature on rheological properties of four dental restorative materials *Journal of Dental Research* **52(6)** 1216-1219.

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.

Youngson CC & Barclay CW (2000) A pilot study of intraoral temperature changes *Clinical Oral Investigations* **4(3)** 183-189.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3**, 307-314

An Energy Conversion Relationship Predictive of Conversion Profiles and Depth of Cure for Resin-Based Composite

RH Halvorson • RL Erickson • CL Davidson

Clinical Relevance

Conversion throughout resin-based composite can be predicted at various light-curing conditions by using an energy conversion relationship. In addition, using a modified ISO standard for depth of cure, the conversion at 1/2 the scrape-back length was correlated to approximately 90% of the maximum measured conversion.

SUMMARY

Predicting the polymerization throughout resin-based composite (RBC) has been reduced to a set of variables involving irradiance of the light source, exposure duration and RBC transmission properties, together with an energy-conversion relationship (ECR) derived from Fourier Transform Infrared Spectroscopic analysis (FTIR) of a single shade of photo-polymerized RBC. The ECR describes the localized energy density required to achieve a desired conversion independent of shade. Using this ECR, conversion was predicted and experimentally verified throughout different opacities of RBC based on knowledge of their transmission properties and the incident radiant energy density (irradiance times exposure time). Also, using RBC transmission properties, a critical scrape-back energy of approximately 32 mJcm^{-2} was determined from cylindrical samples of photo-polymerized RBC in which the poorly polymerized material was removed. This value correlates to approximately 22% conversion. The critical scrape-back energy was then used to predict scrape-back lengths obtained from samples polymerized at various energy densities. These results confirm the logarithmic relationship between depth of cure and energy of exposure and the reciprocal relationship between irradiance and time of exposure.

*Rolf H Halvorson, 3M ESPE, St Paul, MN

Robert L Erickson, DDS, PhD, associate professor, Creighton University School of Dentistry, Omaha, NE

Carel L Davidson, PhD, professor, ACTA, University of Amsterdam, The Netherlands

*Reprint request: 3M Center Building 260-2B-09, St Paul, MN 55144-1000; e-mail: rhhalvorson@mmm.com

INTRODUCTION

A number of methods have been explored to characterize depth of polymerization of photoactivated resin-based composite (RBC) and understand the variables involved. The "scrape-back" technique (Cook, 1980) is perhaps the simplest of such methods and essentially delineates a polymerization boundary beyond which the resin is either grossly underpolymerized or completely unpolymerized. The length of the remaining composite has a logarithmic dependence for both the intensity of the light source and exposure time for UV (Cook, 1980) and visible light (Cook & Standish, 1983) polymerized RBC. The logarithmic relationship was

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

predicted using a mathematical model for depth of cure based on the rate of initiation of free radical polymerization and incorporates the exponential attenuation of light intensity through composite thickness. This attenuation severely limits the length of the scrape-back sample that can be obtained as revealed in a study that reported only a modest increase in length (<25%) upon doubling the exposure time (Ruyter & Øysaed, 1982). Similar depths of cure (scrape-back) have also been obtained when the product of the irradiance and the exposure time is kept constant (Cook, 1982; Nomoto, Uchida & Hirasawa, 1994). It was suggested that the depth of cure corresponds to the minimum amount of energy required to initiate polymerization.

To determine polymerization throughout composite requires more extensive methods, such as hardness measurements (Cook, 1980; De Lange, Bausch & Davidson, 1980) or infrared spectroscopy (Eliades, Vougiouklakis & Caputo, 1987; Dewald & Ferracane, 1987). These techniques generally reveal a rapid decrease in hardness or conversion of methacrylate double bonds beyond a certain depth. Consistent with studies utilizing the scrape-back technique, the irradiance of the light source, the exposure time and light transmission of composite are significant variables that affect the hardness or conversion profile (variation with depth). It has been shown that similar conversion profiles (via FTIR) were obtained when an RBC was exposed under reciprocal irradiance-exposure time relationships (Nomoto & others, 1994). This suggests that the conversion at any point within the RBC is dependent upon the radiant energy available at that point. It is, therefore, useful to construct the relationship between the conversion of photopolymerized RBC and the exposure energy (the energy-conversion relationship or ECR). This has been performed for various commercial RBCs in a thin film via transmission FTIR, together with confirmation of the reciprocal nature of irradiance and exposure time (Halvorson, Erickson & Davidson, 2002). This ECR is applicable toward the goal of predicting conversion at the surface of photopolymerized RBC given the irradiance and time of exposure. Similarly, prediction of conversion at any point within an RBC may be accomplished from an ECR for bulk curing and knowledge of the light transmission of the RBC. The transmission curves are readily determined radiometrically, and, based on prior work, it should be possible to define a unique ECR by measuring the conversion versus depth for a single shade of RBC at maximum irradiance. This data, combined with the transmission data, can relate conversion to energy, thereby, providing the ECR.

The goals of this investigation were to 1) determine the energy-dependent conversion relationship (ECR) of commercial RBC and confirm that this describes a reciprocal relationship between irradiance and exposure time, 2) show that this relationship, together with transmission properties, can be used to predict the conversion profile for various exposure energies and RBC opacities and 3) define a critical exposure energy that is predictive of scrape-back length for various exposure energies and RBC opacities.

METHODS AND MATERIALS

Energy Conversion Relationship

Two small particle hybrid resin-based composites of similar shade (A3.5) were examined to construct the ECRs: XRV Herculite (Kerr Corp, Orange, CA, USA) and 3MZ100 Restorative (3M, St Paul, MN, USA). The composition of these RBC materials has previously been described (Halvorson & others, 2002). Cylindrically-shaped samples were prepared by packing RBC into a split stainless steel mold with an approximate 6-mm diameter by 16-mm length. The mold was assembled to include two stainless steel wedges positioned along the length of the mold, on opposite sides, with their internal edges protruding into the cylinder approximately one-half millimeter and their outside edges extending outside the sides of the mold. Two machine screws held the assembly in place during the packing and polymerization phase. Transparent polyester film was placed over the ends of the cylinder to confine the composite within the mold. The mold was then placed on a white background and positioned directly under the 7-mm diameter light guide of a tungsten-halogen lamp (3MXL 3000 Curing Lamp, 3M) with a nominal power density of 600mWcm^{-2}. This lamp was checked periodically throughout the experiment to monitor any deviations in its output. Samples were exposed for 30 seconds (18 Jcm-^{2}) and kept in the dark at room temperature for 24 hours. The screws were than removed, and one of the wedges was gently tapped with a hammer, splitting the sample lengthwise down its center. The two halves were then separated, carefully teasing the unpolymerized end of the sample apart with a scalpel.

To determine conversion with depth, microscopic specimens were dissected with a scalpel at selected intervals down the length of each half using a binocular microscope. The microscope's reticle was used to determine the depth along the cylinder at which the specimen was dissected and its lamp was filtered to prevent additional polymerization. Dissection was confined to approximately the central-third of the sample. Conversion of the dissected specimens was measured using transmission FTIR microspectroscopy. Specimens were placed on a KBr disc and measured in transmission with a Nic-Plan Microscope combined with a Magna-IR 750 spectrometer (Nicolet, Madison, WI, USA) co-adding 90 scans at a resolution of 4 cm^{-1}. Three cylinders were prepared and analyzed for each group with three-to-five specimens measured at each

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

depth from each cylinder. Conversion was determined by measuring the decreasing absorbance of the methacrylate carbon double-bond vibration at 1638 cm^{-1}, using the aromatic skeletal absorbance from BIS-GMA at 1582 cm^{-1} as an internal reference. Integrated areas of both peaks were determined using a standard baseline technique. The radiation energy density of the curing lamp was determined using a power meter (Power Max 500D Laser Power Meter, Molectron Detector Inc, Portland, OR, USA) that integrated the radiant power density over the 30-second exposure time. Power density was determined by dividing the measured power by the cross-sectional area of the light guide.

The transmittance ($T=P/P_o$) at thicknesses for each RBC shade was determined by polymerizing the respective materials in 6-mm diameter stainless steel molds of various lengths. The polymerized sample, together with its mold, was placed on the detector of a power meter (351 Power Meter, UDT Instruments, Baltimore, MD, USA), centering the light guide of the curing lamp over the mold and in contact with the sample. The power measured in this fashion (P) was divided by the unattenuated power (P_o) obtained by placing the light guide in direct contact with the detector head. A minimum of three replications was done for each condition and a mean value was determined. Transmission as a function of thickness was determined by regression analysis of the data. Small errors may be introduced by using only transmission data from cured composite, but the benefits in simplifying the analysis justify this procedure. The energy exposure at depths (E_d) where FTIR specimens were dissected was determined from the incident energy (E_o) and the transmittance ($E_d=\%T_d x E_o$). This permitted conversion to be related to energy and, thereby, define an ECR for the Z100 and Herculite RBC materials.

Predicted Conversion Profiles

Predicted conversion profiles were obtained by determining the energy density transmitted to various depths from the transmittance curves and the incident energy density. The corresponding conversions obtained from the Z100 ECR were plotted as a function of depth that yielded the predicted conversion profiles. Profiles for Z100, shades A1, A3.5 and CY were predicted at various exposure conditions (Figure 4). The curves were experimentally verified by FTIR microspectroscopy using methods described above.

Scrape-Back Length: Measured and Predicted

Identical molds as those described for FTIR sampling (without wedges) were used to determine the depth of cure via the scrape-back technique. The samples were prepared as described above and exposed to the curing light at various energy densities. After 24 hours at room temperature, the molds were disassembled and the poorly polymerized material gently scraped off with a rigid plastic spatula. Three replicates were prepared and the length along the cylinder axis was measured to the nearest 0.01 mm. Average scrape-back lengths for Herculite and Z100 (A3.5 shade) exposed to 18 Jcm^{-2} were used to define a critical energy density associated with the scrape-back lengths utilizing the transmission data and incident energy density. The critical energy density, together with transmission data, was subsequently used to predict scrape-back lengths for other materials and curing conditions. Conversions at the scrape-back lengths were determined from the ECR for the critical energy density.

