OPERATIVE DENTISTRY

May/Jun 2015
Volume 40
Number 3
227–338

Academy of Operative Dentistry • American Academy of Gold Foil Operators • Academy of R.V. Tucker Study Clubs

OPERATIVE DENTISTRY

Volume 40 / Number 3
May/June 2015

www.jopdent.org

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

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 via email at editor@jopdent.org. Payment must be in USD and accompany orders. Online payment by credit card (American Express, Discover, Mastercard, and Visa) is available on our website.

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.

Author Instructions

Please refer to author instructions at www.jopdent.org in the preparation of manuscript submissions and for journal policies.

Journal Policies

The Operative Dentistry Policy Manual which details journal policies, including late fees and claims, is available online at:

https://www.jopdent.com/journal/policies.pdf

Permissions

For permission to reproduce material from Operative Dentistry please apply to Operative Dentistry at the Editorial Office address or via email at editor@jopdent.org.

Online Access

Register for online access, manage subscriptions, save favorite articles and searches, get email alerts, and more at:

http://www.jopdentonline.org/action/registration

Editorial Board

Reviewer names available at: www.jopdent.com/journal/editorial_board.html

We thank all our reviewers for their time and dedication to Operative Dentistry.

On The Cover

Ring of Fire, Bryce Canyon National Park, UT USA. Photo provided by Lawrence Blank of Bethesda, MD USA. Photo taken with a Canon Rebel T1, Tamron 28-300 lens. © Operative Dentistry, Inc.

We welcome the submission of pictures for consideration for use on the cover of Operative Dentistry! All photographs should be submitted via the forms at:

https://www.jopdent.com/journal/journal.html

Editorial Office

The views expressed in Operative Dentistry do not necessarily represent those of the academies or the editors.

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

Editorial Staff

©Operative Dentistry, 2015, **40-3,** 227-234

Clinical Technique/Case Report

Tooth Fragment Reattachment: A Case Report

CMC Taguchi • JK Bernardon • G Zimmermann
LN Baratieri

Clinical Relevance

The reattachment of a fractured tooth fragment offers a viable option for the dental clinician. Function and esthetics may be restored with the use of this conservative and low-cost approach.

SUMMARY

The aim of this article is to present a case report for the multidisciplinary treatment of anterior tooth fractures with invasion of the biologic width and pulpectomy. Successful esthetic and functional results were achieved by bonding the crown fragment, without any form of preparation or the utilization of intra-canal posts.

INTRODUCTION

Crown fractures are the most common consequences of traumatic injuries that mainly occur in the anterior permanent dentition. It is estimated that a quarter of the population suffers a minimum of one dental traumatic injury related to coronal fractures of the anterior teeth before the age of 18 years, the most common of which are attributed to falls, high-impact sports, and motor vehicle accidents.[1, 2]

Most traumatic tooth injuries merely involve damage to the enamel and dentin without pulp exposure.[3] Crown-root fractures represent only 0.3% to 5% of these injuries and require a complex and multidisciplinary treatment.[4,5] Choosing the correct treatment to be followed is based on the age of the dental patient; the extent of the fracture (severity and location of the invasion of the biologic width); the presence or absence of endodontic involvement; the presence/absence of the tooth fragment and its condition of use; the occlusion and esthetics; and time and patient expectations.[6-12] A review of the possible treatments can be found in Table 1.

In the case of fractures that involve invasion of the biologic width, restorations of the biological distance and access to the remnant's margins are required for the purpose of allowing for the correct isolation of the operatory field and determination of the fracture extent.[7,11,13,14] Advantages and disadvantages of the different treatments are listed in Table 2.

The choice of the esthetic restorative treatment of fractured anterior teeth remains the biggest challenge for the dentist. Treatment options include composite resin restoration, fragment reattachment, and ceramic restorations (full crowns, laminate veneers, or ceramic fragments). When the

*Carolina Mayumi Cavalcanti Taguchi, DDS, UFSC Operative Dentistry, Santa Catarina, Brazil

Jussara Karina Bernardon, PhD, UFSC Operative Dentistry, Santa Catarina, Brazil

Gláucia Zimmermann, PhD, UFSC CCS/STM Campus, Santa Catarina, Brazil

Luiz Narciso Baratieri, PhD, UFSC Operative Dentistry, CCS/STM Campus, Santa Catarina, Brazil

*Corresponding author: Rua Almirante Lamengo, 910 ap.1001B, Florianopolis, Santa Catarina 88015-600, Brazil; e-mail: cm.taguchi@gmail.com

DOI: 10.2341/14-034-T

Table 1: *Type of Fractures, Treatment of the Pulp, and Restorative Protocol*

Type of Fracture	Tissue Involved	Pulp Treatment	Restorative Protocol
Enamel fracture	Enamel	None	Protocol 1: incisal edge wear Protocol 2: direct adhesive restoration
Enamel and dentin fracture (without pulp involvement)	Enamel Dentin	None	Protocol 1: direct adhesive restoration Protocol 2: fragment reattachment
Enamel and dentin fracture (with pulp involvement)	Enamel Dentin Pulp	Pulp vitality and root apex in formation 1. Small exposure, up to 2 h after trauma, hemostasis: direct pulp capping 2. Small to medium exposure, more than 2 h after trauma, hemostasis: pulp curettage 3. Large exposure, more than 2 h after trauma, hemostasis: pulpotomy Pulp vitality and root apex formed 1. Presence of hemostasis: pulpotomy 2. Absence of hemostasis: pulpectomy Absence of vitality 1. Pulpectomy	Protocol 1: direct adhesive restoration Protocol 2: fragment reattachment Protocol 3: ceramic restorations (ceramic veneers, fragments, crowns)
Crown-root fracture (with or without invasion of the biologic width)	Enamel Dentin Cementum Periodontal ligament Alveolar bone	Pulp may or may not be involved Pulp involvement: following the above protocol	Protocol 1: direct adhesive restoration Protocol 2: fragment reattachment Protocol 3: ceramic restorations

tooth fragment is present and in good working condition, the best option for the treatment of a coronal fracture fragment is reattachment.[15] Proposed as a simple and conservative option, fragment reattachment restores the morphological, functional, and esthetic aspects of the dentition, while maintaining the shape, contour, texture, color, and alignment of the natural teeth. Furthermore, fragment reattachment can be considered a fast and low-cost treatment solution, creating a positive emotional and psychological response in the patient.[3,4,9,11,16-19]

Table 2: *Treatments of Crown-root Fractures*

Clinical Situation	Type of Treatment	Advantages	Disadvantages
Crown-root fracture without involvement of the biologic width	Gingivectomy: margin exposure with removal of excess gum tissue	Easy to perform Rapid healing	Change in the gingival level and alignment
	Gingival flap: margin exposure without removing gum tissue	Facilitates isolation of the operative field Easy to perform Rapid healing	Gingival alteration in the esthetic area
Crown-root fracture with involvement of the biologic width	Flap technique: gingival flap displacement + osteotomy and osteoplasty	Safe and effective technique	Compromised bone support of the adjacent tooth Increased clinical crown Reducing the cervical diameter
	Extrusion dental: orthodontic extrusion of the apical portion until the fracture margin is exposed	No change in the gingival level and alignment No removal of bone tissue	Slow technique Stabilization time Appearance of black space Edge wear to incisal length adjustment Loss of shape and optical characteristics of the tooth
	Flapless technique: osteotomy without gingival flap	Safe and effective technique Rapid healing	Change in the gingival level and alignment

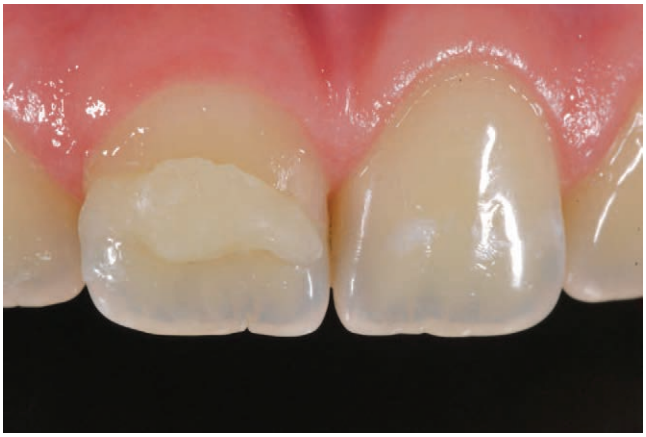

Figure 1. *Initial view of the fractured element.*

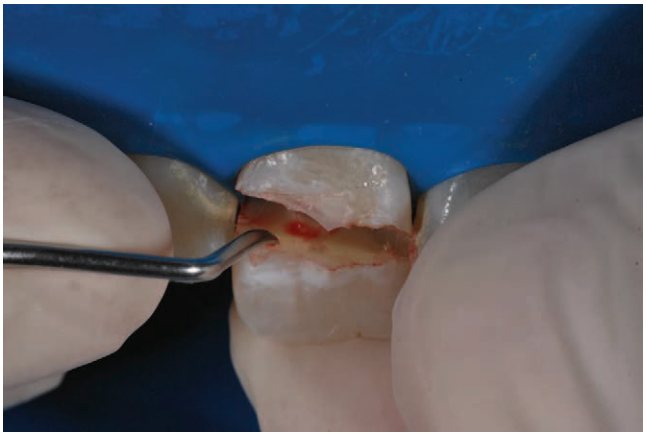

Figure 2. *Fragment dislocation, followed by bleeding.*

This technique was first published in 1964, when Chosak and Eidelman described a case involving the reattachment of a natural tooth fragment. Since then, different preparation techniques (bevel, circumferential chamfer, buccal chamfer, overcontour, internal dentin groove) as well as adhesive materials have been described throughout the literature, designed to increase the chemical and mechanical retention of fragments.[20,21] However, dentists are still seeking consensus over which preparation method and which materials are best to achieve the best results using the fragment reattachment technique.[22]

The following case report describes the management of a crown-root fracture of a maxillary central incisor treated in a multidisciplinary manner, in which the dental fragment was used as the main restorative material.

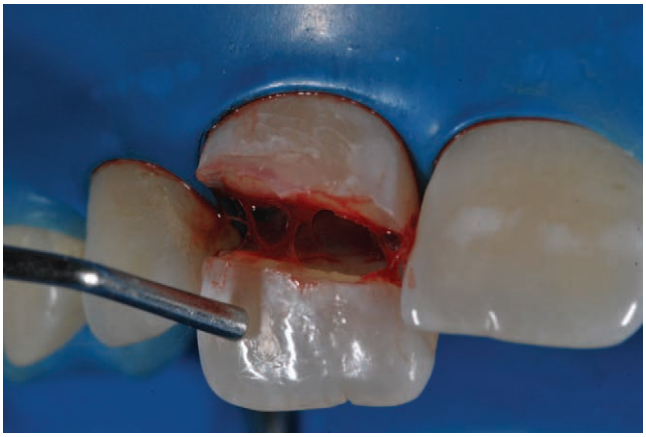

Figure 3. *Fragment retained by palatal gingival tissue.*

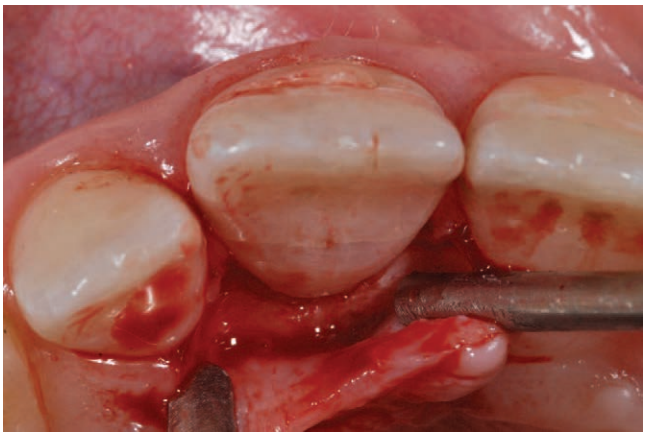

Figure 4. *Gingival flap allowing visualization of the fracture line.*

CLINICAL CASE REPORT

A 21-year-old patient presented to the clinic with a coronal fracture of the maxillary central incisor caused by a domestic fall. Through clinical evalua-

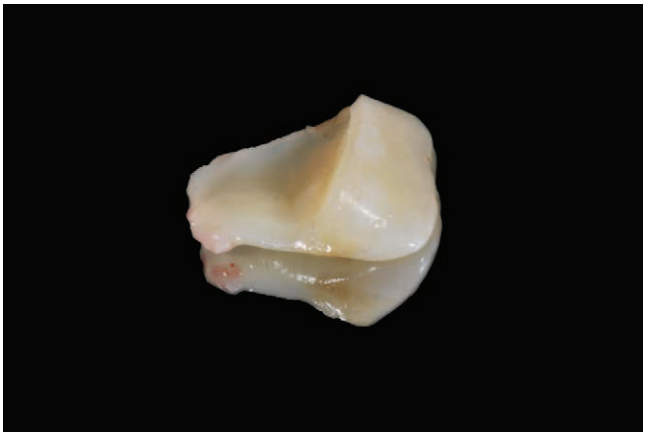

Figure 5. *Dental fragment.*

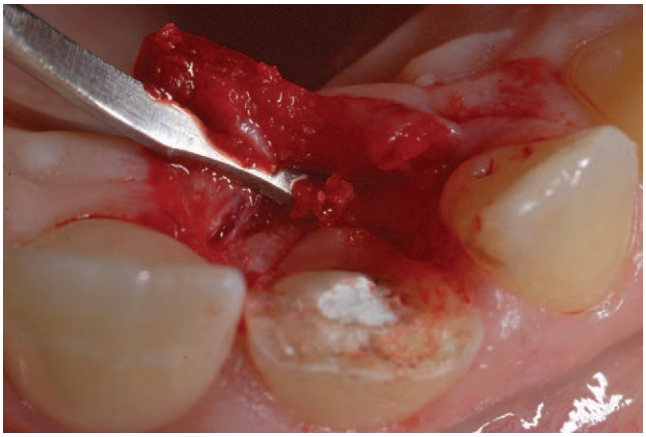

Figure 6. *Osteotomy and osteoplasty of the palatal region.*

tion it was observed that the dental fragment was in position, stabilized by an increment of composite resin that had been placed by a dentist in an emergency office. There were no signs of pulp involvement, and the fracture line was subgingival (Figure 1). Under rubber dam isolation, the composite resin was removed and the tooth fragment was displaced, causing pulp exposure followed by bleeding (Figure 2). During its removal, it was observed that the fragment was retained by the palatal gingival tissue (Figure 3). Thus, it was necessary to perform an intrasulcular incision followed by a gingival flap in order to remove the fragment (Figure 4). The tooth fragment was then frozen in distilled water during the clinical examination, for a period of about six hours (Figure 5). When exposing the fracture margin it was observed that the location of the fracture line was located intraosseously, invading the biological space. Therefore, it was necessary to perform osteotomy and osteoplasty of the palatal region, removing approximately 1 mm of bone tissue (Figure 6).

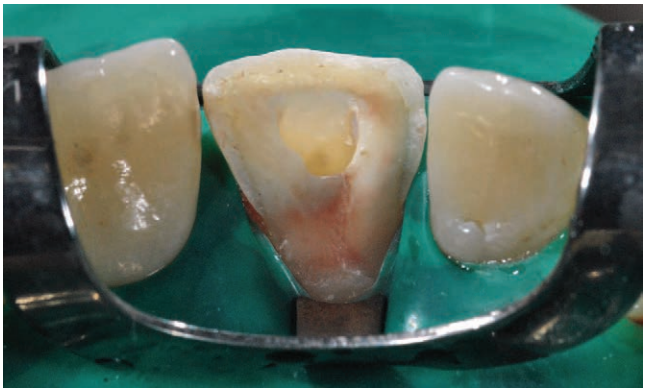

Figure 7. *Isolation of the operative field.*

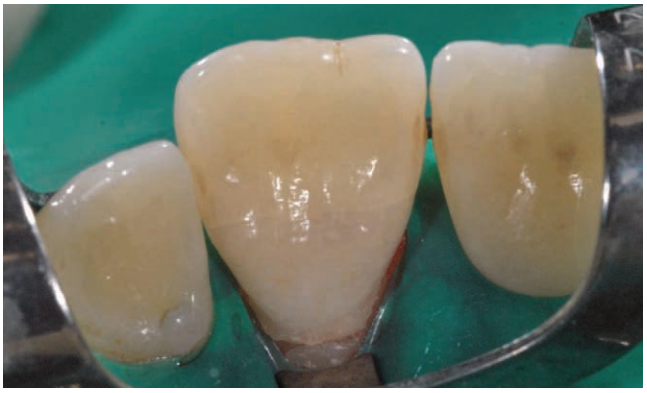

Figure 8. *Dental fragment repositioned and exhibiting excellent adaptation.*

Rubber dam isolation was performed using a #212 retraction clamp in order to expose the fracture margin and to keep the area clean and dry, providing favorable conditions for the restorative treatment (Figure 7). The clinician opted to perform an invasive procedure of the pulp (pulpectomy) followed by a single-session endodontic treatment. The selection of a less invasive treatment would prejudice the process of the functional recovery of dental pulp.

The tooth fragment was thawed in water for 30 minutes before being repositioned. After repositioning the tooth fragment it was possible to observe excellent adaptation. Considering this result, the best treatment choice was the fragment reattachment technique (Figure 8). The tooth fragment was positioned and stabilized with an increment of composite resin and then the adjacent teeth were isolated with petroleum jelly. Lastly, an acrylic resin guide was fabricated (Figure 9). This technique allows for the correct insertion and cementation of

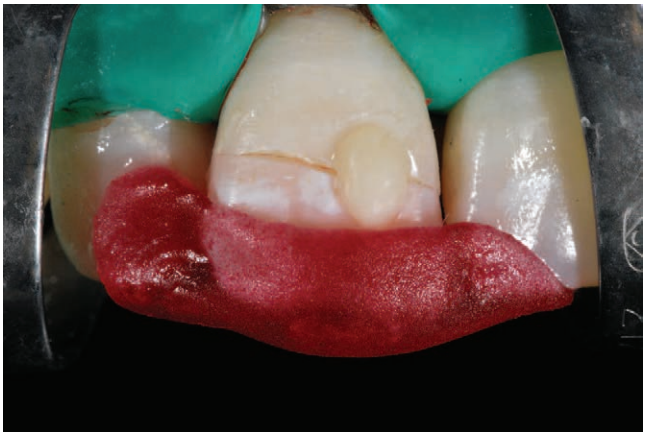

Figure 9. *Acrylic guide made with the fragment in position.*

Table 3: *Materials Used*	
Rubber dam	SSWhite (Rio de Janeiro, Brazil)
#212 Retractor clamp	SSWhite (Rio de Janeiro, Brazil)
DuraLay (acrylic resin)	Reliance Dental Mfg (Worth, IL, USA)
Phosphoric acid 37% (etching gel)	BM4 (Florianópolis, SC, Brazil)
Adper Single Bond (adhesive)	3M ESPE (St Paul, MN, USA)
Empress Direct (composite resin)	Ivoclar Vivadent (São Paulo, Brazil)
Variolink Veneer (resin cement)	Ivoclar Vivadent (São Paulo, Brazil)
Bluephase (LED unit)	Ivoclar Vivadent (São Paulo, Brazil)
Sof-lex (polishing discs)	3M ESPE (St Paul, MN, USA)
Diamond Flex (felt disc)	FGM (Joinville, SC, Brazil)
Diamond Excel (polishing paste)	FGM (Joinville, SC, Brazil)

the fragment, facilitating its manipulation and adaptation. The fragment was etched with 37% phosphoric acid beyond the margins for 15 seconds and rinsed with air/water spray. After being dried, two layers of the adhesive system were applied and thinned with air jets. The fragment was preserved, without light activation, protected from the ambient light.

The tooth and the root canal were both etched with 37% phosphoric acid for 15 seconds. After being rinsed for 30 seconds, the enamel surface was left completely dry, while dentin was left slightly moist. Two layers of an adhesive (Table 3) were applied and mild air jets were applied until a shiny appearance was observed on the uncured surface. After light curing the adhesive, the root canal opening was sealed with an increment of composite resin in close contact with the filling material, without interfering with the repositioning of the fragment (Figure 10).

A small amount of dual resin cement was applied over the whole surface of the tooth fragment. Then it was correctly positioned with the aid of the acrylic guide (Figure 11). After the excesses were removed, the resin cement was light cured for 20 seconds using an LED unit (900 mW/cm^2 output). The guide was then removed and a final light curing was performed for 60 seconds on each aspect of the tooth. Finishing and polishing of the buccal and palatal surface were carried out with abrasive discs, felt discs, and polishing pastes. After the removal of the rubber dam, the gingival flap was repositioned and the papillae sutured (Figure 12).

In a follow-up clinical evaluation conducted four months after the trauma, the fracture line was not visibly observable and satisfactory periodontal health was exhibited (Figure 13).

POTENTIAL PROBLEMS

The development of adhesive restorative materials has provided new perspectives for the treatment of fractured teeth. Common restorative treatments, such as ceramic laminates or crowns, tend to sacrifice large amounts of tooth structure, making the color matching to the adjacent teeth difficult.[23] The variety of materials, such as adhesive systems

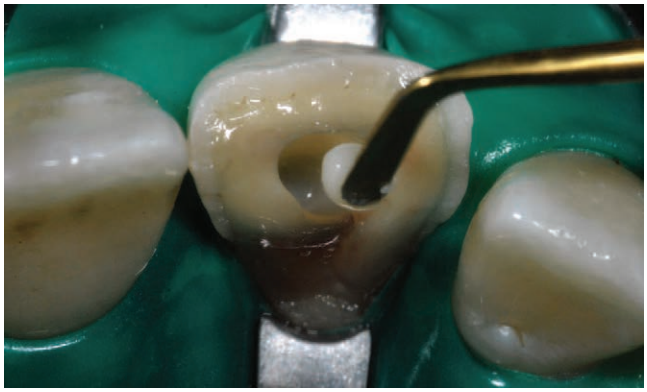

Figure 10. *Root canal opening being sealed.*

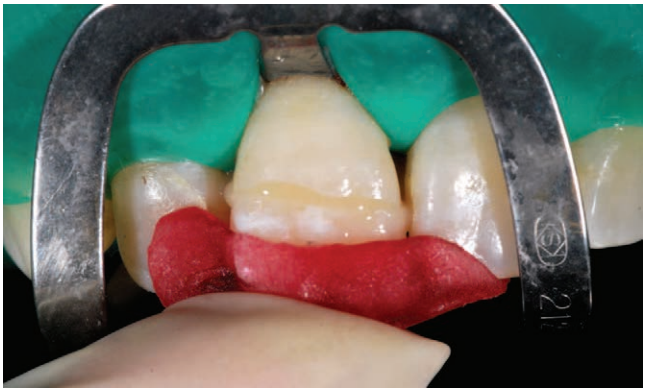

Figure 11. *Fragment reattachment.*

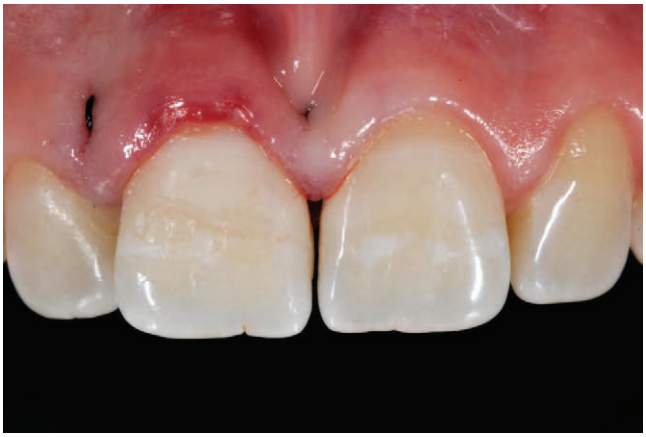

Figure 12. *View after the papillae were sutured.*

and composite resins, combined with the skill and knowledge required to mimic the shape, color, and texture of a tooth make the realization of direct composite resin restorations difficult.[8] Thus, fragment reattachment becomes a fast, simple, and conservative technique that provides excellent rehabilitation of the esthetics and function.[16] However, in order to make feasible the most favorable long-term results, the dental clinician needs to have knowledge of the materials and follow the correct treatment protocol by first performing periodontal procedures, followed by the endodontic treatment, and, lastly, by facilitating fragment reattachment.[14]

Dehydration of the fragment may result in a change in dental color and a decrease in the fracture strength of the tooth. Proper rehydration of the fragment has the capability of restoring both color and strength.[8,12] Farik and others[24] evaluated the fracture resistance of dehydrated and rehydrated teeth over different periods of time. When the fragment remains dehydrated for more than one hour, the fracture resistance decreases significantly. In the same study, the authors observed that when the specimen remains dehydrated for more than 24 hours, the optimal rehydration time given was a period equal to, but not less than, 24 hours. This will ensure the maintenance of the adhesive strength. Similar results were observed by Shirani and others[25] in their latest study. The authors evaluated the adhesive strength of dental fragments dehydrated for different time periods with posterior rehydration time periods of 30 minutes or 24 hours. They concluded that the final results were independent of the dehydration time and that 24-hour rehydration periods result in greater adhesive strength when compared to the 30-minute rehydration periods.

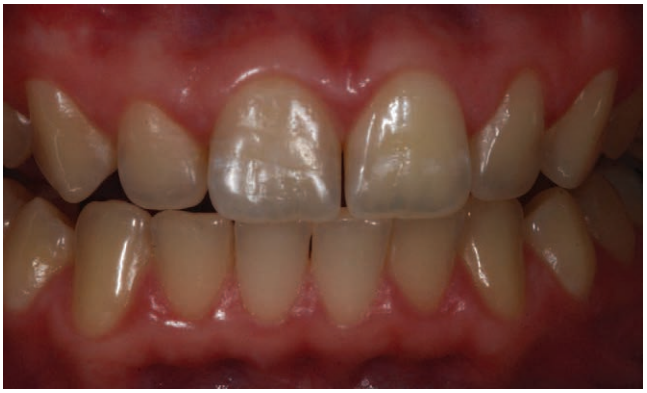

Figure 13. *Clinical view four months after the trauma.*

However, when the fragment dehydration time is 30 minutes or less, rehydration for 30 minutes is sufficient enough to obtain significant improvement of the adhesive strength.[25]

When endodontic treatment is indicated, the use of intraradicular posts, with the aim of reinforcing the tooth structure, becomes dubious. A recent study[21] evaluating the fracture strength of three fragment bonding techniques associated or not associated with fiber glass posts concluded that the use of intraradicular posts in endodontically treated teeth did not provide any reinforcement of the dental structure, making it unnecessary when the bonding technique is chosen.

There is not any consensus in the literature regarding the realization of any type of preparation and regarding the long-term effectiveness of the strengthening of the dental structure after fragment reattachment. It has been found that making a preparation increases the fracture strength of the tooth when compared to direct bonding without any type of preparation.[20,21,26-28] However, neither direct bonding nor the use of preparations reaches the initial fracture strength of the dental element. In addition, it has been argued that the adhesive is ultimately responsible for the bond strength of the fragment to the tooth and that the preparation is less important.[22,29] In the case presented, the fragment was well adapted, and for this reason there was not any additional preparation and the retention of the fragment was achieved only through hybridization of the dental tissue. We opted for the use of resin cement and not composite resin, considering that the shade, viscosity, and dual-cure mechanism of these cements facilitate the insertion and polymerization, while the innermost portions of the luting interface may not be light cured. If the fragment does not exhibit good

adaptability and if there is a loss of continuity, composite resin is indicated for filling any volume loss.

It is the dentist's responsibility to undertake periodic follow-up consultations and to perform clinical, radiographic, and periodontal examinations as well as pulp vitality tests in order to ensure the integrity, the esthetics, and the functional health of the fractured element.[6]

Advantages

The advantages of fragment reattachment are as follows:

- It offers faithful reproduction of the shape, contour, and texture of the natural tooth;
- It offers unchanged color and optical characteristics; and
- It is a predictable, quick, conservative, and low-cost method.

Disadvantages

The disadvantages of fragment reattachment are as follows:

- It may result in a change in color due to inadequate rehydration of the fragment; and
- It carries the possibility of detachment of the fragment.

CONCLUSION

- Complex coronary fractures require a specialized interdisciplinary treatment and must be carefully assessed by the dental clinician to achieve the best possible outcome.
- Bearing in mind that it is a simple, fast, affordable, and esthetically predictable technique, tooth fragment reattachment should always be the treatment method of choice when the fragment is present and is in good condition, even if a perfect adaptation is not observable.

Human Subjects Statement

This work was completed at the Universidade Federal de Santa Catarina, Campus Universitário Trindade, Florianópolis, Brazil.

Conflict of Interest

The authors have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

(Accepted 8 August 2014)

REFERENCES

1. Andreasen JO (1970) Etiology and pathogenesis of traumatic injuries. A clinical study of 1,298 cases *Scandanavian Journal of Dental Research* **78(1-4)** 329-342.
2. Andreasen JO, Andreasen FM, & Andersson L (2007) *Textbook and Color Atlas of Traumatic Injuries to the Teeth* Blackwell, Oxford, UK.
3. Badami V, & Reddy SK (2011) Treatment of complicated crown-root fracture in a single visit by means of rebonding *Journal of the American Dental Association* **142(6)** 646-650.
4. Olsburgh S, Jacoby T, & Krejci I (2002) Crown fractures in the permanent dentition: Pulpal and restorative considerations *Dental Traumatology* **18(3)** 103-115.
5. Terry DA (2003) Adhesive reattachment of a tooth fragment: The biological restoration *Practical Procedures & Aesthetic Dentistry* **15(5)** 403-409.
6. Baratieri LN, Monteiro S Jr, Andrada MAC, Vieira LCC, Cardoso AC, & Ritter AV (1995) *Estética: Restaurações Adesivas Diretas em Dentes Anteriores Fraturados* Quintessence, São Paulo, Brazil.
7. Chu FC, Yim TM, & Wei SH (2000) Clinical considerations for reattachment of tooth fragments *Quintessence International* **31(6)** 385-391.
8. Maia EA, Baratieri LN, de Andrada MA, Monteiro S Jr, & de Araújo EM Jr (2003) Tooth fragment reattachment: Fundamentals of the technique and two case reports *Quintessence International* **34(2)** 99-107.
9. Macedo GV, Diaz PI, de O Fernandes CA, & Ritter AV (2008) Reattachment of anterior teeth fragments: A conservative approach *Journal of Esthetic and Restorative Dentistry* **20(1)** 5-20.
10. Macedo GV, & Ritter AV (2009) Essentials of rebonding tooth fragments for the best functional and esthetic outcomes *Journal of Pediatric Dentistry* **31(2)** 110-116.
11. de Castro JC, Poi WR, Pedrini D, Tiveron AR, Brandini DA, & de Castro MA (2011) Multidisciplinary approach for the treatment of a complicated crown-root fracture in a young patient: A case report *Quintessence International* **42(9)** 729-735.
12. Krastl G, Filippi A, Zitzmann NU, Walter C, & Weiger R (2011) Current aspects of restoring traumatically fractured teeth *European Journal of Esthetic Dentistry* **6(2)** 124-141.
13. Baratieri LN, Monteiro S Jr, Cardoso AC, & de Melo Filho JC (1993) Coronal fracture with invasion of the biologic width: A case report *Quintessence International* **24(2)** 85-91.
14. Lise D, Vieira LC, Araújo E, & Lopes G (2012) Tooth fragment reattachment: The natural restoration *Operative Dentistry* **37(6)** 584-590.
15. Alvares I, Sensi LG, Araujo EM Jr, & Araujo E (2007) Silicone index: An alternative approach for tooth fragment reattachment *Journal of Esthetic and Restorative Dentistry* **19(5)** 240-246.

16. Baratieri LN, Monteiro S Jr, & de Andrada MA (1990) Tooth fracture reattachment: Case reports *Quintessence International* **21(4)** 261-270.

17. Vâlceanu AS, & Stratul SI (2008) Multidisciplinary approach of complicated crown fractures of both superior central incisors: A case report *Dental Traumatology* **24(4)** 482-486.

18. Kina M, Ribeiro LG, Monteiro S Jr, & de Andrada MA (2010) Fragment bonding of fractured anterior teeth: Case report *Quintessence International* **41(6)** 459-461.

19. Ojeda-Gutierrez F, Martinez-Marquez B, Rosales-Ibanez R, & Pozos-Guillen AJ (2011) Reattachment of anterior teeth fragments using a modified Simonsen's technique after dental trauma: Report of a case *Dental Traumatology* **27(1)** 81-85.

20. Reis A, Francci C, Loguercio AD, Carrilho MR, & Rodriques Filho LE (2001) Re-attachment of anterior fractured teeth: Fracture strength using different techniques *Operative Dentistry* **26(3)** 287-294.

21. Loguercio AD, Leski G, Sossmeier D, Kraul A, Oda M, Patzlaff RT, & Reis A (2008) Performance of techniques used for re-attachment of endodontically treated crown fractured teeth *Journal of Dentistry* **36(4)** 249-255.

22. Pusman E, Cehreli ZC, Altay N, Unver B, Saracbasi O, & Ozgun G (2010) Fracture resistance of tooth fragment reattachment: Effects of different preparation techniques and adhesive materials *Dental Traumatology* **26(1)** 9-15.

23. Murchison DF, Burke FJ, & Worthington RB (1999) Incisal edge reattachment: Indications for use and clinical technique *British Dental Journal* **186(12)** 614-619.

24. Farik B, Musksgaard EC, Andreasen JO, & Kreiborg S (1999) Drying and rewetting anterior crown fragments prior to bonding *Endodontics and Dental Traumatology* **15(3)** 113-116.

25. Shirani F, Malekipour MR, Manesh VS, & Aghaei F (2012) Hydration and dehydration periods of crown fragment prior to reattachment *Operative Dentistry* **37(5)** 501-508.

26. De Santis R, Prisco D, Nazhat SN, Riccitiello F, Ambrosio L, Rengo S, & Nicolais L (2001) Mechanical strength of tooth fragment reattachment *Journal of Biomedical Materials Research* **55(4)** 629-636.

27. Demarco FF, Fay RM, Pinzon LM, & Powers JM (2004) Fracture resistance of re-attached coronal fragments—Influence of different adhesive materials and bevel preparation *Dental Traumatology* **20(3)** 157-163.

28. Bruschi-Alonso RC, Alonso RC, Correr GM, Alves MC, Lewgoy HR, Sinhoreti MA, Puppin-Rontani RM, & Correr-Sobrinho L (2010) Reattachment of anterior fractured teeth: Effect of materials and techniques on impact strength *Dental Traumatology* **26(4)** 315-322.

29. Rajput A, Ataide I, Lambor R, Monteiro J, Tar M, & Wadhawan N (2010) In vitro study comparing fracture strength recovery of teeth restored with three esthetic bonding materials using different techniques *European Journal of Esthetic Dentistry* **5(4)** 398-411.

©Operative Dentistry, 2015, **40-3,** 235-240

White Diet: Is It Necessary During Tooth Whitening?

BA Matis • G Wang • JI Matis
NB Cook • GJ Eckert

Clinical Relevance

Ingestion of coffee/tea during bleaching did not minimize the effect of tooth whitening. Subjects who drank large amounts of coffee/tea had a greater effect of bleaching because their teeth were initially darker. Ingestion of red wine/dark fruit did not limit the effect of tooth whitening.

SUMMARY

Patients are sometimes blamed for a reduced effect of bleaching when they do not adhere to a dentist's prescribed white diet. This study aimed to determine whether a white diet is necessary by evaluating the effects of coffee, tea, wine, and dark fruits on the potential tooth whitening during the bleaching process. Each of the effects of discoloration was categorized as "yes" or "no" based on a patient questionnaire. Data from five published studies were included in the analyses. Outcomes were based on the color change between baseline and the end of bleaching. The relationships between color changes were measured subjectively and objectively. A nonwhite diet was not significantly associated with less tooth whitening, and there was only a weak positive association between tooth whitening and diet for subjects who drank large amounts of coffee/tea.

Bruce A. Matis, DDS, MSD, Indiana University School of Dentistry, Department of Restorative Dentistry, Indianapolis, Indiana

*Ge Wang, DDS, MD, PhD, Hubei-MOST KLOS & KLOBM, School & Hospital of Stomatology, Wuhan University, Wuhan, China

Jeremy I. Matis, DDS, US Air Force, Eielson Air Force Base, Alaska

N. Blaine Cook, DDS, MSD, Indiana University School of Dentistry, Department of Restorative Dentistry, Indianapolis

George J. Eckert, MAS, Indiana University School of Medicine, Department of Biostatistics, Indianapolis

*Corresponding author: 237 Louyo Rd. Wuhan, Hubei 430079 China; e-mail: wanggeinus@aliyun.com

DOI: 10.2341/14-019-LIT

INTRODUCTION

Cosmetic dentistry has become a very important part of today's restorative dental practice. The esthetic appearance of patients' teeth is very important to them; as white teeth are believed to be associated with health and beauty. Cosmetic procedures have become more available because of improved standards of living. Although more patients are demanding esthetic treatments, it is the responsibility of dentists to offer treatments to help patients safely achieve their goals.[1]

Dentists often instruct patients to refrain from smoking and drinking coffee, tea, or red wine during the active bleaching procedures, as some manufacturers ask patients to stay on a white diet during that time. However, no current clinical studies have determined whether refraining from these substanc-

Table 1: *Inclusion and Exclusion Criteria*
Inclusion Criteria
• Patient has all six maxillary anterior teeth.
• None of the maxillary anterior teeth has more than one-sixth of the labial surfaces of the natural tooth covered with a restoration, and the location must not interfere with colorimeter placement.
• All six anterior teeth must be darker than B54 and lighter than B85 on the Trubyte Bioform Color Ordered Shade Guide.
• None of the maxillary anterior teeth is excessively rotated, such as mesiorotation or distorotation, which interferes with colorimeter placement
• Patient is willing to sign a consent form.
• Patient is at least 18 years old.
• Patient is able to return for periodic examinations.
Exclusion Criteria
• A history of any medical condition that may interfere with the study and other conditions left up to the judgment of the principal investigator.
• Patient used professionally applied or prescribed tooth whiteners, whether in-office or at-home, in the preceding five years.
• Gross pathology in the oral cavity (excluding caries).
• Gingival index score greater than 1.0.
• Intrinsic discolored teeth due to tetracycline staining.
• Pregnant or lactating women.

es during the process of tooth whitening is necessary.