RESULTS

Figure 1 shows the conversion profiles, as measured with FTIR microspectroscopy for shade A3.5 of Herculite and Z100 at an exposure energy of 18 Jcm^{-2}. Maximum conversion for Herculite is greater than for Z100 and reflects differences in the formulation of these

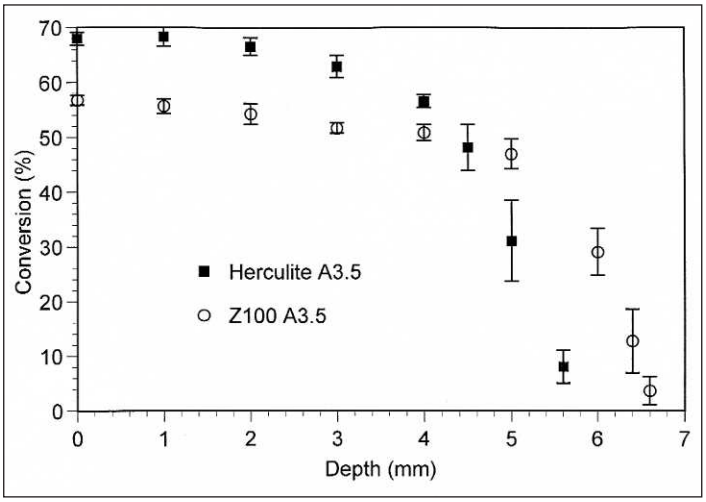


Figure 1: *Conversion profiles for shade A3.5 of Herculite and Z100 exposed with 18 Jcm^{-2} (30s/600 $mWcm^{-2}$).*

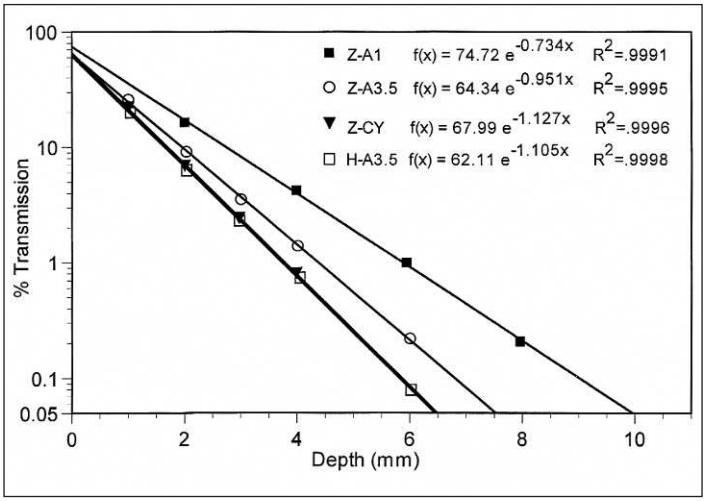


Figure 2: *Percent transmittance versus depth. H:Herculite, Z:Z100.*

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

two RBC materials. Although both materials are designated as A3.5 shades, there is a greater depth of cure for Z100 because of its lower opacity (Figure 2). Figure 2 shows the percent transmittance curves for the materials investigated and describes the expected exponential decrease in energy with depth. Regression analysis reveals an exponential relationship between transmittance and depth with the attenuation coefficient defined by the slope of the line and R^2 values very close to 1.000. Using the regression equations and the incident light energy density, the energy density at depths corresponding to the measured conversion in Figure 1 was calculated and ECRs were plotted as shown in Figure 3. In this comparison, the conversion for both materials has been expressed relative to their maximum measured conversion, and it is apparent that the ECR curves on a relative conversion basis are very similar.

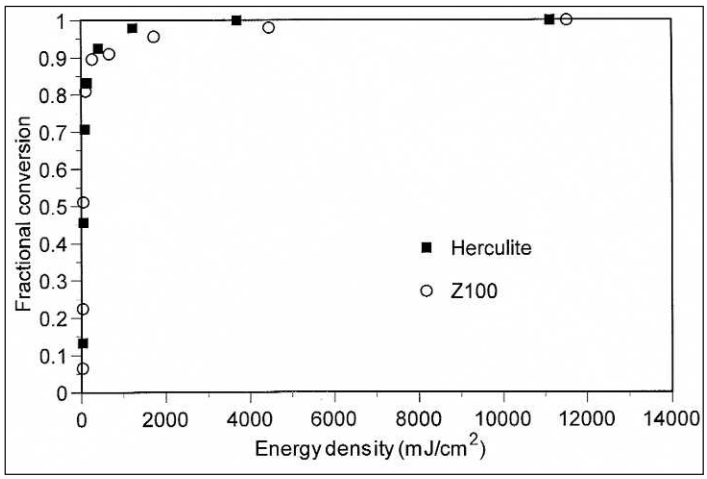


Figure 3: *Energy conversion relationship (ECR) for Herculite and Z100 derived from their respective conversion profiles and transmittance curves. Conversion is expressed relative to the maximum 24-hour conversion.*

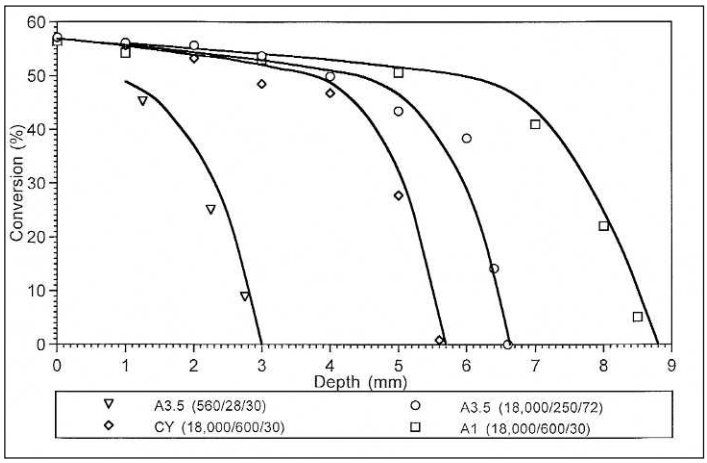


Figure 4: *Predicted conversion profiles (solid lines) for the indicated shades and exposure conditions for Z100 together with experimental values from FTIR analysis. Legend: (incident energy density,mJcm^{-2}/irradiance, mWcm^{-2}/ exposure time(s)).*

Using the ECR for Z100, together with transmittance curves and the incident energy density, conversion profiles for Z100 shades A1, A3.5 and CY were predicted as shown in Figure 4. Experimental values obtained by FTIR microspectroscopy are also shown in Figure 4 and depict a reasonably good fit to the predicted curves. The variability of the experimental values is similar to the variability of the average values for the conversion profiles depicted in Figure 1 and is greatest at the steepest portion of the curve. The precision of the predicted curves will be affected mostly by the precision represented in the conversion profile for Z100 (Figure 1) from which the ECR was derived.

The scrape-back lengths and exposure conditions for shade A3.5 of both materials are shown in Table 1. Comparison of conversion profiles in Figure 1 to their respective scrape-back values in Table 1 reveal that the latter are several tenths of millimeters shorter than the extrapolated depth at zero conversion and that conversion at the scrape-back depth is approximately 20% for Herculite and 22% for Z100. These conversions represent a local exposure of approximately 32 mJcm^{-2} for each of the materials as determined from the ECR. This energy density will be defined as the critical scrape-back energy density. Figure 5 presents a photograph of material Z100-A3.5 prepared as described for sample dissection after 24 hours, together with the corresponding conversion-depth profile from Figure 1. To enhance the contrast between cured and uncured material, the split sample has been stained with a dye (Astra Blue) that has an affinity for the dimethacrylate monomers (de Gee, ten Harkel-Hagenaar & Davidson, 1984). The scrape-back length and associated conversion (Table 1) are indicated in the figure. Beyond the scrape-back depth, there is a region that exhibits very low cohesion and terminates as a granular appearing zone with a gelatinous consistency. The terminus of this zone, at approximately 6.7 mm, corresponds to the extrapolated depth at zero conversion. The corresponding energy density was determined from the ECR to be 21 mJcm^{-2}.

Predicted scrape-back lengths are shown in Table 2, together with experimentally derived values. Predicted values were determined using the critical scrape-back energy (32 mJcm^{-2}). The good agreement between the predicted and measured values suggests

Table 1: *Scrape-Back Lengths*

Material	Scrape-Back Length (mm)	Conversion at Scrape-Back (%)	Energy Density (mJ/cm^2)	Irradiance (mW/cm^2)	Exposure Time (s)
Herculite-A3.5	5.27 (0.07)	20	18,000	600	30
Z100-A3.5	6.19 (0.10)	22	18,000	600	30

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

that the critical energy is unique for both RBC materials and applies to varying shades of material and curing conditions. Figure 6 shows predicted curves relating scrape-back length and exposure energy for Z100 shades A1, A3.5 and CY. Measured scrape-back lengths for selected exposure energies are superimposed. Corresponding results for Herculite are shown in Figure 7. The experimental scrape-back values agree with the predicted curves and verify the logarithmic dependence between scrape-back length and exposure energy.

Figure 8 shows conversion profiles, each for an incident energy density of 18 Jcm^{-2} but with different incident irradiance and time of exposure for Z100-A3.5. The excellent overlap of these two profiles confirms a reciprocity relationship between irradiance and time. Similar confirmations of reciprocity are seen in Table 2, where similar scrape-back lengths are observed when total energy density is conserved. One-factor ANOVA showed that the scrape-back values obtained with constant energy densities were equivalent.