Many people drink coffee, tea, and red wine and eat dark-colored fruit as a part of daily life. Some investigators have reported that coffee, tea, and wine can lead to tooth discoloration.[2-4] In an *in vitro* study, Attia and others[5] found that the stability of a dental whitening treatment could be compromised by the use of coffee during home bleaching procedures. Gerlach and Zhou[6] recognized that drinkers of coffee and tea may require a specialized post-treatment maintenance plan. However, it is not known if the patients' behavior influences the effectiveness and stability of dental whitening during bleaching or if the dentist should recommend restricting the consumption of coffee, tea, wine, or dark fruit during the tooth whitening process.

This study addresses those questions with a review of five *in vivo* studies where patients responded to a questionnaire regarding their ingestion of coffee, tea, red wine, and dark fruit during tooth whitening. The objective was to determine if a patient should restrict the consumption of coffee, tea, red wine, and dark fruit during tooth whitening. The reviewed studies were conducted at the same facility by the same faculty, and they have been previously reported in the scientific literature.

METHODS AND MATERIALS

This study is based on five published *in vivo* studies[7-11] in peer reviewed publications that include a total of 185 subjects. The studies were approved by the Institutional Review Board at Indiana University Purdue University Indianapolis. The studies used different products and methods and came to different conclusions regarding color change. In each study, the manufacturers' instructions for handling and use were followed for each product. Four of the five studies were half-mouth design studies; the remaining study used parallel groups. All studies, except one, had the same inclusion and exclusion factors (Table 1), which required the lightest color tooth in the maxillary arch to be at least a B65 shade on the Trubyte Bioform Color Ordered Shade Guide (Dentsply International, York, PA, USA), which equates to an A-3 on the Vita Classical Shade Guide (Vita Zahnfabrik, Bad Sackingen, Germany). One study evaluating over-the-counter tooth whitening products did not exclude smokers from participating;[7] however, because of the small number of subjects, smoking was not evaluated in this study. All five studies asked questions regarding the number of cups of coffee or tea and the number of glasses of red wine each participant drank. Four of the studies asked about the number of servings of dark-colored fruits (blueberries, blackberries) consumed during the study.

In all of the studies, color was evaluated both subjectively and objectively. In four studies, a faculty member who is experienced in color matching used the Trubyte Bioform Color Ordered Shade Guide to evaluate color. The fifth study used a Vita Classical Shade Guide; the results of that study were mapped to the Trubyte shade guide for comparison purposes in the current study. The color evaluation was performed in an area that was shielded from direct sunlight and lit using color-corrected overhead lighting. The objective evaluation of color in all of the studies was accomplished using a colorimeter (Chroma Meter CR 321, Minolta, Osaka, Japan) that was calibrated to a color standard.

The colorimeter measured the color of the teeth based on the CIE L*a*b* color space system. This system, defined by the International Commission on Illumination in 1978, is referred to as CIELAB.[12] L* represents the value (lightness or darkness), a* is the measurement along the red-green axis, and b* is the measurement along the yellow-blue axis. A

Table 2: *Mean (Standard Deviation) and Range Responses to Dietary Questions*

Study (Ref. No.)	N	Cups Coffee/Tea per Day	Red Wine/Dark Fruit per Week	White Diet (%)
All	185	1.5 (1.8), 0-10	0.9 (1.6), 0-8	30 (16)
1 (7)	75	0.5 (0.6), 0-3	0.8 (1.6), 0-8	14 (19)
2 (8)	32	2.9 (2.3), 0-10	1.3 (1.9), 0-7	2 (6)
3 (9)	36	2.0 (2.1), 0-8	0.9 (1.7), 0-7	6 (17)
4 (10)	19	1.2 (1.0), 0-3	1.1 (2.1), 0-7	3 (16)
5 (11)	23	2.0 (1.7), 0-5	0.2 (0.4), 0-1	5 (22)

positive L* indicates a lighter color tooth. A negative a* indicates a decrease in the intensity of red, and a negative b* indicates a decrease in the intensity of yellow. Total color differences, or distances between two colors (ΔE), were calculated at the end of each study using the following formula: $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.[12] This formula is only valid for increased lightness of tooth color if the L* value increases and a* and b* are in the red and yellow colors, respectively, and decrease in number.

No restrictions on dietary habits were imposed during the course of the studies. Each subject was evaluated based on the diet questions, and a subject was classified as following a white diet if he or she did not drink any coffee, tea, or wine or consume any dark fruits. Analysis of the white diet was performed using mixed-model analysis of variance, with study and product included as covariates and a random subject effect included to account for the half-mouth design used in most of the studies. Interactions with baseline color were also examined to determine if the effect of the white diet varied by baseline tooth color. The Spearman rank correlations for color change were also examined, and the number of cups of coffee/tea and the number of wine/fruit servings were used to evaluate a dose effect. Because the timing of postbleaching visits varied too much to make strong conclusions from that data, analyses in the present study were restricted to the color change between baseline and the end of bleaching. Also, by focusing only on the bleaching period, a strict analysis of the effects of diet on bleaching itself was performed and not an evaluation of additional staining caused by diet after bleaching.

RESULTS

Sixteen percent of the subjects followed a white diet during the study period (Table 2). The baseline color and color-change results from each study are summarized in Table 3. The baseline color and color change results for the white and nonwhite groups are summarized in Table 4.

The number of cups of coffee and tea consumed per day was significantly and positively associated with ΔE (r=0.32, p<0.0001; Figure 1) and negatively associated with ΔShade (r=–0.30, p<0.0001; Figure 2). A positive correlation in ΔE and a negative correlation in ΔShade Guide signify that the number of cups of coffee and tea consumed per day were positively associated with more tooth whitening (greater consumption equals greater bleaching).

The correlations indicate that subjects who drank more coffee/tea had greater color change. These associations are statistically significant but not large enough to be clinically relevant. Color change, as measured by the colorimeter parameters and shade guide, was not significantly different between subjects who followed a white diet and those who did not ($p \geq 0.65$). Given the significance of the number of cups of coffee/tea, this result indicates that a subject's diet needs to be fairly severe to affect the color change.

A significant interaction was found between the white diet and baseline L* when ΔL* was the outcome (Figure 3) and a significant interaction between white diet and baseline a* when Δa* was the outcome (Figure 4). No significant interaction was found between white diet and baseline b* when Δb* was the outcome (Figure 5). Subjects on a white diet who had low baseline L*, or darker teeth, had less change in L*, and those who had high baseline L*, or lighter teeth, had more change in L* compared with subjects not on a white diet. However, subjects on a white diet who had low baseline a*, or less red, had more change in a* and those who had high

Table 3: *Mean (Standard Deviation) Color and Color Change for Each Study*

Study (Ref. No.)	L*	a*	b*	Shade	ΔL*	Δa*	Δb*	ΔE	ΔShade
1 (7)	66.2 (2.9)	0.4 (0.6)	14.2 (3.3)	19.3 (2.7)	2.4 (1.2)	–0.8 (0.4)	–2.5 (1.3)	3.7 (1.5)	–7.9 (3.6)
2 (8)	49.1 (2.8)	–0.5 (0.7)	4.2 (3.0)	20.5 (2.5)	6.5 (3.0)	–1.1 (0.5)	–4.1 (1.9)	8.8 (2.8)	–15.6 (3.8)
3 (9)	47.7 (2.4)	–0.5 (0.5)	3.1 (2.2)	18.2 (2.9)	5.9 (2.6)	–0.9 (0.6)	–3.8 (1.5)	7.2 (2.8)	–10.8 (4.4)
4 (10)	45.3 (2.7)	–0.5 (0.7)	4.4 (2.7)	18.0 (2.9)	7.3 (4.0)	–1.0 (0.6)	–4.3 (2.3)	8.8 (4.3)	–13.3 (4.4)
5 (11)	46.8 (3.7)	–0.6 (0.5)	4.2 (2.4)	17.8 (4.0)	7.7 (3.8)	–1.5 (0.6)	–5.4 (1.8)	10.1 (3.3)	–15.9 (4.0)

Table 4: *Mean (Standard Error) Color and Color Change by Diet*

	L*	a*	b*	Shade	ΔL*	Δa*	Δb*	ΔE	ΔShade
White diet	51.1 (0.5)	–0.3 (0.1)	5.7 (0.5)	18.9 (0.6)	6.1 (0.4)	–1.1 (0.1)	–4.2 (0.2)	7.7 (0.4)	–13.0 (0.7)
Nonwhite Diet	51.0 (0.3)	–0.3 (0.1)	6.0 (0.3)	18.7 (0.3)	6.0 (0.2)	–1.1 (0.1)	–4.3 (0.1)	7.7 (0.2)	–12.7 (0.3)
p-Value	0.93	0.91	0.54	0.74	0.85	0.99	0.70	0.99	0.65

baseline a*, or more red, had less change in a* compared with subjects not on a white diet.

We repeated the analyses using more lenient definitions of white diet that included subjects who had up to one, up to two, or up to three servings of coffee/tea/fruit in the white diet group. The conclusions using these other white diet definitions did not differ from the results presented earlier using the pure white diet definition.

DISCUSSION

The effectiveness of bleaching has been related to peroxide concentration and time of contact with the dental tissues.[13] Because nonwhite diets have colorants that cause extrinsic stain, it is a worthwhile exercise to determine whether a nonwhite diet may influence the effectiveness of bleaching.

Determining color changes is challenging. Most shade tabs do not have even color spacing. When using the CIELAB system, the accepted standard for noticeable color change under close examination is a ΔE of 1.0.[14] A ΔE of 2.0 is detectable[15] by visual observation, and a ΔE of 3.3 is unacceptable[16] from an esthetic standpoint.

The findings of this study demonstrated that those subjects who drank a greater amount of coffee/tea had teeth that were initially darker and therefore had a greater amount of color change during bleaching compared with those whose teeth were initially lighter because they did not drink coffee/tea. Attia and others[5] found no affect of coffee on tooth bleaching during the bleaching process; however, the stability of dental whitening treatment was compromised by the use of coffee after bleaching. Bleached teeth were found to be more susceptible to staining with coffee after bleaching. Attia and others[5] concluded that tooth contact with brown staining agents should be avoided during the whitening procedure. Alternatively, Attin and others[3] found that extrinsic staining does not significantly affect postbleaching staining when studying tea-staining on previously bleached enamel. Liporoni and others[17] found that coffee had little effect on color change after bleaching, but wine staining susceptibility was increased after bleaching. Berger and others[18] also found that staining susceptibility increased when wine was applied to enamel surfaces after bleaching. Bazzi and others[19] reported that enamel stained with coffee was more susceptible to restaining than was enamel stained with cigarette smoke.

A recent study by Cortes and others[20] reported that bleaching is effective in preventing staining

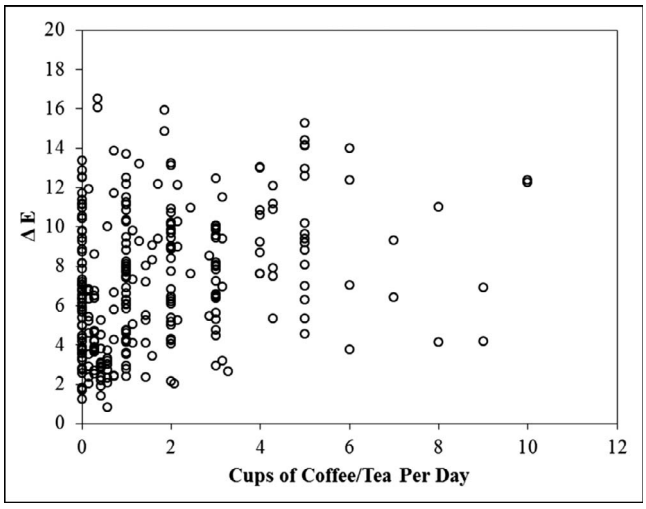

Figure 1. *Association of Number of Cups of Coffee/Tea With Total Color Difference (ΔE).*

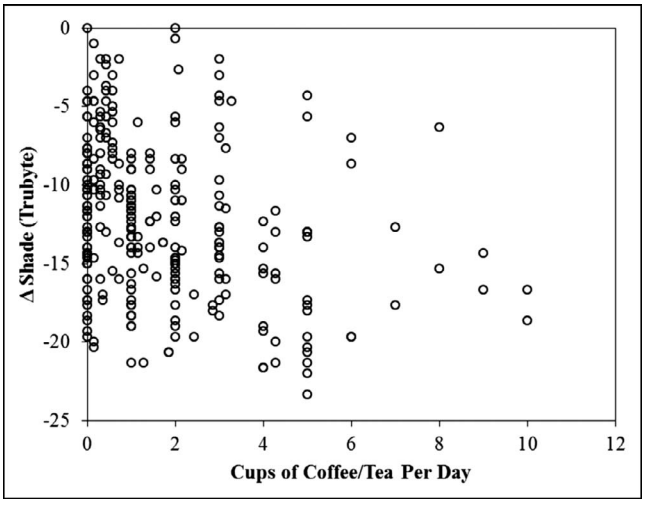

Figure 2. *Association of Number of Cups of Coffee/Tea With Change in Shade.*

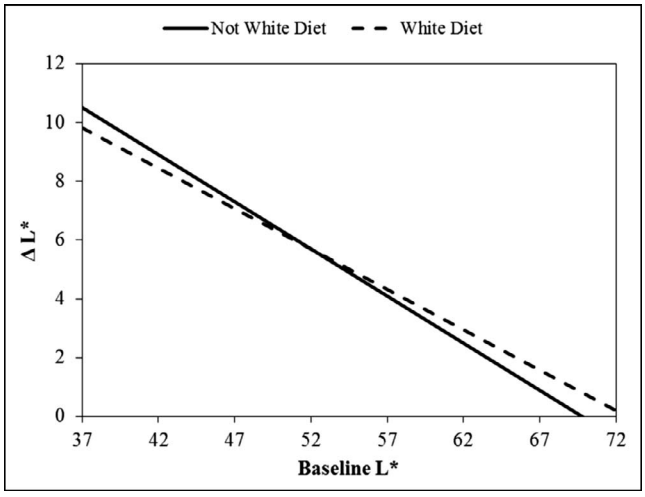

Figure 3. *Associations of Baseline L* With Change in L* Between White Diet and Not White Diet.*

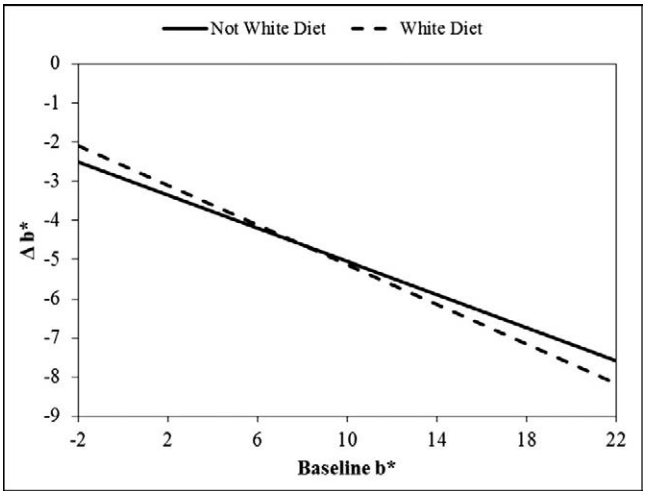

Figure 5. *Associations of Baseline b* With Change in b* Between White Diet and Nonwhite Diet.*

but that both coffee and wine caused enamel color changes after bleaching, though wine caused greater staining than coffee. Ley and others[4] recommend that applying topical fluorides to bleached enamel before exposure to a potentially staining and erosive beverage could be beneficial for maintaining tooth color by preventing extrinsic discoloration.

This present study evaluated the effect of coffee, tea, wine, and dark fruits on tooth bleaching during the process of tooth whitening. Previous research has evaluated the staining capacity of different agents *in vitro*. This is the first *in vivo* study that addresses the concern of adhering to a white diet during bleaching.

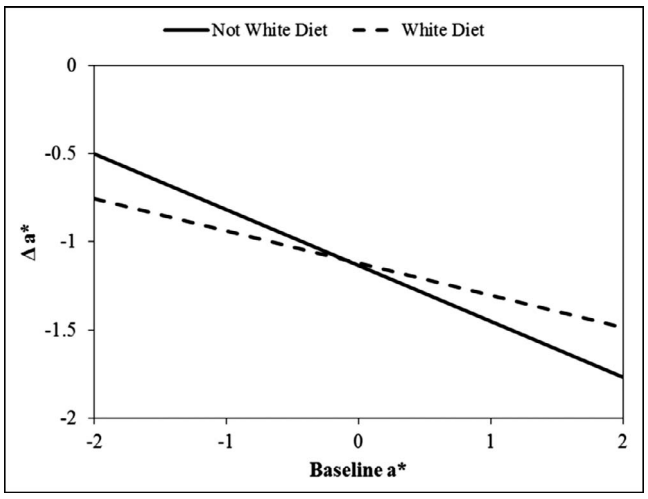

Figure 4. *Associations of Baseline a* With Change in a* Between White Diet and Nonwhite Diet.*

An evaluation of the five *in vivo* studies indicates that nonadherence to a white diet before bleaching results in a greater color change after bleaching treatment. Additionally, those same studies also indicated that the consumption of beverages/foods that are not included in a white diet does not negatively affect the bleaching process. As a result, strict adherence to a white diet during dental bleaching is not necessary during the bleaching process. However, care should be taken after bleaching, as extrinsic staining to bleached enamel will occur from the consumption of such agents as coffee/tea/wine/dark fruits.

CONCLUSIONS

The degree of tooth whitening increased as the number of cups of coffee/tea consumed during tooth whitening increased, although the change was not clinically relevant. Subjects that consumed red wine/dark fruit had no difference in ΔL* compared with the subjects that did not. Adhering to a white diet during the process of tooth whitening did not improve the esthetic outcome.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 29 September 2014)

References

1. Barghi N (1998) Making a clinical decision for vital tooth bleaching: At-home or in-office? *Compendium of Continuing Education in Dentistry* **19(8)** 831-838.

2. Watts A, & Addy M (2001) Discoloration and staining: A review of the literature *British Dental Journal* **190(6)** 309-316.

3. Attin T, Manolakis A, Buchalla W, & Hannig C (2003) Influence of tea on intrinsic colour of previously bleached enamel *Journal of Oral Rehabilitation* **5(5)** 488-494.

4. Ley M, Wagner T, & Bizhang M (2006) The effect of different fluoridation methods on the red wine staining potential on intensively bleached enamel *in vitro American Journal of Dentistry* **19(2)** 80-84.

5. Attia ML, Aguiar FH, Mathias P, Ambrosano GM, Fontes CM, & Liporoni PC (2009) The effect of coffee solution on tooth color during home bleaching *American Journal of Dentistry* **22(3)** 175-179.

6. Gerlach RW, & Zhou X (2001) Vital bleaching with whitening strips: Applications *American Journal of Dentistry* **22(3)** 175-179.

7. Matis BA, Cochran M, Wang G, Franco M, Eckert GJ, Carlotti RJ, & Bryan CA (2005) Clinical evaluation of bleaching using whitening wraps and strips. *Operative Dentistry* **30(5)** 588-592.

8. Matis BA, Cochran MA, Eckert GJ, & Matis JI (2007) In vivo study of two carbamide peroxide gels with different desensitizing agents *Operative Dentistry* **32(6)** 549-555.

9. Matis BA, Cochran MA, Wang G, & Eckert GJ (2009) A clinical evaluation of two in-office bleaching regimens with and without tray bleaching *Operative Dentistry* **34(2)** 142-149.

10. Zekonis R, Matis BA, Cochran MA, Al Shetri SE, Eckert GJ, & Carlson TJ (2003) Clinical evaluation of in-office and at-home bleaching treatments *Operative Dentistry* **28(2)** 114-121.

11. Matis BA, Mousa HN, Cochran MA, & Eckert GJ (2000) Clinical evaluation of bleaching agents of different concentrations *Quintessence International* **31(5)** 303-310.

12. International Commission on Illumination (1978) *Recommendations on Uniform Color Spaces, Color Difference Equations, Psychometric Color Terms* Supplement 2 to CIE, publication 15 Bureau Central de la CIE, Paris.

13. Sulieman M (2004) An overview of bleaching techniques 1.History chemistry, safety and legal aspects *Dentistry Update* **31(10)** 608-616.

14. Kuehni RG, & Marcus RT (1979) An experiment in visual scaling of small color differences *Color* **4(12)** 83-91.

15. Seghi RR, Hewlett ER, & Kim J (1989) Visual and instrumental colorimetric assessments of small color differences on translucent dental porcelain *Journal of Dental Research* **68(12)** 1760-1764.

16. Ruyter IE, Niler K, & Moller B (1987) Color stability of dental composite resin materials for crown and bridge veneers *Dental Materials* **3(5)** 246-251.

17. Liporoni PCS, Souto CMC, Pazinatto RB, Cesar ICR, de Rego MA, Mathias P, & Cavalli V (2010) Enamel susceptibility to coffee and red wine staining at different intervals elapsed from bleaching: A photoreflectance spectrophotometry analysis. *Photomedicine and Laser Surgery* **23(Supplement 2)** S105-S109.

18. Berger SB, Coelho AS, Oliveira VA, Cavalli V, & Giannini M (2008) Enamel susceptibility to red wine staining after 35% hydrogen peroxide bleaching *Journal of Applied Oral Sciences* **16(3)** 201-204.

19. Bazzi JZ, Bindo MJFB, Rached RN, Mazur RF, Viera S, & de Souza M (2012) The effect of at-home bleaching and toothbrushing on removal of coffee and cigarette smoke stains and color stability of enamel *Journal of the American Dental Association* **143(5)** e1-e7.

20. Cortes G, Pini NP, Lima DANL, Liporoni PCS, Munin E, Ambrosano GMB, Aguiar FHB, & Lovadino JR (2013) Influence of coffee and red wine on tooth color during and after bleaching *Acta Odontologica Scandinavica* **71(6)** 1475-1480.

©Operative Dentistry, 2015, **40-3,** 241-246

Digital Workflow for Virtually Designing and Milling Ceramic Lithium Disilicate Veneers: A Clinical Report

A Zandinejad • WS Lin • M Atarodi
T Abdel-Azim • MJ Metz • D Morton

Clinical Relevance

Digital impressions and virtual CAD/CAM design of ceramic veneers may facilitate the implementation of digital dentistry and virtual design as a viable option for all ceramic restorations.

SUMMARY

Laminate veneers have been routinely used to restore and enhance the appearance of natural dentition. The traditional pathway for fabricating veneers consisted of making conventional polyvinyl siloxane impressions, producing stone casts, and fabricating final porcelain prostheses on stone dies. Pressed ceramics have successfully been used for laminate veneer fabrication for several years. Recently, digital computer-aided design/computer-aided manufacturing scanning has become commercially available to make a digital impression that is sent electronically to a dental laboratory or a chairside milling machine. However, technology has been developed to allow digital data acquisition in conjunction with electronically transmitted data that enables virtual design of restorations and milling at a remote production center.

Following the aforementioned workflow will provide the opportunity to fabricate a physical cast-free restoration. This new technique has

*Amirali Zandinejad, DDS, MSc, assistant professor, Department of Oral Health and Rehabilitation, School of Dentistry, University of Louisville, Louisville, KY, USA

Wei-Shao Lin, DDS, assistant professor, Department of Oral Health and Rehabilitation, School of Dentistry, University of Louisville, Louisville, KY, USA

Merila Atarodi, DDS, assistant professor, Department of Pediatric Dentistry, School of Dentistry, University of Louisville, Louisville, KY, USA

Tamer Abdel-Azim, DDS, assistant professor, Department of Oral Health and Rehabilitation, School of Dentistry, University of Louisville, Louisville, KY, USA

Michael J. Metz, DMD, MSD, MS, MBA, assistant professor and vice-chair, Department of General Dentistry, School of Dentistry, University of Louisville, Louisville, KY, USA

Dean Morton, BDS, MS, professor and chair, Department of Oral Health and Rehabilitation, School of Dentistry, University of Louisville, Louisville, KY, USA.

*Corresponding author: 501 S. Preston Street, Room 312, Louisville, KY 40202; e-mail: a0zand01@louisville.edu

DOI: 10.2341/13-291-S

been reported recently for all-ceramic IPS e.max full-coverage pressed-ceramic restorations. However, laminate veneers are very delicate and technique-sensitive restorations when compared with all-ceramic full-coverage ones made from the same material. Complete digital design and fabrication of multiple consecutive laminate veneers seems to be very challenging. This clinical report presents the digital workflow for the virtual design and fabrication of multiple laminate veneers in a patient for enhancing the esthetics of his maxillary anterior teeth. A step-by-step process is presented with a discussion of the advantages and disadvantages of this novel technique. Additionally, the use of lithium disilicate ceramic as the material of choice and the rationale for such a decision is discussed.

INTRODUCTION

Ceramic materials have been used to mimic the appearance of natural teeth in dental restorations for years now. Several materials and fabrication methods are available on the market.[1] The intention of ceramics has been to closely imitate the optical properties of natural teeth while maintaining acceptable biomechanical and biocompatibility characteristics.

Lithium disilicate glass ceramic using a pressed technique and its applications in clinical dentistry were introduced in 1998 by Brodkin and others.[2] It is composed of 65% lithium disilicate in the form of crystalline structures,[3] which results in relatively strong ceramic[4] with high flexural strength of about 400 MPa, a fracture toughness of 3.3 MPa $m^{0.5}$, and a good translucency. Lithium disilicate glass ceramic can be etched and bonded to both enamel and dentin. A solution of 10% hydrofluoric acid is used to microetch the bonding surface to increase the bond strength.[5] Different clinical applications are suggested for lithium disilicate ceramics including veneers, anterior and posterior single crowns, and anterior fixed dental prostheses.[6]

By definition, porcelain laminate veneers are thin bonded ceramic restorations that restore the facial and part of the proximal surfaces of teeth requiring esthetic restoration.[7] Introduction of the acid-etch technique by Buonocore in 1955 and porcelain etching in 1983 resulted in long-lasting veneers after adhesive bonding.[8,9] According to the published literature, the veneer material with the most clinical data is feldspathic porcelain. However, new research on ceramics, including hot pressed, is becoming available in the literature. Lithium disilicate glass ceramic has been used for veneers, but there are limited clinical data regarding its outcome.[10] Porcelain laminate veneer preparations have the advantage of being more conservative than full-coverage crowns and can address some of the limitations of metal-ceramic full-coverage restorations, such as superior optics and color control, supragingival margins, and bonding with improved tissue response.[11]

Digital technology is emerging quickly and has introduced many new aspects to contemporary dental practice. Digital impressions have become an alternative to conventional polyvinyl siloxane (PVS) impression techniques and materials. Clinical evaluation of intraoral digital impressions has shown very promising results. It has been claimed that all ceramic crowns fabricated using chairside scanners have superior marginal fit and improved proximal contact points compared with those fabricated using conventional impressions.[12]

Therefore, the purpose of this paper is to present the esthetic outcome of multiple consecutive lithium disilicate ceramic veneers using a digital impression, virtual computer-aided design/computer-aided manufacturing (CAD/CAM) design, and model-free fabrication. Shortcomings of the novel technique will be presented and discussed in this case report.

CLINICAL REPORT

A 43-year-old healthy man was referred for a restorative consultation. His chief complaint was large clinical diastemas between his maxillary anterior teeth, 7-10 (Figure 1). Also, the patient was not satisfied with the color and shape of his natural teeth. A comprehensive oral examination and a full-mouth radiographic series were completed. It was determined the patient had a low caries risk with no active dental caries or signs of periodontal disease. Medical history was reviewed and revealed no contraindication for elective dental treatment.

Treatment plan options were discussed with the patient to include vital whitening therapy followed by either direct resin-composite bonding or laminate ceramic veneers. After careful consideration by the patient, vital whitening therapy followed by laminate ceramic veneers was selected as the treatment of choice. Minor soft tissue crown lengthening was recommended to the patient before veneer preparations to correct slight tissue asymmetry. The patient

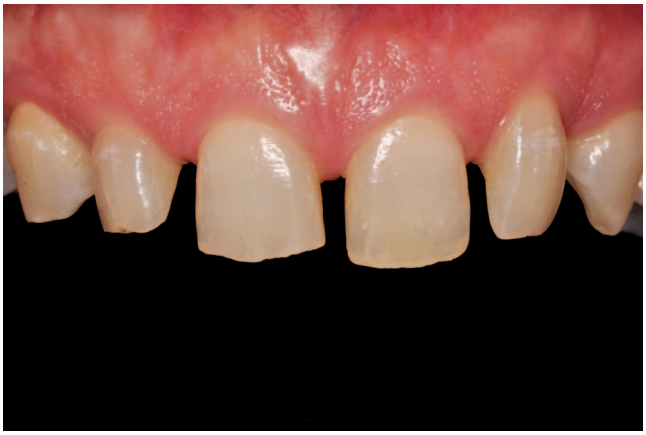

Figure 1. *Pretreatment frontal view of maxillary anterior teeth.*

declined surgical intervention because he has a low smile line that would not affect the social or esthetic outcomes of his treatment. Vital whitening provides the operator with the opportunity to use a translucent glass ceramic, allowing the stump shade to control the final color. Porcelain laminate veneers are considered a conservative treatment with predictable clinical results.[10,13]

Initial diagnostic impressions were taken for treatment planning using irreversible hydrocolloid impression material (Jeltrate Fast Set, Dentsply Caulk, Milford, DE, USA) and poured with type III dental stone (Buff Stone, Whip Mix Corp, Louisville, KY, USA). Casts were articulated on a semi adjustable articulator (Model 2240, Whip Mix Corp) with a face-bow transfer (Model 8645, Whip Mix Corp). A diagnostic wax-up was completed by the laboratory for patient presentation of proposed shape and contour of final laminate ceramic veneers and provisional stent fabrication (Figure 2).

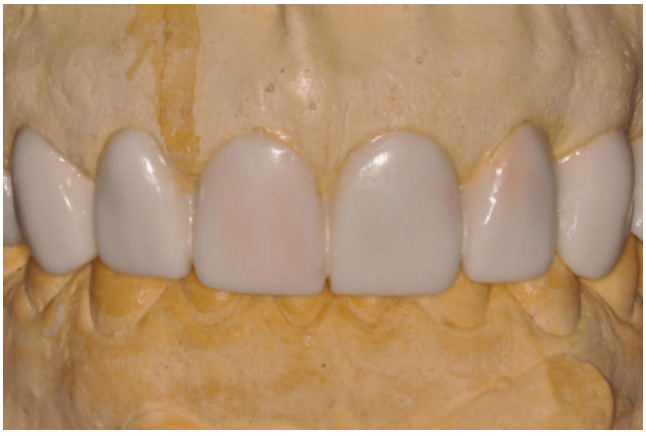

Figure 2. *Diagnostic wax-up of maxillary anterior teeth mounted in semiadjustable articulator.*

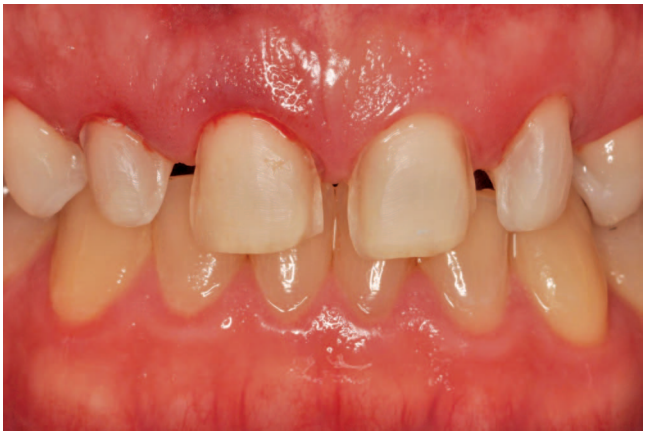

Figure 3. *Frontal view of maxillary anterior teeth after preparation for ceramic veneers.*

Teeth whitening treatment started with an at-home whitening kit using 15% carbamide peroxide (Opalescence PF 15%, Ultradent Corp, South Jordan, UT, USA) overnight daily for six weeks. The patient's initial tooth shade was Vita A3.5. Preparations were started for laminate ceramic veneers after achieving an acceptable shade (Vita A1) and waiting 14 days post whitening for oxygen free-radical dissipation and possible color regression (Figure 3). The maxillary anterior teeth were prepared with a butt-joint margin on the lingual surfaces, 1.5-mm reduction of the incisal edges and 0.3-mm (gingival) to 0.8-mm (incisal) reduction on facial surfaces using a round-ended diamond cutting instrument (Brasseler USA, Savannah, GA, USA) and a reduction guide (Figure 3).

Soft tissue management and marginal exposure was performed using a single-cord technique (#0 Ultrapak, Ultradent Inc) . A CAD digital impression of the prepared maxillary teeth and a CAD digital scan of the opposing mandibular teeth were taken following application of the spray contrast medium (Lava COS, 3M ESPE, St Paul, MN, USA). A closed-jaw record was then taken with the same intraoral digital scanner using the spray contrast media (Figure 4). The CAD software (Lava COS, 3M ESPE) overlapped the digital information obtained from the previously acquired maxillary and mandibular scans with the closed-bite scan to form a virtual bite registration and articulation. The completed CAD data were sent electronically to the scan center at a local commercial dental laboratory to mark the laminate veneer margins and perform a virtual ditching process for marginal design and fabrication (Figure 5). Provisional veneers were virtually designed (Figure 6) using CAD/CAM design software

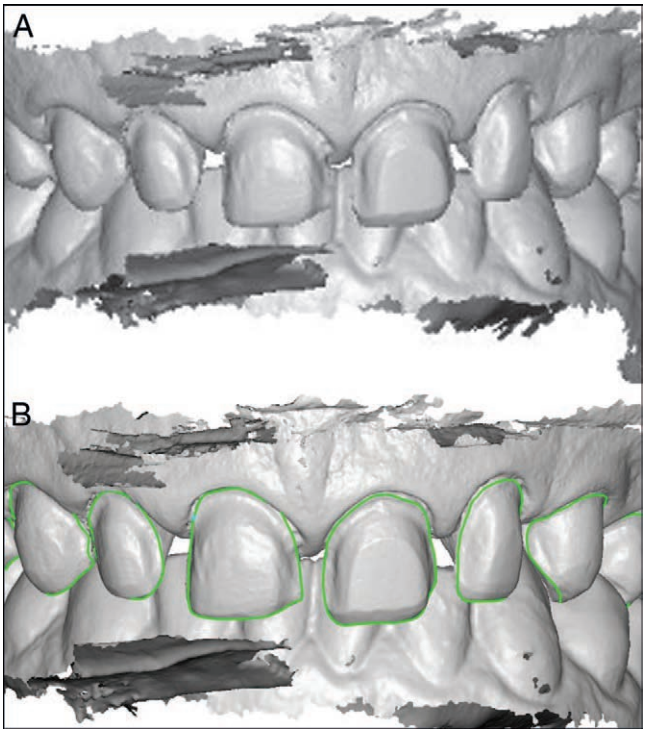

Figure 4. *(a): Digital impression and (b): marked finishing margins.*

(Dental Wings Inc, Montreal, QC, Canada). The provisional veneers were designed to be splinted for better retention and subsequently milled from polymethyl-methacrylate (PMMA) Vita A1 blocks within a custom milling center (Figure 7 a,b).

The provisional veneers were tried intraorally for marginal integrity, functionality, occlusion, esthetics, and patient satisfaction. They were temporarily cemented using an acid-etch point technique (midfacial) and bonded with flowable resin composite (Vita A1). Excess composite was removed and polymerized with a VALO Broadband LED Curing Light (Ultradent Corp) on standard setting. The occlusion was

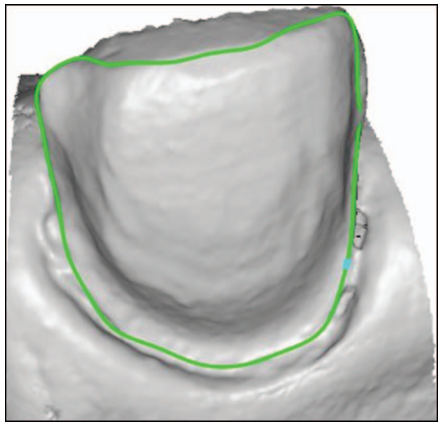

Figure 5. *Margin selection and die trim.*

Figure 6. *Virtual design of connected provisional veneers before milling.*

checked and adjusted. The provisional veneers were reevaluated after a few weeks following the patient's evaluation of form, function, and esthetics. The patient requested modifications that were performed and communicated to the dental laboratory. The definitive laminate veneer designs were modified and milled from low translucency IPS e.max (Vita

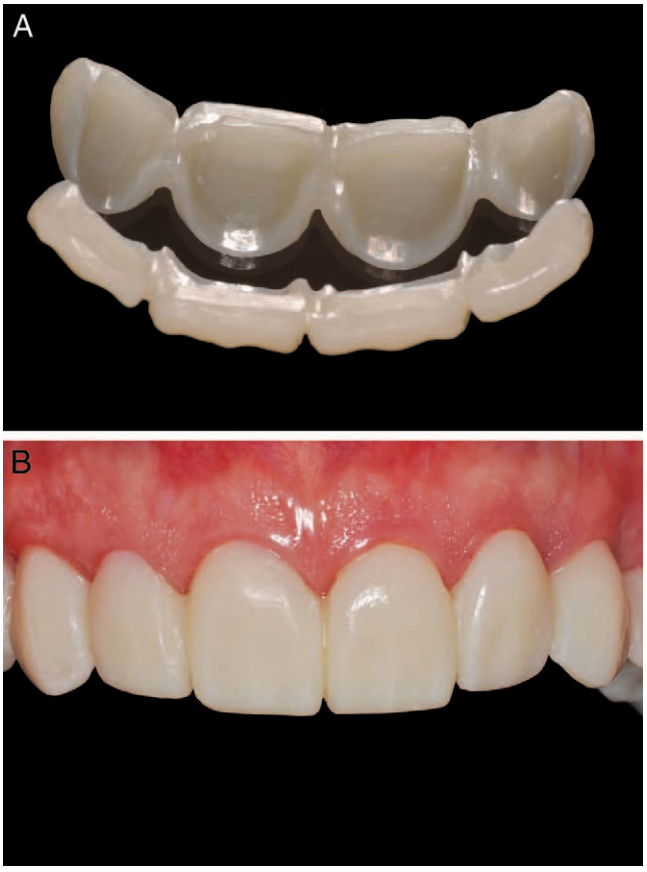

Figure 7. *(a): provisional PMMA veneers and (b): PMMA veneers cemented as a long-term provisional.*

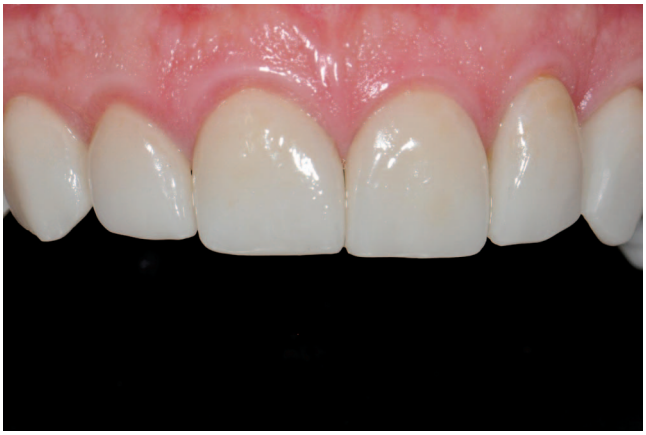

Figure 8. *After cementation of lithium disilicate ceramic veneers.*

A1) milling blocks (Ivoclar-Vivadent, Amherst, NY, USA) at a remote milling center.

At the insertion appointment, marginal adaptation, restoration fit, interproximal contacts, and occlusion were verified individually and collectively using a translucent try-in paste (RelyX Veneer Kit, 3M ESPE). Minor adjustments to the interproximal contacts were made using a fine diamond bur (Brasseler USA) and were polished using a chairside ceramic polishing kit (Brasseler USA). After final approval by the patient, all internal bonding surfaces of the laminate veneers were etched with 10% hydrofluoric acid for 25 seconds and silanated (Ceramic Silane, 3M ESPE). The laminate veneers were cemented one at a time using a translucent light-cure resin cement (RelyX Veneer, 3M ESPE) following manufacturer recommendations. All excess cement was removed from margins and cured using a VALO Broadband LED Curing Light (Ultradent Corp) on standard setting. The final delivered veneers can be seen in Figure 8.

Upon completion of the bonding process, an irreversible hydrocolloid impression (Jeltrate Fast Set, Dentsply Caulk) was taken of the maxillary arch and poured with type III dental stone (Buff Stone, Whip Mix Corp). The stone model was used to fabricate a vacuum-formed, clear bite guard (Soft-Tray Classic Sheets, 0.08 inches, Ultradent Corp) for nocturnal use by the patient. The patient was instructed on home care and hygiene and was placed on a recall system at six-month intervals. A one-year follow-up was completed.

DISCUSSION

Laminate veneers have the advantages of being conservative while providing ultimate optical properties and esthetics. Several techniques and materials have been introduced for fabricating laminate veneers. Upon introduction by Calamia and Horn in 1983, laminate veneers were limited to feldspathic porcelain using the acid-etch technique.[14,15]

Since then, the fabrication techniques and materials have been streamlined by digital dentistry and stronger infiltrated glass ceramics. With the introduction of stronger glass ceramics, especially lithium disilicate pressable glass ceramics, laminate veneers can be fabricated by the lost-wax technique or CAD scanning technology. Due to the emerging CAD scanning technology, complete virtual design (CAD) and milling (CAM) are available.[16,17]

In this clinical case report we designed and fabricated multiple ceramic laminate veneers using a completely digital workflow. The technique chosen provides the opportunity to fabricate precision-milled temporary restorations that represent the proposed final shape, contour, and color of definitive restorations. Long-term provisional restorations can improve the esthetic outcome of the definitive restorations by allowing the patient to critically evaluate them over a period of time.[18] Temporary restorations that were designed and used as long-term interim prostheses provided the opportunity to customize the final restorations further, based on patient feedback, desires, and expectations. There are some disadvantages associated with this technique involving provisional veneer fabrication: the added cost associated with laboratory fabrication, increased chair time, and additional appointments for the patient.

Lithium disilicate glass ceramic was the material of choice in this clinical case. Glass ceramics, especially lithium disilicate, have shown high esthetic potential when treatment planned correctly.[19] The marginal integrity of the milled veneers proved to be clinically adequate. Additionally, the contour, shade, and form of all laminate veneers were all clinically acceptable. However, the breadth and depth of color should be compared with both laboratory hot-pressed and feldspathic ceramic restorations. The aforementioned techniques allow artistic detailing and surface characterization by an experienced laboratory technician or ceramist to provide fine details lacking in CAD design software. The design software used for this cast-free case has minor design limitations for full artistic customization, which may have restricted the esthetic potential of restorations.[16,20] The software does not allow decreasing thickness beyond a certain value that is preset. Furthermore, the shade cannot be

customized prior to fabrication of the restoration. These limitations are software specific and vary by the type of design software used.

Because this is an emerging technology that is constantly being updated, there is a learning curve that involves CAD software developers, clinicians, and dental laboratory technicians. As the dental team members obtain more experience with this innovative technology, additional improvements to the esthetics can be achieved in the near future.

CONCLUSIONS

This clinical report described a cast-free digital workflow for multiple ceramic veneers. The technique provides the opportunity to enhance the esthetics by using milled acrylic provisional and IPS e.max definitive restorations. All restorations fabricated were clinically acceptable in terms of marginal fit, shape, contour, and esthetics. CAD can be altered to achieve the patients' desires and clinical expectations. Implementation of digital dentistry and virtual design can improve communication among the patient, clinician, and commercial dental laboratories and may become a common technique for all ceramic restorations.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 17 December 2013)

REFERENCES

1. Anusavice KJ (2003) *Dental Ceramics. Phillips, Science of Dental Materials* **12th edition** Elsevier Saunders, St. Louis, MO.
2. Brodkin D, Panzera C, & Panzera P (September 24, 2002) Jeneric/Pentron, Inc. Pressable lithium disilicate glass ceramics US patent 6,455,451 B1
3. Albakry M, Guazzato M, & Swain MV (2003) Biaxial flexural strength, elastic moduli, and x-ray diffraction characterization of three pressable all-ceramic materials *Journal of Prosthetic Dentisty* **89(4)** 374-80.
4. Höland W, Schweiger M, Frank M, & Rheinberger V (2000) A comparison of the microstructure and properties of the IPS Empress 2 and the IPS Empress glass-ceramics *Journal of Biomedical Material Research* **53(4)** 297-303.
5. Guarda GB, Correr AB, Gonçalves LS, Costa AR, Borges GA, Sinhoreti MA, & Correr-Sobrinho L (2013) Effects of surface treatments, thermocycling, and cyclic loading on the bond strength of a resin cement bonded to a lithium disilicate glass ceramic *Operative Dentistry* **38(2)** 208-217.
6. Giordano R, & McLaren EA (2010) Ceramics overview: Classification by microstructure and processing methods. *Compendium of Continuing Education in Dentistry* **31(9)** 682-684.
7. The glossary of prosthodontic terms (2005) *Journal of Prosthetic Dentistry* **94(1)** 10-92.
8. Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34(6)** 849-853.
9. Marocho SM, Ozcan M, Amaral R, Bottino MA, & Valandro LF (2013) Effect of resin cement type on the microtensile bond strength to lithium disilicate ceramic and dentin using different test assemblies *Journal of Adhesive Dentistry* **15(4)** 361-368.
10. Layton DM, & Clarke M (2013) A systematic review and meta-analysis of the survival of non-feldspathic porcelain veneers over 5 and 10 years *International Journal of Prosthodontics* **26(2)** 111-124.
11. Pippin DJ, Mixson JM, & Soldan-Els AP (1995) Clinical evaluation of restored maxillary incisors: Veneers vs PFM crowns *Journal of the American Dental Association* **126(11)** 1523-1529.
12. Syrek A, Reich G, Ranftl D, Klein C, Cerny B, & Brodesser J (2010) Clinical evaluation of all-ceramic crowns fabricated from intraoral digital impressions based on the principal of active wavefront sampling *Journal of Dentistry* **38(7)** 553-559.
13. Layton DM, & Walton TR (2012) The up to 21-year clinical outcome and survival of feldspathic porcelain veneers: accounting for clustering *International Journal of Prosthodontics* **25(6)** 604-612.
14. Calamia JR (1983) Etched porcelain facial veneers: A new treatment modality based on scientific and clinical evidence *New York Journal of Dentistry* **53(6)** 255-259.
15. Horn HR (1983) Porcelain laminate veneers bonded to etched enamel *Dental Clinics of North America* **27(4)** 671-684.
16. Lin WS, Harris BT, Ozdemir E, & Morton D (2013) Maxillary rehabilitation with a CAD/CAM-fabricated, long-term interim and anatomic contour definitive prosthesis with a digital workflow: A clinical report *Journal of Prosthetic Dentistry* **110(1)** 144-146.
17. Fritzsche G (2013) Cerec omnicam and the virtual articulator—A case report *International Journal of Computerized Dentistry* **16(1)** 59-67.
18. Gürel G, & Bichacho N (2006) Permanent diagnostic provisional restorations for predictable results when redesigning the smile *Practical Procedure and Aesthetic Dentistry* **18(5)** 281-286.
19. Taskonak B, & Sertgoz A (2006) Two-year clinical evaluation of lithia-disilicate-based all-ceramic crowns and fixed partial dentures *Dental Materials* **22(11)** 1008-1013.
20. Beuer F, Schweiger J, Edelhoff D, & Sorensen JA (2011) Reconstruction of esthetics with a digital approach *International Journal of Periodontics and Restorative Dentistry* **31(2)** 185-193.

©Operative Dentistry, 2015, **40-3,** 247-254

Laboratory Research

CO_2 Laser Glazing Treatment of a Veneering Porcelain: Effects on Porosity, Translucency, and Mechanical Properties

R Sgura • MC dos Reis • MC Salvadori
AC Hernandes • PF Cesar • IS Medeiros

Clinical Relevance

Since laser glazing is a fast and viable procedure it could be considered for chairside application in ceramic restorations, reducing the number of appointments required in the dental office.

SUMMARY

This work tested CO_2 laser as a glazing agent and investigated the effects of irradiation on the porosity, translucency, and mechanical properties of veneering porcelain. Sixty discs (diameter 3.5 × 2.0 mm) of veneering porcelain for Y-TZP frameworks (VM9, VITA Zahnfabrik) were sintered and had one of their faces mirror polished. The specimens were divided into six groups (n=10/group) according to surface treatment, as follows: no treatment–control; auto-glaze in furnace following manufacturer's instructions (G); and CO_2 laser (45 or 50 W/cm^2) applied for four or five minutes (L45/4, L45/5, L50/4, L50/5). Optical microscopy (Shimadzu, 100×) was conducted and the images were analyzed with Image J software for the determination of the following porosity parameters: area fraction, average size, and Feret diameter. The translucency parameter studied was masking ability, determined by color difference (ΔE) over black and white backgrounds (CM3370d, Konica Minolta). Microhardness and fracture toughness (indentation fracture) were measured with a Vickers indenter (HMV, Shimadzu). Contact atomic force microscopy (AFM) (50 × 50 μm^2, Nanoscope IIIA, Veeco) was performed at the center of one sample from

*Ricardo Sgura, University of Sao Paulo, Department of Biomaterials and Oral Biology, Sao Paulo, Brazil

Mariana Cavalcante dos Reis, University of Sao Paulo, Department of Biomaterials and Oral Biology, Sao Paulo, Brazil

Maria Cecília Salvadori, University of Sao Paulo, Institute of Physics, Sao Paulo, Brazil

Antônio Carlos Hernandes, University of Sao Paulo, Sao Carlos Institute of Physics, Sao Paulo, Brazil

Paulo Francisco Cesar, University of Sao Paulo, Department of Biomaterials and Oral Biology, Sao Paulo, Brazil

Igor Studart Medeiros, Sao Paulo, Brazil

*Corresponding author: Av. Professor Lineu Prestes, 2227 – Cidade Universitária. Departamento de Biomateriais e Biologia Oral, Faculdade de Odontologia. São Paulo, SP, 05508000, Brasil; e-mail: risgura@hotmail.com

DOI: 10.2341/14-079-L

each group, except in the case of L45/5. With regard to porosity and translucency parameters, auto-glazed and laser-irradiated specimens presented statistical similarity. The area fraction of the surface pores ranged between 2.4% and 5.4% for irradiated specimens. Group L50/5 presented higher microhardness when compared to the G group. The higher (1.1) and lower (0.8) values for fracture toughness ($MPa.m^{1/2}$) were found in laser-irradiated groups (L50/4 and L45/4, respectively). AFM performed after laser treatment revealed changes in porcelain surface profile at a submicrometric scale, with the presence of elongated peaks and deep valleys.

INTRODUCTION

Glazing is a fundamental step during the production of dental ceramic restorations, which consists of an application of a transparent, viscous, and low-fusion ceramic frit on the porcelain surface in order to increase smoothness and gloss after a new firing cycle (glazing cycle).[1-4] One variation of this type of glaze procedure is the so-called "auto-glaze," which involves an additional firing cycle without the addition of an external ceramic layer to the porcelain piece.[5] The auto-glaze cycle is carried out at low temperatures aiming at softening the outermost superficial layer of the ceramic restoration without changing its shape. Both glazing methods described above decrease the number of surface flaws[6,7] and create surface compressive stresses, increasing the lifetime of the ceramic structure.[8-10]

The reduction and elimination of surface defects is one of the most advantageous aspects of any glazing technique, since surface flaws and pores may act as stress concentrators and lead to the catastrophic failure of the material in the long term.[5,11-14] External surface flaws usually appear during the processing of the prostheses or after intraoral occlusal/interproximal adjustments.[12,15] Pores are different types of flaws that occur in dental porcelains as a consequence of viscous flow sintering. They may also appear in the glassy matrix of a porcelain as a result of bubble formation after the release of dissolved or entrapped insoluble gases that are present in the initial pores.[16]

In addition to the negative effect of surface flaws and pores on the porcelain mechanical strength, they also jeopardize the optical properties of the material. Surface irregularities such as grooves and cracks increase light scattering at the surface and therefore make the material more opaque.[17] Residual pores also affect light transmission, as they act as scattering centers within the material. When light propagates within a material and reaches a pore, it deviates from its normal path as a result of the difference in refraction index between the pore medium (air) and the porcelain material.[18]

Alternative glazing treatments have been suggested in the literature (eg, microwave oven[19] and CO_2 laser sintering[20]), with promising results. Continuous CO_2 laser irradiation of ground porcelain discs resulted in surface roughness similar to that of auto-glazed specimens. In the same study, the color difference (ΔE) between irradiated and glazed specimens was not considered perceivable to the human eye for most experimental groups. The advantages of CO_2 laser glazing are 1) the possibility of reducing the number of appointments that they offer, since laser application is a chairside technique, and 2) the higher processing speed, which is approximately four times faster than that of the conventional technique.[20]

To the authors' knowledge, the effects of CO_2 laser glazing on porosity and translucency have not yet been determined. Therefore, this study evaluated the surface porosity, masking ability, microhardness, and apparent fracture toughness of a commercial veneering porcelain submitted to CO_2 laser glazing. The surface profiles of irradiated specimens were also assessed by atomic force microscopy (AFM). The hypothesis tested was that, for the characterizations cited above, CO_2 laser glazing would be similar to auto-glazing in a conventional furnace.

METHODS AND MATERIALS

The porcelain used in this study was VM9 (VITA-Zahnfabrik, Bad Sackingen, Germany), which is recommended to veneer Y-TZP substrates. Sixty porcelain discs were produced using a metallic device in order to standardize their dimensions to 4.1×2.4 mm (diameter $\times$ thickness). These discs were then sintered (Kerampress, Kota, São Paulo, SP, Brazil) following the heating cycle recommended by the manufacturer (Table 1). The final dimensions of the specimens after sintering were 3.5×2.0 mm. Specimens had one of their faces ground and polished with diamond abrasive suspensions ranging from 15 to 1 μm (Ecomet 3, Buehler, Lake Buff, IL, USA).

Specimens were then divided into 10 groups according to the surface treatment (Table 2). For group G, specimens were submitted to the glaze cycle

Table 1: *Sintering and Glazing Furnace Cycles Applied to VM9 Porcelain*

	Sintering Cycle	Glaze Cycle
Dry, min	6	4
Start temperature, °C	500	500
Heating rate, °C/min	55	80
Maximum temperature, °C	910	900
Vacuum shutdown, °C	910	No vacuum
Sintering time, min	1.5	2
Cooling time, min	6	6

described in Table 1 in a conventional furnace. A 10.6-μm CO_2 laser device (35 W, Coherent, Santa Clara, CA, USA) was used for the surface treatment of specimens in groups L45 and L50. A copper mirror was used to focus the laser beam onto a refractory with a spot size of 0.5 cm, and the distance between the laser tip and the porcelain disc was kept constant during the experiment, as illustrated in Figure 1. The laser outputs tested were 9 and 10 W, which corresponded to the irradiances of 45 and 50 W/cm^2. The laser beam was continuously irradiated for four or five minutes, according to each group. Irradiance and time were chosen based on a previous study.[20]

For surface porosity determination, three images per specimen were obtained at 100× magnification (HMV, Shimadzu, Singapure). Micrographs were analyzed with Image J software (National Institutes of Health, Bethesda, MD, USA) for determination of the area fraction and Feret diameter of pores. Mean values were submitted to Kruskall-Wallis one-way analysis of variance (ANOVA) (p=0.05). The Feret diameter was also used to determine the pore size at the 90% cumulative frequency (D90).

Microhardness and apparent fracture toughness (indentation fracture, IF) were determined by measuring the cracks produced on the specimen surface by a Vickers indenter (HMV, Shimadzu).

For microhardness determination (measured in GPa) the following formula was applied:

$$\left(1.8544\frac{F}{d^2}\right)0.009807,$$

where F is the applied load in Kgf, and

$$d = \frac{d_h + d_v}{2},$$

where d_h and d_n are the horizontal and vertical indentation diagonals, respectively.

The formula proposed by Lawn and others[21] was used to determine the apparent fracture toughness, as follows:

$$K_{IC} = 0.028\left(\frac{E}{H}\right)^{1/2} Hd^{1/2}\left(\frac{c}{d}\right)^{-3/2},$$

where E is the elastic modulus of VM9 porcelain (66.5), c is the crack length observed after indentation, and H is the Vickers microhardness, in GPa.

Indentations were produced with a load of 2.0 Kgf, and the dwell time was 20 seconds. Three indentations were made per specimen, and mean values were compared by ANOVA and Tukey test, with global significance level of 5%.

To determine the effect of laser application on the material's ability to mask a dark substrate, the color difference (ΔE) between specimens positioned over a black or white substrate was calculated with a spectrophotometer (CM-3700d, Konica Minolta, Sakai, Osaka, JP), according to the following equation:

$$\Delta E = \left[(L_B - L_w)^2 + (a_B - a_w)^2 + (b_B - b_w)^2\right]^{1/2},$$

where L, a, and b parameters refer to the color coordinates of lightness, degree of redness/greenness, and degree of yellowness/blueness, respectively, as designated by the Comission International de l'Eclairage.[22] Subscripts B and W represent the black and white backgrounds, respectively.

Black and white standardized cards were used to simulate the substrates. A coupling agent (glycerin)

Table 2: *Groups Distribution (n=10)*

Group		Fluence, J/cm^2	Surface Treatment	Designation
Control		—	Polishing	C
Auto-glaze		—	Polishing + glaze in furnace	G
Laser 45 W/cm^2	4 min	10,800	Polishing + continuous CO_2 laser	L45/4
	5 min	13,500	Polishing + continuous CO_2 laser	L45/5
Laser 50 W/cm^2	4 min	12,000	Polishing + continuous CO_2 laser	L50/4
	5 min	15,000	polishing + continuous CO_2 laser	L50/5

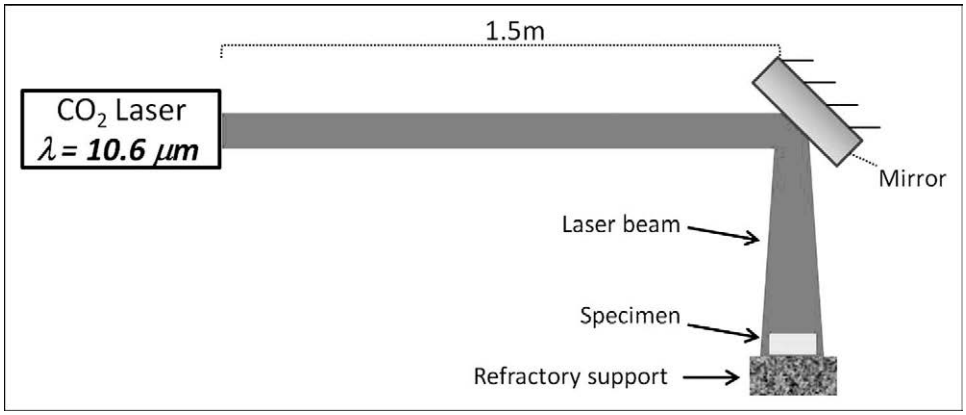

Figure 1. *Optical table disposal for laser irradiation. Laser was continuously irradiated over sintered specimens (0.5-cm spot size).*

with same refractive index of the studied porcelain was applied between the porcelain disc and the background. The means of masking ability were submitted to ANOVA and a Tukey *post hoc* test, with global significance level of 5%. The surface profile of one specimen from each of the following groups—L45/4, L50/4, and L50/5—was assessed by AFM and compared to the profiles obtained for groups C and G. Contact AFM (Nanoscope IIIA, Veeco, Plainview, NY, USA) analysis was conducted using a scanned area of 50 × 50 μm^2 located at the center of the specimen surface. The AFM resolution was 512 pixels. Three-dimensional image processing was carried out with WSxM 5.0 software (Nanotech, Madrid, Spain).[23]

RESULTS

The porosity parameters are described in Table 3. The total area fraction of pores increased significantly after treating the specimens with almost all laser glazing procedures. The only exception was the surface treatment involving the combination of a laser irradiance of 50 W/cm^2 and an application time of five minutes, which resulted in porosity similar to that of the control group. The means of pore sizes, as expressed by the Feret diameter, were similar for all experimental groups and varied from 7.1 to 8.5 μm. The parameter D90 increased, in comparison to the control, from 32% to 52% for the different glazing treatments.

The optical micrographs in Figure 2 show the heterogeneous distribution of the pores described in Table 3. One important aspect that should be highlighted in Figure 2 is the presence of striations on the surface of the specimens glazed at 50 W/cm^2 for five minutes. These striations could also be noticed on the surface of specimens from groups L45/5 and L50/4; however, they were not as clear and numerous as on the surface of the specimens in group L50/5.

Figure 3 shows that the use of laser irradiation as a glazing treatment for the studied porcelain resulted in a significant increase in microhardness in comparison to the auto-glazed group only for the group treated at 50 W/cm^2 for five minutes. The fracture toughness values depicted in Figure 3 varied significantly as a function of the experimental group. The K_{Ic} values obtained for two of the laser-treated groups (L45/5 and L50/5) were similar to that obtained for the auto-glaze group. The other two laser-irradiated groups, L45/4 and L50/4, showed, respectively, lower and higher mean fracture toughness mean values in comparison to the auto-glaze group.

The means of translucency parameter (masking ability, ΔE) obtained for specimens submitted to CO_2

Table 3: *Means and Standard Deviations (in Parentheses) for Porosity. D90 Corresponds to the Feret Diameter of Pores at 90% of Cumulative Frequency. Mean Values of Area Fraction Exhibiting the Same Letters Were Not Significantly Different (p>0.05). There Were Not Statistical Differences Among the Mean Values of Feret Diameter (p=0.58)*

Groups	C	G	L45/4	L45/5	L50/4	L50/5
Area fraction, %	1.2 (0.9) A	3.6 (1.4) BC	4.3 (1.2) BC	5.4 (1.4) C	4.4 (2.4) C	2.4 (1.0) AB
Feret diameter, μm	7.4 (2.1)	7.3 (1.1)	7.8 (2.6)	7.8 (1.1)	7.1 (1.3)	8.5 (2.0)
D90, μm	11.4	16.9	16.0	18.4	15.0	17.0

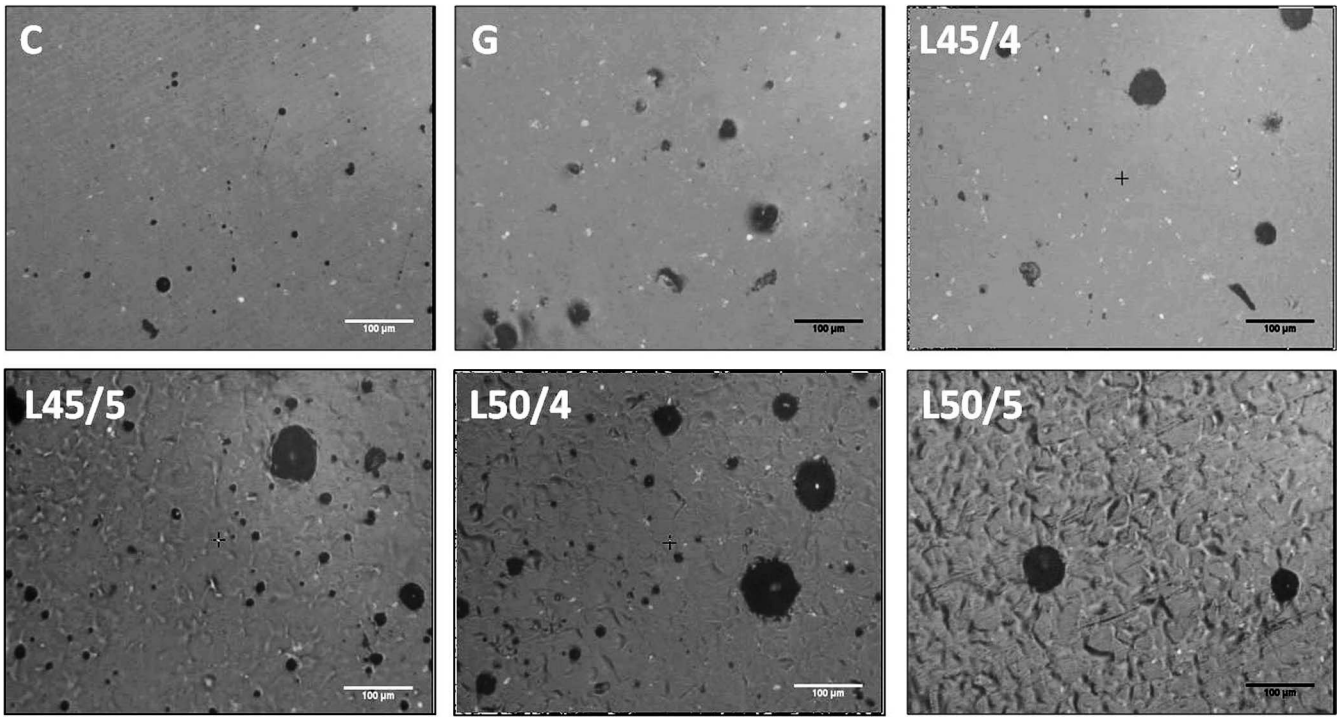

Figure 2. *Optical microscopy images (100×) of studied groups. In figure L50/5 it is possible to observe the striations formation.*

laser were statistically similar to that found for group G. Group L45/5 showed the lowest ΔE value among all groups; however, it was only statistically different from the mean obtained for the control (Figure 3C).

Surface profiles assessed by AFM showed submicrometric surface modifications on the porcelain specimens after laser irradiation. All laser-irradiated surfaces showed elongated, rounded peaks and deep valleys (Figure 4). The surface of the control was flat and showed grooves resulting from the polishing procedure. These polishing grooves were also noticeable on the surface of groups G and L45/4; however, their depth was significantly lower (arrows in Figure 4). Polishing grooves were not found in images from groups L50/4 and L50/5.

DISCUSSION

The hypothesis that CO_2 laser glazing would be similar to auto-glazing in a conventional furnace could only be partially accepted, because although these two surface treatments resulted in similar porosity and translucency, some of the laser parameters tested resulted in significantly different microhardness, apparent fracture toughness, and surface topography.

Although it was expected that the energy provided by the glazing treatments would favor viscous flow and therefore reduce surface porosity, the results indicated that in fact a significantly higher number of pores was found after all types of glazing. Such an increase in porosity observed for most glazed groups in comparison to the control may be related to the displacement of subsurface pores toward the surface as a result of the decrease in the porcelain's viscosity at high temperatures.[16] The observed increase in pore size after heat treatment (see D90 values in Table 3) suggests that moving pores are probably growing together as a consequence of energy increase during the glaze cycle.

The similar masking ability obtained for laser-glazed and oven-glazed specimens is likely directly related to the similar level of porosity found for these groups. It is well known that pores act as scattering centers that reduce the translucency of the material and therefore increase their ability to mask dark backgrounds.[17] Not surprisingly, the group that showed the highest masking ability (lowest ΔE, L45/5) was also the one with the highest porosity.

The observed increase in microhardness for irradiated specimens may be explained by the increase in leucite content and size after continuous CO_2 laser irradiation, as described in a previous study.[20] On the other hand, the differences observed among the mean values of fracture toughness did not follow any

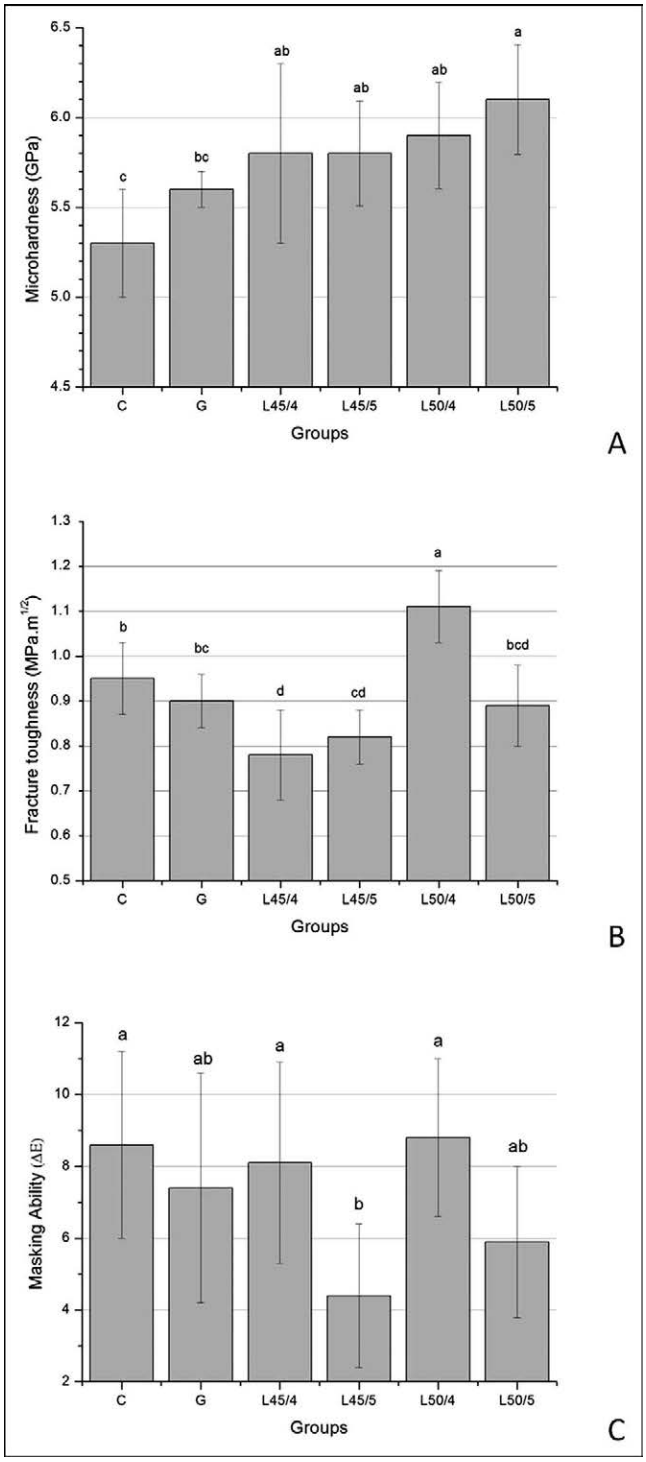

Figure 3. *Graphical representation of microhardness (A), apparent fracture toughness (B), and masking ability (C) means with standard deviations. Means with different letters are significantly different (p<0.05).*

logical trend. It is believed that the different heat treatments applied to the porcelain surface resulted in significantly different distributions of residual stresses, which affected the crack growth that originated from the corner of the Vickers indentations during the IF test. Further studies are necessary to determine the level and distribution of residual stresses as a function of the glaze treatment performed.

AFM proved to be a fundamental tool with which to analyze the surface profile of irradiated specimens. Although a previous study[20] showed that laser-irradiated and oven-glazed porcelain had similar surface roughness when measured with a contact profilometer, the AFM analysis carried out in the present investigation showed important differences at the submicrometric scale (Figure 4). The better performance of AFM in comparison to a stylus perfilometer has already been shown in a previous work,[24] in which subtle changes caused by conventional glazing on the porcelain surface could only be detected by the first method.

The round and elongated peaks observed at the center of the irradiated specimens (AFM analysis, Figure 4) and the lack of polishing grooves for groups L50/4 and L50/5 suggest that the temperature generated by the laser treatment led to melting of the outermost porcelain layer. To the authors' knowledge, these surface features observed after laser treatment in porcelain specimens under AFM have not been demonstrated before. The above-mentioned submicrometric peaks correspond to the striations observed under optical microscopy (Figure 2). The mechanism by which these striations are formed is not yet clear, but one possible explanation would be fluctuations in the distribution of the beam intensity, which can generate thermal gradients, as explained in a previous work[25] for a similar type of striation reported for ceramic pieces after CO_2 laser cutting.

The results of this study indicate that continuous CO_2 laser applied for four minutes with 50 W/cm^2 of irradiance over VM9 porcelain is able to produce a surface comparable to that achieved after an auto-glaze treatment with regard to surface porosity, masking ability, and microhardness, with an increase in the apparent fracture toughness. Further studies should take into account the submicrometric changes observed on the porcelain surface after laser incidence and how they would affect the tribological properties of porcelain pieces during functioning.