DISCUSSION

In this study, predicting the extent of polymerization of RBC material throughout its thickness has been reduced to a set of variables by considering the energy-conversion relationship (ECR), the light transmitting properties of the RBC and the applied radiant energy. The results have shown that an ECR, derived from a single shade of RBC, can be used to predict conversion profiles for a range of shades at various exposure conditions. Specifically, the ECR describes the local energy density required to obtain a given normalized conversion at any depth in the material, independent of shade and reflects the combined polymerization efficiency of the monomer composition and photoinitiating system. The ECR has previously been described for other commercial RBCs using a thin film technique that predicts surface conversion (Halvorson & others, 2002). Predicting conversion within RBC using the thin film technique, however, is limited to exposure conditions that yield near maximum conversion. This limitation is possibly due to an inhibition mechanism and requires further investigation. Both techniques are consistent, though, in describing similar ECRs across different RBC compositions and both confirm reciprocity between time and irradiance. The similar ECRs are likely a consequence of the widespread use of resins based on the dimethacrylate, BIS-GMA and photoinitiator consisting of camphorquinone and amine.

For a given chemistry, the ECR suggests that transmission properties of the RBC ultimately determine the conversion profile and depth of cure. This is shown by the transmission curves for Z100 (Figure 2) and the predicted and experimental conversion profiles obtained with an 18 Jcm^{-2} exposure (Figure 4). The significance of similar ECRs across materials is shown for Herculite A3.5 and Z100 CY, where identical scrape-back lengths at an18 Jcm^{-2} exposure are predicted from their nearly identical transmission curves (Figure 2). The regression equations in Figure 2, that describe the exponential decrease in percent transmittance with depth, conform to the Lambert Law (Christian, 1977) and represent the combined effects of reflection, scattering and absorption. Analysis of the regression equations revealed that surface reflected radiation, identified by the y-intercept, is as much as 38% of the incident radiation. Thus, the maximum fractional conversion, observed in Figure 3 (relating to surface measurements in Figure 1), correspond to energy densities significantly less than the 18 Jcm^{-2} incident energy. This loss is considerable, though less than that measured for similar commercial RBCs

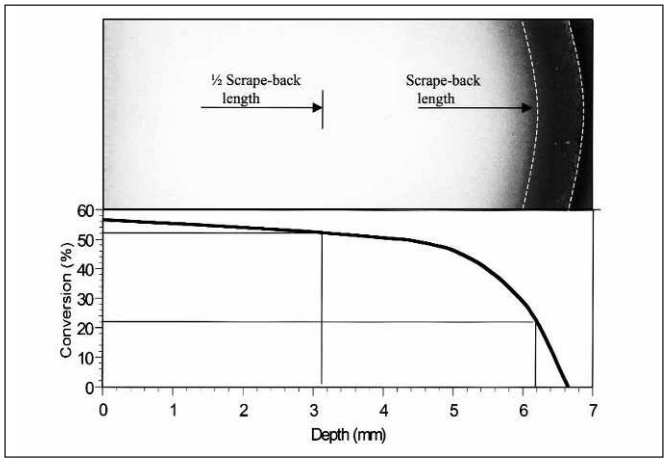


Figure 5: *Sample of Z100 A3.5 exposed with 18,000mJcm^{-3} (600mWcm^{-2}/30s) prepared as described for specimen dissection and FTIR analysis (24 hours). Sample has been stained with a dye (astra blue) that has an affinity for dimethacrylate monomers. The conversion profile for Z100-A3.5 depicted in Figure 1 and exposed under identical conditions is shown for comparison.*

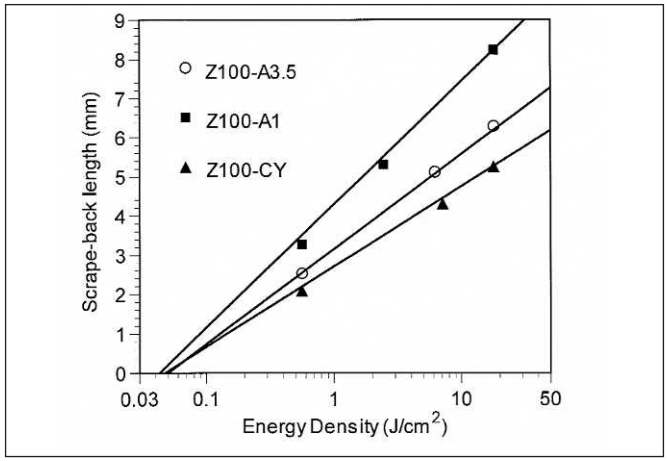


Figure 6: *Predicted scrape back lengths (solid lines) as a function of the incident energy density together with experimental values for the indicated shades of Z100.*

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 2: *Predicted and Experimental Scrape-Back Lengths*

Material	Scrape-Back Length (mm) Predicted	Experimental	Energy Density ($mJcm^{-2}$)	Irradiance ($mWcm^{-2}$)	Exposure Time(s)
H-A3.5	NA	5.27 (0.07)	18,000	600	30
	5.30	5.29 (0.04)	18,000	250	72
	4.33	4.21 (0.08)	6160	560	11
	4.33	4.30 (0.10)	6160	310	20
	4.33	4.31 (0.14)	6160	170	37
	2.16	2.14 (0.08)	560	28	20
Z-A3.5	NA	6.19 (0.01)	18,000	600	30
	6.19	6.29 (0.19)	18,000	250	72
	5.07	5.06 (0.16)	6160	560	11
	5.07	5.14 (0.13)	6160	310	20
	5.07	5.16 (0.09)	6160	170	37
	2.54	2.54 (0.01)	560	28	20
Z-A1	8.23	8.09 (0.08)	18,000	600	30
	5.50	5.31(0.02)	2430	122	20
	3.50	3.27 (0.12)	560	28	20
Z-CY	5.27	5.26 (0.10)	18,000	600	30
	4.45	4.31(0.04)	7110	355	20
	2.20	2.10 (0.06)	560	28	20

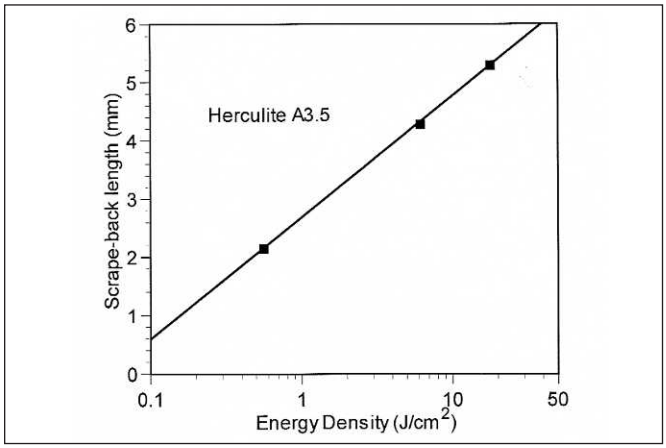


Figure 7: *Predicted scrape back lengths (solid line) as a function of the incident energy density together with experimental values for Herculite A3.5.*

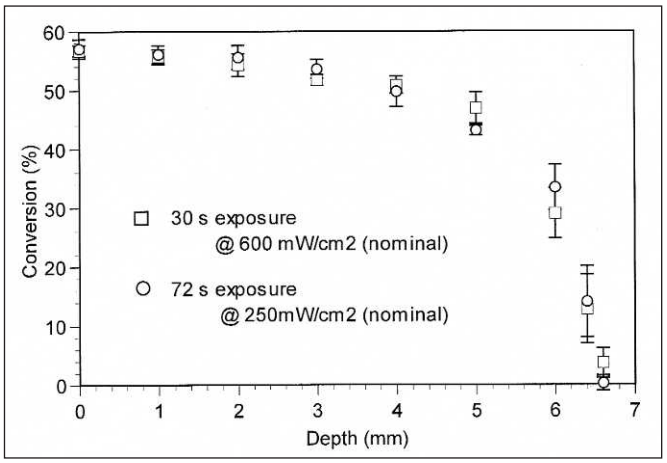


Figure 8: *Conversion profiles for Z100 A3.5 produced from samples exposed with equivalent doses.*

in another study (Watts & Cash, 1994). The effect of shade on attenuation reveals the expected result for Z100, showing progressively decreasing attenuation from the darkest (CY) to the lightest (A1) shade corresponding to a progressive change in opacity. However, shade designations, are not necessarily a predictor of the relative curing potential (Ferracane & others, 1986; Matsumoto & others, 1986). Similar shade designations of various commercial materials may show substantial differences in attenuation and depth of cure due to differences in opacity (Shortall, Wilson & Harrington, 1995) as indicated in Figure 1 for the A3.5 shades of Z100 and Herculite.

The value of the ECR as a predictive tool relies on the dose-dependent conversion and the reciprocal nature of irradiance and exposure time. The dose dependency has previously been described from a kinetic model of the free radical polymerization of methacrylates that relates depth of cure to the product of the intensity and exposure time (Cook, 1980; Cook, 1982). Support for reciprocity in this investigation is noted in the scrape back-values for the A3.5 shade of Herculite and Z100 light-cured with different irradiances and exposure times to yield total exposures of either 18 Jcm^{-2} or 6160 $mJcm^{-2}$ (Table 2), and with depth profiles for Z100 A3.5 shown in Figure 8. The latter compares the data in Figure 1 for Z100 A3.5 with the experimental data of the same material in Figure 4, where a 60% reduction in irradiance has been compensated for with an equivalent increase in exposure time. These results verify similar findings obtained for thin films over a multiple dose range (Halvorson & others, 2002). Additional evidence for the reciprocal irradiance/exposure time relationship has been presented for bulk-polymerized specimens (Nomoto & others, 1994; Miyazaki & others, 1996).