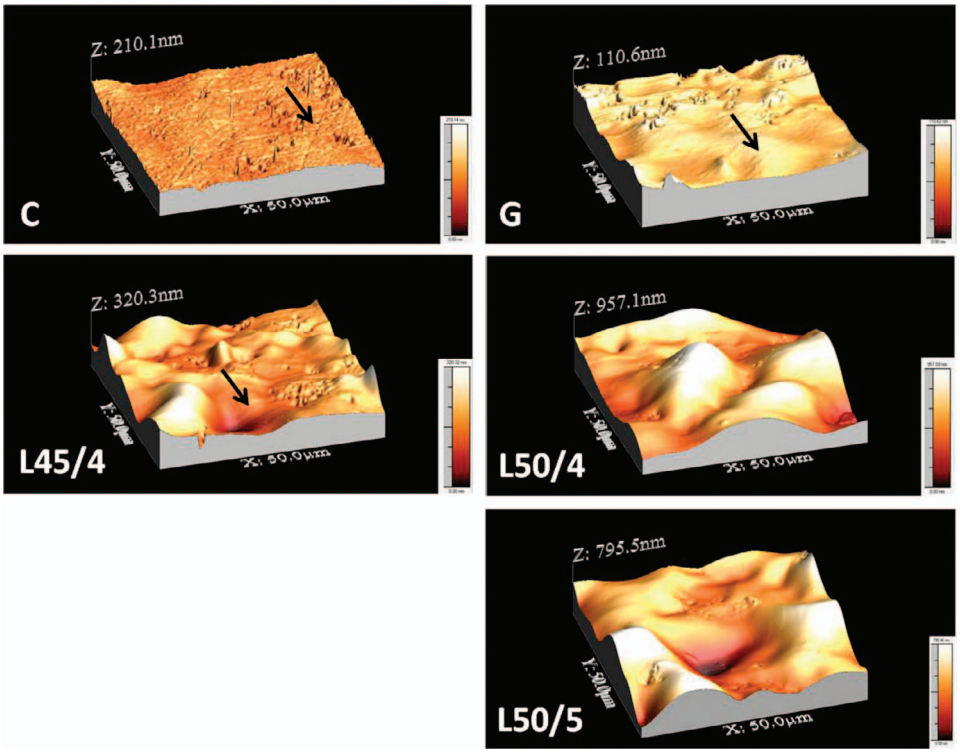

Figure 4. *Atomic force microscopy. Arrow indicates grooves originated from polishing procedure. In groups L50/4 and L50/5 the grooves are not present. For all laser groups (L) it is possible to note the profile, characterized by rounded, elongated peaks and deep valleys.*

CONCLUSIONS

The surface porosity and the masking ability observed in specimens submitted to laser treatment were similar to those of specimens auto-glazed in a furnace. Laser-glazed specimens presented a surface profile characterized by rounded and elongated peaks and deep valleys when assessed by AFM. At lower magnification, those surface changes resulted in striation formation, as observed by optical microscopy. With regard to mechanical properties, CO_2 laser glazing resulted in an increase in microhardness and changed the apparent fracture toughness of porcelain, depending on the irradiance/time tested.

Acknowledgements

The authors would like to thank Laboratório de Filmes Finos/ Institute of Physics – USP, São Paulo, for the AFM facilities and CAPES (Coordination for the Improvement of Higher Education Personnel) for their financial support.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 27 May 2014)

REFERENCES

1. Romero M, Rincon JM, & Acosta A (2002) Effect of iron oxide content on the crystallization of a diopside glass-ceramic glaze *Journal of the European Ceramic Society* **22(6)** 883-890.
2. Raimondo RL Jr, Richardson JT, & Wiedner B (1990) Polished versus autoglazed dental porcelain *Journal of Prosthetic Dentistry* **64(5)** 553-557.
3. Fuzzi M, Zaccheroni Z, & Vallania G (1996) Scanning electron microscopy and profilometer evaluation of glazed and polished dental porcelain *International Journal of Prosthodontics* **9(5)** 452-458.
4. Barghi N, King CJ, & Draughn RA (1975) A study of porcelain surfaces as utilized in fixed prosthodontics *Journal of Prosthetic Dentistry* **34(3)** 314-319.
5. Griggs JA, Thompson JY, & Anusavice KJ (1996) Effects of flaw size and auto-glaze treatment on porcelain strength *Journal of Dental Research* **75(6)** 1414-1417.
6. Clayton JA, & Green E (1970) Roughness of pontic materials and dental plaque *Journal of Prosthetic Dentistry* **23(4)** 407-411.
7. Podshadley AG, & Harrison JD (1966) Rat connective tissue response to pontic materials *Journal of Prosthetic Dentistry* **16(1)** 110-118.
8. Williamson RT, Kovarik RE, & Mitchell RJ (1996) Effects of grinding, polishing, and overglazing on the flexure strength of a high-leucite feldspathic porcelain *International Journal of Prosthodontics* **9(1)** 30-37.

9. Chu FC, Frankel N, & Smales RJ (2000) Surface roughness and flexural strength of self-glazed, polished, and reglazed In-Ceram/Vitadur Alpha porcelain laminates *International Journal of Prosthodontics* **13(1)** 66-71.
10. Fischer H, Schafer M, & Marx R (2003) Effect of surface roughness on flexural strength of veneer ceramics *Journal of Dental Research* **82(12)** 972-975.
11. Albakry M, Guazzato M, & Swain MV (2004) Effect of sandblasting, grinding, polishing and glazing on the flexural strength of two pressable all-ceramic dental materials *Journal of Dentistry* **32(2)** 91-99.
12. de Jager N, Feilzer AJ, & Davidson CL (2000) The influence of surface roughness on porcelain strength *Dental Materials* **16(6)** 381-388.
13. Quinn GD, Hoffman K, & Quinn JB (2012) Strength and fracture origins of a feldspathic porcelain *Dental Materials* **28(5)** 502-511.
14. Albakry M, Guazzato M, & Swain MV (2004) Influence of hot pressing on the microstructure and fracture toughness of two pressable dental glass-ceramics *Journal of Biomedical Materials Research Part B Applied Biomaterials* **71(1)** 99-107.
15. Scherrer SS, Kelly JR, Quinn GD, & Xu K (1999) Fracture toughness (KIc) of a dental porcelain determined by fractographic analysis *Dental Materials* **15(5)** 342-348.
16. Fredericci C, Yoshimura HN, Molisani AL, Pinto MM, & Cesar PF (2011) Effect of temperature and heating rate on the sintering of leucite-based dental porcelains *Ceramics International* **37(3)** 1073-1078.
17. Apetz R, & van Bruggen MPB (2003) Transparent alumina: A light-scattering model *Journal of the American Ceramic Society* **86(3)** 480-486.
18. Yoshimura HN, & Goldenstein H (2009) Light scattering in polycrystalline alumina with bi-dimensionally large surface grains *Journal of the European Ceramic Society* **29(2)** 293-303.
19. Prasad S, Monaco EA Jr, Kim H, Davis EL, & Brewer JD (2009) Comparison of porcelain surface and flexural strength obtained by microwave and conventional oven glazing *Journal of Prosthetic Dentistry* **101(1)** 20-28.
20. Sgura R, Reis MC, Hernandes AC, Fantini MCA, Andreeta MRB, & Medeiros IS (2013) Surface treatment of dental porcelain: CO_2 laser as an alternative to oven glaze *Lasers in Medical Science* http://dx.doi.org/10.1007/s10103-013-1392-4
21. Lawn BR, Evans AG, & Marshall DB (1980) Elastic-plastic indentation damage in ceramics—The median-radial crack system *Journal of the American Ceramic Society* **63(9-10)** 574-581.
22. Vichi A, Louca C, Corciolani G, & Ferrari M (2011) Color related to ceramic and zirconia restorations: A review *Dental Materials* **27(1)** 97-108.
23. Horcas I, Fernandez R, Gomez-Rodriguez JM, Colchero J, Gómez-Herrero J, & Baro AM (2007) WSXM: A software for scanning probe microscopy and a tool for nanotechnology *Review of Scientific Instruments* **78(1)** http://dx.doi.org/10.1063/1.2432410
24. Tholt de Vasconcellos B, Miranda-Junior WG, Prioli R, Thompson J, & Oda M (2006) Surface roughness in ceramics with different finishing techniques using atomic force microscope and profilometer *Operative Dentistry* **31(4)** 442-449.
25. Wee LM, Crouse PL, & Li L (2008) A statistical analysis of striation formation during laser cutting of ceramics *International Journal of Advanced Manufacturing Technology* **36(7)** 699-706.

©Operative Dentistry, 2015, **40-3,** 255-262

Occlusal Caries Extension in Relation to Visual and Radiographic Diagnostic Criteria: Results from a Microcomputed Tomography Study

K Bücher • M Galler • M Seitz
R Hickel • K-H Kunzelmann • J Kühnisch

Clinical Relevance

The use of a combination of visual and radiographic criteria for caries diagnostics results in fewer invasive treatment decisions.

SUMMARY

Objective: This *in vitro* study aimed to evaluate occlusal caries extension in relation to visual and radiographic diagnostic criteria and their clinical value to indicate operative or preventive dental care.

Methods: A total of 196 third molars with clinically sound occlusal fissures or noncavitated lesions were collected. Before microcomputed tomography (μCT) investigation, each tooth was examined visually and radiographically. Kühnisch's μCT-based caries-extension index (CE index) was used to determine the caries depth on a numeric scale (0 = sound; 0.01-0.99 = enamel caries; 1.0-1.99 = dentin caries). Sensitivities (SEs), specificities (SPs), and area under the receiver operating characteristic curve (Az value) were also calculated.

Results: Based on μCT data, the following mean CE index values and standard deviations (SDs) were documented according to the visual criteria: sound = 0.6 (0.4); first visible signs = 0.9 (0.4); established lesions = 1.3 (0.3); microcavities = 1.4 (0.2); dentin exposure = 1.5 (0.2); and large cavities = 1.5 (0.3). The radiographic categories according to Marthaler (enamel

*Katharina Bücher, Dr. med. dent., assistant professor, Ludwig-Maximilians University, Operative Dentistry and Periodontology, Munich, Germany

Matthias Galler, Dr. med. dent., Ludwig-Maximilians University, Operative Dentistry and Periodontology, Munich, Germany

Michael Seitz, Dr. med. dent., Ludwig-Maximilians University, Operative Dentistry and Periodontology, Munich, Germany

Reinhard Hickel, Prof. Dr. med. dent., professor, head of the department, Ludwig-Maximilians University, Operative Dentistry and Periodontology, Munich, Germany

Karl-Heinz Kunzelmann, Prof. Dr. med. dent., professor, Ludwig-Maximilians University, Operative Dentistry and Periodontology, Munich, Germany

Jan Kühnisch, PD Dr. med. dent., assistant professor, Ludwig-Maximilians University, Department of Conservative Dentistry, Munich, Germany

*Corresponding author: Goethestr. 70, Munich, 80336, Germany; e-mail: kbuecher@dent.med.uni-muenchen.de

DOI: 10.2341/13-128-L

caries [D0-2], caries in the outer half of dentin [D3], and caries in the inner half of dentin [D4]) were related to CE index values of 0.9 (0.4), 1.4 (0.2) and 1.6 (0.4), respectively. Caries detected visually or radiographically showed an SE of 84% and an SP of 85% (Az = 0.85). When both methods were used to predict dentin involvement simultaneously, SE = 27%, SP = 100%, and Az = 0.63; this combined visual and radiographic approach was associated with a perfect specificity and no false-negative decisions. The proportion of false-positive diagnoses was moderately high, and lesion extension in these cases was mainly limited to the outer 20% of the dentin.

Conclusions: Our results might be useful for differentiating between preventive and operative dental care for pits and fissures.

INTRODUCTION

Comprehensive caries treatment protocols include preventive and operative measures to reduce ongoing damage of the dental hard tissue. Precise caries diagnostic methods are needed to determine whether remineralization of the damaged tissue is appropriate in a single tooth or if removal of the diseased tissue is necessary. These caries diagnostic methods should reliably predict (dentin) caries lesions and provide valid cutoff values or intervals to support shifting from preventive to operative treatment for each caries site. Most recently, some studies have indicated that caries activity and patient-related risk factors should be used as primary indicators to decide whether restorative treatment is necessary.[1-3] Due to the phenomenon of hidden occlusal caries, which is often linked to a noncavitated but deep caries lesion, it seems worthwhile to rethink using caries activity and risk factors to determine treatment.[4-6] Therefore, it seems to be necessary to discuss the caries extension as another fundamental factor for the decision-making process.

Caries extension is mainly determined semiquantitatively according to the D classification system, which is used for diagnostic and histologic purposes.[7] The D3 score describes dentin caries that ranges from that just penetrating the enamel-dentin junction up to lesions comprising the complete outer half of the dentin and has limitations in differentiated treatment recommendations, such as caries monitoring, pit and fissure sealants, or invasive restorative treatment. Therefore, using a quantitative measure, such as the caries extension (CE) index appears to be a more precise approach because it incorporates caries depth in relation to overall hard tissue thickness.[8] Although high-resolution and nondestructive microcomputed tomography (μCT) has not yet been evaluated for *in vitro* investigation of occlusal caries, it appears to be another innovative method to determine the number or percentage of caries extensions as precisely as possible and may enhance the informative value of validation studies.

Therefore, our investigation aimed to determine the diagnostic performance and caries extension of the routinely used visual and radiographic caries diagnostic criteria and the combination of both methods on occlusal surfaces using μCT and the CE index as reference standards. Based on these data, potential thresholds or cutoff intervals were determined that can be used to distinguish between preventive and operative dental care.

METHODS AND MATERIALS

Sample Size

A sample of 196 seemingly sound or noncavitated third molars was selected from a pool of teeth extracted for surgical or orthodontic reasons. Molar teeth with sealants, fillings, or caries lesions on their smooth surfaces were excluded. After the gross debris was removed, the teeth were carefully cleaned with an airflow device (ProphyFlex, KaVo, Biberach, Germany) and a rotating bristle brush. To prevent bacterial growth, all of the teeth were stored in separate containers with physiologic saline containing 0.02% sodium azide.

Determining the Universal Visual Scoring System Consensus Diagnosis

All of the teeth were examined visually using compressed air and illumination from a dental unit light. The visual inspection was carried out according to the Universal Visual Scoring System (UniViSS)[8,9] under the following principles: for detectable occlusal caries lesions, the severity (first signs, established lesion, microcavity, or dentin exposure) and discoloration (white, brown, or white-brown) were assessed separately with respect to opalescence and enamel translucency as well as tactile inspection with a blunt probe (ISO 21672-2:2012) (Figure 1a).[8,9] Two dentists (M.G. and M.S.) performed visual inspections independently. All of the assessments were cross-checked two weeks later with an experienced dentist (J.K.) to form a consensus diagnosis for each surface. In case of

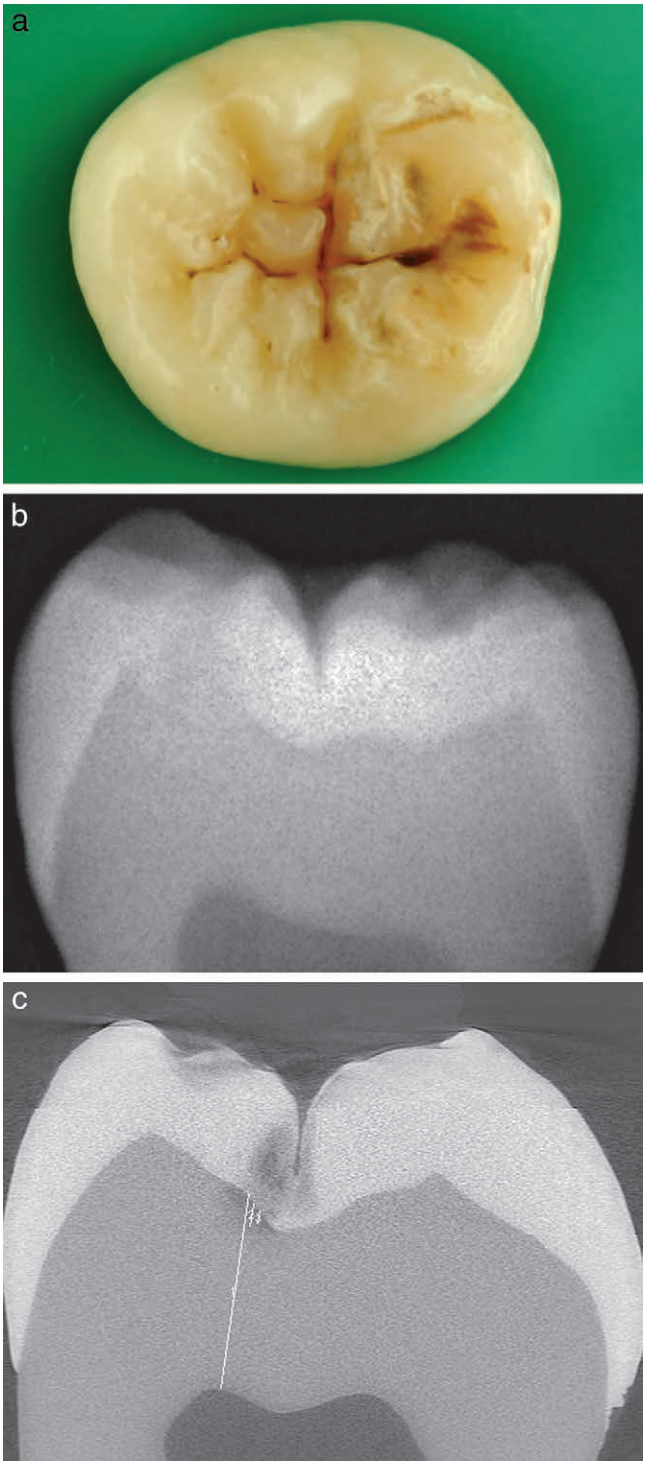

Figure 1. *(a): The occlusal lesion of the central fissures shows a white-brown lesion with dentin exposure that was also detectable with a blunt probe. After use of the Universal Visual Scoring System (UniViSS), preventive measures only were recommended for this tooth. (b): No dentin caries was detectable in the x-ray. The tooth was classified as enamel caries (D0-2 after Marthaler). (c): The micro-computed tomography slice reveals a dentin caries lesion near the enamel-dentin junction (D3 after Marthaler). A maximum score of 1.11 was calculated according to the Caries Extension Index.*

different findings, all of the examiners discussed the discordant findings to reach a consensus.

Determining the Digital Radiography Consensus Diagnosis

All of the digital radiographs were taken using a charged-coupled device system (Sidexis, Sirona, Bensheim, Germany). The exposure time was 0.06 seconds at a cathode voltage of 60 kV and a cathode current of 7 mA. A standardized alignment fixture with a soft-tissue scattering equivalent was used to reproduce the exact object-to-film distance. Two dentists (M.G. and M.S.) evaluated the digital radiographs independently in a darkened room using system-related analysis software (version 2.53, Sirona), with the option to adjust the brightness and contrast. Each examiner determined the caries depth on each radiograph semiquantitatively according to the Marthaler categories of D0-2 for enamel and D3 and D4 for dentin caries and quantitatively by the CE index (Figure 1b).[7,8] All of the radiographic decisions were reassessed two weeks later in collaboration with an experienced examiner (J.K.) to arrive at a consensus diagnosis. When the examiners came to different conclusions, they reassessed the corresponding radiograph, discussed their points, and modified the diagnosis accordingly.

Microcomputed Tomography

A high-resolution μCT system (μCT40, Scanco Medical, Basserdorf, Switzerland) was used to obtain digital three-dimensional (3D) data sets of the teeth. The x-ray source was set at 70 kV and 114 μA. The sample time was 600 milliseconds. Image resolution was fixed at a voxel size of 20 μm. For further evaluation, the reconstructed data sets were imported into ImageJ (ImageJ, US National Institutes of Health, Bethesda, MD, USA; http://rsb.info.nih.gov/ij).[10-12] The analysis of each 3D data set was performed by two blinded dentists (M.G. and M.S.). In the first step they detected the deepest caries progression. In the second step, the caries extension in the enamel and dentin toward the pulp was determined, as well as the enamel and dentin thicknesses, to calculate the CE index (Figure 1c).[8] All of the data sets were reassessed two weeks later by a third examiner (J.K.), and the decisions were finalized for each specimen. In case of different findings, all of the examiners discussed their discordant measures and reached a consensus. Specimens with demarcated defects in the dentin

Table 1: *Visual Caries Diagnosis in Relation to Caries Depth[1]: Number of Surfaces Classified and Mean (SD) CE Index Values*

N (Surfaces) **Mean CE Index** (sd)/ Min-Max		Visual discoloration					
		None	**White**	**White-brown**	**Brown**	**Greyish**	∑
Visual severity	**Sound**	35 **0.6** (0.4)/ 0-1.1	-	-	-	-	35 **0.6** (0.4)/ 0-1.1
	First visible signs	-	29 **0.8** (0.3)/ 0-1.5	13 **1.2** (0.1)/ 1.0-1.4	8 **0.9** (0.3)/ 0.21.4	-	50 **0.9** (0.4)/ 0-1.5
	Established lesion	-	7 **1.1** (0.3)/ 0.4-1.4	54 **1.3** (0.2)/ 0.8-1.7	9 **1.1** (0.3)/ 0.5-1.5	1 **1.2** (-)/ 1.2-1.2	71 **1.3** (0.3)/ 0.4-1.7
	Microcavity	-	2 **1.4** (0.3)/ 1.2-1.6	20 **1.4** (0.2)/ 1.0-1.7	2 **1.6** (0.0)/ 1.6-1.7	1 **1.7** (-)/ 1.7-1.7	25 **1.4** (0.2)/ 1.0-1.7
	Dentin exposure	-	-	8 **1.4** (0.2)/ 1.1-1.7	1 **1.6** (-)/ 1.6-1.6	-	9 **1.5** (0.2)/ 1.1-1.7
	Large cavity	-	-	3 **1.6** (0.4)/ 1.2-1.9	3 **1.8** (0.1)/ 1.6-1.9	-	6 **1.7** (0.3)/ 1.2-1.9
	∑	35 **0.6** (0.4)/ 0-1.1	38 **0.9** (0.4)/ 0-1.6	98 **1.3** (0.2)/ 0.8-1.9	23 **1.2** (0.4)/ 0.2-1.9	2 **1.4** (0.3)/ 1.2-1.7	196 **1.1** (0.4)/ 0-1.9

[1] Caries depth was quantified using microcomputed tomography images and the caries extension index.
[2] The bold/dashed line illustrates the cutoff threshold for caries detection. Based on mean CE index values >1.0, the cutoff threshold (bold line only) for detecting dentin caries lesions was determined.

beneath the enamel-dentin junction were excluded from this investigation.[6]

Statistical Analysis

The data were analyzed using Excel 2007 (Microsoft Corporation, Redmond, WA, USA) and SPSS 15.0 (SPSS Inc, Chicago, IL, USA) to cross-tabulate the findings and calculate the mean (mean), standard deviation (SD), and minimum (min) and maximum (max) values of the CE index for each diagnostic score. A CE index of 0 was associated with sound surfaces, and values between 0.01 and 1.0 corresponded with enamel caries lesions. Dentin caries lesions corresponded with a CE index value between 1.01 and 1.99.[9] Overall diagnostic performance was determined by calculating the sensitivity (SE), specificity (SP), positive predictive value, negative predictive value, and area under the receiver operating characteristic (ROC) curve (Az). An Az value near 1 indicated a high accuracy.[13] Performances of all diagnostic methods were evaluated separately. In addition, two combinations of both methods were tested. First, diagnostic performance was calculated for those teeth in which at least one of the methods had predicted dentin caries (visual or radiographic). Second, the performance was evaluated if both visual and radiographic methods diagnosed dentin caries simultaneously (visual and radiographic).

RESULTS

Table 1 summarizes the CE index based on the μCT data in relation to each UniViSS score. In general, established lesions and microcavities were linked to a dentin caries lesion. In cases of established lesions, a white-brown discoloration indicated deeper caries lesions (CE index = 1.3) compared with white or brown discolorations (CE index = 1.1). Microcavities reached nearly half of the dentin.

The CE index for the radiologic findings is reported in Table 2. Interestingly, 44.2% (53/120) of all radiographically diagnosed D0-2 lesions showed caries penetration into the dentin with μCT. However, the mean CE index of 1.2, which indicates a 20% extension into the dentin below the enamel-dentin junction, suggested minimal dentin involvement. All of the D3-4 lesions observed on radiography were found in the dentin and were correlated with a mean CE index of at least 1.4 according to μCT (40% dentin caries extension, Table 2).

The combined visual and radiographic diagnostic evaluation was also analyzed in relation to the caries extension (Table 3). All of the caries lesions associated with a D0-2 lesion on the dental radiographs

Table 2: *Radiographic Diagnosis for All Occlusal Surfaces in Relation Caries Depth Quantified Using Microcomputed Tomography (μCT) Images and the Caries Extension (CE) Index*

Digital Radiography	CE Index (μCT)[1]					∑
	D0	D1	D2	D3	D4	
No dentin caries	10; **0.0** (0.0)/ 0.0-0.0	12; **0.3** (0.1)/ 0.2-0.5	45; **0.8** (0.1)/ 0.5-1.0	53; **1.2** (0.1)/ 1.0-1.4	-	120; **0.9** (0.4)/ 0-1.4
D3	1; **0.0** (0.0)/ 0.0-0.0	-	-	39; **1.4** (0.1)/ 1.1-1.5	21; **1.6** (0.1)/ 1.5-1.7	61; **1.4** (0.2)/ 0.0-1.7
D4	-	-	-	4; **1.4** (0.2)/ 1.2-1.3	11; **1.7** (0.1)/ 1.6-1.9	15; **1.6** (0.2)/ 1.2-1.9
∑	11; **0.0** (0.0)/ 0.0-0.0	12; **0.3** (0.1)/ 0.2-0.5	45; **0.8** (0.1)/ 0.5-1.0	96; **1.2** (0.1)/ 1.0-1.5	32; **1.6** (0.1)/ 1.5-1.9	196; **1.1** (0.4)/ 0-1.9

[1] For each Marthaler category (D0–D4), values shown are number of classified surfaces, mean (SD) CE index value (boldfaced)/minimum-maximum.

showed minimal caries extension into the dentin and were associated with a maximum CE index of 1.2. Radiographically detectable D3 and D4 lesions were linked to a mean CE index of at least 1.4 and 1.6, respectively (Table 3).

The overall diagnostic performances of the visual, radiographic, and visual-radiographic techniques for (dentin) caries detection are reported in Table 4. The Az values under the ROC curves were found to be greater than 0.75, with the exception of the combined visual and radiographic approach, which had an Az value of 0.63. The overall specificity was near 1.0.

DISCUSSION

The main finding of our study was that the overall diagnostic performances for visual caries detection and diagnosis ranged in a good order of magnitude (Table 4). This finding is remarkable because most of the lesions were noncavitated caries lesions, which

Table 3: *Combined Visual and Radiographic Assessment for All Occlusal Surfaces in Relation to Caries Depth Quantified Using Microcomputed Tomography (μCT) Images and the Caries Extension (CE) Index*

Severity	Discoloration	Digital Radiography[1]			∑
		No Dentin Caries	D3	D4	
Sound	None	34; **0.6** (0.4)/0.0-1.1	1; **0.0** (-)/0.0-0.0	-	35; **0.6** (0.4)/0.0-1.1
First visible signs	White	28; **0.8** (0.3)/0.0-1.2	1; **1.3** (-)/1.3-1.3	-	29; **0.8** (0.3)/ 0.0-1.5
	White-brown	8; **1.2** (0.1)/1.0-1.3	5; **1.3** (0.1)/1.2-1.4	-	13; **1.2** (0.1)/1.0-1.4
	Brown	8; **0.9** (0.3)/0.2-1.4	-	-	8; **0.9** (0.3)/0.2-1.4
Established lesion	White	4; **1.0** (0.4)/0.4-1.2	3; **1.4** (0.1)/1.3-1.4	-	7; **1.1** (0.3)/0.4-1.4
	White-brown	23; **1.1** (0.1)/0.8-1.3	28; **1.4** (0.1)/1.1-1.7	3; **1.6** (0.1)/1.5-1.7	54; **1.3** (0.2) /0.8-1.7
	Brown	7; **1.0** (0.3)/0.5-1.3	2; **1.4** (0.1)/1.4-1.5	-	9; **1.1** (0.3)/ 0.5-1.5
	Greyish	1; **1.2** (-)/1.2-1.2	-	-	1; **1.2** (-)/1.2-1.2
Microcavity	White	1; **1.2** (-)/1.2-1.2	1; **1.6** (-)/1.6-1.6	-	2; **1.4** (0.3)/1.2-1.6
	White-brown	5; **1.1** (0.2)/1.0-1.4	9; **1.5** (0.1)/1.3-1.7	6; **1.5** (0.2)/1.2-1.7	20; **1.4** (0.2)/1.0-1.7
	Brown	-	1; **1.6** (-)/1.6-1.6	1; **1.7** (-)/ 1.7-1.7	2; **1.6** (0.0)/1.6-1.7
	Greyish	-	1; **1.7** (-) /1.7-1.7	-	1; **1.7** (-)/1.7-1.7
Dentin exposure	White	-	-	-	-
	White-brown	1; **1.1** (-)/ 1.1-1.1	6; **1.4** (0.1)/1.3-1.7	1; **1.7** (-)/1.7-1.7	8; **1.4** (0.2)/1.1-1.7
	Brown	-	1; **1.6** (-)/1.6-1.6	-	1; **1.6** (-)/1.6-1.6
	Greyish	-	-	-	-
Large cavity	White	-	-	-	-
	White-brown	-	2; **1.5** (0.3)/1.2-1.7	1; **1.9** (-)/1.9-1.9	3; **1.6** (0.4)/1.2-1.9
	Brown	-	-	3; **1.7** (0.1)/1.6-1.9	3; **1.7** (0.1)/1.6-1.9
∑		120; **0.9** (0.4)/0.0-1.4	61;**1.4** (0.2)/0.0-1.7	15; **1.6** (0.2)/1.2-1.9	196; **1.1** (0.4)/0.0-1.9

[1] For each group classified according to UniViSS and Marthaler, values shown are number of classified surfaces, mean (SD) CE index values (boldfaced)/minimum-maximum.

Table 4: *Both Investigated Visual-Radiographic Approaches (Visual or Radiographic; Visual and Radiographic) Resulted in Different Findings*

	SE	SP	NPV	PPV	Az Value
Caries detection level (D0 versus D1-4)					
Visual (UniViSS)	0.86	0.91	0.29	0.99	0.82
Digital radiographic Visual or radiographic Visual and radiographic	*Impossible to calculate because of the limited ability to detect enamel caries lesion on dental radiographs*				
Dentin caries detection level (D0-2 versus D3-4)					
Visual (UniViSS)	0.87	1.00	0.28	1.00	0.89
Digital radiographic	0.59	0.99	0.56	0.99	0.79
Visual or radiographic[1]	0.84	0.85	0.74	0.92	0.85
Visual and radiographic[2]	0.27	1.00	0.42	1.00	0.63

Abbreviations: SE, sensitivity; SP, specificity; NPV, negative predictive value; PPV, positive predictive value; Az value, area under the receiver operating characteristic curve; UniViSS, Universal Visual Scoring System.
[1] A true positive test result was assumed if at least one of the test methods indicated a dentin caries lesion and the microcomputed tomography revealed a caries extension index value >1.00.
[2] A true positive test result was assumed if both of the test methods indicated a dentin caries lesion and the microcomputed tomography revealed a caries extension index value >1.00.

are occasionally difficult to classify correctly. Established caries lesions and lesions that were more severe were always associated with dentin involvement. Lesions that had been diagnosed visually as established were found mostly in the outer third of the dentin according to μCT (mean CE index between 1.1 and 1.3; Table 1). More severe lesions were always connected with caries progression into the middle of the dentin (CE index >1.4; Table 1).

Another interesting finding involved the visually detectable discoloration of the caries lesion. Surfaces with white discolorations showed less caries extension compared to surfaces with brown discolorations. In addition, lesions with white-brown discolorations had progressed farther than other lesions (Table 1). Therefore, it can be argued that discoloration might be used as a clinical predictor of caries activity and should be included generally into visual assessments.

For digital radiography, a high sensitivity (0.99) and reduced specificity (0.59) were recorded. These values were in line with previously published studies and illustrated the high ability of these techniques to correctly classify sound occlusal surfaces as healthy (true negatives).[14-16] In contrast, identification of dentin caries lesions (true positives) is limited when using low-dose digital radiography. In 53 teeth evaluated as D0-2 lesions on digital radiographs, the real caries extension had progressed into the dentin and therefore farther than predicted (Table 2). However, when taking into account the CE index for these lesions, most of the lesions were located near the enamel-dentin junction (mean CE index 1.2; Table 2). Overall, digital radiography underestimated the real dentin caries extension.

A unique feature of our study was the analysis of the diagnostic outcome of the combined visual-radiographic diagnostics in relation to the quantitative caries extension determined according to μCT (Tables 3 and 4). Both investigated visual-radiographic approaches (visual or radiographic; visual and radiographic) resulted in different findings (Table 4). The best diagnostic performance was found when pit and fissure caries were diagnosed either visually or radiographically (SE=84%; SP=85%; Az=0.85). The second approach, which involved accepting dentin caries only when visual investigation and digital radiography predicted dentin involvement simultaneously, had a lower SE (27%) and Az value (0.63) but classified all sound teeth correctly (SP=100%; positive predictive value = 100%) (Table 4). This diagnostic approach would avoid overtreatment best because these false-negatives that were verified by μCT did not need operative care as they showed either enamel caries or only small dentin involvement of only 20% (mean CE index 1.2; Tables 2 and 3). Hence, nonoperative preventive dental care, including options such as pit and fissure sealing and/or regular fluoride applications, would be proposed as the treatment option of choice to reduce caries progression effectively.[17-19] However, other reports have recommended early operative intervention strategies for these lesions.[20] Therefore, studies should be initiated to investigate this ongoing discussion and dilemma.

The strength of this validation study lies in the large number of samples: 196 molars. To our knowledge, this number has been exceeded by only one previously published *in vitro* study.[21] Another unique feature was the use of μCT technology, with ~400 separate scans for each surface. This enabled a quantitative measurement of caries extension for each diagnostic score, which added valuable new data (Tables 1 through 3) to the conventional analysis of the diagnostic performance (Table 4). A possible limitation of the present study was that all of the teeth were third molars, which are generally characterized by a much more irregular fissure pattern than first or second permanent molars and premolars. Sample groups with different compositions should be taken into consideration when comparing our results with others and translating the documented experiences into clinical practice.

CONCLUSION

Simultaneous visual and radiographic prediction of dentin caries seems to result in a more conservative approach in comparison to visual or radiographic diagnostic only. Nevertheless, it has to be accepted that small dentin caries lesion will not be detected.

Acknowledgement

This study was supported by a grant from the German Society of Conservative Dentistry (DGZ-GABA-Wissenschaftsfond, AZ: DGZ-GABA-WF/Bücher 2010-1).

Conflict of Interest

The Authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

(Accepted 27 November 2013)

REFERENCES

1. Fejerskov O, & Kidd EA (2008) *Dental Caries. The Disease and Its Clinical Management*. Blackwell Publishing Ltd, Oxford **2nd edition**.
2. Pitts NB, Richards D, & International Caries Detection and Assessment System Committee (2009) Personalized treatment planning *Monographs in Oral Science* **21** 128-143, http://dx.doi.org/10.1159/000224217.
3. Pitts NB (2009) How the detection, assessment, diagnosis and monitoring of caries integrate with personalized caries management *Monographs in Oral Science* **21** 1-14, http://dx.doi.org/10.1159/000224211.
4. Weerheijm KL, Gruythuysen RJ, & van Amerongen WE (1992) Prevalence of hidden caries *Journal of Dentistry for Children* **59(6)** 408-412.
5. Ricketts D, Kidd E, Weerheijm K, & de Soet H (1997) Hidden caries: what is it? Does it exist? Does it matter? *International Dental Journal* **47(5)** 259-265.
6. Kühnisch J, Galler M, Seitz M, Hickel R, Kunzelmann KH, & Bücher K (2012) Developmental defects below the enamel-dentine-junction could explain the early onset of occlusal caries lesions *Journal of Dental Research* **91(11)** 1066-1070.
7. Marthaler TM (1966) A standardized system of recording dental conditions *Helvetica Odontologica Acta* **10(1)** 1-18.
8. Kühnisch J, Bücher K, Henschel V, Albrecht A, Garcia-Godoy F, Mansmann U, Hickel R, & Heinrich-Weltzien R (2011) Diagnostic performance of the Universal Visual Scoring System (UniViSS) on occlusal surfaces *Clinical Oral Investigations* **15(2)** 215-223.
9. Kühnisch J, Goddon I, Berger S, Senkel H, Bücher K, Oehme T, Hickel R, & Heinrich-Weltzien R (2009) Development, methodology and potential of the new universal visual scoring system (UniViSS) for caries detection and diagnosis *International Journal of Environmental Research and Public Health* **6(9)** 2500-2509.
10. Abramoff MD, Magalhaes PJ, & Ram SJ (2004) Image processing with ImageJ *Biophotonics International* **11(7)** 36-42.
11. Rasband WS (1997-2013) ImageJ, U.S National Institutes of Health, Bethesda, Maryland; retrieved online November 2013 from: http://imagej.nih.gov/ij/
12. Schneider CA, Rasband WS, & Eliceiri KW (2012) NIH Image to ImageJ: 25 years of image analysis *Nature Methods* **9** 671-675.
13. Matthews DE, & Farewell VT (2007) *Using and understanding medical statistics*. S. Karger GmbH, Freiburg **4th edition**.
14. Kühnisch J, Ifland S, Tranæus S, & Heinrich-Weltzien R (2009) Comparison of different radiographic methods for dentine caries detection on occlusal surfaces *Dentomaxillofacial Radiology* **38(7)** 452-457.
15. Ashley PF, Blinkhorn AS, & Davies RM (1998) Occlusal caries diagnosis: an in vitro histological validation of the Electronic Caries Monitor (ECM) and other methods *Journal of Dentistry* **26(2)** 83-88.
16. Young SM, Lee JT, Hodges RJ, Chang TL, Elashoff DA, & White SC (2009) A comparative study of high-resolution cone beam computed tomography and charged-coupled device sensors for detecting caries *Dentomaxillofacial Radiology* **38(7)** 445-451.
17. Welbury R, Raadal M, & Lygidakis NA (2004) EAPD guidelines for the use of pit and fissure sealants *European Journal of Paediatric Dentistry* **5(3)**179-184.
18. Griffin SO, Oong E, Kohn W, Vidakovic B, Gooch BF, CDC Dental Sealant Systematic Review Work Group, Bader J, Clarkson J, Fontana MR, Meyer DM, Rozier RG, Weintraub JA, & Zero DT (2008) The effectiveness of

sealants in managing caries lesions *Journal of Dental Research* **87(2)** 169-174.

19. Tubert-Jeannin S, Auclair C, Amsallem E, Tramini P, Gerbaud L, Ruffieux C, Schulte AG, Koch MJ, Rège-Walther M, & Ismail A (2011) Fluoride supplements (tablets, drops, lozenges or chewing gums) for preventing dental caries in children *Cochrane Database of Systematic Reviews* **7(12)** http://dx.doi.org/10.1002/14651858.CD007592
20. Betrisey E, Rizcalla N, Krejci I, & Ardu S. Caries diagnosis using light fluorescence devices: VistaProof and DIAGNOdent *Odontology* prepublished March 7, 2013, doi:10.1007/s10266-013-0105-6
21. Grossman ES, Cleaton-Jones PE, Cortes DF, Daya NP, Parak RB, Fatti LP, & Hargreaves JA (2002) Accurate diagnosis of occlusal carious lesions—a stereo microscope evaluation of clinical diagnosis *Journal of the South African Dental Association* **57(6)** 215-220.

©Operative Dentistry, 2015, **40-3,** 263-270

Effectiveness of Different Mechanical Methods on Dentin Caries Removal: Micro-CT and Digital Image Evaluation

C Ferraz • AR Freire • JS Mendonça
CAO Fernandes • JC Cardona • M Yamauti

Clinical Relevance

Selective caries removal is an important goal in dentistry, and caries-affected dentin should be preserved. Polymer burs could represent an important minimally invasive tool to preserve remineralizable dentin substrate during caries excavation.

SUMMARY

Purpose: To determine the caries removal effectiveness (CRE) and minimal invasive potential (MIP) of caries excavation methods using digital imaging and microtomography analyses.

Methods: Twelve human molars with occlusal caries lesions in dentin were randomly divided into three groups (carbide bur, exacavator, and polymer bur). They were sectioned mesiodistally, and standardized digital and computed microtomography x-ray (micro-CT) images were taken from each section before and after caries excavation. On each image, initial carious dentin (IC), prepared cavity (PC), and residual caries (RC) were defined according to visual criteria using ImageJ software. CRE was determined based on the RC/IC ratio, whereas MIP was determined by the PC/IC ratio. Data were analyzed using one-way analysis of variance and Student *t*-test or with Kruskal-Wallis and Student-Newman-Keuls test. The level of significance was set at 0.05.

Camila Ferraz, DDS, MS, Department of Restorative Dentistry, Faculty of Pharmacy, Dentistry and Nursing, Federal University of Ceará, Fortaleza, Brazil

Alexandre Rodrigues Freire, DDS, MS, Department of Morphology-Anatomy, Piracicaba Dental School, University of Campinas, Piracicaba, Brazil

Juliano Sartori Mendonça, DDS, MS, PhD, Department of Restorative Dentistry, Faculty of Pharmacy, Dentistry and Nursing, Federal University of Ceará, Fortaleza, Brazil

Carlos AO Fernandes, DDS, MS, PhD, Department of Restorative Dentistry, Faculty of Pharmacy, Dentistry and Nursing, Federal University of Ceará, Fortaleza, Brazil

Juan de la Cruz Cardona, DDO, PhD, Department of Optics, Facultad de Ciencias, University of Granada, Granada, Spain

*Monica Yamauti, DDS, MS, PhD, visiting professor, Department of Restorative Dentistry, Faculty of Pharmacy, Dentistry and Nursing, Federal University of Ceará, Fortaleza, Brazil

*Corresponding author: Fortaleza, 60430-355, Brazil; e-mail: myamauti@gmail.com

DOI: 10.2341/13-278-L

Results: For both digital image and micro-CT analysis, the carbide bur showed higher CRE values than the excavator (p=0.0063 and p=0.0263, respectively) and the polymer bur (p=0.0028 and p=0.0005, respectively). The latter two presented similar results (p>0.05). Regarding MIP, for the digital image analysis, the polymer bur was different from the carbide bur (p=0.0030) but was not different from that of the excavator (p=0.1240). For micro-CT analysis, the MIP values of all the groups were significantly different, and the polymer bur was the most conservative method (p<0.05).

Conclusions: The carbide bur was the most effective method for caries removal but was not completely conservative. The polymer bur and excavator presented low invasive potential but were not able to remove all of the carious dentin.

INTRODUCTION

In light of the trend toward minimally invasive dentistry, caries excavation has become more conservative.[1] Theoretically, only the superficial layer (ie, infected dentin that is strongly infected with viable microorganisms and presents irreversible denaturation and disorganization of the collagen fibers),[1] should be removed. Clinically, this is a difficult task to achieve. Traditionally, carious dentin is removed mechanically with excavator and/or slow-speed round diamond, tungsten carbide, or carbon-steel burs.[2,3] However, these techniques are indiscriminant and nonselective in removing carious tissues. Often during excavation, clinicians tend to include all soft, discolored, and stained tissue to ensure complete elimination of the infected layer.[4,5] In such cases, tactile and optical judgment is used to evaluate the consistency and color of the dental tissue. These criteria were shown to be adequate to ensure the removal of most of the infected dentin,[5] but they are still clinical and subjective parameters, dependent on the operators' experience.[1] Thus, their use frequently results in unnecessary removal of sound dentin or tissue with reduced mineral content, such as caries-affected dentin,[3,4] that is still a remineralizable organic matrix of the lesion and therefore must be preserved.[1,4]

Alternative caries removal methods such as polymer burs, air abrasion, and chemomechanical methods have been proposed to allow a less invasive/less destructive dentin caries excavation and to conserve tooth substance. A gentler, more comfortable and conservative caries excavation will provide a minimal thermal change, less vibration and pain, and removal of the infected dentin only.[6-10]

Polymer burs were first described by Boston in 2003.[10] They are made of a polyamide–imide polymer and present slightly lower mechanical properties than sound dentin. The blade design was developed to remove dentin by locally depressing the carious tissue and pushing it forward along the surface until it ruptures and is carried out of the cavity.[10] According to the manufacturer, this material is harder than infected dentin but softer than normal and sclerotic dentin, thus allowing a very selective caries removal. However, the efficacy of these new burs on selectively and efficiently removing natural carious dentin in permanent teeth is not well established.

X-ray computed microtomography (micro-CT) is a microscopic version of computed tomography that uses an x-ray–focused beam. It constitutes a nondestructive imaging method in which individual projections (radiographs) can be re-created in any plane[11] and images may be assessed qualitatively and quantitatively. More recently, studies have been developed regarding using this technique in the evaluation of enamel lesion caries, detection of proximal carious lesions, caries-excavation techniques, and restorative treatments. This seems to be promising in cariology.[2,6,11-14] Despite its many advantages, micro-CT is not yet a reliable method for caries detection, and for this reason, more studies are necessary to evaluate and define its feasibility as a diagnostic method.

The digital image analysis also represents a nondestructive imaging method and is a powerful tool for the study of a wide range of materials and parameters. Therefore, the use of digital imaging and subsequent image analysis is expected to successfully assess the performance of different types of burs in the carious excavation process.[15]

This study aimed to assess the caries removal effectiveness (CRE) and the minimal invasiveness potential (MIP) of mechanical excavation methods on dentin caries using microtomography and digital imaging analyses. The null hypotheses tested were (1) all mechanical methods will be equally effective at removing dentin caries and (2) none of the methods will remove sound dentin.

METHODS AND MATERIALS

Sample Preparation

Twelve extracted human molars with dentin caries on their occlusal surfaces were collected. Immedi-

ately after extraction, the teeth were properly cleaned and stored in a 0.1% (w/v) thymol solution for a period of no longer than three months. The roots of all the teeth were embedded in acrylic resin in a mold with dimensions of $2 \times 2 \times 2$ cm. They were then sectioned longitudinally in half through the center of the carious lesion using a low-speed diamond saw[15] (IsoMet Low Speed Saw, Buehler, Lake Bluff, IL, USA) under running water. A visual inspection was performed to determine the limits of the caries lesion. All of the teeth presented occlusal dentin caries. Teeth were randomly allocated to three experimental groups of four samples each.

Each section of tooth was digitally photographed[15] (Sony α300, Sony Corp, Tokyo, Japan), and micro-CTs (Skyscan 1174 high-resolution desktop micro-CT scanner, Skyscan, Kontich, Belgium) were taken before caries excavation. All images were assigned as "pre-rank." Tooth halves were reassembled with cyanoacrylate glue (Loctite Superbond, Henkel, São Paulo, Brazil) applied on the external surface of the crown, matching both halves together to remove the carious lesion.[15] Only one operator performed the caries removal. Training sessions were carried out by a principal investigator to calibrate the operator to remove caries using the three methods. The following describes the groups.

Group 1 (n=4)—Conventional round carbide bur. Burs 4, 6, and 8 (Dentsply Maillefer, Ballaigues, Switzerland) were used in a slow-speed hand piece without water cooling. Carious dentin was excavated with circular movements starting from the periphery to the center of the lesion.[16] Caries removal ended when hard dentin was detected using a nonflexible probe (SS White Duflex, Rio de Janeiro, Brazil). Dentin was considered hard when, under a firm pressure, the probe was not able to penetrate into the tissue.[4] For each tooth, a new carbide bur was used.[15]

Group 2 (n=4)—Excavator. Carious dentin was removed using excavators 14 and 19 (SS White Duflex). During excavation, the dentin hardness was checked and the carious dentin removal was completed when hard tissue was detected with the probe (SS White Duflex), as described for group 1. For each tooth, a new excavator was used.

Group 3 (n=4)—Polymer bur. SmartBurs II 4, 6, and 8 (SS White, Lakewood, NJ, USA) were used with a slow-speed handpiece without water cooling. Carious dentin was removed with circular movements starting from the center of the lesion to the periphery as recommended by the manufacturer. Excavation ended when the instrument became macroscopically abraded and blunted and was no longer able to remove tissue.[9,15] A probe was also used to check the hardness of the remaining tissue. For each tooth, new burs were used.[11]

During the time between procedures, the samples were stored in a physiological solution with a relative humidity of H=100%. After finishing the caries excavation, tooth halves were separated. Digital images and micro-CTs were taken and assigned as "post-rank."

Digital Images

The specimens were placed over a 45° tilted base inside a viewing cabinet (VeriVide CAC60, Verivide Ltd, UK) under constant CIE D65 illumination. For each sample, both before and after the caries removal procedure, 20 consecutive digital images were taken using a digital single lens reflex commercial camera (α300, Sony Corp) operating in manual mode with fixed parameters. The camera was calibrated using a spectroradiometer (PR704, Photo Research Inc, Chatsworth, CA, USA) and a Color Checker (Gretag Macbeth, New Windsor, NY, USA). The geometry of measuring/illumination used for the camera calibration was 0°/diffuse, and the CIE 1931 2° standard observer (CIE Bureau, 2004) was used to calculate the color.[15]

X-ray Micro-CT

The surfaces of all the halves were scanned before and after caries removal at 50 kV, 800 μA, and 14.1 μm pixel size using a 0.5-mm Al filter. This eliminates low-energy x-rays in a Skyscan 1174 high-resolution desktop micro-CT scanner (Skyscan, Kontich, Belgium). The rotation step was set to 0.70°, resulting in 264 two-dimensional projections over a 180° rotation of the specimen. A flat-field reference was taken before the start of time section scanner to improve the acquisition settings. The reconstruction program NRecon (NRecon, SkyScan) reads angular shadow 16-bit projection images saved by the control program and reconstructs virtual slices (cross sections). Before the reconstruction, beam-hardening correction was performed at 20%, with the same scanning parameters aiming at input of optimal contrast limits, based on prior scanning and reconstruction of the tooth.

After reconstruction, Dataviewer software (SkyScan, Kontich, Belgium) was used to select the two-dimensional image most similar to the respective digital image sample, and it was saved as a single image for further analyses and comparisons.

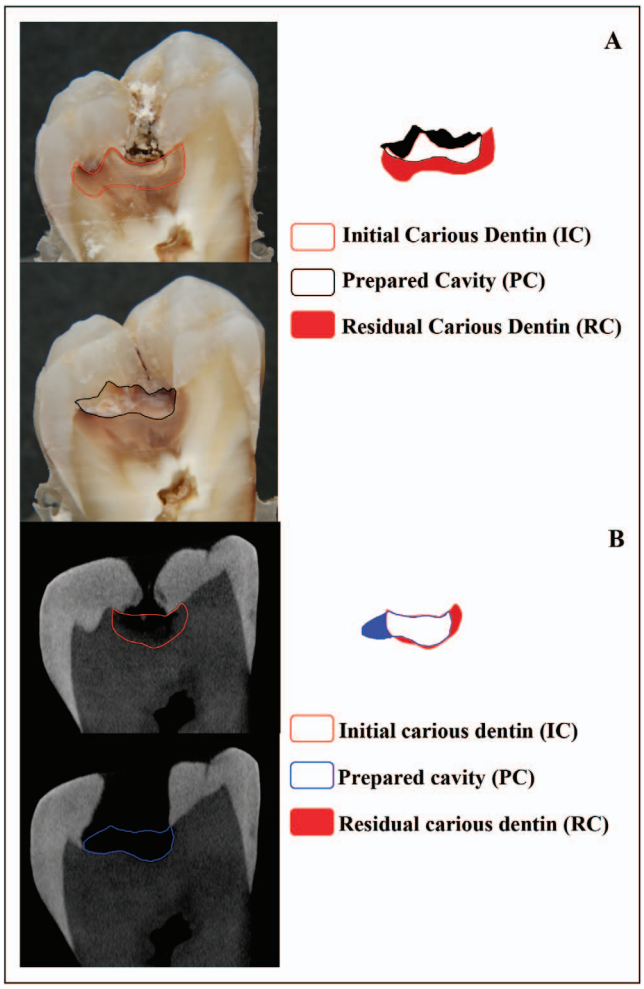

Figure 1. *Schematic representation of digital image and micro-CT analysis.*

Image Analysis

All of the steps, including the image processing, image analysis, and area measurement, were performed using the software ImageJ (National Institutes of Health, Bethesda, MD, USA). For each specimen, two areas of interest (initial carious dentin [IC] from pre-rank and prepared cavity [PC] from post-rank) were measured (Figure 1). The residual carious dentin (RC) and removed sound dentin (RS) areas were also determined (Figure 1). Digital image scaling and calibration were used to calculate the area values. The final result for each sample was the average over 20 images.[15]

Performance Assessment of Caries Removal Procedures

After obtaining all area measurements, performance assessment of the three different caries removal procedures was carried out by evaluating the CRE and the MIP indexes, supported in Neves and others.[6]

1. CRE: The effectiveness of caries removal of the three excavation methods was evaluated by means of the RC/IC ratio. The values of this parameter vary from 0 to 1, and high values represent less effective caries excavation.
2. MIP: The invasiveness of sound dentin of the three excavation methods was evaluated by means of the PC/IC ratio. The values of this parameter vary from 0 to 1. The higher the MIP value, the more invasive is the method.

Statistical Analysis

Means and standard deviations were determined for the described parameters (CRE and MIP). After testing for normality (Shapiro-Wilk test), those that did not pass were given nonparametric tests (Kruskall-Wallis and Student-Newman-Keuls). Those that did pass were given one-way analysis of variance and Student *t*-test. A value of $p<0.05$ was considered to be statistically significant.

RESULTS

Results of CRE and MIP are shown in Table 1 and Figure 2.

There were statistical differences for CRE between the groups ($p=0.0034$) according to digital imaging analysis. The carbide bur was statistically more effective in removing caries (0.00 ± 0.01) than was the excavator ($p=0.0063$) and the polymer bur ($p=0.0028$). The excavator and the polymer bur showed similar effectiveness ($p=0.8957$). Statistical differences were found for MIP between the groups ($p=0.0122$). The carbide bur and the excavator did not show significant differences regarding their MIP to remove sound dentin ($p=0.2092$). The MIP values of the polymer bur (0.57 ± 0.27) were statistically different from those of the carbide bur ($p=0.0030$) but were not statistically different from the excavator ($p=0.1240$).

There was a statistical difference for CRE between the groups ($p=0.0020$) according to micro-CT image analysis. The carbide bur presented greater effectiveness (0.00 ± 0.00) than did the excavator ($p=0.0263$) and the polymer bur ($p=0.0005$). There was a statistically significant difference for MIP between the groups investigated ($p=0.0001$). The carbide bur was the most invasive method (1.12 ± 0.14), and its MIP values were statistically different from those of the excavator ($p=0.0030$) and the

Table 1: *Mean ± SD of Caries Removal Effectiveness (CRE) and Minimal Invasive Potential (MIP) According to Different Carious Removal Methods Using Digital Image and Micro-CT*[a]

Caries Removal Method	CRE (Mean ± SD)	MIP (Mean ± SD)
Digital image		
Carbide bur	0.00 ± 0.01 A	1.09 ± 0.11 a
Excavator	0.33 ± 0.37 B	0.84 ± 0.44 a,b
Polymer bur	0.41 ± 0.32 B	0.57 ± 0.27 b
Micro-CT		
Carbide bur	0.00 ± 0.00 α	1.12 ± 0.14 γ
Excavator	0.30 ± 0.29 β	0.69 ± 0.27 δ
Polymer bur	0.73 ± 0.16 β	0.26 ± 0.14 ε

[a] *In the same column, different letters or symbols indicate significant difference (p<0.05).*

polymer bur ($p<0.001$). The polymer bur presented the lowest MIP values which were significantly different from the excavator ($p=0.0031$).

DISCUSSION

On both the digital image and the micro-CT analysis, the excavator and the polymer bur showed similar CRE, which was lower than that of the carbide bur (Table 1; Figure 2). Thus, the first hypothesis was rejected. A CRE ratio higher than 0 means that the excavation method could not completely remove the caries. In other words, the excavation methods were not sufficiently effective. The similarities in CRE of the excavator and the polymer bur could be attributed to the carious dentin's characteristics. Carious dentin, especially caries-infected dentin, presents a low cohesive strength,[2] a low degree of mineralization, and a high collagen matrix disorganization,[9,17] which are more easily removed. In the current study, it was speculated that carious dentin left by the polymer bur could be the effect of its self-limiting capacity, as previously reported.[15] In fact, it is possible that the polymer bur is not really able to remove caries-affected dentin because of its self-limiting effect. As a consequence, the low effectiveness of this method compared with the carbide bur represents an advantage toward dentin preservation and conservative caries removal. Toledano and others[15] demonstrated that the polymer bur was the excavation method that most preserved the carious-affected dentin. However, some authors consider remaining

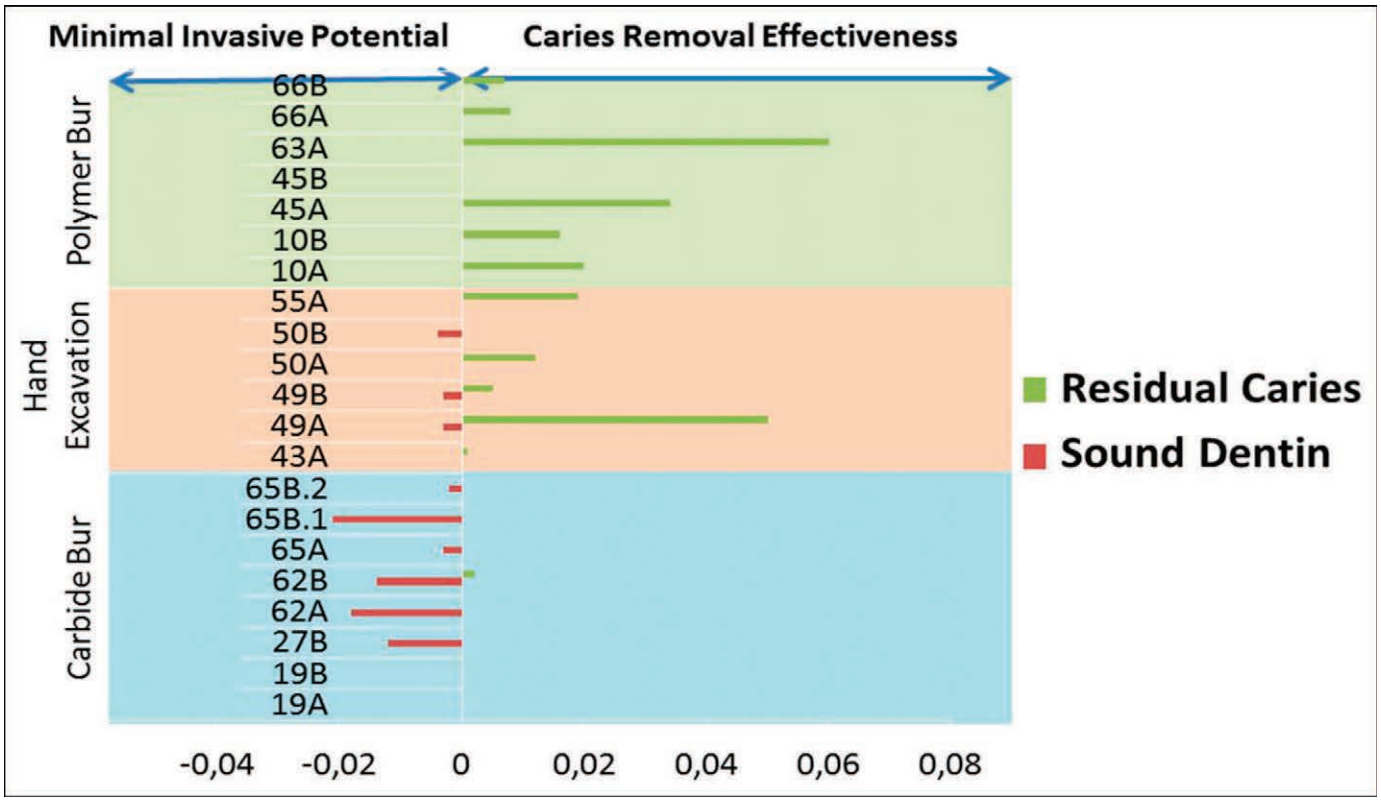

Figure 2. *Schematic representation of caries removal effectiveness (CRE) and minimal invasive potential (MIP) according to different carious removal methods using digital photography and micro-CT for each specimen.*

caries-affected dentin as unremoved and under-excavated caries.[16]

The hardness of the polymer bur (50 KHN) is higher than the hardness attributed to carious dentin (0 to 30 KHN) but lower than that of sound dentin (70 to 90 KHN). The bur could be damaged when touching sound dentin resulting in the inability to the remove all carious tissue.[9] On the other hand, some studies report that caries-affected dentin hardness is about 30.7 to 33.9 KHN.[18] Considering these hardness values, the polymer bur is not able to preserve caries-affected dentin and is not precise enough to distinguish the slight differences between the remineralizable dentin and that with minimal collagen degradation.[19]

In addition, the polymer bur presented a lower MIP than the carbide bur and this value was lower than 0 (Table 1; Figure 2). When the MIP ratio is lower than 1, it means that the prepared cavity was smaller than the initial carious lesion, and residual carious dentin could have been left behind. This could be more evidence that the polymer bur does not excavate into sound dentin tissue and was not able to totally remove the caries. In fact, these results are in agreement with other studies[10,15,16] that showed that the polymer bur excavation led to one of the largest coincidences between the caries removed and the caries lesion limits; they also exhibited the biggest underprepared area of all methods evaluated. Residual carious dentin might be left at the cavity walls due to the presence of a compact and thick smear layer, produced during the use of the polymer bur.[15] This is the so-called compacting effect on carious dentin, and it is likely that the carbide bur produces the same effect.[3,19] Such an effect could hinder and compromise the action of the polymer bur despite its self-limiting properties. The authors speculate that this similar effect could also happen during tooth storage, which is different from the clinical situation. Vital teeth contain dentin fluid that exudes from the pulp tissue to the dentin surfaces that have been prepared to keep it moist.

For digital and micro-CT analysis, the excavator presented MIP values that were somewhere between both extremes of the burs (Table 1; Figure 2). Specifically, in the digital image analysis, there was no significant difference between these values and those of the carbide and polymer burs (Table 1). According to the current findings, it seems that the excavator is not as invasive as the carbide bur, but it is more able to remove carious dentin beneath the compact smear layer than the polymer bur. Since dentin hardness may vary between teeth and even locally in the same tooth, its assessment usually varies between operators, and erroneous removal of dentin (over- or underpreparation) may occur.[11] The excavator is considered a suitable excavation method combining good excavation time and effective caries removal.[16] But, optimal effectiveness depends on clinical judgment and the operator's experience.[3]

On the other hand, the carbide bur presented the lowest CRE (0.00 for both digital and micro-CT images; Table 1; Figure 2). These low values mean that the carbide bur left almost no residual carious dentin, which is effective when removing caries. Prior studies also reported that the steel and carbide burs showed similar demineralized dentin removal effectiveness as the excavator.[16]

The caries removal endpoint has still not been well established and continues to be subjective. Determining the endpoint with a dental probe and dentin hardness is no longer the most reliable method as it does not take caries-affected dentin into account. Indeed, overexcavation can occur when probes are used in conjunction with traditional caries removal methods (ie, carbide burs).[2,3] An MIP ratio higher than 1 means that the prepared cavity was greater than the initial carious lesion (Table 1; Figure 2). In this case, sound dentin was probably removed. According to the present results, when the carbide bur was used, initial carious dentin was overexcavated, and the MIP was higher (1.09 and 1.12, respectively, for digital and micro-CT images) than when the polymer bur (0.57 and 0.26, respectively, for digital and micro-CT images) was employed (Table 1). Hence, the second tested hypothesis must be rejected. The carbide bur usually overprepares carious dentin, also removing the caries-affected dentin.[3,16,20] The carbide bur presents a negative rake angle, which means that its blade is ahead of the perpendicular line to the surface being cut. To keep the blade in contact with this surface, increased downward pressure is needed, and this can lead to a less controlled movement of the instrument on the surface.[16] The speed of this rotary instrument and its mode of function may make this technique less sensitive and more difficult to control.[16]

Micro-CT is becoming increasingly popular in dental research, as it enables collecting detailed quantitative and qualitative data of the substrate before and after a specific treatment.[12,14,21-24] Its application in studying caries excavation techniques has recently been demonstrated by comparing the internal tooth structure before and after caries removal.[6,13,22,25] However, the use of micro-CT as an accurate tool for caries detection is controversial.

Mitropoulos and others[26] reported that micro-CT diagnosis was strongly correlated with some visual criteria but was not a reliable alternative to histological examination for caries research. The lack of correlation between these two diagnostic methods was attributed to the inherent deficiency of microtomography technology, similar to radiography, to detect early signs of demineralization.[26] Current results show that micro-CT is able to detect caries lesions before and after excavation, although high precision in lesion limits could not be observed.

Improvement in digital camera technology has allowed this technique to be used in several fields of science, as obtained images contain information that can be processed and analyzed.[15] Digital imaging presents several advantages. It is a low cost, easily available, nondestructive research technique that allows hard tissues to be measured and evaluated many times with samples remaining available after scanning for additional biological and mechanical testing.[15,27] Therefore, to assess caries removal methods, the digital image analysis tool was used to diagnose carious lesions. The present results demonstrate that digital photography seems to be a viable method for analyzing mineralized and carious dental tissues, as well as color alterations before and after caries excavation.

Although this study did not aim to compare caries detection methods, it is possible to note that there was a slight tendency of similarity between the digital image and micro-CT analysis.

Development of new conservative technology to remove caries and the improvement of those already available in the market represent a great challenge for researchers and manufacturers. Polymer burs represent important tools to preserve caries-affected dentin but still lack clinical evaluation and follow-up after caries removal and the restorations are placed. Diagnostic methods must also be studied and improved. The search for methods of diagnosis that are more precise, less invasive, and cheaper has been a constant concern in dentistry. More extensive analysis of both digital and micro-CT images is necessary to consolidate them as feasible and reliable diagnostic methods. New micro-CT devices and three-dimensional reconstruction seems to be very promising.

CONCLUSIONS

Within the limitations of this study, when either digital imaging or micro-CT analysis were employed, it was concluded that the carbide bur was the most effective method to remove carious dentin. In addition, the polymer bur presented low invasive potential but was not able to remove all carious dentin.

Acknowledgements

The authors thank Professor Francisco Haiter Neto, Department of Oral Diagnosis, Piracicaba Dental School, University of Campinas, and Adriano L. Martins for their assistance with the micro-CT apparatus. We are indebted to Professor Nelson RF Silva and to SS White (Lakewood, NJ, USA) for their support and polymer bur donation.

Human Subject Statement

This study was conducted in accordance with all the provisions of the human subject oversight committee guidelines and policies at Universidade Federal do Ceará. The approval code for this study was 108/12 under protocol 54/12. This study was conducted at the Federal University of Ceará.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 14 January 2014)

REFERENCES

1. 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.
2. Neves AA, Coutinho E, Munck JD, Lambrechts P, & Meerbeek BV (2011) Does diagnodent provide a reliable caries-removal endpoint? *Journal of Dentistry* **39(5)** 351-360.
3. Banerjee A, Kidd EAM, & Watson T (2000) *In vitro* evaluation of five alternative methods of carious dentine excavation *Caries Research* **34(2)** 144-150.
4. Banerjee A, Kidd EAM, & Watson T (2003) *In vitro* validation of carious dentin removed using different excavation criteria *American Journal of Dentistry* **16(4)** 228-230.
5. Kidd EAM, Joyston-Bechal S, & Beighton D (1993) Microbiological validation of assessments of caries activity during cavity preparation *Caries Research* **27(5)** 402-408.
6. Neves AA, Coutinho E, Munck JD, & Meerbeek BV (2011) Caries-removal effectiveness and minimal-invasiveness potential of caries-excavation techniques: a micro-CT investigation *Journal of Dentistry* **39(2)** 154-162.
7. Neuhaus KW, Ciucchi P, Donnet M, & Lussi A (2010) Removal of enamel caries with an air abrasion powder *Operative Dentistry* **35(5)** 538-546.
8. Motisuki C, Lima LM, Bronzi ES, Spolidorio DM, & Santos-Pinto L (2006) The effectiveness of alumina powder on carious dentin removal *Operative Dentistry* **31(3)** 371-376.

9. Silva NRFA, Carvalho RM, Pegoraro LF, Tay FR, & Thompson VP (2006) Evaluation of a self-limiting concept in dentinal caries removal *Journal of Dental Research* **85(3)** 282-286.
10. Boston DW (2003) New device for selective dentin caries removal *Quintessence International* **34(9)** 678-685.
11. Hamba H, Nikaido T, Sadr A, Nakashima S, & Tagami J (2012) Enamel lesion parameter correlations between polychromatic micro-CT and TMR *Journal of Dental Research* **91(6**) 586-591.
12. Soviero VM, Leal SC, Silva RC, & Azevedo RB (2012) Validity of microCT for *in vitro* detection of proximal carious lesion in primary molars *Journal of Dentistry* **40(1)** 35-40.
13. Neves AA, Coutinho E, Cardoso MV, Jaecques SV, & Meerbeek BV (2010) Micro-CT based quantitative evaluation of caries excavation *Dental Materials* **26(6)** 579-588.
14. Meleo D, Manzon L, Pecci R, Zuppante F, & Bedini R (2012) A proposal of microtomography evaluation for restoration interface gaps *Annali Dell'istituto Superiore Di Sanità* **48(1)** 83-88.
15. Toledano M, Ghinea R, Cardona JC, Yamauti M, Pérez MM, & Osorio R (2013) Digital image analysis method to assess the performance of conventional and self-limiting concepts in dentin caries removal *Journal of Dentistry* **41(supplement 3)** e31-e38.
16. Celiberti P, Francescut P, & Lussi A (2006) Performance of four dentine excavation methods in deciduous teeth *Caries Research* **40(2)** 117-123.
17. Toledano M, Cabello I, Yamauti M, & Osorio R (2012) Differential resin-dentin bonds created after caries removal with polymer burs *Microscopy and Microanalysis* **18(3)** 497-508.
18. Almahdy A, Downey FC, Sauro S, Cook RJ, Sherriff M, Richards D, Watson TF, Banerjee A, & Festy F (2012) Microbiochemical analysis of carious dentine using raman and fluorescence spectroscopy *Caries Research* **46(5)** 432-440.
19. Meller C, Welk A, Zeligowski T, & Splieth C (2007) Comparison of dentin caries excavation with polymer and conventional tungsten carbide burs *Quintessence International* **38(7)** 565-569.
20. Yip HK, Stevenson AG, & Beeley JA (1994) The specificity of caries detector dyes in cavity preparation *British Dental Journal* **176(11)** 417-421.
21. Lee HS, Berg JH, Garcia-Godoy F, & Jang KT (2008) Long-term evaluation of the remineralization of interproximal carieslike lesions adjacent to glass-ionomer restorations: a micro-CT study *American Journal of Dentistry* **21(2**) 129-132.
22. Clementino-Luedemann TN, & Kunzelmann KH (2006) Mineral concentration of natural human teeth by a commercial micro-CT *Dental Materials Journal* **25(1)** 113-119.
23. Olejniczak AJ, & Grine FE (2006) Assessment of the accuracy of dental enamel thickness measurements using microfocal X-ray computed tomography *Anatomical Record Part A: Discoveries in Molecular, Cellular, and Evolutionary Biology* **288(3**) 263-275.
24. Dowker SE, Elliott JC, Davis GR, & Wassif HS (2003) Longitudinal study of the three-dimensional development of subsurface enamel lesions during *in vitro* demineralisation *Caries Research* **37(4)** 237-245.
25. Willmott NS, Wong FSL, & Davis GR (2007) An x-ray microtomography study on the mineral concentration of carious dentine removed during cavity preparation in deciduous molars *Caries Research* **41(2)** 129-134.
26. Mitropoulos P, Rahiotis C, Stamatakis H, & Kakaboura A (2010) Diagnostic performance of the visual caries classification system ICDAS II versus radiography and micro-computed tomography for proximal caries detection: an *in vitro* study *Journal of Dentistry* **38(11)** 859-867.
27. Swain MV, & Xue J (2009) State of the art of micro-ct applications in dental research *International Journal of Oral Science* **1(4)** 177-188.

©Operative Dentistry, 2015, **40-3,** 271-281

Color Stability Behavior of Methacrylate-based Resin Composites Polymerized with Light-emitting Diodes and Quartz-Tungsten-Halogen

C Sabatini

Clinical Relevance

Ensuring optimal color match of composite restorations to the surrounding tooth structure is critical to any esthetic restorative procedure. A better understanding of the color shifts composites undergo after polymerization and storage can greatly minimize shade mismatch issues.

SUMMARY

Despite significant developments in improving the optical properties of resin composite materials, their color stability remains a challenge. This study aimed to evaluate the shade stability of light-polymerized, methacrylate-based resin composites with different filler particle composition (microfill, minifill, nanohybrids, and microhybrids) polymerized with quartz-tungsten-halogen (QTH) and light-emitting diodes (LED).

Methods and Materials: Composite discs were fabricated from Tetric EvoCeram, Premise, Artiste, and Beautifil II (nanohybrids); Filtek Supreme Plus and Vit-l-escence (microhybrids); Heliomolar (microfill); and Estelite Sigma Quick (minifill) using a Teflon mold. The specimens were irradiated either with QTH (Elipar 2500; 600 mW/cm^2) for 40 seconds or with LED (Bluephase G2; 1200 mW/cm^2) for 20 seconds. Color parameters were measured with a colorimeter before and after polymerization and at 24 hours, one week, one month, and three months. Color change was calculated among the different storage periods.

Results: There was a significant effect of the composite, time, and their interaction ($p<0.001$) but no effect of the polymerization unit on the color stability. Color changes immediately after polymerization and at 24 hours (4.22 and 3.88 for LED; and 4.08 and 3.82 for QTH) were not significantly different from each other but were both significantly higher than changes after one week (0.96 and 0.78),

*Camila Sabatini, DDS, MS, University at Buffalo, Restorative Dentistry, Buffalo, NY, USA

*Corresponding author: 3435 Main Street, 215 Squire Hall, Buffalo, NY 14214, USA; e-mail: cs252@buffalo.edu

DOI: 10.2341/14-080-L

one month (1.12 and 1.02), and three months (1.27 and 1.11) for LED and QTH, respectively ($p<0.001$).

Conclusions: Color changes were observed for all the materials that were dependent on the type of composite but not on the polymerization unit. These color shifts took place primarily immediately after polymerization and after 24 hours and were additive in nature.

INTRODUCTION

Despite efforts to improve the color stability of current composite resin materials, their color remains unstable. Composite resins' optical properties are dependent on the degree of polymerization,[1,2] which is in turn a function of aspects relative to the material's composition and the amount of energy delivered on polymerization.[3-5] The type, size, amount of inorganic filler loading,[6,7] resin matrix composition,[8-12] and type and concentration of photoinitiator[13-15] as well as the wavelength of the emitted light, bulb intensity, and exposure time[16] are all known to affect the material's degree of polymerization.