The usefulness of the ECR in predicting conversion profiles for various shades of RBC and various incident-curing exposures is demonstrated by the results shown in Figure 4. The predicted conversion profiles mostly agree with the measured values. An implicit assumption in predicting profiles for various shades of material is that there are no changes in the formulations of

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

monomer content or photoinitiator levels. This is generally a good assumption for commercially available materials.

The extent of cure at the terminus of the scrape-back sample has generally been considered to be significantly less than the maximum attained conversion. In studies characterizing the hardness or conversion profile through RBC, extrapolated depths at zero hardness or conversion were felt to correspond favorably to the length remaining after gently removing the uncured material (Cook, 1980; Nomoto & others, 1994). Under a kinetic model (Cook, 1980), the exposure energy at this depth relates to the minimum energy required to initiate polymerization. However, in this study, the scrape-back length corresponds to approximately 20% conversion and a related unique critical scrape-back energy of 32m Jcm^{-2}. This length obviously does not identify the minimum required polymerization energy. The latter can be identified by the split sample shown in Figure 5, where the sample terminates at a clearly visible delineation. This length corresponds to an energy density of approximately 21 $mJcm^{-2}$ for all the materials investigated. The difference in results between the current and the studies referenced above with respect to the extent of cure at the scrape-back terminus are, perhaps, related to a small inaccuracy in extrapolating conversion to the zero point (Nomoto & others, 1994) and the definition of the scrape-back conversion in the kinetic model (Cook, 1980). It is expected that even with great care to keep it intact, the gelled material, identified in Figure 5, will be readily lost during scrape-back. It is likely that the scrape-back length is determined by a degree of polymerization, where sufficient mechanical properties are developed to resist moderate abrasive forces and, in this study, this is characterized to be about 20-22% conversion. The scrape-back measurements have also demonstrated that the scrape-back length is logarithmically related to the exposure as shown in Figures 6 and 7, where the predicted cures and experimental values agree. This is reflective of the logarithmic attenuation of light intensity and its affect on free radical generation as described by the model referenced above (Cook, 1980).

Though the minimum cure required for maintenance of acceptable clinical performance of an RBC material is not known, a recommendation has evolved, based on comparative analysis of scrape-back, hardness, solubility and sorption measurements (Fan & others, 1986). From these measurements, the depth at one-half the scrape-back length corresponded to the depth at which the relative solubility started to increase and was marginally less than the depth corresponding to 80% of the maximum Knoop Hardness. The current ISO standard also defines an acceptable cure depth as one-half the scrape-back length as measured immediately after curing (International Organization for Standardization, 2000). In this study, this value corresponds to approximately 90% of the maximum measured conversion at 24 hours for the materials investigated. It should be noted that the test method defined by this standard was modified in this investigation to conform to the sample preparation for FTIR analysis. Scrape-back lengths determined using a 4-mm diameter mold as per the standard have been observed to be around 1/2 mm shorter than values described in this report (personal observation). Similar mold effects have been reported previously (Fan & others, 1984). While mold geometry may have an impact on scrape-back length, the cohesion at the scrape-back length is expected to represent a unique conversion independent of mold geometry. As shown, this conversion is approximately 20% for both materials and is expected to be similar for other RBCs formulated with similar chemistry.

Some results of this investigation are expected to depend on certain experimental conditions. Light transmission through the composite is likely to include interactions with the walls of the metal mold. In this investigation, such interactions are equalized by using the same mold materials and geometry throughout. Consequently, predictions from the described ECRs would be accurate only for samples prepared in similar molds; for different molds, new transmission curves would be needed for use with the ECR. The reaction temperature will have an impact on the final conversion attained in free-radical polymerization of RBC (Maffezzoli & others, 1994). Hence, ECRs derived from samples prepared at different temperatures may not correspond to those described here. The impact of different reaction temperatures may be minimized, however, by expressing the conversion relative to the maximum attained conversion as in Figure 3. However, it is expected that conversion at scrape-back will be unaffected by these experimental factors. Finally, ECRs derived from samples cured with plasma arc lamps or devices based on light-emitting diodes may be different due to radiated heat associated with the former and possibly greater polymerization efficiency of the latter.

CONCLUSIONS

This study has shown that an energy-conversion relationship that is predictive of the conversion profiles for a family of RBC materials under variable light-curing conditions can readily be determined. It has further been confirmed that depth of cure is logarithmically related to the energy of exposure and that reciprocity between time and irradiance still exists. From these results it is suggested that scrape-back lengths are correlated with about 20% to 22% conversion.

(Received 6 June 2002)

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

References

Christian GD (1977) *Analytical Chemistry* New York John Wiley & Sons.

Cook WD (1980) Factors affecting the depth of cure of UV-polymerized composites *Journal of Dental Research* **59(5)** 800-808.

Cook WD (1982) Depth of cure in the UV photopolymerization of dimethacrylate-based dental filling materials *Journal of Macromolecular Science-Chemistry* **A17(1)** 99-111.

Cook WD & Standish PM (1983) Cure of resin based restorative materials II White light photopolymerized resins *Australian Dental Journal* **28(5)** 307-311.

Eliades GC, Vougiouklakis GJ & Caputo AA (1987) Degree of double bond conversion in light-cured composites *Dental Materials* **3(1)** 19-25.

de Gee AJ, ten Harkel-Hagenaar E & Davidson CL (1984) Color dye for identification of incompletely cured composite resins *Journal of Prosthetic Dentistry* **52(5)** 626-631.

De Lange C, Bausch JR & Davidson CL (1980) The curing pattern of photo-initiated dental composites *Journal of Oral Rehabilitation* **7(5)** 369-377.

DeWald JP & Ferracane JL (1987) A comparison of four modes of evaluating depth of cure of light-activated composites *Journal of Dental Research* **66(3)** 727-730.

Fan PL, Stanford CM, Stanford WB, Leung R & Stanford JW (1984) Effects of backing reflectance and mold size on polymerization of photo-activated composite resin *Journal of Dental Research* **63(10)** 1245-1247.

Fan PL, Knoeppel R, Kamagai T, Tosaki S, Leung RL & Stanford JW (1986) Composite resin depth of cure parameters: Hardness, sorption, solubility *Journal of Dental Research* **65** Special Issue Abstract #775 p 225.

Ferracane JL, Aday P, Matsumoto H & Marker VA (1986) Relationship between shade and depth of cure for light-activated dental composite resins *Dental Materials* **2(2)** 80-84.

Halvorson RH, Erickson RL & Davidson CL (2002) Energy dependent polymerization of resin-based composite *Dental Materials* **18(6)** 463-469.

International Organization for Standardization (ISO4049:2000E) Dentistry—Polymer-based filling, restorative and luting materials.

Maffezzoli A, Della Pietra A, Rengo S, Nicolais L & Valletta G (1994) Photopolymerization of dental composite matrices *Biomaterials* **15(15)** 1221-1228.

Matsumoto H, Gres JE, Marker VA, Okabe T, Ferracane JL & Harvey GA (1986) Depth of cure of visible light-cured resin: Clinical simulation *Journal of Prosthetic Dentistry* **55(5)** 574-578.

Miyazaki M, Oshida Y, Moore BK & Onose H (1996) Effect of light exposure on fracture toughness and flexural strength of light-cured composites *Dental Materials* **12(6)** 328-332.

Nomoto R, Uchida K & Hirasawa T (1994) Effect of light intensity on polymerization of light cured composite resins *Dental Materials Journal* **13(2)** 198-205.

Ruyter IE & Øysaed H (1982) Conversion in different depths of ultraviolet and visible light activated composite materials *Acta Odontological Scandanavia* **40(3)** 179-192.

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.

Watts DC & Cash AJ (1994) Analysis of optical transmission by 400-500 nm visible light into aesthetic dental biomaterials *Journal of Dentistry* **22(2)** 112-117.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3,** 315-320

Influence of Curing Tip Distance on Resin Composite Knoop Hardness Number, Using Three Different Light Curing Units

DBM Caldas • JB Almeida • L Correr-Sobrinho
MAC Sinhoreti • S Consani

Clinical Relevance

The correlation between light intensity and exposure distance to curing light promoted an adequate resin composite cure. Adequate curing of resin composite is necessary to achieve good mechanical properties and, consequently, a better restoration.

SUMMARY

This *in vitro* study evaluated the influence of curing tip distance on the Knoop Hardness Number (KHN) of a resin composite when using three different light curing units: (1) a halogen light (XL 1500 curing unit–3M), (2) a "softstart-polymerization" (Elipar Trilight curing in an exponential mode-ESPE) and (3) a PAC (Apolo 95E curing unit-DMD). The resin composite, Filtek Z250 (3M), was cured by these curing units at three light-tip distances from the resin composite: 0 mm, 6 mm and 12 mm. The resin composite specimens were flattened to their middle portion and submitted to 18 KHN measurements *per* specimen. The results showed that for the Elipar Trilight unit, the hardness of the resin composite decreased as the light tip distance increased. The XL 1500 unit presented a significant decrease in hardness as the depth of cure of the resin composite increased. Apolo 95E caused a decrease in the resin composite hardness values when the depth of cure and light tip distance increased.