Light-activated composite resins polymerize by free radical polymerization, whereby methacrylate carbon-carbon double bonds (C=C) become available for cross-linking monomers into polymeric chains.[17,18] Most light-activated systems use camphorquinone (CQ) as their photoinitiator. When exposed to blue light of wavelengths in the range of 400 to 500 nm, CQ reacts with an amine activator to form free radicals, initiating the polymerization reaction.[19] Approximately 75% of the polymerization reaction takes place during the first 10 minutes,[20,21] after which the free radicals undergo a postirradiation polymerization reaction that lasts up to 24 hours.[22] This dark polymerization can be quite extensive, with as much as 19-26% of the final monomer conversion taking place during this period.[23] However, the conversion of C=C is not complete, ranging from 55% to 75%,[21] resulting in a heterogeneous structure of densely cross-linked and poorly cross-linked areas.[24] Quartz-tungsten-halogen (QTH) and light-emitting diodes (LEDs) represent the most commonly used light-curing units (LCUs) for the polymerization of light-activated composite resins. QTH's broad emission spectrum allows polymerization of a wide range of composite materials. However, drawbacks associated with the degradation of their filters have been reported to result in inadequately polymerized restorations.[25] LEDs, with a narrower wavelength spectrum that matches more closely the absorption peak of CQ,[26] allow reduced polymerization times due to their higher irradiances.[27] In general, the same degree of conversion can be obtained with a fixed energy density, independent from variations in light irradiance and exposure time.[28] Issues derived from insufficient polymerization and residual unreacted monomers are known to compromise the polymer mechanical properties,[29,30] resulting in premature degradation, wear, and staining[9,31] as well as in a compromised color stability.[32]

The International Commission on Illumination developed the CIE L*a*b* scale, which can be used to describe the color characteristics of composite resins based on three parameters: lightness/darkness (+L*/–L*), red/green (+a*/–a*), and yellow/blue (+b*/–b*). Previous studies have demonstrated that color changes occurring in composite resins are primarily within the L* and b* parameters.[33-38] Color change is described quantitatively in delta E (ΔE^*) units, which represent the "linear distance" between two colors located in the CIE L*a*b* color space and combine changes in the L*, a*, and b* parameters. The smallest color difference that the human eye can detect has been a subject of debate. Different ΔE^* values have been proposed to determine "unacceptable" color changes with values set at $\Delta E^* \geq 2$,[39] $\Delta E^* \geq 3.3$,[10,40-42] and $\Delta E^* \geq 3.7$.[43,44]

As improved formulations of materials are developed, it is essential that studies investigate their optical properties when polymerized with different LCUs. Therefore, the aim of this study was to evaluate the polymerization-dependent color change and shade stability of eight commercially available light-polymerized, methacrylate-based composite resins with different filler particle composition (microfill, minifill, nanohybrids, and microhybrids) polymerized with QTH or LED immediately after polymerization and after different storage periods. The null hypotheses evaluated were the following: 1) There is no influence of the polymerization unit, when delivering equivalent energy densities, on the color stability of eight methacrylate-based composite resin materials. 2) There is no influence of the type of composite resin on the color stability following polymerization with an LED or QTH LCU.

METHODS AND MATERIALS

Polymerization-dependent color changes and shade stability of eight light-polymerized, methacrylate-based resin composites were evaluated in this study. The composition and energy requirements of the materials evaluated, per the manufacturers' descrip-

tions, are summarized in Table 1. Tetric EvoCeram, Premise, Artiste, and Beautifil II (nanohybrids); Filtek Supreme Plus and Vit-l-escence (microhybrids); Heliomolar (microfill); and Estelite Sigma Quick (minifill) were polymerized with either LED or QTH. Five discs, in shade A3 dentin, were fabricated for each combination of composite resin–LCU (n=5), for a total of 80 specimens, as determined by preliminary power analysis. Two LCUs were used for photoactivation of the composite resin specimens: An LED unit (Bluephase G2; Ivoclar-Vivadent, Amherst, NY, USA; 1200 mW/cm^2) and a QTH unit (Elipar 2500; 3M ESPE, St Paul, MN, USA; 600 mW/cm^2) with light probe diameters of 10 mm and 8 mm. The irradiances of the LCUs were measured using a hand-held LED radiometer (Demetron; Kerr, Orange, CA, USA). The total energy requirement for optimal polymerization of the materials, referred to as radiant exposure, was calculated as the product of the irradiance and irradiation time recommended by the manufacturer. The radiant exposure values for the different materials evaluated ranged from 4.5 to 24 J/cm^2 (Table 1). For standardization of the amount of energy delivered, all specimens received 24 J/cm^2. The irradiation time was set to 20 seconds for the LED (1200 mW/cm^2×20 seconds) and 40 seconds for the QTH (600 mW/cm^2×40 seconds). The specimens were prepared by condensing the composite resin into a white polytetrafluoroethylene mold (5 mm diameter × 2 mm height) against two microscope glass slabs, with Mylar strips between the glass slabs and the mold to avoid oxygen inhibition. Glass slabs were used to provide flat specimens with a uniform surface that would be less likely to introduce variations in the color measurements.[6]

Color measurements were recorded before polymerization, immediately after polymerization, and after 24 hours, one week, one month, and three months of storage (100% humidity at 37°C) with a colorimeter (Minolta Chroma Meter model CR-321; Minolta Corp, Ramsey, NJ, USA). Calibration of the device was performed against a white calibration tile provided by the manufacturer. A measuring area of 3 mm in diameter with 45° circumferential illumination and 0° viewing angle was used. The colorimeter device measures the color of a specimen against a black background and exposed to a standard light source (D65 or regular daylight). All color measurements were recorded through the glass slabs to eliminate the potential effect of the glass specular and diffuse reflectance. Because the diameter of the colorimeter optical geometry was smaller than the diameter of the specimens, three overlapping measurements were taken and averaged to determine a single color value. Between testing periods, the specimens were stored individually in hermetically sealed containers containing distilled water in an incubator at 37°C. Color values were expressed according to the CIE L*a*b* scale color coordinates: lightness-darkness (L*), red-green (a*), and yellow-blue (b*). The overall color change (ΔE*) was calculated using the equation $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. Mean ΔE* values were calculated between baseline and immediate polymerization, immediate polymerization and 24 hours, 24 hours and one week, one week and one month, and one month and three months. The total net color change after three months relative to baseline was also calculated. Color change values $\Delta E^* \geq 3.3$ were considered unacceptable based on thresholds designated by previous studies.[10,40-42] Further analysis of changes to the individual color parameters (ΔL*, Δa*, Δb*) was also conducted only for values $\Delta E^* \geq 3.3$.

A three-way analysis of variance (ANOVA) was used to evaluate the effect of the main variables—composite resin, time, and LCU—and their interactions on the color stability. Since no effect was detected for the variable LCU, individual two-way ANOVAs were conducted to evaluate the effect of the variables composite resin, time, and their interactions on the color stability of both LED- and QTH-polymerized samples. Post hoc Tukey tests were used for pairwise multiple comparisons of group means. For color changes above the critical threshold of 3.3, a two-way ANOVA and Tukey tests were used to evaluate the effect of the variables composite resin and LCU on each of the corresponding changes in L*, a*, and b* parameters. A significance level of $p < 0.05$ was used for all tests. All statistical analyses were performed with SigmaStat version 3.5 (San Jose, CA, USA).

RESULTS

The three-way ANOVA revealed a significant effect of the composite resin, time, and their interactions ($p<0.001$) but no effect of the LCU on the color stability (Table 2). Overall, the color change observed for specimens polymerized with LED was not significantly different from that of specimens polymerized with QTH ($p<0.162$). Individual two-way ANOVAs revealed a significant effect of the composite resin, time, and their interactions ($p<0.001$) on color stability for both LED- and QTH-polymerized samples (Table 3). Pairwise multiple comparisons revealed that color changes immediately after polymerization and at 24 hours (4.22 and 3.88 for LED, and 4.08 and 3.82 for QTH) were not significantly different from each other but were both significantly

Table 1: *Composite Resin Brands, Categories, and Composition, Per Manufacturer's Description*

Product (Manufacturer)	Lot	Category	Matrix	Photoinitiator	Energy Required,[a] (J/cm^2)
Estelite Sigma Quick (Tokuyama, Tokyo, Japan)	E674	Minifill	Bis-GMA, TEGDMA	CQ/RAP	4.5-6
Heliomolar (Ivoclar-Vivadent, Amherst, NY, USA)	M00783	Microfill	Bis-GMA, UDMA, decandiol dimethacrylate	CQ/amine	20
Tetric EvoCeram (Ivoclar-Vivadent, Amherst, NY, USA)	N58533	Nanohybrid	Dimethacrylates	CQ/amine	10
Premise (Kerr, Orange, CA, USA)	3204934	Nanohybrid	Bis-EMA, TEGDMA	CQ/amine	10
Artiste (Pentron, Wallingford, CT, USA)	167373	Nanohybrid	PCBisGMA/BisGMA/UDMA/ HDDMA	Not reported	8-12
Beautifil II (Shofu, Kyoto, Japan)	051026-51	Giomer nanohybrid	Bis-GMA, TEGDMA	CQ/amine	10
Filtek Supreme Plus (3M-ESPE, St Paul, MN, USA)	8EA	Microhybrid	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA	CQ/amine	24
Vit-l-escence (Ultradent, South Jordan, UT, USA)	B4869	Microhybrid	Bis-GMA	CQ/amine	9.2

Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, bisphenol A glycidyl dimethacrylate; CQ, camphorquinone; HDDMA, hexanediol dimethacrylate; PCBis-GMA, polycarbonate modified Bis-GMA; PEGDMA, poly (ethylene glycol) dimethacrylate; PPF, pre-polymerized filler; RAP, radical amplified photopolymerization; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] The energy requirement was calculated based on the information provided from the manufacturer regarding time and light curing unit recommended for polymerization.

higher than the changes observed after one week (0.96 and 0.78), one month (1.12 and 1.02), and three months (1.27 and 1.11) for LED and QTH ($p<0.001$), respectively. With only a few exceptions, color changes immediately after polymerization and after 24 hours surpassed the critical threshold of 3.3, and the changes after one week, one month, and three months remained below this threshold. Figure 1A and B summarizes the color changes for each material at the different testing periods for LED and QTH. The total net color changes after three months, relative to baseline, are also represented in Figure 1A and B for all the materials, which were ranked from greatest to least amount of color change as follows: Artiste (11.33) > Vit-l-escence (11.32) > Premise (10.81) > Tetric Evo Ceram (7.98) > Supreme (7.54) > Beautifil (7.51) > Estelite (4.96) > Heliomolar (4.51) for LED-polymerized specimens; and Vit-l-escence (10.56) > Premise (9.44) > Tetric Evo Ceram (7.76) > Artiste (7.36) > Beautifil (7.08) > Heliomolar (6.47) > Supreme (5.33) > Estelite (4.44) for QTH-polymerized specimens.

Subsequent analysis of the contribution of the individual parameters L*, a*, and b* to the overall color change was performed immediately after polymerization and after 24 hours only since these values exceeded the critical threshold of 3.3. Two-way ANOVA immediately after polymerization (Table 4) revealed that there was a significant effect of both composite resin and LCU on parameters ΔL^* ($p<0.001$ and $p=0.018$) and Δa^* ($p<0.001$ and

Table 2: *Three-way Analysis of Variance (ANOVA) for Color Change in Delta E (ΔE^*) Units*

Source of Variation	df	SS	MS	*F*	*P*
Composite	7	48.250	6.893	8.179	<0.001
Light	1	1.651	1.651	1.960	0.163
Time	4	841.988	210.497	249.769	<0.001
Composite × light	7	6.582	0.940	1.116	0.353
Composite × time	28	136.960	4.891	5.804	<0.001
Light × time	4	0.178	0.0445	0.0528	0.995
Composite × light × time	28	23.379	0.835	0.991	0.482
Residual	317	267.157	0.843		
Total	396	1326.172	3.349		

Abbreviations: MS, mean squares; SS, sum of squares.

Table 1: Extended.

Product (Manufacturer)	Particle Size, μm (Mean)	Filler Type	Filler content	
			%wt	%vol
Estelite Sigma Quick (Tokuyama, Tokyo, Japan)	0.1-0.3 (0.2)	Zirconia-silica, composite filler	82	71
Heliomolar (Ivoclar-Vivadent, Amherst, NY, USA)	0.04-0.2	Silicon dioxide, ytterbium trifluoride, pre-polymers	66.7	46
Tetric EvoCeram (Ivoclar-Vivadent, Amherst, NY, USA)	0.04-3.0 (0.55)	Barium glass, ytterbium trifluoride, mixed oxide, pre-polymers	75-76	53-55
Premise (Kerr, Orange, CA, USA)	PPF, 30-50; silica, 0.02; barium, 0.4	Pre-polymerized filler, barium glass, silica filler	84	70
Artiste (Pentron, Wallingford, CT, USA)	0.02-0.7	Barium boro-alumino silicate glass, nano-particulated silica, zirconium silicate	75	66
Beautifil II (Shofu, Kyoto, Japan)	0.01-4.0 (0.8)	Glass filler, S-PRG filler (fluoroboroaluminosilicate glass)	83.3	68.6
Filtek Supreme Plus (3M-ESPE, St Paul, MN, USA)	Clusters, 0.6-1.4; silica, 0.02	Silica filler, zirconia filler, aggregated zirconia/silica	78.5	59.5
Vit-l-escence (Ultradent, South Jordan, UT, USA)	0.7	Barium alumina silicate	75	58

$p<0.001$). Only the composite resin variable demonstrated a significant effect on parameter Δb* ($p<0.001$). Two-way ANOVA after 24 hours (Table 5) demonstrated that only the composite resin variable had a significant effect on parameters ΔL* ($p=0.013$) and Δa* ($p<0.001$). Neither composite resin nor LCU had an effect on parameter Δb*. The contribution of the individual parameters L*, a*, and b* to the overall color change for specimens polymerized with LED and QTH is summarized in Figure 2A and B for values obtained immediately after polymerization and in Figure 3A and B for values obtained 24 hours following polymerization.

DISCUSSION

The present study evaluated the shade stability of a number of light-polymerized methacrylate-based composite resins with different filler particle composition polymerized with QTH and LEDs. The first null hypothesis was accepted since there was no influence of the LCU on the color stability of the different materials evaluated. Although monomer conversion ratios were not determined in this study, it is possible that an equivalent degree of conversion was obtained with both LCUs, explaining, at least in part, the observed results. This is coincident with previous findings,[28] which have shown an equivalent degree of conversion with a fixed energy density, independent from variations of light irradiance and exposure time.

A wide range of color change was observed for the different materials evaluated, thus leading to rejection of the second null hypothesis. Aspects relative to the material composition, such as the type, size, amount of inorganic filler loading, resin matrix composition, and type and concentration of photoinitiator, have all been reported[14] to affect the material's degree of polymerization and may help explain the differences in color stability of the materials evaluated. Light-activated composite resins commonly use CQ/amine as their photoinitiator system. Although present in small amounts, CQ can significantly affect the material's color.[13,15] Enhanced color stability has been reported with

Table 3: *Two-way Analysis of Variance (ANOVA) for Color Change in Delta E (ΔE*) Units*

Source of Variation	LED					QTH				
	df	SS	MS	*F*	*P*	df	SS	MS	*F*	*P*
Composite	7	23.597	3.371	5.348	<0.001	7	31.111	4.444	4.228	<0.001
Time	4	413.237	103.309	163.912	<0.001	4	429.038	107.260	102.028	<0.001
Composite × time	28	73.086	2.610	4.141	<0.001	28	87.333	3.119	2.967	<0.001
Residual	157	98.953	0.630			160	168.205	1.051		
Total	196	608.240	3.103			199	715.687	3.596		

Abbreviations: MS, mean squares; SS, sum of squares.

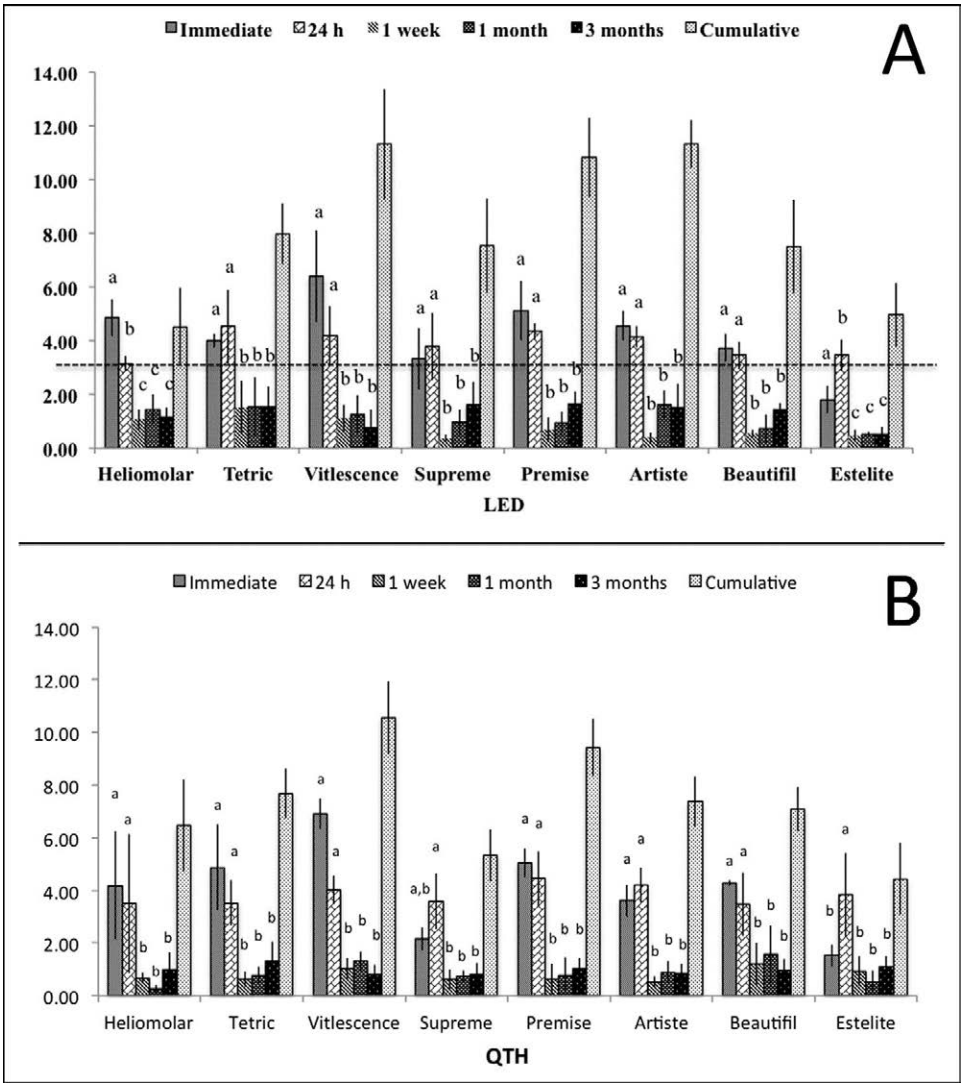

Figure 1. *(A) Mean (standard deviation [SD]) color change (ΔE*) values immediately after polymerization, at 24 hours, and after one week, one month, and three months and total net color change after three months relative to baseline for LED-polymerized specimens. Different superscript letters denote significant differences among the different 'time' intervals within each composite resin group (Tukey test,* $p<0.05$*). (B) Mean (SD) ΔE* values immediately after polymerization, at 24 hours, and after one week, one month, and three months and total net color change after three months relative to baseline for QTH-polymerized specimens. Different superscript letters denote significant differences among the different 'time' intervals within each composite resin group (Tukey test,* $p<0.05$*).*

Table 4: *Two-way Analysis of Variance (ANOVA) for ΔL*, Δa*, and Δb* Immediately After Polymerization*

Source of Variation	df	ΔL*				Δa*				Δb*			
		SS	MS	F	P	SS	MS	F	P	SS	MS	F	P
Composite	7	384.800	54.971	63.066	<0.001	16.111	2.302	16.128	<0.001	86.901	12.414	10.683	<0.001
Light	1	5.126	5.126	5.881	0.018	2.702	2.702	18.933	<0.001	0.615	0.615	0.530	0.469
Composite × light	7	12.170	1.739	1.995	0.069	1.852	0.265	1.854	0.092	15.355	2.194	1.888	0.086
Residual	65	56.657	0.872			9.276	0.143			75.536	1.162		
Total	80	465.015	5.813			29.767	0.372			178.446	2.231		

Abbreviations: MS, mean squares; SS, sum of squares.

Table 5: *Two-way Analysis of Variance (ANOVA) for ΔL*, Δa*, and Δb* After 24 Hours*

Source of Variation	df	ΔL*				Δa*				Δb*			
		SS	MS	*F*	*P*	SS	MS	*F*	*P*	SS	MS	*F*	*P*
Composite	7	17.964	2.566	2.803	0.013	2.146	0.307	10.401	<0.001	15.691	2.242	1.532	0.173
Light	1	2.032	2.032	2.220	0.141	0.0162	0.0162	0.551	0.461	2.574	2.574	1.759	0.189
Composite × light	7	4.449	0.636	0.694	0.677	0.516	0.0737	2.500	0.025	9.613	1.373	0.938	0.484
Residual	64	58.590	0.915			1.886	0.0295			93.662	1.463		
Total	79	83.034	1.051			4.564	0.0578			121.540	1.538		

Abbreviations: MS, mean squares; SS, sum of squares.

increased filler loading[6,7] and less resin volume fraction.[10,11] The nature of the matrix is also known to affect the color stability of composite resins with more hydrophilic monomers, resulting in greater water absorption and thus greater color change,[8] and with more hydrophobic monomers resulting in less water sorption and enhanced color stability.[8-10] The amount of triethylene glycol dimethacrylate (TEGDMA) present in the resin matrix has also been reported to affect the extent of postirradiation polymerization. [12] As TEGDMA increases, the amount of postirradiation polymerization decreases

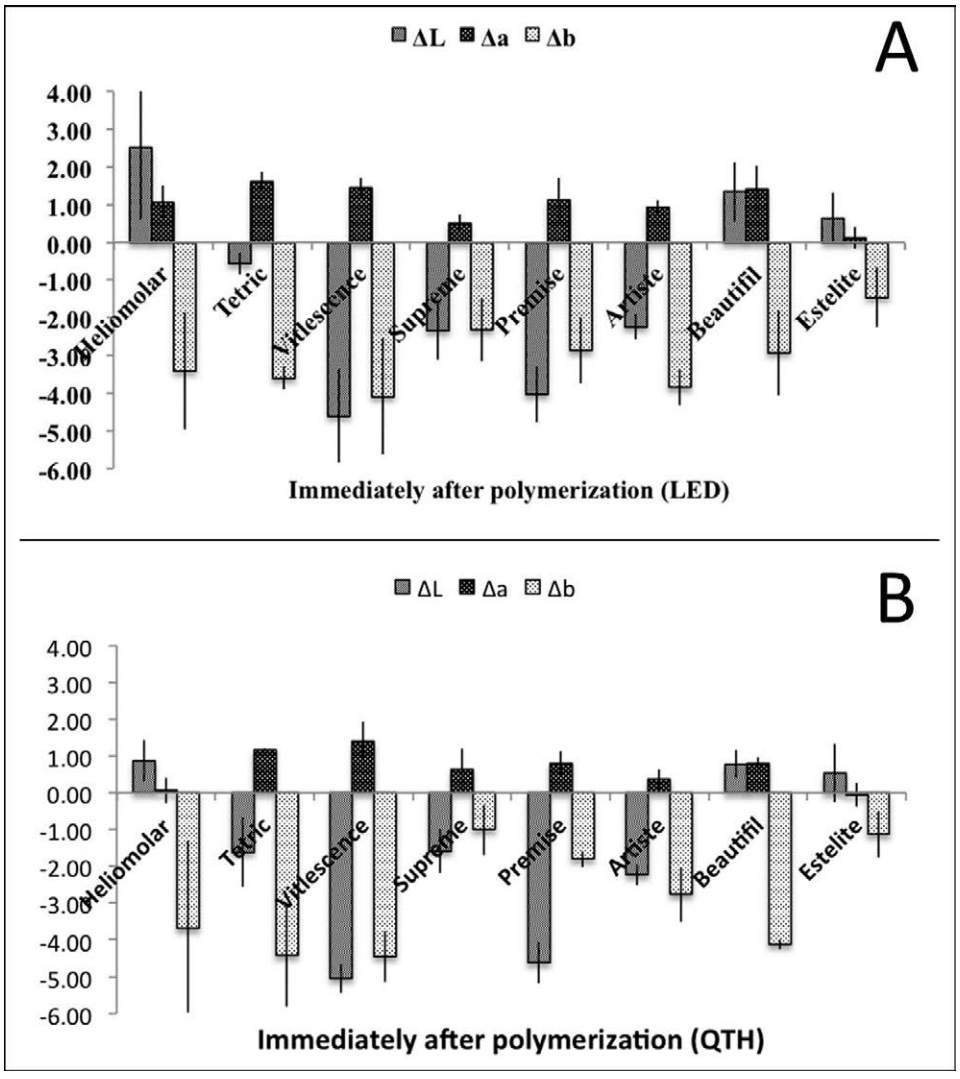

Figure 2. *(A) Mean change on ΔL*, Δa*, and Δb* parameters immediately after polymerization for LED-polymerized specimens. (B) Mean change on ΔL*, Δa*, and Δb* parameters immediately after polymerization for QTH-polymerized specimens.*

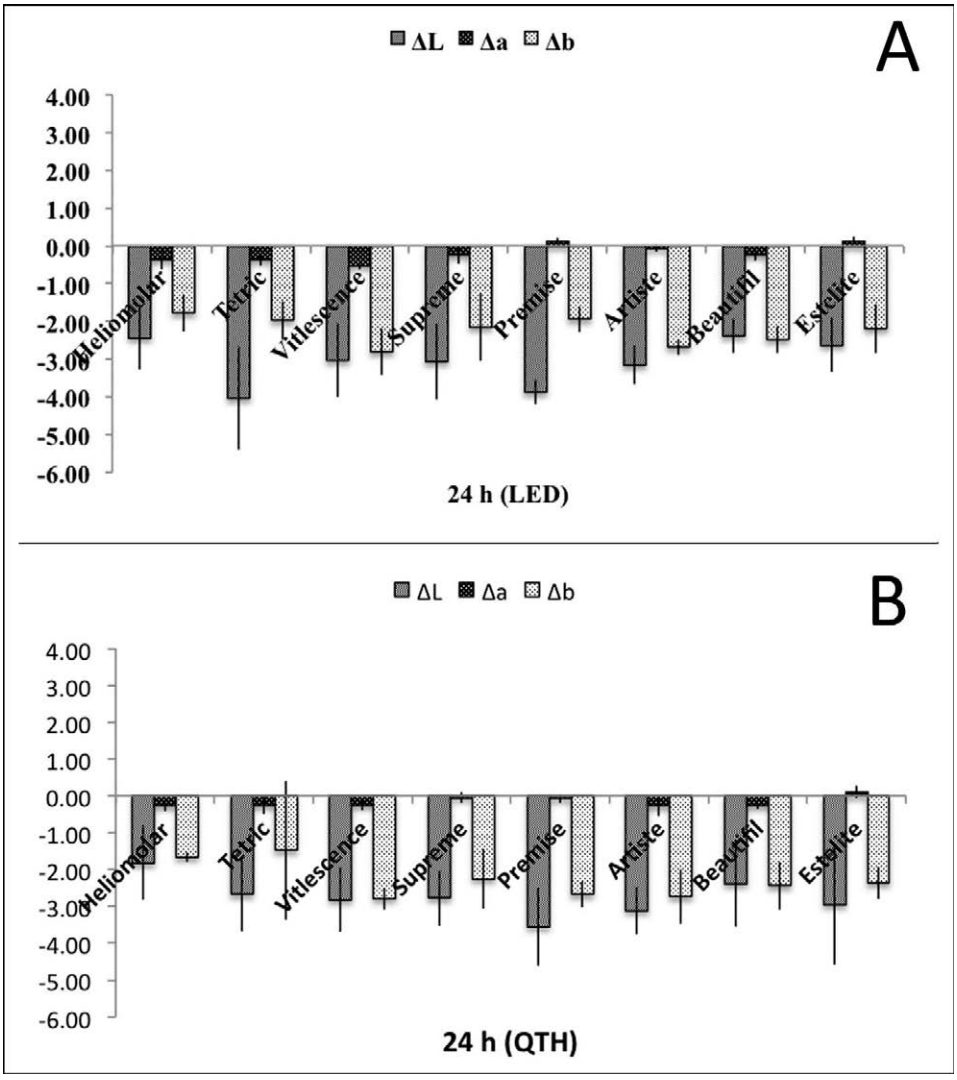

Figure 3. *(A) Mean change on ΔL*, Δa*, and Δb* parameters 24 hours following polymerization for LED-polymerized specimens. (B) Mean change on ΔL*, Δa*, and Δb* parameters 24 hours following polymerization for QTH-polymerized specimens.*

because TEGDMA generates higher initial conversion. However, correlations between the amount of color change and TEGDMA content cannot be established, since detailed compositional information of these materials is proprietary.

The color change of materials with different compositions was followed between storage periods to evaluate behavioral patterns inherent in each material. A common trend was observed, whereby all materials exhibited large color shifts immediately after polymerization and 24 hours following polymerization, but changes at subsequent intervals were negligible, indicating that they only played a minor role in the total net color change result after three months. For most materials, the degree of polymerization on initial light irradiation and 24 hours following irradiation with light was enough to generate clinically relevant color shifts above the threshold of 3.3 (Figure 1A,B). Polymerization-dependent color changes can be attributed to shifts in the resin optical properties, which take place during cross-linking of the monomers into polymeric chains.[17] The degree of polymerization is in turn dependent on factors such as the radiant intensity of the polymerization unit and polymerization time.[3,4] To standardize the amount of energy delivered to the different materials, all specimens received 24 J/cm^2. Although monomer conversion ratios were not calculated as a part of this study, it is possible that the degree of polymerization may have been maximized for materials such as Estelite Sigma, which received 24 J/cm^2 despite the recommended manufacturer energy requirement of 4.5-6 J/cm^2, perhaps leading to an enhanced color stability relative to

other brands. Clinically, a color mock-up is indicated for shade selection to account for color changes that may take place during polymerization. Changes taking place 24 hours following polymerization are even more critical to understand, as accurate predictions of these changes can significantly help minimize the shade discrepancies that may occur during this postirradiation polymerization reaction. Under the testing conditions evaluated in this study, color changes after one week, one month, and three months could be attributed to the aging conditions, such as water absorption and polymer swelling, which are in turn a function of the initial extent of polymerization of the material.[2] The negligible color changes observed at these testing intervals are suggestive of an improved chemistry of contemporary composite resin materials and indicate that, provided that an accurate color match has been obtained after 24 hours of placement, shade discrepancies after some time of intraoral function may be attributed to extrinsic factors.

The total net color change after three months, relative to baseline, was also unacceptable for all the composite materials evaluated, as it surpassed the threshold of 3.3 (Figure 1A,B). Materials with the largest net color change, Artiste (11.33), Vit-l-escence (11.32), and Premise (10.81) polymerized with LED; and Vit-l-escence (10.56) and Premise (9.44) polymerized with QTH, can be explained by the additive nature of the changes that took place immediately after polymerization and after 24 hours (Figures 2A,B and 3A,B). Vit-l-escence and Premise are heavily filled materials and use CQ/amine as their photoinitiator system. Estelite Sigma Quick, which showed the least color change of all materials, is also heavily filled, but it uses a radical amplified photo-polymerization system as the catalyst to initiate the setting reaction, allowing less CQ to be used, explaining, at least partially, its improved color stability relative to other materials.

More in-depth information regarding the behavior of the different materials was derived from an analysis of the contribution of the individual parameters L*, a*, and b* to the overall color change. This evaluation was conducted immediately after polymerization and after 24 hours, since these were the only values that exceeded the critical threshold of 3.3, and hence were considered the main changes responsible for total net color change after three months, relative to baseline. Parameters L* and b* were responsible for most of the observed changes, whereas changes to the a* parameter were negligible. Coincident with our results, previous studies have reported the a* parameter to contribute the least to the overall color change.[33-35] Analysis of the individual L*, a*, and b* parameters immediately after polymerization (Figure 2A,B) revealed similar behavior for most materials. The color change was derived predominantly from shifts to the dark (−L) and blue (−b) region. The only exceptions were Heliomolar, Beautifil, and Estelite, which shifted to the light (+L) rather than the dark (−L) region. Changes to the a* parameter were considerably smaller and almost always directed to the red (+a) region. After 24 hours (Figure 3A,B), all materials displayed the same behavior. Color shifts were primarily derived from shifts to the dark (−L) and blue (−b) region, the same direction as the changes immediately after polymerization, indicating that these changes were additive in nature, rather than neutralizing each other, as demonstrated in a previous study.[35] Changes to the a* parameter were negligible and, in most cases, neutralized the changes immediately after polymerization, since they were directed to the green (−a) region. To an extent, immediate and 24-hour changes to the L* parameter for Heliomolar, Beautifil, and Estelite neutralized each other, since these shifts took place in opposite directions but were not exactly of the same extent. Based on the additive nature of the color shifts observed immediately after polymerization and after 24 hours, and the relatively minor changes under the threshold of 3.3 observed at subsequent time intervals, it is safe to conclude that the total net color change result after three months was primarily derived from changes to the dark and blue region, which took place primarily immediately after polymerization and 24 hours following polymerization. This is in agreement with results from previous studies, which have shown a decrease in the L*[6,36,37] and b* [6,13,37,38] coordinates after polymerization, regardless of the brand and shade. Materials that use CQ as photoinitiator are known to become less yellow as the photoinitiator is consumed.[13] However, no estimations can be made based solely on the compositional information, since manufacturers do not typically disclose the amount of CQ.

Newer formulations of composite resin materials offer great potential provided their color stability behavior is understood. The present study aimed to investigate intrinsic material- and polymerization-dependent factors, which may affect the color stability of materials with different composition, by providing an in-depth analysis of the extent and nature of the color shifts taking place during initial setting and storage. By gaining a better understanding of the changes taking place at the different

stages in the maturation process, behavioral trends inherent to specific materials can be established, allowing clinicians to make more accurate predictions regarding the direction and magnitude of the changes expected to take place. A better understanding of the material of their choice is critical to assisting clinicians to minimize shade mismatching issues, thereby improving the long-term esthetic results of their restorations. Once reproducible behavioral trends for specific materials have been established, further research should investigate the behavior of the different materials when they are exposed to various staining solutions over longer incubation periods. Moreover, future color stability studies should be conducted in a clinical setting using intraoral color measurement devices and following the thresholds of color perceptibility (ΔE^* of 2.6) and acceptability (ΔE^* of 5.5) established by Douglas and others.[45] Similar to other *in vitro* studies, our study reported color stability of monochromatic composite samples taken benchtop under perfect lighting conditions. A threshold of 3.3 was used to indicate unacceptable color changes, as determined by previous studies.[41] This threshold is applicable to laboratory conditions such as those described above and cannot be extrapolated as a threshold for "clinical unacceptability."

CONCLUSIONS

Within the limitations of this *in vitro* study, it can be concluded that when delivering equivalent energy densities, polymerization with LED or QTH did not have a significant influence on the color change. A significant effect of the type of composite resin on the color change was shown regardless of the LCU. The overall color shift after three months was primarily derived from changes to the dark and blue regions, which took place immediately after polymerization and after 24 hours. These changes were additive in nature. Color changes after one week, one month, and three months were negligible, and thus their contribution to the total net color change after three months was considered minor.

Acknowledgements

The author wishes to thank the University at Buffalo Biomaterials Research Laboratory for facilitating the equipment and Miss Kristin Lawson for her assistance with the preparation and testing of the specimens.

Note

This work was presented at the American Association for Dental Research Annual Meeting, Tampa, FL, USA, March 2012.

Conflict of Interest

The Authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

(Accepted 11 June 2014)

REFERENCES

1. Tseng WY, Huang CH, Chen RS, Lee MS, Chen YJ, Rueggeberg FA, & Chen MH (2007) Monomer conversion and cytotoxicity of dental composites irradiated with different modes of photoactivated curing *Journal of Biomedical Materials Research Part B Applied Biomaterials* **83(1)** 85-90.
2. Ferracane JL (2006) Hygroscopic and hydrolytic effects in dental polymer networks *Dental Materials* **22(3)** 211-222.
3. Arikawa H, Kanie T, Fujii K, Takahashi H, & Ban S (2007) Effect of filler properties in composite resins on light transmittance characteristics and color *Dental Materials Journal* **26(1)** 38-44.
4. Emami N, Sjodahl M, & Soderholm KJM (2005) How filler properties, filter fraction, sample thickness and light source affect light attenuation in particulate filled resin composites *Dental Materials* **21(8)** 721-730.
5. Falkensammer F, Arnetzl GV, Wildburger A, & Freudenthaler J (2013) Color stability of different composite resin materials *Journal of Prosthetic Dentistry* **109(6)** 378-383.
6. Eldiwany M, Friedl KH, & Powers JM (1995) Color stability of light-cured and post-cured composites *American Journal of Dentistry* **8(4)** 179-181.
7. Schulze KA, Marshall SJ, Gansky SA, & Marshall GW (2003) Color stability and hardness in dental composites after accelerated aging *Dental Materials* **19(7)** 612-619.
8. Buchalla W, Attin T, Hilgers RD, & Hellwig E (2002) The effect of water storage and light exposure on the color and translucency of a hybrid and a microfilled composite *Journal of Prosthetic Dentistry* **87(3)** 264-270.
9. Dietschi D, Campanile G, Holz J, & Meyer JM (1994) Comparison of the color stability of ten new-generation composites—An in-vitro study *Dental Materials* **10(5-6)** 353-362.
10. Inokoshi S, Burrow MF, Kataumi M, Yamada T, & Takatsu T (1996) Opacity and color changes of tooth-colored restorative materials *Operative Dentistry* **21(2)** 73-80.
11. Reis AF, Giannini M, Lovadino JR, & Ambrosano GM (2003) Effects of various finishing systems on the surface roughness and staining susceptibility of packable composite resins *Dental Materials* **19(1)** 12-18.
12. Tarumi H, Imazato S, Ehara A, Kato S, Ebi N, & Ebisu S (1999) Post-irradiation polymerization of composites containing bis-GMA and TEGDMA *Dental Materials* **15(4)** 238-242.
13. Janda R, Roulet JF, Kaminsky M, Steffin G, & Latta M (2004) Color stability of resin matrix restorative materials as a function of the method of light activation *European Journal of Oral Sciences* **112(3)** 280-285.