Danilo Biazzetto de Menezes Caldas, DDS, MSD, PhD student, Dental Materials, Piracicaba Dental School, Piracicaba–São Paulo–Brazil

Janaina Bertoncelo de Almeida, DDS, MSD, PhD student, Dental Materials, Piracicaba Dental School, Piracicaba–São Paulo–Brazil

*Lourenço Correr-Sobrinho, DDS, MSD, PhD, titular professor, Dental Materials Department, Piracicaba Dental School, Piracicaba–São Paulo–Brazil

Mário Alexandre Coelho Sinhoreti, DDS, MSD, PhD, associate professor, Dental Materials Department, Piracicaba Dental School, Piracicaba–São Paulo–Brazil

Simonides Consani, DDS, PhD, titular professor, Dental Materials Department, Piracicaba Dental School, Piracicaba–São Paulo–Brazil

*Reprint request: Faculdade de Odontologia de Piracicaba–UNICAMP, Departamento de Odontologia Restauradora–Area Materiais Dentários, Av Limeira, 901, Piracicaba–São Paulo–Brazil, 13414-903; e-mail: sobrinho@fop.unicamp.br

INTRODUCTION

Light curing units have been used since the 1970s, when photo-curing resin composites were introduced to the market. The first resin composites were cured by UV light, however, today, UV light has been replaced with visible light activation. Visible light activating systems are more capable of curing thicker resin layers at depths up to 2 mm (Anusavice, 1996).

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Several light curing unit technologies are available. The conventional light-curing unit, which generates light by a quartz tungsten halogen bulb, is most widely used. Other examples include the plasma arc unit (PAC), which generates light by a gaseous mixture of ionized molecules and electrons, and the stepped-light intensity or "soft-start polymerization" unit, which uses a low initial light output followed by a higher intensity.

An increasing demand by dental professionals, who are requesting faster curing units, has led to the marketing of new models and PAC units with very high light intensity. The PAC units have a narrow wave length range of around 470 nm, and some units have a low energy density that may decrease the resin composite depth of cure (Peutzfeldt, Sahafi & Asmussen, 2000). The use of high intensity light to cure resin composites is universally recommended. A higher degree of conversion is primarily related to curing light intensity and exposure time (Feilzer & others, 1995). However, as the degree of conversion increases, shrinkage augments and, consequently, an increase in gap formation and microleakage is observed. To overcome this problem, the use of a stepped-light intensity has been suggested. Some studies have shown less microleakage and marginal gap, without decreasing monomer conversion, with this method (Bouschlicher, Rueggeberg & Boyer, 2000; Koran & Kürschner, 1998; Yoshikawa, Burrow & Tagami, 2001).

The light-curing unit tip should ideally be in direct contact with the resin composite; however, this is not always clinically possible. In proximal restorations, distances of more than 8 mm between the light tip and the bottom of the proximal cavity have been demonstrated (Hansen & Asmussen, 1997). Sobrinho and others (2000) observed that resin composite Knoop Hardness Number (KHN) decreased when the distance between the curing light tip and the resin composite increased, supporting the law of the Inverse Square (Feilzer & others, 1995).

Thus, this study evaluated the influence of curing tip distance on the Knoop Hardness Number (KHN) of a resin composite when using three different light curing units.

METHODS AND MATERIALS

For purposes of this study, the resin composite Filtek Z-250 (3M, Dental Products Division, St Paul, MN, USA) was used with three light curing units: (1) a conventional unit, XL-1500 (3M); (2) a plasma arc, Apolo 95E (DMD, Fleury d'Aude, France) and (3) a soft-start polymerization unit, Elipar Trilight (ESPE, Dental Medizin-Germany) with an exponential mode. The resin composite was incrementally placed inside a circular split brass mold and covered with a Mylar matrix. The brass mold was 5 mm in diameter and 2.5 mm in depth. Brass jigs were used to position the light-curing tips of each curing unit at 0 mm, 6 mm and 12 mm over the resin composite. Four resin composite specimens were made for each light-curing unit at each curing distance. The light-curing unit XL-1500 used 20 seconds of light exposure as recommended by the resin composite manufacturer. The Apolo 95E unit used three seconds of light exposure, also as recommended. The Elipar Trilight unit used 40 seconds of light exposure, recommended by its manufacturer, when the exponential mode is employed; during the first 16 seconds the light intensity increased exponentially and, in the final 24 seconds, the light intensity was stable at 800 mW/cm^2. The output of each curing unit was checked prior to each procedure. Table 1 lists the curing units and their respective manufacturers. All specimens were prepared in a temperature-controlled room at 23°C by the same operator.

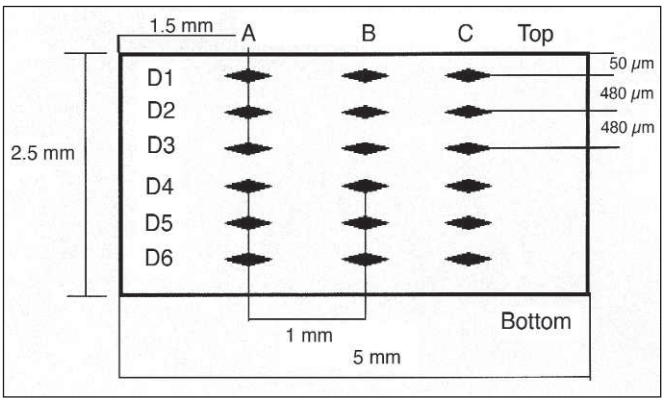


Figure 1: *Schematic representation of the sites evaluated for KHN.*

After the polymerization procedures were completed, the specimens were removed from the brass molds and stored at 37°C and 95±5% relative humidity for 24 hours. They were then placed in a vertical position and embedded in a polyester resin (Resapol T208, São Paulo, SP, Brazil). The cured resin composites were ground and polished to the center of each specimen using 280, 320, 600, 1000 and 1200 grit sandpaper (Norton SA, São Paulo, SP, Brazil) on an automated rotary grinder (Metalserv-Rotary Pregrinder, London, England). The procedures were followed by hand polishing with 3 μm diamond paste (Metadi II Diamond Compound, Buehler, Lake Bluff, IL, USA) mixed with water over a glass slab covered with a soft polishing cloth.

The Knoop Hardness Numbers (KNH) were measured across the section of resin composite using an HMV 2 microhardness tester (Shimadzu Corporation, Japan) in an automatic procedure with a load of 50g applied for 15 seconds. Three measurement positions (A, B and C) were made with six indentations each (D1 to D6) from the top to the bottom of the specimen section, totaling 18 measurements for each specimen. Figure 1 shows this layout.

All statistical analysis was conducted at a significance level of $p<.05$. Two-way analysis of variance (ANOVA)

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

and Tukey's test were performed on hardness data for each depth of cure and light tip distance in each light curing system.

Table 1: *Light Curing Units and Their Respective Manufacturers*

Light Cure Unit	Manufacturer	mW/cm²	Energy Density
Trilight	ESPE, Dental Medizin, Germany	800 mW/cm²	>19.2mJ/cm²
XL-1500	3M, Dental Products Division, St Paul, MN, USA	430 mW/cm²	8.6 mJ/cm²
Apolo 95E	DMD, Fleury d'Aude, France	1370 mW/cm²*	4.1 mJ/cm²

**Manufacturer information*

RESULTS

Table 2 shows the mean Knoop Hardness Numbers (KHN) for the Elipar Trilight curing unit at different depths cured at three distinct distances. The hardness values for the curing tip distance of 0 mm were significantly higher than those obtained at 12 mm for the depths D1, D3 to D6; and the values for the 6 mm distance were significantly higher than 12 mm for the depths D5 and D6 ($p<0.05$). No statistically significant difference in KHN was observed when the cure tip distance of the Elipar Trilight unit was tested at 0, 6 and 12 mm, or at any depth.

Table 2: *Mean Hardness Knoop Number (KHN) and Standard Deviation (SD) for the Light Curing Unit Elipar Trilight at Different Depths of Cure From Three Distances*

Depth	Distance								
	0 mm	SD		6 mm	SD		12 mm	SD	
D1	73.88	0.88	a, A	70.13	1.58	a, AB	63.73	2.75	a, B
D2	75.77	0.68	a, A	74.05	1.26	a, A	70.22	1.03	a, A
D3	78.15	0.32	a, A	74.28	2.47	a, AB	67.72	0.71	a, B
D4	76.94	0.71	a, A	72.95	1.36	a, AB	65.85	1.49	a, B
D5	76.98	0.85	a, A	71.42	1.86	a, A	60.65	2.79	a, B
D6	66.33	3.19	a, A	65.42	4.66	a, A	48.33	4.04	a, B

Means followed by the same small letter in the column and capital letter in the row indicate no statistical difference at the 95% confidence level (Tukey's test, p>0.05).

Table 3: *Mean Hardness Knoop Number (KHN) and Standard Deviation (SD) for the Light Curing Unit XL 1500 at Different Depths of Cure From Three Distances*

Depth	Distance								
	0 mm	SD		6 mm	SD		12 mm	SD	
D1	80.35	1.11	a, A	75.30	3.51	a, A	75.20	2.80	a, A
D2	75.65	0.91	ab, A	73.39	0.78	a, A	73.40	1.17	ab, A
D3	73.50	1.63	ab, A	71.18	0.45	a, A	69.77	1.04	ab, A
D4	72.63	1.04	abc, A	67.77	0.39	a, A	68.77	1.58	ab, A
D5	65.37	3.38	bc, A	66.00	1.25	a, A	60.17	0.69	b, A
D6	61.65	3.35	c, A	52.68	5.65	b, A	25.30	4.38	c, B

Means followed by the same small letter in the column and capital letter in the row indicate no statistical difference at the 95% confidence level (Tukey's test, p>0.05).

Table 4: *Mean Hardness Knoop Number (KHN) and Standard Deviation (SD) for the Light Curing Unit Apolo 95E at Different Depths of Cure From Three Distances*

Depth	Distance								
	0 mm	SD		6 mm	SD		12 mm	SD	
D1	74.32	1.54	a, A	66.70	2.25	ab, B	67.20	3.13	a, B
D2	76.72	0.98	a, A	73.00	2.56	a, AB	66.27	1.59	a, B
D3	79.92	0.82	a, A	69.78	1.80	ab, B	65.48	3.53	a, B
D4	78.35	1.43	a, A	66.33	3.48	ab, B	60.62	4.39	ab, B
D5	73.27	1.55	a, A	60.63	3.62	ab, B	56.50	4.33	ab, B
D6	53.98	4.92	b, A	32.10	2.23	c, B	25.65	2.88	c, B

Means followed by the same small letter in the column and capital letter in the row indicate no statistical difference at the 95% confidence level (Tukey's test, p>0.05).

Table 3 shows the KHN for the XL 1500 curing unit at different depths and three curing distances. The hardness values for the curing tip distance of 0 mm and 6 mm were significantly higher than for the 12-mm distance for the D6 depth. For the D1 to D5 depths, no statistically significant difference was observed. The XL 1500 unit demonstrated significantly higher KHN at D1 depth for 0 mm and 12 mm compared to the D5 and D6 depths ($p<0.05$). For the 6-mm distance, the D1 to D5 depths presented higher KHN than the D6 depth ($p<0.05$).

Table 4 shows the mean KHN for the Apolo 95E curing unit at all depths and three distinct curing distances. The hardness values for a curing tip distance of

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Table 5: *Mean Hardness Knoop Number (KHN) and Standard Deviation (SD) of the Light Curing Units Elipar Trilight, XL 1500 and Apolo 95E at Different Depths of Cure at 0 mm Light Tip Distance*

Depth	Light Curing Units								
	Trilight	SD		XL 1500	SD		Apolo 95E	SD	
D1	73.88	0.88	A	80.35	1.11	A	74.32	1.54	A
D2	75.77	0.68	A	75.65	0.91	A	76.72	0.98	A
D3	78.15	0.32	A	73.50	1.63	A	79.92	0.82	A
D4	76.94	0.71	A	72.63	1.04	A	78.35	1.43	A
D5	76.98	0.85	A	65.37	3.38	B	73.27	1.55	AB
D6	66.33	3.19	A	61.65	3.35	A	53.98	4.92	B

Means followed by the same capital letter in the row indicate no statistical difference at the 95% confidence level (Tukey's test, p>0.05).

Table 6: *Mean Hardness Knoop Number (KHN) and Standard Deviation (SD) of the Light Curing Units Trilight, XL 1500 and Apolo 95E at Different Depths of Cure 6 mm from the Surface of the Resin Composite*

Depth	Light Curing Units								
	Trilight	SD		XL 1500	SD		Apolo 95E	SD	
D1	70.13	1.58	A	75.30	3.51	A	66.70	2.25	A
D2	74.05	1.26	A	73.39	0.78	A	73.00	2.56	A
D3	74.28	2.47	A	71.18	0.45	A	69.78	1.80	A
D4	72.95	1.36	A	67.77	0.39	A	66.33	3.48	A
D5	71.42	1.86	A	66.00	1.25	AB	60.63	3.62	B
D6	65.42	4.66	A	52.68	5.65	B	32.10	2.23	C

Means followed by the same capital letter in the row indicate no statistical difference at the 95% confidence level (Tukey's test, p>0.05).

Table 7: *Mean Hardness Knoop Number (KHN) and Standard Deviation (SD) for the Light Curing Units Trilight, XL 1500 and Apolo 95E at Different Depths of Cure 12 mm From the Surface of the Resin Composite*

Depth	Light Curing Units								
	Trilight	SD		XL 1500	SD		Apolo 95E	SD	
D1	63.73	2.75	B	75.20	2.80	A	67.20	3.13	B
D2	70.22	1.03	A	73.40	1.17	A	66.27	1.59	A
D3	67.72	0.71	A	69.77	1.04	A	65.48	3.53	A
D4	65.85	1.49	A	68.77	1.58	A	60.62	4.39	A
D5	60.65	2.79	A	60.17	0.69	A	56.50	4.33	A
D6	48.33	4.04	A	25.30	4.38	B	25.65	2.88	B

Means followed by the same capital letter in the row indicate no statistical difference at the 95% confidence level (Tukey's test, p>0.05).

0 mm were significantly higher than for the 6 mm distance at the depths D1, D3-to-D6 and also for the 12-mm distance at all depths ($p<0.05$). For all three curing tip distances to the surface, the resin composite polymerized with Apolo 95E had significantly higher KHN for the D1-to-D5 depths than the D6 depth ($p<0.05$).

Tables 5, 6 and 7 show mean KHN for the curing units Elipar Trilight, XL 1500 and Apolo 95E at the different depths cured at three distances: 0, 6 and 12 mm. For a curing tip distance of 0 mm the Elipar Trilight unit demonstrated statistically higher results than the XL 1500 unit for the D5 depth and higher values than Apolo 95E for the D6 depth. For the 6 mm curing tip distance, the Elipar Trilight unit was statistically superior to XL 1500 and Apolo 95E for the D6 depth. For the 12 mm curing tip distance, Elipar Trilight was statistically superior to XL 1500 and Apolo 95E for the D6 depth ($p<0.05$). The curing unit XL 1500 was statistically superior to Elipar Trilight and Apolo 95E for the D1 depth ($p<0.05$).

DISCUSSION

Filtek Z250 (3M) resin composite is the successor to Z100 (3M) resin composite and the manufacturer states that Filtek Z250 is a microhybrid resin composite with the majority of its TEGDMA replaced by UDMA and BIS-EMA. Both are high molecular weight monomers, resulting in the softening of the resin composite and a decreased number of double bond *per* molecular unit, resulting in decreased polymerization shrinkage. In addition, Filtek Z250 resin composite is softened by the smaller standard deviation of the particle size distribu-

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

tion, ranging from 0.01 µm to 3.5 µm, with an average particle size of 0.6 µm (according to the manufacturer) when compared with Z100 resin composite. The decrease in standard deviation of the particle size distribution results in large particle surface areas for wetting by the monomers, leading to a decrease in filler content.

It has been reported that the hardness test provides an estimation of the degree of conversion of a resin composite (Balland, Gukllard & Andre, 1984; Asmussen, 1982; Ferracane, 1985; Rueggeberg & Craig, 1988). The higher the degree of conversion, the better the mechanical properties, hardness, biocompatibility, water sorption, color stability and wear resistance of the resin composites (Bouschlicher & others, 2000; Ferracane & others, 1997; Hinoura & others, 1995). An important aspect for daily base practice is to adjust the time required for a procedure with adequate quality. Three methods of curing are utilized by light curing units, a conventional halogen light, a "soft-start polymerization" and the PAC high intensity light. These methods of curing, with distinct light curing times and intensities, are intended to fulfill the needs of restorative procedures.

In regard to curing a resin composite, aspects such as the blue light spectrum, light intensity, distance of the light source from the resin restorative material and energy density, require special attention. The majority of resin composites employ camphoroquinone as their initiator. Camphoroquinone has a large spectrum of light excitation, ranging from 400 to 500 nm (Anusavice, 1996), however, the most effective band lies between 460 nm and 480 nm, with an optimum at 468 nm (Hofmann & others, 2000). Thus, light cure units with a narrow light spectrum may be outside the range of maximum sensitivity of the camphoroquinone/amine complex, jeopardizing polymerization of some available resin composites (Peutzfeldt & others, 2000).

It has been demonstrated that increased monomer conversion is achieved with high light intensity (Rueggeberg & others, 1993). The light intensity that excites the initiator of a resin composite can be considered equal to that of the light source rationed by the power of two of the distance between the light source and the resin. Therefore, the greater the distance, the weaker the light intensity.

Energy density can be calculated by multiplying the light intensity by the time of light exposure. The higher the energy density, the higher the degree of conversion of a monomer (Peutzfeldt & others, 2000).

It was observed that when using the Elipar Trilight unit the hardness of the resin composite decreased with the increase in light tip distance. However, no effect upon the depth of cure was observed. Most probably, the energy density of this unit was enough to polymerize resin at all tested depths even with the decrease in light intensity resulting from the increase in light tip distance.

XL1500 has a lower energy density than Elipar Trilight, which contributes to the decrease in hardness as the depth increased. However, the three variations in light tip distance had almost no effect on hardness values, which was probably due to the decrease in hardness as the depth increased in a similar way for all three groups.

Apolo 95 E had the lowest energy density of the light-curing units tested and demonstrated a decrease in resin hardness values when the depth of cure and light tip distance increased. The D6 position, at 6 mm and 12 mm, demonstrated the greatest decrease in hardness values, most probably because these areas had the lowest light energy density.

When comparing light curing units at tip distances of 0 mm, 6 mm and 12 mm, levels D5 and D6 at 6 mm and 12 mm had the lowest resin hardness values when XL1500 and Apolo 95E were used. These two light curing units had the lowest light energy densities of the units tested.

On some occasions, it was observed that the D1 depth level had lower hardness values than deeper levels and, occasionally, the lowest hardness values of the group together with the D6-depth level. It may be speculated, as mentioned by Hofmann and others (2000), that the percentage of unreacted double bonds is up to twice as high at the surface protected by a matrix band as compared to the bulk of the material, even when the specimens are prepared in an argon atmosphere. This phenomenon may be explained by the fact that in the bulk of the material, a free radical is surrounded three-dimentionally by possible reaction partners, while a radical located at the surface can find possible reaction partners only at one side. Analysis of polymerized composites by infrared spectroscopy showed the amount of unconverted double bonds to be between 29% and 48%. This conversion is limited to a surface layer of about 20 µm in thickness and usually does not reveal any information as to the quality of the bulk material. At depths greater than 60 µm, the amount of unconverted double bonds ranges from 18% to 32%. Samples containing more than 35% of unconverted double bonds tend to be highly susceptible to discoloration; the removal of these layers improves color stability (Reinhardt, 1991). Thus, hardness is lower at the surface than at depths of 80-100 mm.