14. Leonard DL, Charlton DG, Roberts HW, & Cohen ME (2002) Polymerization efficiency of LED curing lights *Journal of Esthetic and Restorative Dentistry* **14(5)** 286-295.
15. Shin DH, & Rawls HR (2009) Degree of conversion and color stability of the light curing resin with new photo-initiator systems *Dental Materials* **25(8)** 1030-1038.
16. Aravamudhan K, Rakowski D, & Fan PL (2006) Variation of depth of cure and intensity with distance using LED curing lights *Dental Materials* **22(11)** 988-994.
17. Lee YK, & Powers JM (2001) Color and optical properties of resin-based composites for bleached teeth after polymerization and accelerated aging *American Journal of Dentistry* **14(6)** 349-354.
18. Peutzfeldt A (1997) Resin composites in dentistry: The monomer systems *European Journal of Oral Sciences* **105(2)** 97-116.
19. Rueggeberg F (1999) Contemporary issues in photocuring *Compendum of Continuing Education in Dentistry Supplement* **(25)** S4-S15.
20. Chung KH, & Greener EH (1990) Correlation between degree of conversion, filler concentration and mechanical properties of posterior composite resins *Journal of Oral Rehabilitation* **17(5)** 487-494.
21. Ferracane JL, & Greener EH (1986) The effect of resin formulation on the degree of conversion and mechanical properties of dental restorative resins *Journal of Biomedical Materials Research* **20(1)** 121-131.
22. Eliades GC, Vougiouklakis GJ, & Caputo AA (1987) Degree of double bond conversion in light-cured composites *Dental Materials* **3(1)** 19-25.
23. Halvorson RH, Erickson RL, & Davidson CL (2002) Energy dependent polymerization of resin-based composite *Dental Materials* **18(6)** 463-469.
24. Sideridou I, Tserki V, & Papanastasiou G (2002) Effect of chemical structure on degree of conversion in light-cured dimethacrylate-based dental resins *Biomaterials* **23(8)** 1819-1829.
25. Mills RW, Uhl A, Blackwell GB, & Jandt KD (2002) High power light emitting diode (LED) arrays versus halogen light polymerization of oral biomaterials: Barcol hardness, compressive strength and radiometric properties *Biomaterials* **23(14)** 2955-2963.
26. Uhl A, Sigusch BW, & Jandt KD (2004) Second generation LEDs for the polymerization of oral biomaterials *Dental Materials* **20(1)** 80-87.
27. Fleming GJ, Awan M, Cooper PR, & Sloan AJ (2008) The potential of a resin-composite to be cured to a 4mm depth *Dental Materials* **24(4)** 522-529.
28. Emami N, & Soderholm KJ (2003) How light irradiance and curing time affect monomer conversion in light-cured resin composites *European Journal of Oral Sciences* **111(6)** 536-542.
29. 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.
30. 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.
31. Doray PG, Wang X, Powers JM, & Burgess JO (1997) Accelerated aging affects color stability of provisional restorative materials *Journal of Prosthodontics* **6(3)** 183-188.
32. Micali B, & Basting RT (2004) Effectiveness of composite resin polymerization using light-emitting diodes (LEDs) or halogen-based light-curing units *Brazilian Journal of Oral Research* **18(3)** 266-270.
33. Janda R, Roulet JF, Latta M, Steffin G, & Ruttermann S (2005) Color stability of resin-based filling materials after aging when cured with plasma or halogen light *European Journal of Oral Sciences* **113(3)** 251-257.
34. Kim IJ, & Lee YK (2007) Changes in color and color parameters of dental resin composites after polymerization *Journal of Biomedical Materials Research Part B Applied Biomaterials* **80B(2)** 541-546.
35. Sabatini C, Campillo M, & Aref J (2012) Color stability of ten resin-based restorative materials *Journal of Esthetic and Restorative Dentistry* **24(3)** 185-199.
36. Paravina RD, Ontiveros JC, & Powers JM (2002) Curing-dependent changes in color and translucency parameter of composite bleach shades *Journal of Esthetic and Restorative Dentistry* **14(3)** 158-166.
37. Seghi RR, Gritz MD, & Kim J (1990) Colorimetric changes in composites resulting from visible-light-initiated polymerization *Dental Materials* **6(2)** 133-137.
38. Sidhu SK, Ikeda T, Omata Y, Fujita M, & Sano H (2006) Change of color and translucency by light curing in resin composites *Operative Dentistry* **31(5)** 598-603.
39. O'Brien WJ, Groh CL, & Boenke KM (1990) A new, small-color-difference equation for dental shades *Journal of Dental Research* **69(11)** 1762-1764.
40. Kim HS, & Um CM (1996) Color differences between resin composites and shade guides *Quintessence International* **27(8)** 559-567.
41. Ruyter IE, Nilner K, & Moller B (1987) Color stability of dental composite resin materials for crown and bridge veneers *Dental Materials* **3(5)** 246-251.
42. Vichi A, Ferrari M, & Davidson CL (2004) Color and opacity variations in three different resin-based composite products after water aging *Dental Materials* **20(6)** 530-534.
43. Kim BJ, & Lee YK (2009) Influence of the shade designation on the color difference between the same shade-designated resin composites by the brand *Dental Materials* **25(9)** 1148-1154.
44. Paravina RD, Kimura M, & Powers JM (2005) Evaluation of polymerization-dependent changes in color and translucency of resin composites using two formulae *Odontology* **93(1)** 46-51.
45. Douglas RD, Steinhauer TJ, & Wee AG (2007) Intraoral determination of the tolerance of dentists for perceptibility and acceptability of shade mismatch *Journal of Prosthetic Dentistry* **97(4)** 200-208.

©Operative Dentistry, 2015, **40-3,** 282-292

In Vitro Longevity of Bonding Properties of Universal Adhesives to Dentin

MA Muñoz • I Luque-Martinez • P Malaquias
V Hass • A Reis • NH Campanha
AD Loguercio

Clinical Relevance

Clinicians should opt to use methacryloyloxydecyl dihydrogen phosphate–containing universal adhesives to improve the bonding longevity of dentin interfaces.

SUMMARY

Purpose: To evaluate the immediate and 6-month resin-dentin bond strength (μTBS) and nanoleakage (NL) of universal adhesives that contain or do not contain methacryloyloxydecyl dihydrogen phosphate (MDP) and are used in the etch-and-rinse and self-etch strategies.

Methods and Materials: Forty caries-free extracted third molars were divided into eight groups for μTBS (n=5). The groups were bonded with the Clearfil SE Bond (CSE) and Adper Single Bond 2 (SB) as controls; Peak Universal, self-etch (PkSe) and etch-and rinse (PkEr); Scotchbond Universal Adhesive, self-etch (ScSe) and etch-and-rinse (ScEr); and All Bond Universal, self-etch (AlSe) and etch-and-rinse (AlEr). After composite restorations, specimens were longitudinally sectioned to obtain resin-dentin bonded sticks (0.8 mm^2). The μTBS of the specimens was tested immediately (IM) or after 6 months of water storage (6M) at 0.5 mm/min. Some sticks at each storage period were immersed in silver nitrate and photo developed, and the NL was evaluated with scanning electron microscopy. Data were analyzed with two-way repeated-

Miguel Angel Muñoz, DDS, MS, PhD, Universidad de Valparaiso, Operatoria Dental, Valparaiso, Chile

Issis Luque-Martinez, DDS, MS, PhD candidate, Ponta Grossa State University, Restorative Dentistry, Ponta Grossa, Brazil; Universidad de Valparaiso, Operatoria Dental, Valparaiso, Chile

Pamela Malaquias, DDS, MS candidate, Ponta Grossa State University, Restorative Dentistry, Ponta Grossa, Brazil

Viviane Hass, DDS, MS, PhD candidate, Ponta Grossa State University, Restorative Dentistry, Ponta Grossa, Brazil

Alessandra Reis, DDS, PhD, Universidade Estadual de Ponta Grossa, Restorative Dentistry, Ponta Grossa, Brazil

Nara Hellen Campanha, Ponta Grossa State University, Restorative Dentistry, Ponta Grossa, Brazil

*Alessandro D. Loguercio, DDS, MS, PhD, Ponta Grossa State University, Restorative Dentistry, Ponta Grossa, Brazil

*Corresponding author: Rua Carlos Cavalcanti, 4748, Bloco M, Sala 64A-Uvaranas, Ponta Grossa, PR 84030-900, Brazil; e-mail: aloguercio@hotmail.com

DOI: 10.2341/14-055-L

measures analysis of variance and Tukey test (α=0.05).

Results: At the IM period, PkSe and PkEr showed μTBS similar to the control adhesives ($p>0.05$) but increased NL pattern and lower μTBS after 6M ($p<0.05$). ScSe and ScEr showed intermediary μTBS values at the IM period but remained stable after 6 months ($p>0.05$). AlSe showed the lowest μTBS ($p<0.05$), but μTBS and NL remained stable after 6M ($p>0.05$). AlEr showed higher IM μTBS but showed higher degradation after 6M ($p<0.05$).

Conclusions: Universal adhesives that contain MDP showed higher and more stable μTBS with reduced NL at the interfaces after 6 months of water storage.

INTRODUCTION

Current adhesive materials simplify bonding procedures by reducing the number of application steps and time required for application. This simpler protocol makes them less technique sensitive and allows for better application standardization.[1] All of these factors are responsible for the large increase in the use of self-etch adhesives among clinicians.[2]

Self-etch materials (Se; also known as nonrinsing adhesives or etch-and-dry adhesives) do not require a separate acid step, as demineralization and priming occur simultaneously.[3] The preliminary use of phosphoric acid increases the probability of clinical errors due to the need of rinsing and adequate management of dentin moisture.[4] Contrary to the etch-and-rinse approach (Er), Se adhesives do not remove but incorporate the smear layer in the hybridized complex. Although a complete and thorough resin infiltration is not observed for some acidic Se systems,[5,6] some studies report a lower incidence of postoperative sensitivity after placement of direct composite posterior restorations.[7]

On the other hand, some drawbacks may be listed for these Se materials. They do not produce an enamel conditioning pattern that is as retentive as that produced by phosphoric acid,[8,9] which is likely responsible for the higher rates of marginal discoloration in the enamel margins of cervical restorations.[10] Selective enamel etching on the enamel margins with phosphoric acid is the most recently accepted technique to solve this problem, showing good results in both *in vitro*[11,12] and *in vivo* studies.[13]

Keeping this concept in mind, a novel family of bonding systems, known as "universal" or "multi-mode" adhesives,[14,15] was recently launched in the market. They are one-step Se adhesives that can be associated with phosphoric acid etching, mainly for enamel etching,[16] which gives the dentist a more versatile adhesive system.[17]

Universal adhesive differs from the current Se systems by the incorporation of monomers that are capable of producing chemical adhesion to the dental substrates.[14,15] It is believed that this incorporation may increase the durability of the bonds produced with simplified Se adhesives, which was shown to be limited for the current Se under *in vitro* and *in vivo* studies.[10,18] To the extent of our knowledge, the literature is still scarce with regard to the longevity of bonds produced by universal adhesives.[19]

Thus, the aim of this study was to evaluate the immediate and six-month resin-dentin bond strength (μTBS) and nanoleakage (NL) of universal adhesive systems used in the Er and Se approaches. The following null hypotheses were tested: 1) the immediate and six-month resin-dentin μTBS of universal adhesives is not influenced by the adhesive strategy selected (Er or Se) and 2) the immediate and six-month NL of universal adhesives is not influenced by the adhesive strategy selected.

METHODS AND MATERIALS

Tooth Selection and Preparation

Forty extracted caries-free human third molars were used. The teeth were collected after obtaining the respective patients' informed consent under a protocol approved by the local Ethics Committee Review Board. The teeth were disinfected in 0.5% chloramine, stored in distilled water, and used within six months of extraction. A flat dentin surface was exposed after wet grinding the occlusal enamel on a No. 180-grit SiC paper. The exposed dentin surfaces were further polished on wet No. 600-grit silicon-carbide paper for 60 seconds to standardize the smear layer.

Experimental Design

A total of five adhesive systems were evaluated. As control materials, the two-step Er, Adper Single Bond 2 (SB; 3M ESPE, St Paul, MN, USA), and the two-step Se, Clearfil SE Bond (CSE; Kuraray, Okayama, Japan), were used. The following three universal adhesive systems were tested in both the Er and Se strategies: Peak Universal Adhesive System (Peak LC Bond and Peak SE Primer Ultradent Products Inc, South Jordan, UT, USA), applied as a two-step Er (PkEr) and two-step Se (PkSe);

Scotchbond Universal Adhesive (3M ESPE), applied as a two-step Er (ScEr) and one-step Se (ScSe); and All Bond Universal (Bisco Inc, Shaumburg, IL, USA), applied as a two-step Er (AlEr) and one-step Se (AlSe). A total of eight experimental conditions were tested in this study, and five teeth were randomly assigned for each group.

Restorative Procedure and Specimen Preparation

The adhesive systems were applied as per the manufacturer's instructions (Table 1). After the bonding procedures, all teeth received a microhybrid composite restoration (Opallis, FGM Produtos Odontológicos, Joinville, SC, Brazil) in two increments of 2 mm. Each increment was light polymerized for 40 seconds using an LED light-curing unit set at 1200 mW/cm^2 (Radii-cal, SDI Limited, Bayswater, Victoria, Australia).

After the restored teeth had been stored in distilled water at 37°C for 24 hours, the specimens were sectioned longitudinally in the mesiodistal and buccal-lingual directions across the bonded interface using a slow-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to obtain 25-30 resin-dentin sticks with a cross-sectional area of approximately 0.8 mm^2 as measured with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan). All sticks from each tooth were divided for μTBS and NL evaluation. Usually, six sticks per tooth were used for NL, three in each storage time; the remaining sticks were used for μTBS, half in the immediate time and half after six months of water storage time (37°C).

Microtensile Bond Strength

Resin-dentin bonded sticks were attached to a Geraldeli jig[20] with cyanoacrylate adhesive and tested under tension (Kratos Dinamometros, Cotia, SP, Brazil) at 0.5 mm/min until failure. The μTBS values were calculated by dividing the load at failure by the cross-sectional bonding area.

The failure mode of the specimens was classified as cohesive (C; failure exclusive within dentin or resin composite), adhesive (A; failure at the resin-dentin interface), or mixed (M; failure at the resin-dentin interface, which included cohesive failure of the neighboring substrates). The classification was performed under a stereomicroscope at 100× magnification (Olympus SZ40, Tokyo, Japan). Specimens with premature failures were included in the tooth mean.

Nanoleakage

Three resin-bonded sticks from each tooth at each period were not tested in tension and were prepared for NL evaluation. The sticks were placed in an ammoniacal silver nitrate solution[21] in darkness for 24 hours, rinsed thoroughly in distilled water, and immersed in photo-developing solution for eight hours under a fluorescent light to reduce silver ions into metallic silver grains within voids along the bonded interface. Specimens were polished down to 2500-grit SiC paper and 1 and 0.25 μm diamond paste (Buehler Ltd) using a polishing cloth. They were ultrasonically cleaned, air dried, mounted on stubs, and coated with carbon-gold (Shimadzu IC 50, Tóquio, Japão). Resin-dentin interfaces were analyzed in a field-emission scanning electron microscope (SEM) operated in the backscattered mode (SSX-550, Shimadzu).

Three images were captured of each resin-dentin stick. The relative percentage of NL in the adhesive and hybrid layers was measured by a blinded operator (UTHSCSA ImageTool 3.0 software, Department of Dental Diagnostic Science at The University of Texas Health Science Center, San Antonio, TX, USA). Values originating from the same specimen were averaged for statistical purposes. The mean NL of all sticks from the same tooth was taken for statistical purposes.

Statistical Analysis

The experimental unit in the current study was the hemi-tooth. The μTBS and NL of all sticks from the same hemi-tooth were averaged for statistical purposes. The μTBS (MPa) and NL (%) means for every testing group were expressed as the average of five hemi-teeth used per group. The premature failures during specimen preparation were not included in the tooth mean. The μTBS (MPa) and NL (%) data were subjected to a two-way repeated-measure analysis of variance (adhesive vs storage time) and a post hoc test (Tukey post hoc test at $\alpha=0.05$) for pairwise comparisons.

RESULTS

The percentage of specimens with premature failure and the frequency of each fracture pattern mode are shown in Table 2. Few premature (5.7% on average) and cohesive failures (4.6% on average) were observed. Most of the specimens showed adhesive or adhesive/mixed failures.

Regarding μTBS, the cross-product interaction adhesive vs storage time was statistically significant

(p=0.001). PkSe and PkEr showed higher immediate μTBS, which was statistically similar to the control adhesives (CSE and SB; p>0.05; Table 3). However, a significant decrease in μTBS was observed for these materials after six months of water storage (p<0.05).

The adhesives ScSe, ScEr, AlSe, and AlEr showed lower immediate μTBS compared with the control adhesives (CSE and SB; p<0.05; Table 3). In the Se mode, only ScSe and AlSe showed no significant decrease of the μTBS after six months (p>0.05). In the Er mode, only Al showed significantly lower μTBS after six months (p<0.05).

The cross-product interaction adhesive vs storage time was statistically significant (p=0.001; Table 4). PkSe and PkEr in both bonding strategies showed the highest NL at the immediate time (p<0.05; Table 4), which significantly increased after six months (p<0.05; Table 4). Sb and Al, when applied in Se and Er mode, showed lower NL at both storage periods, which was statistically similar to the control adhesives (CSE and SB; p>0.05; Table 4).

Representative backscattered SEM images of the resin-dentin interfaces for all experimental conditions are depicted in Figure 1. Specimens of the Peak Universal Adhesive System applied as Er and Se (Figure 1B,J) showed a thicker area of silver nitrate deposition throughout the hybrid and adhesive layer at the immediate period. This deposition resembles the classic images of water trees[21,22] (Figure 1). A higher amount of NL was detected for this adhesive after six months (Fig. 1F,N).

For both control adhesives, as well as the universal adhesives Scotchbond Universal Adhesive tested in Er and Se strategies, a thinner deposition of silver nitrate was observed, mainly restricted to the base of the hybrid layer at the immediate time (Figure 1). This NL remained stable after six months of water storage (Figure 1).

DISCUSSION

The results of the present study demonstrated that the universal adhesive tested had a heterogeneous behavior, since some adhesives diminished the bonding performance over the course of time and some did not.

Although the adhesive Peak showed high immediate μTBS values, this material produced an adhesive interface with high deposition of NL at the immediate time. NL represents the location of defects at the resin-dentin interface that may serve as pathways for degradation of the resin-dentin bond over time.[22] Silver nitrate is capable of occupying nanometric-sized spaces present around the exposed collagen fibrils where the monomers were unable to infiltrate or where residual water was not displaced by the adhesive or even in areas with incomplete monomer conversion,[22] factors preponderant for the degradation of the bond interface.

PkSe can be categorized as an aggressive Se,[23,24] as this material has a very low pH (Peak SE Primer, pH=1.2) when compared with the other adhesives (Sb, pH=3.0; Al, pH=2.4).[25] This might be why this material showed the highest NL at the immediate period. It was already reported that acid and unpolymerized monomers are more present in acidic adhesive infiltrate than are polymerizable monomers.[5,6] Also, the hydrolysis of the ester bond of acidic monomer results in a strong phosphoric acid[5] that continues to demineralize the surrounding dentin.

Only the Peak material recommends the application of an extra layer of Peak LC Bond. Various *in vitro*[12,26,27,28] and *in vivo* studies[29,30] have shown that the application of an additional layer increases the performance of one-step Se adhesives, provided that this is a layer with a hydrophobic nature. This additional layer incorporates nonsolvated hydrophobic monomers at the bonding interface, which diminishes the relative concentration of solvents retained and nonreacted monomers in the adhesive layer,[31] making it less permeable[32,33] and less prone to the effects of degradation over the course of time.[34,35]

However, Peak LC Bond appears to be as hydrophilic as Peak SE Primer, since there are no hydrophobic monomers listed in the composition of Peak LC Bond (Table 1). In this way, the material does not take advantage of having a second adhesive layer; the high level of hydrophilicity must be responsible for the degradation of the adhesive interface.

Peak LC Bond is hydrophilic and is the recommended material to be used in the Er strategy. In the Er approach, the primer was not applied; the low pH of the Peak LC Bond (pH=2.0) might have caused an additional etching of the dentinal substrate. This probably resulted in an increase in the demineralization and collagen exposure,[24,36] thereby increasing the NL[37] even when used in the Er strategy, as can be seen in Figure 1.

Sc and Al are one-step Se adhesives and are therefore highly hydrophilic. In three of the four groups tested with these two adhesives, no degrada-

Table 1: *Adhesive System (Batch Number), Composition,[a] and Application Mode of the Adhesive Systems Used According to the Manufacturer's Instructions*

Adhesive (Batch Number)	Composition	Self-Etch Strategy (Se)	Etch-and-Rinse Strategy (Er)
Adper Single Bond 2 (BPBR)	1. Etchant: 35% phosphoric acid (Scotchbond Etchant) 2. Adhesive: Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and polyitaconic acids, 10% by weight of 5-nm-diameter spherical silica particles	NA	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Blot excess water 4. Apply 2-3 consecutive coats of adhesive for 15 s with gentle agitation 5. Gently air thin for 5 s 6. Light-cure for 10 s at 1200 mW/cm^2
Clearfil SE Bond (Primer: 00954A - Bond: 01416[a])	1. Primer: water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate 2. Bonding: MDP, Bis-GMA, HEMA, camphorquinone, hydrophobic dimethacrylate, N,N-diethanol p-toluidine bond, colloidal silica	1. Apply primer to tooth surface and leave in place for 20 s 2. Dry with air stream to evaporate the volatile ingredients 3. Apply bond to the tooth surface and then create a uniform film using a gentle air stream 4. Light-cure for 10 s at 1200 mW/cm^2	NA
Peak Universal Adhesive System (Peak SE Primer: 0N062–Peak LC Bond: Y062)	1. Etchant: 35% phosphoric acid (Ultraetch) 2. Primer: ethyl alcohol, methacrylic acid, 2-hydroxyethyl methacrylate (Peak SE Primer) 3. Adhesive: Ethyl alcohol, 2-hydroxyethyl methacrylate (Peak LC Bond)	1. Initial use of Peak SE requires activation of the two components separated in the syringe 2. Application of the Peak SE with microbrush for 20 s using continuous scrubbing on dentin; do not scrub enamel 3. Thin/dry for 3 s using air/water syringe or high-volume suction directly over preparation 4. Apply a puddle coat of Peak LC Bond with gently agitate for 10 s 5. Thin/dry 10 s using to air pressure 6. Light polymerize for 10 s at 1200 mW/cm^2	1. Apply etchant for 20 s 2. Rinse for 5 s 3. Air dry 2 s 4. Apply a puddle coat of Peak LC Bond with gently agitate for 10 s 5. Thin/dry 10 s using to air pressure 6. Light-cure for 10 s at 1200 mW/cm^2
Scotchbond Universal Adhesive (D-82229)	1. Etchant: 34% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminum oxide. (Scotchbond Universal Etchant) 2. Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	1. Apply the adhesive to the entire preparation with a microbrush and rub it in for 20 s; if necessary, rewet the disposable applicator during treatment 2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent is evaporated completely 3. Light-cure for 10 s at 1200 mW/cm^2	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Air dry 2 s 4. Apply adhesive as for the self-etch mode

Table 1: *Adhesive System (Batch Number), Composition,[a] and Application Mode of the Adhesive Systems Used According to the Manufacturer's Instructions (cont.)*

Adhesive (Batch Number)	Composition	Self-Etch Strategy (Se)	Etch-and-Rinse Strategy (Er)
All-Bond Universal (1200006111)	1. Etchant Uni-Etch: 32%phosphoric acid, benzalkonium chloride 2. Adhesive: MDP, Bis-GMA, HEMA, ethanol, water, initiators	1. Apply two separate coats of adhesive, scrubbing the preparation with a microbrush for 10-15 s per coat; do not light cure between coats; do not light polymerize between coats 2. Evaporate excess solvent by thoroughly air-drying with an air syringe for at least 10 s—there should be no visible movement of the material; the surface should have a uniform glossy appearance 3. Light cure for 10 s at 1200 mW/cm^2	1. Apply etchant for 15 s 2. Rinse thoroughly 4. Apply adhesive as for the self-etch mode 3. Remove excess water with absorbent pellet or high volume suction for 1-2 s

[a] *bis-GMA, bisphenol glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, methacryloyloxydecyl dihydrogen phosphate.*

tion of the resin-dentin bonds was observed. This must be attributed to the presence of monomers capable of producing a chemical bond to the hard structures of teeth,[14,15] as opposed to the lack of this compound in Pk.

Sc and Al contain methacryloyloxydecyl dihydrogen phosphate (MDP) in their composition, as does CSE, which was the first Se adhesive to incorporate this component. Studies with CSE have demonstrated that MDP allows for a stable chemical bond to dentin over the course of time, both *in vitro*[30,38-40] and *in vivo*.[13,41,42] This monomer forms a stable nanolayer together with a deposition of stable MDP-Ca salts at the adhesive interface,[43] which increases its mechanical strength.[43,44] However, regardless of the MDP, the adhesives showed different behaviors.

The μTBS values of ScSe and AlSe at the immediate time were not equivalent to those of the control CSE. ScSe and AlSe are one-step adhesives, and this probably leads to the concentration of MDP being lower than it is in CSE, which has MDP incorporated into both the primer and the

Table 2: *Number of Specimens (%) According to Fracture Mode and the Premature Failure of All Experimental Groups*

Adhesive System	Application Mode	Time	Fracture Pattern			
			A	C	A/M	PF
Adper Single Bond 2	Er control	Immediate	51 (73.9)	10 (14.5)	6 (8.7)	2 (2.9)
		6 mo	49 (73.1)	4 (6.0)	9 (13.4)	5 (7.5)
Clearfil SE Bond	Se control	Immediate	50 (74.6)	3 (4.5)	10 (14.9)	4 (6.0)
		6 mo	53 (80.3)	2 (3.0)	8 (12.1)	3 (4.6)
Peak Universal	Er	Immediate	56 (80)	0 (0)	10 (14.3)	4 (5.7)
		6 mo	52 (75.4)	2 (2.9)	12 (17.4)	3 (4.3)
	Se	Immediate	58 (82.8)	3 (4.3)	7 (10)	2 (2.9)
		6 mo	48 (69.6)	1 (1.5)	13 (18.8)	7 (10.1)
Scotchbond Universal	Er	Immediate	53 (79.1)	2 (3.0)	10 (14.9)	2 (3.0)
		6 mo	50 (78.1)	0 (0)	13 (20.3)	1 (1.6)
	Se	Immediate	47 (71.2)	3 (4.6)	11 (16.7)	5 (7.5)
		6 mo	51 (73.9)	1 (1.4)	14 (20.3)	3 (4.4)
Allbond Universal	Er	Immediate	49 (71)	8 (11.6)	7 (10.1)	5 (7.3)
		6 mo	49 (73.1)	2 (3.0)	12 (17.9)	4 (6.0)
	Se	Immediate	51 (72.85)	4 (5.7)	9 (12.9)	6 (8.6)
		6 mo	47 (69.1)	5 (7.4)	10 (14.7)	6 (8.8)

Abbreviations: A, adhesive fracture mode; C, cohesive fracture mode; A/M, adhesive/mixed fracture mode; PF, premature failure.

Table 3: *Microtensile Bond Strength (μTBS) Values (Means ± Standard Deviations) of the Different Experimental Groups (*)*

Time	Adhesive System							
	Adper Single Bond 2	Clearfil SE Bond	Peak Universal Se	Peak Universal Er	Scotchbond Universal Se	Scotchbond Universal Er	Allbond Universal Se	Allbond Universal Er
Immediate	47.6 ± 5.5 a	42.9 ± 4.4 a, b	39.5 ± 5.1 b	44.3 ± 1.6 a, b	33.3 ± 3.2 c	34.7 ± 4.6 b, c	20.9 ± 4.1 e	38.5 ± 4 b
6 mo	38.8 ± 5.7 b	36.2 ± 2.7 b, c	27.9 ± 4.9 d	34.2 ± 4.2 c	33.6 ± 5.8 c	34.6 ± 6.2 c	20.4 ± 4.8 e	28.1 ± 4.3 c

(*) Means identified with identical lower case letters are statistically similar ($p > 0.05$)

bond.[43] Moreover, it has been demonstrated that the presence of 2-hydroxyethyl methacrylate, a component of Sc and Al, may compete with MDP by bonding to the calcium of hydroxyapatite, thereby harming the chemical bond of MDP to dentin.[43]

Many other variables in the composition of the ScSe and AlSe may account for the differences observed between these materials as, for instance, in the presence of the polyalkenoic acid copolymer (PAC) in Sc and the high concentration of solvent of Al. Sc contains specific PACs used in resin-modified glass ionomer Vitrebond (3M ESPE). PAC bonds chemically and spontaneously to hydroxyapatite in glass ionomer materials,[45] and a recent study demonstrated that the presence of PAC showed more bond strength than a PAC-free adhesive with the same composition.[41,45] Yoshida and others[43] hypothesized that PAC may compete with the MDP present in Sc. However, if we compare the longevity results of Sc (MDP+PAC) with SB (PAC), two materials with similar compositions, the only difference being the presence of MDP in the former, it seems that the association MDP-PAC enhanced the bonding ability, since ScSe and ScEr showed stable bonds even after six months of water storage.

Al contains more solvent than Sc (30-60 wt% and 10-15 wt%, respectively).[46,47] This leads to more residual solvent retained in the hybrid layer and adhesive layer,[48] preventing the formation of a polymer with high reticulation.[49-51] As a consequence, a reduced degree of conversion[32] and μTBS values[52-55] is produced, making the adhesive interface more permeable after polymerization[56,57] and more prone to degradation over time.[3,58]

This may explain the lower results of AlSe in the immediate time interval in comparison with the other adhesives. This is in agreement with a recent study published by Munoz and others,[16] even when applied actively; whereas in the mentioned study, Al was applied passively.[16] Active/vigorous application improves the immediate and long-term results of the bond to dentin of the one-step SE adhesive systems,[59-62] because it increases the penetration of monomers into dentin and solvent evaporation. Agitation will also improve the efficacy of polymerization by improving the chemical interaction of the adhesive with the dental substrate, particularly for the acid Se adhesives.[37,63] In addition, unreacted acid monomers present in the superficial layer of the adhesive may be taken to a basal area of dentin, increasing demineralization of the substrate and diffusion of monomers and improving the interaction with the smear layer and subjacent dentin.[60-62]

As only AlEr demonstrated degradation of the μTBS values over the course of time after six months of evaluation, we could hypothesize that the presence of PAC is more important for Er adhesives than for Se. Some authors have indicated that the function of PAC is to improve the stability to humidity,[64,65] a crucial factor for Er adhesives, which, due to dentin demineralization, has a more sensitive technique when compared with that of the Se adhesives.[2]

We reject the first and second null hypotheses, given that the μTBS and NL values of universal

Table 4: *Nanoleakage (NL) Values (Means ± Standard Deviations) of the Different Experimental Groups (*)*

Time	Adhesive System							
	Adper Single Bond 2	Clearfil SE Bond	Peak Universal Bond Se	Peak Universal Bond Er	ScotchBond Universal Se	ScotchBond Universal Er	Allbond Universal Se	Allbond Universal Er
Immediate	13.1 ± 2.0 b	7.5 ± 2.9 a, b	31.6 ± 3.1 c	23.9 ± 4.2 c	5.1 ± 2.1 a	5.3 ± 1.1 a	6.0 ± 3.9 a	9.4 ± 1.8 b
6 mo	14.7 ± 4.1 b	8.6 ± 4.1 a, b	42.2 ± 2.6 d	34.4 ± 4.1 d	4.7 ± 2.8 a	5.4 ± 2.0 a	5.9 ± 1.4 a	8.9 ± 3.1 a, b

(*) Means identified with identical lower case letters are statistically similar ($p > 0.05$)

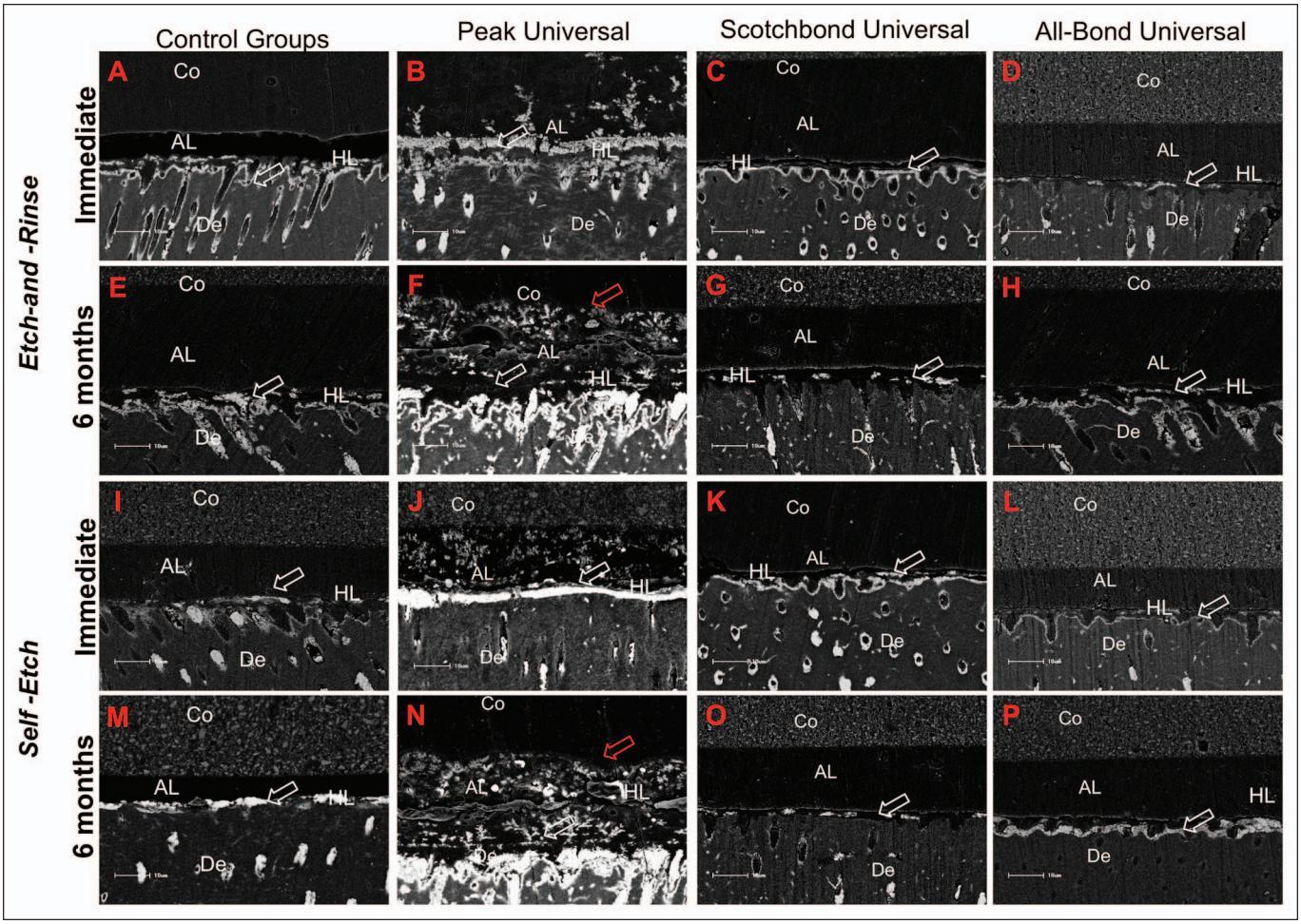

Figure 1. *Representative backscatter SEM images of the resin-dentin adhesive interfaces of each experimental group for immediate and six-month periods. Control etch-and-rinse group=Adper Single Bond 2 and control self-etch group=Clearfil SE Bond. For the Peak Universal Adhesive System, the amount of nanoleakage was higher (red and white arrows) than for the other materials and increased after six months for strategies Er (B, F) and Se (J, N). The amount of nanoleakage was lower and stable after six months within the hybrid layer for Scotchbond Universal (C, G, K, O), AllBond Universal (D, H, L, P), and controls (white arrows) (A, E. I, M). Co indicates composite; De, dentin; HL, hybrid layer; AL, adhesive layer; Se, self-etch; Er, etch-and-rinse.*

adhesives showed different results according to the Er or Se strategies employed.

CONCLUSIONS

Universal adhesives that contain MDP (Scochbond Universal Adhesive Er and Se and All Bond Universal Se) showed stable bond strengths and reduced NL, similar to the two-step SE adhesive tested (Clearfil SE Bond) after six months of water storage.

Acknowledgement

This study was performed by Miguel Angel Muñoz as partial fulfillment of her PhD degree at the State University of Ponta Grossa (UEPG), Brazil. This study was partially supported by CAPES and National Council for Scientific and Technological Development (CNPq) under grants #301937/2009-5 and #301891/2010-9. The authors are very grateful to the manufacturers for the generous donation of the adhesives used in the present study.