Another hypothesis would be that the resin composite organic matrix concentrates at the surface following mylar strip pressure on the specimen or during the packing of the resin composite into the mold. Considering these parameters, the D6 level would be affected. Thus, a clinical situation could occur where

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

the deepest depth level of a resin composite is condensed against a cavity floor, and further investigation is necessary to analyze this possibility and its clinical implications.

Light energy density is fundamental to an adequate depth of cure. XL 1500 and Apolo 95E presented problems related to curing the resin composite at deep levels, 6 and 12 mm light tip distances, when compared to Elipar Trilight. As mentioned, the higher the degree of conversion, the better the mechanical properties, the hardness, the biocompatibility, water sorption, color stability, and wear resistance of a resin composite (Bouschlicher & others, 2000; Ferracane & others, 1997; Hinoura & others, 1995). Considering that the D5 and D6 depth levels were the most affected by decreased hardness and, consequently, monomer conversion, special care must be taken since dental procedures at similar depth conditions and light tip distances may present biocompatibility-related problems due to lower monomer conversion. A solution would be to increase curing time and, consequently, increase the light energy density.

These findings are clinically significant. The unit (Elipar Trilight) with the highest energy density appeared to produce a more homogeneous layer of polymerized resin, considering the depth of cure, even at high light tip distances, suggesting a higher predictability of mechanical properties of the resin composite. Conversely, the layer of resin composite cured by the XL 1500 and APOLO 95E was more heterogeneous, particularly at the greater light tip distances, possibly leading to a decrease in resin composite properties and biocompatibility, as already mentioned.

CONCLUSIONS

Based on the results of this *in vitro* study, it can be concluded:

1. For the Elipar Trilight unit, the hardness of the resin composite decreased as the light tip distance increased.
2. The XL 1500 unit presented a significant decrease in hardness as the depth of cure of the resin composite increased.
3. Apolo 95E caused a decrease in resin composite hardness values when the depth of cure and light tip distance increased.

(Received 6 June 2002)

References

Anusavice KJ (1996) *Philip's Science of Dental Materials* 10th edition Chapter 12 Philadelphia Saunders.

Asmussen E (1982) Restorative resins: Hardness and strength vs quality of remaining double bonds *Scandinavian Journal of Dental Research* **90(6)** 484-489.

Balland D, Gukllard R & Andre JC (1984) Experimental studies and modeling of light distribution in photopolymerizable composite materials *Dental Materials Journal* **3(1)** 93-98.

Bouschlicher MR, Rueggeberg FA & Boyer DB (2000) Effect of stepped light intensity on polymerization force and conversion in a photoactivated composite *Journal of Esthetic Dentistry* **12(1)** 23-32.

Feilzer AJ, Dooren LH, de Gee AJ & Davidson CL (1995) Influence of light intensity on polymerization shrinkage and integrity of restoration-cavity interface *European Journal of Oral Science* **103(5)** 322-326.

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, 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 (1997) Visible-light curing units: Correlation between depth of cure and distance between exit window and resin surface *Acta Odontologica Scandinavica* **55(3)** 162-166.

Hinoura K, Akiyama Y, Miyazaki M, Kuroda T & Onose H (1995) Influence of irradiation sequence on dentin bond of resin inlays *Operative Dentistry* **20(1)** 30-33.

Hofmann N, Hugo B, Schubert K & Klaiber B (2000) Comparison between a plasma arc light source and conventional halogen curing units regarding flexural strength, modulus, and hardness of photoactivated resin composites *Clinical Oral Investigations* **4(3)** 140-147.

Koran P & Kürschner R (1998) Effect of sequential versus continuous irradiation of a light cured resin composite on shrinkage, viscosity, adhesion and degree of polymerization *American Journal of Dentistry* **11(1)** 17-22.

Peutzfeldt A, Sahafi A & Asmussen E (2000) Characterization of resin composites polymerized with plasma arc curing units *Dental Materials* **16(5)** 330-336.

Reinhardt KJ (1991) Unconverted double bonds and interface phenomena in composite materials *Deutsche Zahnarztliche Zeitschrift* **46(3)** 204-208.

Rueggeberg FA & Craig RG (1988) Correlation of parameters used to estimate monomer conversion in a light cured composite *Journal of Dental Research* **67(6)** 932-937.

Rueggeberg FA, Caughman WF, Curtis JW Jr & Davis HC (1993) Factors affecting cure at depths within light-activated resin composites *American Journal of Dentistry* **6(2)** 91-95.

Sobrinho LC, de Lima AA, Consani S, Sinhoreti MA & Knowles JC (2000) Influence of curing tip distance on composite Knoop hardness values *Brazilian Dental Journal* **11(1)** 11-17.

Yoshikawa T, Burrow MF & Tagami J (2001) A light curing method for improving marginal sealing and cavity wall adaptation of resin composite restorations *Dental Materials* **17(4)** 359-366.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

©Operative Dentistry, 2003, **28-3,** 321-323

Clinical Technique/Case Report

Conservative Restoration of Proximal-Cervical Lesions

V Setien • SR Armstrong • MA Vargas

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Clinical Relevance

This technique provides a simplified approach to the conservative treatment of cervical interproximal lesions with a fluoride-releasing restorative.

PURPOSE

Improvements in healthcare and health awareness have lengthened life spans and decreased edentulism, while at the same time presenting significant challenges to the dental community in the form of root caries. The incidence of root caries is dramatically high in spite of the vast amount of preventive tools available to patients and dentists (Cochran & Matis, 2000). Relative to coronal caries, root caries is not as easily diagnosed and treated. Diagnosis, access and isolation for restorative material placement can be extremely difficult, especially in the posterior regions of the mouth. When using the traditional Class II approach to restoring proximal root caries, a significant amount of sound tooth structure may have to be removed.

Glass ionomers (glass ionomer cements and resin-modified glass ionomers) are biocompatible restorative materials that chemically bond to tooth structure while serving as rechargeable fluoride delivery devices (Williams, Billington & Pearson, 2002; Creanor & others, 1995; Forsten, 1995). Recent advances in glass ionomer materials have improved not only the physical properties but also their ease of use. This paper presents an expedient clinical application for applying encapsulated glass ionomer cement when restoring proximal root decay in the presence of the contiguous tooth.

Victor Setien, DDS, MS, PhD, associate professor, Universidad de los Andes, Facultad de Odontologia, Catedra de Operatoria Dental

*Steven R Armstrong, DDS, PhD, assistant professor, Operative Dentistry Department, The University of Iowa, Iowa City, IA

Marcos A Vargas, DDS, MS, associate professor, Operative Dentistry Department, The University of Iowa, Iowa City, IA

*Reprint request: College of Dentistry, S-229 Dental Science Building, Iowa City, IA 52242; e-mail: steven-armstrong@uiowa.edu

DESCRIPTION OF TECHNIQUE

There are several options for restoring posterior interproximal caries: 1) the traditional Class II approach through the occlusal marginal ridge, 2) posterior modification of the traditional anterior tooth Class III approach (Strassler & Buchness, 1990), 3) direct assess during the placement of an adjacent Class II restoration and 4) the tunnel approach (McComb, 2001). Each technique has advantages and disadvantages regarding access for caries removal, placement and finishing of restorative material, conservation of tooth structure and strength of remaining structures and materials. The following technique is recommended when posterior proximal root caries does not involve the proximal contact.

Pre-operatively, the extent of carious involvement should be assessed clinically and radiographically, and the pulpal status should be addressed both subjectively during patient interview and objectively with palpation, percussion and pulp vitality testing. Once it has been determined that the carious lesion does not involve the contact point, and the surrounding structures are sound,

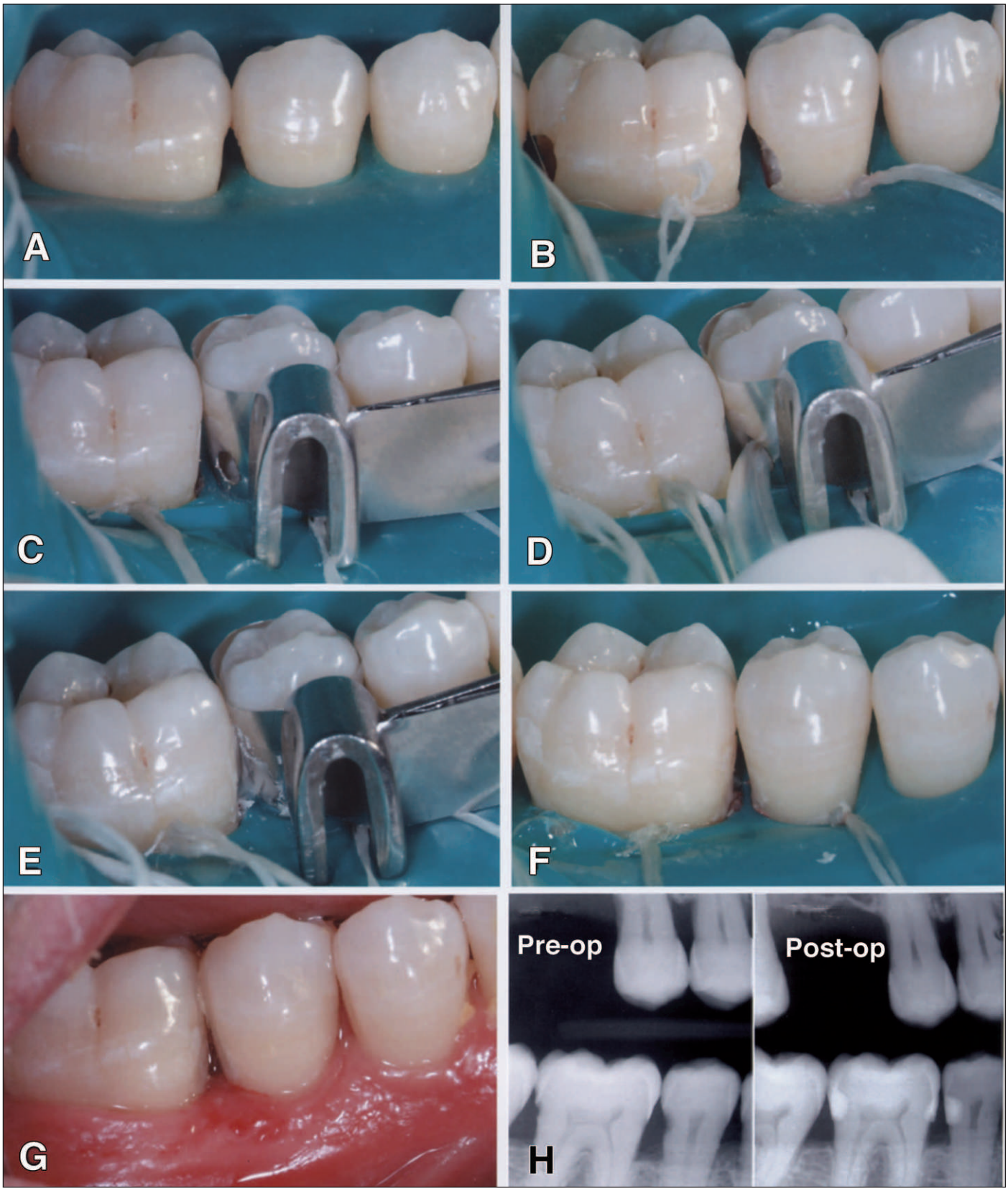


Figure 1. *Figure 1. A. Rubber dam placement (retainer on 2nd molar not visible in photograph). B. Placement of floss ligatures and cavity preparation on tooth #29. C. Matrix in place with access hole. D. Tip of the glass ionomer capsule in position. E. Material placed in preparation. F. Contoured restoration on the first day. G. Finished restoration one week later. H. Pre- and post-operative radiographs.*

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

the following technique should be considered as a viable alternative.

The rubber dam is highly encouraged (Figure 1A) and, often, a floss ligature can aid in the isolation and further displaces the gingival tissues (Figure 1B). If rubber dam isolation is not possible, cotton and retraction cords are indicated. Depending on where the lesion is located, labial or lingual access is begun below the contact point directly over the carious lesion with a small round (#2) or pear-shaped (#329) bur. One may also consider small "safe-sided" cutting instruments, such as the SonicSys (Setien & others, 2001; Liebenberg, 1998). Once access is gained, caries removal continues in the typical manner; however, due to the conservative nature of this approach, careful attention must be given to complete caries removal. The use of caries indicating dyes may be of value (Thomas & others, 2000), and tactile sensation with the side of the explorer rather than the tip is encouraged (Kidd, Joyston-Bechal & Beigton, 1993) or use of a spoon excavator.

Glass ionomers are self-adhesive and the formal placement of retention is usually not necessary; however, if deemed necessary, small retentive points or grooves should be positioned in the gingival and occlusal walls. When caries is removed and the margins sound and no grossly undermined tooth structure remains, the manufacturer's cavity conditioner is used to clean the cavity and remove the smear layer. A Toffelmier #2 metal matrix band (Teledyne Water Pik, Fort Collins, CO) is now placed and isolation is rechecked for completeness. To avoid bonding the glass ionomer to the matrix band while also leaving a protective coating over the material, applying and light-curing a thick coat of adhesive resin to the matrix is recommended. The matrix is placed around the tooth and beyond the gingival margin of the preparation similar to the Class II situation, but with the matrix holder typically opposite the cavity access. At this point a small hole with a high-speed mounted #4 round bur is be made where the access was created initially (Figure 1C) to a size slightly larger than the GIC applicator tip (Figure 1D). If the preparation becomes contaminated with oral fluids during the matrix placement, it is thoroughly rinsed with water and lightly dried with a short blast of air through the matrix access hole. After discarding a small amount of the initial mixed material, the applicator tip is placed in the access hole and filled until GIC is expressed back out of the access hole (Figure 1E). It is important to note that the dispensing tip should not fit snugly to the matrix access opening. This may create too much pressure on the dispensed GIC material and force it to flow beyond the cavity, requiring unnecessary contouring and finishing. After the manufacturer's recommended setting time has elapsed, the matrix is removed and additional protective adhesive resin is applied and light cured. The restoration can be contoured and finished at the same appointment or finishing can be delayed until a subsequent appointment to allow for more maturation time. Surgical blades (#12), flame finishing burs and periodontal scalers can be used to improve contours if gingival excess is present. Finishing strips such as Compo Strip (Premiere Dental Inc, Markhan, Ontario) or Epitex (GC America Inc) can be used to create a smooth surface (Figure 1F-H).

SUMMARY

One of the authors has used this technique successfully for the past three years in various clinical situations that involve both difficult access anterior and posterior teeth. This is a tooth-structure conserving clinical procedure that can provide a simplified approach for restoring otherwise difficult clinical lesions.

(Received 22 October 2002)

References

Cochran MA & Matis BA (2000) Diagnosis and treatment of root caries in Summit JB, Robbins JW & Schwartz RS (eds) *Fundamentals of Operative Dentistry A Contemporary Approach* Chicago Quintessence Publishing Co p 365-376.

Creanor SL, Saunders WP, Carruthers LM, Strang R & Foye RH (1995) Effect of extrinsic fluoride concentration on the uptake and release of fluoride from two glass ionomer cements *Caries Research* **29** 424-426.

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

Kidd EA, Joyston-Bechal S & Beighton D (1993) The use of a caries detector dye during cavity preparation: A microbiological assessment *British Dental Journal* **174** 245-248.

Liebenberg WH (1998) Sonicsys approx: An innovative addition to the restorative continuum *Practical Periodontics & Aesthetic Dentistry* **10** 913-922.

McComb D (2001) Systematic review of conservative operative caries management strategies *Journal of Dental Education* **65** 1154-1161.

Setien VJ, Cobb DS, Denehy GE & Vargas MA (2001) Cavity preparation devices: Effect on microleakage of Class V resin-based composite restorations *American Journal of Dentistry* **14** 157-162.

Strassler HE & Buchness GF (1990) Class III acid-etch light polymerized composite resin restoration [erratum appears in *Journal of Esthethetic Dentistry* 1990 **2(5)** 158.] *Journal of Esthetic Dentistry* **2** 13-16.

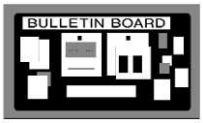
Thomas CC, Land MF, Albin-Wilson SM & Stewart GP (2000) Caries detection accuracy by multiple clinicians and techniques *General Dentistry* **48** 334-338.

Williams JA, Billington RW & Pearson GJ (2002) The glass ionomer cement: The sources of soluble fluoride *Biomaterials* **23** 2191-2200.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

Departments

Announcements



Funding for Students' Research in Operative Dentistry

Students wanting to carry out research related to Operative Dentistry may apply for a Ralph Phillips Research Award, sponsored by the Founder's Fund of the Academy of Operative Dentistry.

The application should consist of a protocol (and 15 copies) outlining the background, aim/hypothesis to be tested, the methodology to be employed, a time schedule and the expected outcome of the study. The protocol should not exceed three double-spaced type-written pages and a budget page (including where the funds should be sent provided the Award is granted). The budget may not exceed $6,000.

If an abstract, based on the research and acknowledging support from the Academy of Operative Dentistry, is accepted for presentation at the IADR/AADR meeting in 2004, additional travel funds not exceeding $1,000 will be made available to the recipient.

A Faculty Advisor should be named, and he/she should co-sign the application. The application must be submitted by December 11, 2003 to:

Academy of Operative Dentistry,
Research Committee
c/o Dr Ivar A Mjör, Chairman
UFCD, Box 100415
Gainesville, FL 32610

Applications may also be submitted by e-mail to: imjor@dental.ufl.edu followed by one signed original mailed to the above address. Award recipients will be announced during the Annual Meeting of the Academy of Operative Dentistry, February 18-20, 2004.

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.

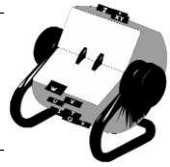
Affiliates: provides links to the American Academy of Gold Foil Operators, the Academy of Operative Dentistry, the AADS-Operative Section, and our Corporate Sponsors. In addition, membership applications for the journal's parent academies are available for downloading.

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: password-protected 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/

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

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.

SDI
SOUTHERN DENTAL INDUSTRIES

Den-Mat®

DENTSPLY
CAULK

BISCO
Specialist In Adhesive and Composite Technology
Bringing Science to the Art of Dentistry™

ULTRADENT

3M ESPE

sds Kerr
SYBRON DENTAL SPECIALTIES

ivoclar vivadent®

Please view the Corporate Sponsors Page at our website (http://www.jopdent.org) for direct links to these companies.

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access

MAY/JUNE 2003 • VOLUME 28 • NUMBER 3 • 213-324

©OPERATIVE DENTISTRY, Inc.

EDITORIAL

BUONOCORE MEMORIAL LECTURE

CLINICAL RESEARCH

LABORATORY RESEARCH

CLINICAL TECHNIQUE/CASE REPORT

DEPARTMENTS

10-9385
Operative Dentistry
Indiana University School of Dentistry, Rm S411
1121 West Michigan Street
Indianapolis, IN 46202-5186 USA

Periodicals

Downloaded from https://prime-pdf-watermark.prime-prod.pubfactory.com/ at 2025-09-01 via free access