Human Subjects Approval

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies. The approval code for this study is 17878/13. This study was conducted at State University of Ponta Grossa, Paraná, Brazil.

Conflict of Interest

The authors have no proprietary, financial, or other personal interests of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 4 August 2014)

REFERENCES

1. Van Meerbeek B, Van Landuyt K, De Munck J, Hashimoto M, Peumans M, Lambrechts P, Yoshida Y, Inoue S, & Suzuki K (2005) Technique-sensitivity of contemporary adhesives *Dental Materials Journal* **24(1)** 1-13.
2. Perdigao J (2007) New developments in dental adhesion *Dental Clinics of North America* **51(2)** 333-357.
3. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, & Van Meerbeek B (2005) A critical review of the durability of adhesion to tooth tissue: methods and results *Journal of Dental Research* **84(2)** 118-132.
4. Reis A, Loguercio AD, Azevedo CL, de Carvalho RM, da Julio Singer M, & Grande RH (2003) Moisture spectrum of demineralized dentin for adhesive systems with different solvent bases *Journal of Adhesive Dentistry* **5(3)** 183-192.
5. Wang Y, & Spencer P (2005) Continuing etching of an all-in-one adhesive in wet dentin tubules *Journal of Dental Research* **84(4)** 350-354.
6. Carvalho RM, Chersoni S, Frankenberger R, Pashley DH, Prati C, & Tay FR (2005) A challenge to the conventional wisdom that simultaneous etching and resin infiltration always occurs in self-etch adhesives *Biomaterials* **26(9)** 1035-1042.
7. Unemori M, Matsuya Y, Akashi A, Goto Y, & Akamine A (2004) Self-etching adhesives and postoperative sensitivity *American Journal of Dentistry* **17(3)** 191-195.
8. Pashley DH, & Tay FR (2001) Aggressiveness of contemporary self-etching adhesives. Part II: etching effects on unground enamel *Dental Materials* **17(5)** 430-444.
9. Kanemura N, Sano H, & Tagami J (1999) Tensile bond strength to and SEM evaluation of ground and intact enamel surfaces *Journal of Dentistry* **27(7)** 523-530.
10. Hafer M, Schneider H, Rupf S, Busch I, Fuchss A, Merte I, Jentsch H, Haak R, & Merte K (2013) Experimental and clinical evaluation of a self-etching and an etch-and-rinse adhesive system *Journal of Adhesive Dentistry* **15(3)** 275-286.
11. Rotta M, Bresciani P, Moura SK, Grande RH, Hilgert LA, Baratieri LN, Loguercio AD, & Reis A (2007) Effects of phosphoric acid pretreatment and substitution of bonding resin on bonding effectiveness of self-etching systems to enamel *Journal of Adhesive Dentistry* **9(6)** 537-545.
12. Frankenberger R, Perdigao J, Rosa BT, & Lopes M (2001) "No-bottle" vs "multi-bottle" dentin adhesives—a microtensile bond strength and morphological study *Dental Materials* **17(5)** 373-380.
13. Peumans M, De Munck J, Van Landuyt KL, Poitevin A, Lambrechts P, & Van Meerbeek B (2010) Eight-year clinical evaluation of a 2-step self-etch adhesive with and without selective enamel etching *Dental Materials* **26(12)** 1176-1184.
14. Perdigao J, Sezinando A, & Monteiro PC (2012) Laboratory bonding ability of a multi-purpose dentin adhesive *American Journal of Dentistry* **25(3)** 153-158.
15. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Ende A, Van Meerbeek B, & De Munck J (2012) Bonding effectiveness of a new "multi-mode" adhesive to enamel and dentine *Journal of Dentistry* **40(6)** 475-484.
16. Perdigao J, & Loguercio AD (2014). Universal or multi-mode adhesives: why and how? *Journal of Adhesive Dentistry* **16(2)** 193-193.
17. Perdigao J, Munoz M, Sezinando A, Luque-Martinez I, Staichak R, Reis A, & Loguercio A (2014) Immediate adhesive properties to dentin and enamel of a universal adhesive associated with a hydrophobic resin coat *Operative Dentistry* **39(5)** 489-499.
18. Heintze SD, Ruffieux C, & Rousson V (2010) Clinical performance of cervical restorations—a meta-analysis *Dental Materials* **26(10)** 993-1000.
19. Marchesi G, Frassetto A, Mazzoni A, Apolonio F, Diolosa M, Cadenaro M, Pashley DH, Tay F, & Breschi L (2014) Adhesive performance of a multi-mode adhesive system: 1-year *in vitro* study *Journal of Dentistry* **42(5)** 603-612.
20. Perdigao J, Geraldeli S, Carmo AR, & Dutra HR (2002) *In vivo* influence of residual moisture on microtensile bond strengths of one-bottle adhesives *Journal of Esthetic and Restorative Dentistry* **14(1)** 31-38.
21. Tay FR, Pashley DH, Suh BI, Carvalho RM, & Itthagarun A (2002) Single-step adhesives are permeable membranes *Journal of Dentistry* **30(7-8)** 371-382.
22. Sano H (2006) Microtensile testing, nanoleakage, and biodegradation of resin-dentin bonds *Journal of Dental Research* **85(1)** 11-14.
23. Tay FR, & Pashley DH (2001) Aggressiveness of contemporary self-etching systems. I: depth of penetration beyond dentin smear layers *Dental Materials* **17(4)** 296-308.
24. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, & Vanherle G (2003) Buonocore memorial lecture: adhesion to enamel and dentin: current status and future challenges *Operative Dentistry* **28(3)** 215-235.
25. Munoz MA, Luque I, Hass V, Reis A, Loguercio AD, & Bombarda NH (2013) Immediate bonding properties of universal adhesives to dentine *Journal of Dentistry* **41(5)** 404-411.
26. Feitosa VP, Leme AA, Sauro S, Correr-Sobrinho L, Watson TF, Sinhoreti MA, & Correr AB (2012) Hydrolytic degradation of the resin-dentine interface induced by the simulated pulpal pressure, direct and indirect water ageing *Journal of Dentistry* **40(12)** 1134-1143.
27. Reis A, Albuquerque M, Pegoraro M, Mattei G, Bauer JR, Grande RH, Klein-Junior CA, Baumhardt-Neto R, & Loguercio AD (2008) Can the durability of one-step self-etch adhesives be improved by double application or by an extra layer of hydrophobic resin? *Journal of Dentistry* **36(5)** 309-315.
28. Albuquerque M, Pegoraro M, Mattei G, Reis A, & Loguercio AD (2008) Effect of double-application or the application of a hydrophobic layer for improved efficacy of one-step self-etch systems in enamel and dentin *Operative Dentistry* **33(5)** 564-570.

29. Loguercio AD, & Reis A (2008) Application of a dental adhesive using the self-etch and etch-and-rinse approaches: an 18-month clinical evaluation *Journal of the American Dental Association* **139(1)** 53-61.

30. Reis A, Leite TM, Matte K, Michels R, Amaral RC, Geraldeli S, & Loguercio AD (2009) Improving clinical retention of one-step self-etching adhesive systems with an additional hydrophobic adhesive layer *Journal of the American Dental Association* **140(7)** 877-885.

31. Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, & De Stefano Dorigo E (2008) Dental adhesion review: aging and stability of the bonded interface *Dental Materials* **24(1)** 90-101.

32. Cadenaro M, Antoniolli F, Sauro S, Tay FR, Di Lenarda R, Prati C, Biasotto M, Contardo L, & Breschi L (2005) Degree of conversion and permeability of dental adhesives *European Journal of Oral Sciences* **113(6)** 525-530.

33. Breschi L, Cadenaro M, Antoniolli F, Sauro S, Biasotto M, Prati C, Tay FR, & Di Lenarda R (2007) Polymerization kinetics of dental adhesives cured with LED: correlation between extent of conversion and permeability *Dental Materials* **23(9)** 1066-1072.

34. Reis A, Grande RH, Oliveira GM, Lopes GC, & Loguercio AD (2007) A 2-year evaluation of moisture on microtensile bond strength and nanoleakage *Dental Materials* **23(7)** 862-870.

35. Reis A, de Carvalho Cardoso P, Vieira LC, Baratieri LN, Grande RH, & Loguercio AD (2008) Effect of prolonged application times on the durability of resin-dentin bonds *Dental Materials* **24(5)** 639-644.

36. Liu Y, Tjaderhane L, Breschi L, Mazzoni A, Li N, Mao J, Pashley DH, & Tay FR (2011) Limitations in bonding to dentin and experimental strategies to prevent bond degradation *Journal of Dental Research* **90(8)** 953-968.

37. Wang Y, & Spencer P (2003) Hybridization efficiency of the adhesive/dentin interface with wet bonding *Journal of Dental Research* **82(2)** 141-145.

38. Toledano M, Osorio R, Osorio E, Aguilera FS, Yamauti M, Pashley DH, & Tay F (2007) Durability of resin-dentin bonds: effects of direct/indirect exposure and storage media *Dental Materials* **23(7)** 885-892.

39. Waidyasekera K, Nikaido T, Weerasinghe DS, Ichinose S, & Tagami J (2009) Reinforcement of dentin in self-etch adhesive technology: a new concept *Journal of Dentistry* **37(8)** 604-609.

40. Inoue S, Koshiro K, Yoshida Y, De Munck J, Nagakane K, Suzuki K, Sano H, & Van Meerbeek B (2005) Hydrolytic stability of self-etch adhesives bonded to dentin *Journal of Dental Research* **84(12)** 1160-1164.

41. Perdigao J, Kose C, Mena-Serrano A, De Paula E, Tay L, Reis A, & Loguercio A (2014) A new universal simplified adhesive: 18-month clinical evaluation *Operative Dentistry* **39(2)** 113-127.

42. Mena-Serrano A, Kose C, De Paula EA, Tay LY, Reis A, Loguercio AD, & Perdigao J (2013) A new universal simplified adhesive: 6-month clinical evaluation *Journal of Esthetic and Restorative Dentistry* **25(1)** 55-69.

43. Yoshida Y, Yoshihara K, Nagaoka N, Hayakawa S, Torii Y, Ogawa T, Osaka A, & Meerbeek BV (2012) Self-assembled nano-layering at the adhesive interface *Journal of Dental Research* **91(4)** 376-381.

44. Yoshihara K, Yoshida Y, Nagaoka N, Fukegawa D, Hayakawa S, Mine A, Nakamura M, Minagi S, Osaka A, Suzuki K, & Van Meerbeek B (2010) Nano-controlled molecular interaction at adhesive interfaces for hard tissue reconstruction *Acta Biomaterialia* **6(9)** 3573-3582.

45. Mitra SB, Lee CY, Bui HT, Tantbirojn D, & Rusin RP (2009) Long-term adhesion and mechanism of bonding of a paste-liquid resin-modified glass-ionomer *Dental Materials* **25(4)** 459-466.

46. Chen L, & Suh BI (2013) Effect of hydrophilicity on the compatibility between a dual-curing resin cement and one-bottle simplified adhesives *Journal of Adhesive Dentistry* **15(4)** 325-331.

47. 3M ESPE (2013) Material Safety Data Sheet; Retrieved online December 6, 2013 from: http://multimedia.3m.com/mws/mediawebserver?mwsId=SSSSSuUn_zu8l00xMY_eMYt1Nv70k17zHvu9lxtD7SSSSSS--

48. Yiu CK, Pashley EL, Hiraishi N, King NM, Goracci C, Ferrari M, Carvalho RM, Pashley DH, & Tay FR (2005) Solvent and water retention in dental adhesive blends after evaporation *Biomaterials* **26(34)** 6863-6872.

49. Ye Q, Spencer P, Wang Y, & Misra A (2007) Relationship of solvent to the photopolymerization process, properties, and structure in model dentin adhesives *Journal of Biomedical Materials Research Part A* **80(2)** 342-350.

50. Loguercio AD, Loeblein F, Cherobin T, Ogliari F, Piva E, & Reis A (2009) Effect of solvent removal on adhesive properties of simplified etch-and-rinse systems and on bond strengths to dry and wet dentin *Journal of Adhesive Dentistry* **11(3)** 213-219.

51. Paul SJ, Leach M, Rueggeberg FA, & Pashley DH (1999) Effect of water content on the physical properties of model dentine primer and bonding resins *Journal of Dentistry* **27(3)** 209-214.

52. Hass V, Folkuenig MS, Reis A, & Loguercio AD (2011) Influence of adhesive properties on resin-dentin bond strength of one-step self-etching adhesives *Journal of Adhesive Dentistry* **13(5)** 417-424.

53. Reis A, Ferreira SQ, Costa TR, Klein-Junior CA, Meier MM, & Loguercio AD (2010) Effects of increased exposure times of simplified etch-and-rinse adhesives on the degradation of resin-dentin bonds and quality of the polymer network *European Journal of Oral Sciences* **118(5)** 502-509.

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

55. Cho BH, & Dickens SH (2004) Effects of the acetone content of single solution dentin bonding agents on the adhesive layer thickness and the microtensile bond strength *Dental Materials* **20(2)** 107-115.

56. Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR, Yiu CK, & Carrilho MR (2006)

Water sorption/solubility of dental adhesive resins *Dental Materials* **22(10)** 973-980.

57. Ito S, Hashimoto M, Wadgaonkar B, Svizero N, Carvalho RM, Yiu C, Rueggeberg FA, Foulger S, Saito T, Nishitani Y, Yoshiyama M, Tay FR, & Pashley DH (2005) Effects of resin hydrophilicity on water sorption and changes in modulus of elasticity *Biomaterials* **26(33)** 6449-6459.
58. Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, & De Stefano Dorigo E (2008) Dental adhesion review: aging and stability of the bonded interface *Dental Materials* **24(1)** 90-101.
59. do Amaral RC, Stanislawczuk R, Zander-Grande C, Michel MD, Reis A, & Loguercio AD (2009) Active application improves the bonding performance of self-etch adhesives to dentin *Journal of Dentistry* **37(1)** 82-90.
60. Miyazaki M, Platt JA, Onose H, & Moore BK (1996) Influence of dentin primer application methods on dentin bond strength *Operative Dentistry* **21(4)** 167-172.
61. Pleffken PR, de Almeida Lourenco AP, Torres CR, & Buhler Borges A (2011) Influence of application methods of self-etching adhesive systems on adhesive bond strength to dentin *Journal of Adhesive Dentistry* **13(6)** 517-525.
62. Loguercio AD, Stanislawczuk R, Mena-Serrano A, & Reis A (2011) Effect of 3-year water storage on the performance of one-step self-etch adhesives applied actively on dentine *Journal of Dentistry* **39(8)** 578-587.
63. Zhang Y, & Wang Y (2013) Effect of application mode on interfacial morphology and chemistry between dentine and self-etch adhesives *Journal of Dentistry* **41(3)** 231-240.
64. Van Meerbeek B, Conn LJ Jr, Duke ES, Eick JD, Robinson SJ, & Guerrero D (1996) Correlative transmission electron microscopy examination of nondemineralized and demineralized resin-dentin interfaces formed by two dentin adhesive systems *Journal of Dental Research* **75(3)** 879-888.
65. Spencer P, Wang Y, Walker MP, Wieliczka DM& Swafford JR (2000) Interfacial chemistry of the dentin/adhesive bond *Journal of Dental Research* **79(7)** 1458-1463.

©Operative Dentistry, 2015, **40-3,** 293-303

Bond Durability of Different Resin Cements to Caries-Affected Dentin Under Simulated Intrapulpal Pressure

MF Mohamed • HA El Deeb • IE Gomaa
EH Mobarak

Clinical Relevance

Resin cement with self-etch adhesive containing methacryloyloxydecyl dihydrogen phosphate shows promise in obtaining homogenous and durable bonding to normal and caries-affected dentin.

SUMMARY

Objective: To evaluate the durability of the bond of different resin cement systems to normal dentin (ND) and caries-affected dentin (CAD) with and without simulated intrapulpal pressure (IPP).

Methods and Materials: Molars with midcoronal caries were used. Occlusal enamel was cut to expose both dentin substrates (ND and CAD). Dentin substrates were differentiated using visual, tactile, caries-detecting dye, and dye-permeability methods. Prepared crown segments were equally divided according to the tested resin cement systems: etch-and-rinse resin cement, self-etch resin cement containing methacryloyloxydecyl dihydrogen phosphate (MDP), and self-adhesive resin cement. In addition to the dentin substrates and the resin cement types, the effect of application/storage conditions (with or without simulated IPP and with or without thermocycling) were tested. A microtensile bond strength test was done using a universal testing machine. Failure modes were determined using a scanning electron microscope.

Results: Etch-and-rinse resin cement strength values were significantly affected by the difference in the dentin substrates as well as the different application/storage conditions. Self-etch adhesive containing MDP bonded equally to ND and CAD and remained stable under all tested conditions. Self-adhesive resin cement revealed a similar bond to ND and CAD; however, its values were the lowest, especially

Mona F Mohamed, Operative Dentistry, Modern Science and Arts University, 6th October City, Egypt

Heba A El Deeb, Restorative Dentistry Department, Faculty of Oral and Dental Medicine, Cairo University, Cairo, Egypt

Iman E Gomaa, Department of Pharmaceutical Biology and Biotechnology, German University in Cairo–GUC, Cairo, Egypt

*Enas H Mobarak, Restorative Dentistry Department, Faculty of Oral and Dental Medicine, Cairo University, Cairo, Egypt

*Corresponding author: 14 El Zahra Street, Cairo 12311 Egypt; e-mail: enasmobarak@hotmail.com

DOI: 10.2341/14-035-L

when IPP and thermocycling were combined. Mixed failure was the predominant failure mode.

Conclusions: Etch-and-rinse resin cement was sensitive to dentin substrate and application/storage conditions. Resin cement with self-etch adhesive containing MDP revealed more reliable bonding to ND/CAD even when IPP and thermocycling were combined. The bonding of the self-adhesive resin cement could not compete with other resin cements.

INTRODUCTION

Advances in adhesive dentistry have provided solutions to many esthetic challenges faced by clinicians. In certain clinical situations, indirect resin composite restorations represent an alternative to direct ones due to some advantages such as the ease of developing and maintaining occlusal surface anatomy, contours, and contacts.[1] Nevertheless, one of the prime requirements for achieving successful indirect composite restorations is to gain proper bonding to the tooth structure.[2] Resin cements are increasingly used for bonding indirect restorations due to their better bond strengths, excellent mechanical properties, and improved esthetics when compared with conventional cements.[3] Currently, resin cements are used with etch-and-rinse or self-etch adhesives. However, the increased tendency to simplify and reduce the bonding steps has led to combining the adhesive system and the cement in a single application through the use of self-adhesive resin cements.[4]

At the same time, bonding to tooth structure depends not only on adhesive systems but also on the bonding substrates.[5] Many of the dentin bonding studies were done on normal human dentin. However, this is not the substrate frequently encountered in clinical dentistry.[6] Caries-affected dentin (CAD) has different structural and compositional characteristics compared with normal dentin (ND). CAD also has shown different permeability with intrapulpal pressure.[7] Thus, bonding to this dynamic substrate is crucial and appears to influence the long-term durability of adhesive systems. Consequently, it would be of interest to evaluate the bonding performance of the different resin cement systems with different adhesive strategies to ND and CAD after the application of simulated intrapulpal pressure with and without thermocycling.

The null hypotheses were 1) the different types of dentin substrates (ND and CAD) had no impact on dentin/resin cement bond strength; 2) there was no difference in the microtensile bond strength among the different resin cement systems of different adhesive strategies; and 3) the different application/storage conditions (intrapulpal pressure simulation or thermocycling alone or in combination) had no effect on the bond strength of different resin cements to dentin.

METHODS AND MATERIALS

Specimen Preparation

A total of 120 molars with midcoronal caries (site and size = 1.2) were used.[8] The collected teeth were stored in phosphate buffer saline containing 0.2% sodium azide at 4°C for not more than two weeks until being used.[9] The study was accomplished in accord with local human participants' oversight committee guidelines.

Occlusal enamel was trimmed perpendicularly to the long axis of each tooth using a slow-speed diamond-saw sectioning machine (Buehler Isomet Low Speed Saw, Lake Bluff, IL, USA) under water coolant to expose flat ND and CAD surfaces. Ground dentin surfaces were examined for any signs of exposure to be discarded. Another cut was made parallel to the occlusal surface, 2 mm below the cementoenamel junction, exposing the pulp chamber. Remnants of pulp tissue in the pulp chamber were removed using a discoid excavator (Carl Martin GmbH, Solingen, Germany) without touching the walls of the pulp.[10] Each crown segment was mounted on a polymethacrylate plate containing a 19-gauge needle in the center using cyanoacrylate adhesive (Rocket Heavy, Dental Ventures of America, Corona, CA, USA) and subsequently embedded in chemically cured polyester resin (Polyester resin #2121, Hsein, Taiwan) up to the level of the cementoenamel junction.[10]

CAD Identification

CAD was differentiated with the aid of visual and tactile methods.[11] In addition, caries-detecting dye [12] and the dye permeability test [13] were used. In the dye permeability test, 10% methylene blue was permeated into the tooth through the pulp chamber under pressure. Caries-detecting dye appears to stain partially demineralized collagen matrices in CAD.[14] The selective staining of ND with methylene blue was attributed to decreased permeability of CAD in relation to ND due to the presence of peritubular and intertubular crystal formation into the dentinal tubules. Thus, the ND was stained

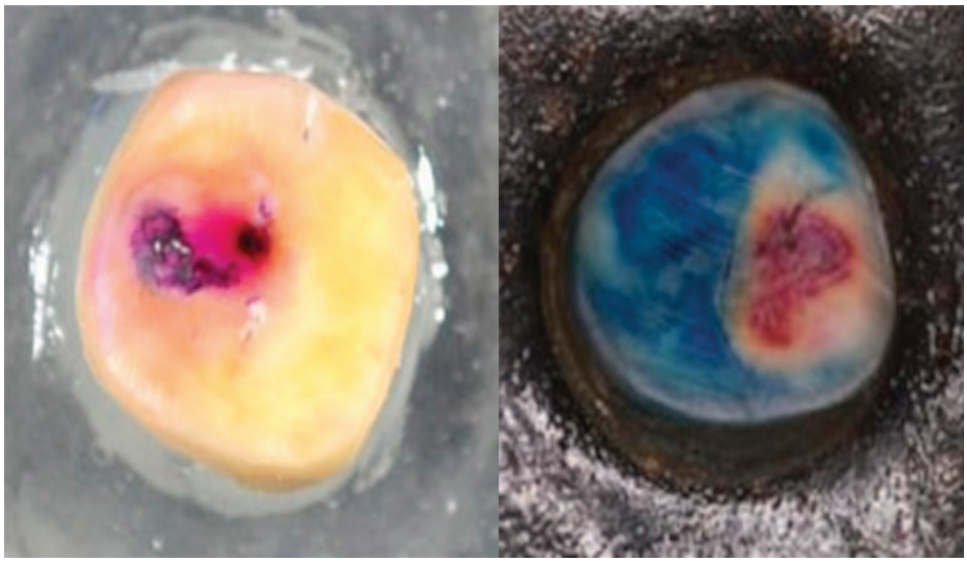

Figure 1. *Caries-affected dentin identification using caries-detecting dye and methylene-blue dye permeability test.*

blue, whereas the CAD was stained a pale pink (Figure 1). The dentin surface was flattened using a rotary grinding machine under water coolant and wet hand polished using 600-grit silicon carbide paper (MicroCut, 8 inch, Buehler Ltd, Lake Bluff, IL, USA) for 10 seconds to produce a standardized smear layer.[13]

Restorative Procedures

The crown segments with ND and CAD were divided equally according to the type of resin cement system (resin cement with etch-and-rinse adhesive [Variolink II/Adper Scotch Bond Multi-Purpose, VL, 3M ESPE, St Paul, MN, USA], resin cement with self-etch adhesive containing methacryloyloxydecyl dihydrogen phosphate [MDP; Panavia F2.0/ED primer II, PF, Kuraray Medical, New York, NY, USA], or self-adhesive resin cement [RelyX Unicem 2, RX, 3M ESPE]). Table 1 shows materials specifications, manufacturers, compositions, and batch numbers. The prefabricated resin composite blocks (4-mm thickness) were made using microhybrid resin composite (Filtek Z250, 3M ESPE) and light cured for 40 seconds from the top and the bottom using an LED light-curing unit (Guilin Woodpecker Medical Instrument Company, London, UK) with an intensity of 800 mW/cm^2. The resin composite blocks were subjected to additional light and heat curing at 110°C for seven minutes, using an oven (Coltene/Whaledent, Inc. DI-500, Cuyahoga Falls, OH, USA) to ensure complete polymerization. The bonding surface of each block was sandblasted with 50 μm alumina particles for 20 seconds using the Renfert sandblasting device (US Dental Depot, Fort Lauderdale, FL, USA). To standardize the distance to 1 cm and the angle to be 90° between the resin composite block and the nozzle handpiece of the sandblasting device, a specially constructed assembly was used. Following sandblasting, chemical surface treatment was done using Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) silane coupling agent. The silane was applied to the sandblasted surface using a microbrush, left for one minute, and then air dried using an air spray syringe.

The cementation procedure was carried out according to manufacturers' instructions (Table 2) while the specimens were still connected to the intrapulpal pressure assembly under 15 mm Hg (P_1), reproducing the effect of the local vasoconstrictor in local anesthetics,[15] or not (P_0). After bonding of the P_1 specimens, the intrapulpal pressure was raised to 20 mm Hg. The specimens of P_0 and P_1 were then inserted in plastic containers containing artificial saliva. The intrapulpal pressure assembly and the specimens in the plastic containers were placed in a specially constructed large incubator at 37°C for 24 hours (Figure 2). After incubation, the specimens were sectioned to obtain multiple sticks (0.9 ± 0.01 mm^2). A precise digital caliper (Proficraft, Mebschieber, Germany) was used to check the cross-sectional area and length of the sticks. Sticks (n=2 for each dentin substrate/specimen) of the same cross-sectional area and length were selected. Then, half of the sticks (ND and CAD) were subjected to 10,000 thermocycling cycles between 5°C and 55°C with a dwell time of 30 seconds and a transfer time of 10 seconds[16] (T_1) and the other half were not (T_0).

Table 1: *Material Specifications, Manufacturers, Compositions, and Batch Numbers*

Material Specification	Manufacturer	Composition	Batch No.
Etch-and-rinse resin cement system Variolink II resin cement	Ivoclar Vivadent Schaan, Liechtenstein	Monomer matrix: Bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate.	39409
		Fillers: barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, spheroid mixed oxide.	
Etch-and-rinse adhesive system Adper Scotch Bond MultiPurpose	3M ESPE Dental Products St Paul, MN, USA	Additional contents: catalysts, stabilizers, pigments. The base contains 26.3% wt monomer and 73.4% wt filler, whereas the catalyst (high viscosity) contains 22% wt monomer and 77.2% wt filler.	75405
		Etchant: 37% phosphoric acid (pH $<$ 1)	
		Primer: ethanol, HEMA and polyalkenoic acid	
		Adhesive: HEMA and Bis-GMA	
Self-etch resin cement Panavia F2.0	Kuraray Medical, New York, NY, USA	Paste A: MDP, hydrophobic aromatic dimethacrylate, hydrophobicaliphatic dimethacrylate, hydrophilic aromatic dimethacrylate, silanated silica filler, silanated colloidal silica, di-camphorquinone, catalysts, intiators	0256AA
		Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, surface-treated sodium fluoride, catalysts, accelerators, pigments	
Single-step self-etch adhesive system ED primer II	Kuraray Medical, New York, NY, USA	Liquid A: HEMA, MDP, water, 5-NMSA, accelerators	0509AA
		Liquid B: 5-NMSA, water, catalysts, accelerators	
		Oxyguard II: glycerol, polyethyleneglycol catalysts, accelerators, dyes.	
Self adhesive resin cement RelyX Unicem 2	3M ESPE Dental Products St Paul, MN, USA	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers initiators, stabilizers	424967
		Catalyst paste: methacrylate monomers, alkaline fillers, silanated fillers, initiators, stabilizers, pigments	
Filtek Z250	3M ESPE Dental Products St Paul, MN, USA	Resin: Bis-GMA, UDMA, bis-EMA	138842
		Fillers: (60%) zirconia/silica with particle size 0.01-3.5 μm	

Abbreviations: Bis-EMA, bisphenol ethoxylated dimethacrylate; Bis-GMA, bisphenol glycidyl methacrylate; 5-NMSA, N-methacryloyl-5-aminosalicylic acid; HEMA, 2-hydroxyethylmethacrylate; MDP, methacryloyloxydecyl dihydrogen phosphate; UDMA, urethane dimethacrylate.

Microtensile Bond Strength Testing

Each stick was fixed to the modified Academisch Centrum Tandheelkunde Amsterdam (ACTA) microtensile strength jig[17] with a cyanoacrylate adhesive (Rocket Heavy) and stressed in tension using a universal testing machine (Lloyd Instruments Ltd, Ametek Company, Bognor Regis, West Sussex, UK) at a cross-head speed of 0.5 mm/min until failure. The tensile force at failure was recorded and converted to tensile stress in megapascal units using computer software (Nexygen-MT, Lloyd Instruments). Sticks that failed before testing were counted as 0 MPa.[10,18] Bond strength values were submitted to a three-way analysis of variance (ANOVA) with repeated measures to determine the effect of dentin substrates, the resin cements systems, and application/storage conditions. It was also used to detect any significant interactions among these three variables. One-way ANOVA was

Table 2: *Adhesive Systems Application and Resin Cements Manipulation*

Resin Cement System	Adhesive Systems Application Steps	Resin Cements Manipulation
Etch-and-rinse resin cement system (Adper Scotch Bond MultiPurpose and Variolink II resin cement)	-Etchant: applied for 15 seconds, rinsed with water spray for 15 seconds, then dried with gentle air flow for 5 seconds. -Adper Scotch Bond MultiPurpose primer: applied, left undisturbed for five seconds, then gently thinned with a mild oil-free air stream for five seconds and at a distance from the dentin surface of 2 cm. -Adper Scotch Bond MultiPurpose adhesive: applied, left undisturbed for 15 seconds, and light cured for 10 seconds	The base and catalyst pastes of Variolink II were applied in a 1:1 ratio and mixed for 10 seconds then applied on treated dentin surface. The resin composite onlay was then cemented under a specified load, and the excess cement was removed using a sponge followed by light curing for 20 seconds.
Single-step self-etch resin cement system (ED primer II and Panavia F2.0 resin cement)	- One drop of liquid A and B of ED Primer was mixed on a glass slab, applied on the dentin surface, and left undisturbed for 30 seconds. -The excess was removed with a sponge, then dried with gentle air flow for five seconds and at distance of 2 cm from dentin surface.	-Equal amounts of paste A and paste B of resin cement was mixed on a glass slab for 20 seconds and applied on the treated surface of composite inlay. -Resin composite onlay was then cemented under a specified load, and the excess cement was removed using a sponge followed by light curing for 20 seconds -Oxyguard II was applied directly to the margins of the restoration, left for three minutes, and then removed.
Self-adhesive resin cement RelyX Unicem 2	No dentin surface pretreatment was done	-Equal amounts of base and catalyst pastes were dispensed, then mixed for 20 seconds. -The mix was applied on the treated dentin surface. -The resin composite onlay was then cemented under a specified load, and the excess cement was removed using a sponge followed by light curing for 20 seconds.

used to test the significant difference among the tested resin cements bonded to each dentin substrate with each application/storage condition as well as for the significant difference among the application/storage conditions for each resin cement bonded to each dentin substrate. Pairwise comparisons were calculated using the Bonferroni test. The *t*-test was used to test the significant difference between the bond strength values to ND and CAD with each resin cement under each application/storage condition. A probability test set at $\alpha=0.05$ was used for statistical significance. All statistical calculations were done using computer program SPSS version 15 (IBM SPSS statistics, Armonk, NY, USA) for Microsoft Windows.

The failure modes of all specimens were evaluated using a scanning electron microscope (SEM) at 100× magnification. Failure modes were classified as type 1: adhesive failure along the dentin side; type 2: cohesive failure in the adhesive layer; type 3: cohesive failure in the resin cement; type 4: mixed failure (adhesive failure along the dentin side and cohesive failure in the adhesive layer); type 5: mixed failure (adhesive failure along the dentin side and cohesive failure in the resin cement); type 6: mixed failure (adhesive failure along the dentin side, cohesive failure in the adhesive layer and cohesive failure in the resin cement); and type 7: mixed failure (cohesive failure in the adhesive layer and cohesive failure in the resin cement).

RESULTS

The three-way repeated measures ANOVA results showed a statistically significant effect for the dentin substrates (ND and CAD) ($p<0.001$), the resin cement systems (VL, PF, and RX) ($p<0.001$), and the application/storage conditions (P_0T_0 [control], P_1T_0, P_0T_1, P_1T_1) ($p=0.021$). The interaction between resin cement systems and application/storage conditions was also significant ($p=0.002$). Nevertheless, the interactions among dentin substrates × resin cement systems, dentin substrates × applica-

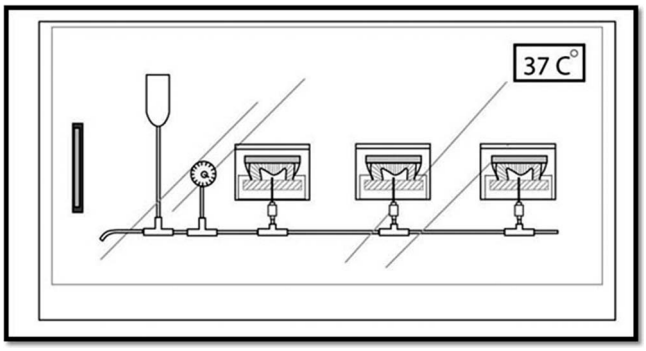

Figure 2. *A diagram showing the specimens in the incubator while immersed in artificial saliva and subjected to intrapulpal pressure simulation.*

tion/storage conditions, and dentin substrates × resin cement systems × application/storage conditions were not significant ($p>0.05$). Means and standard deviations (SD) of microtensile bond strength (μTBS) values of all tested variables are presented in Table 3. VL showed higher bond strength to ND compared with CAD after 24 hours with P_0T_0 ($p=0.02$) and with P_0T_1 ($p=0.04$); whereas, with the other application/storage conditions no significant differences were recorded. Also, the bond strength values of PF and RX to ND were not significantly different from those to CAD under any application/storage condition.

Regarding the bond strength among the different resin cements with each application/storage condition, one-way ANOVA revealed a statistically significant difference among them whether bonded to ND or to CAD when subjected to P_0T_0 and P_1T_1 conditions (Table 3). For ND with the P_0T_0 condition, VL bond strength values showed statistically significantly higher value compared with PF and RX, although no statistically significant difference was recorded between PF and RX values (Table 3). For the P_1T_1 condition, RX revealed the lowest value. For CAD with the P_0T_0 and P_1T_1 conditions, RX also revealed the lowest value.

VL bond strength values to ND and CAD were significantly different with the tested application/storage conditions; RX bond strength values significantly decreased after P_1T_1 application/storage condition. Nevertheless, values for PF bond strength to both dentin substrates were not statistically changed with all tested application/storage conditions (Table 3).

Regarding the failure modes, Figure 3 shows the percentages of the recorded failure modes. VL bonded to ND and CAD showed predominately type 6 mode of failure, whereas PF specimens displayed type 5. RX bonded to ND commonly showed type 1 failure, whereas with CAD, type 5 was the most frequent failure mode. Representative SEM micrographs for some failure modes of different resin cements bonded to either ND or CAD are presented in Figure 4.

DISCUSSION

Results of the present study indicate the rejection of the three null hypotheses because the difference in dentin substrates (ND and CAD) has a significant effect on dentin/resin cement bond strength. Also, there was a statistically significant difference among the three types of resin cements. Moreover, the difference in application/storage conditions showed a

Table 3: *Mean and Standard Deviation (SD) of the Microtensile Bond Strength (μTBS) Values (MPa) of the Tested Variables*[a]

Dentin Substrates	Application/Storage Conditions	Resin Cement Systems			*p*-Value
		Variolink II	Panavia F2.0	Rely X Unicem 2	
Normal dentin (ND)	Artificial saliva (control) P_0T_0	35.2 ± 5.8*Aa	24.9 ± 6.1 Ba	20.5 ± 2.6 Ba	*<.01*
	IPP simulation P_1T_0	26.3 ± 6.0 Ab	24.4 ± 4.6 Aa	19.5 ± 5.3 Aa	*.07*
	Thermocycling P_0T_1	26.6 ± 6.7**Ab	26.0 ± 5.6 Aa	21.2 ± 7.1 Aa	*.26*
	IPP simulation/thermocycling P_1T_1	20.3 ± 4.0 Ac	19.6 ± 1.9 Aa	16.4 ± 2.3 Ba	.04
p-value		*<.001*	.07	.27	
Caries-affected dentin (CAD)	Artificial saliva (control) P_0T_0	28.8 ± 2.8*Aa	23.7 ± 6.0 Aa	17.9 ± 4.7 Ba	<.01
	IPP simulation P_1T_0	27.0 ± 8.0 Aa	20.2 ± 7.0 Aa	18.6 ± 7.1 Aa	.10
	Thermocycling P_0T_1	20.3 ± 3.2 **Ab	21.4 ± 3.6 Aa	22.5 ± 2.5 Aa	.41
	IPP simulation/thermocycling P_1T_1	22.3 ± 3.0 Ab	22.9 ± 2.1 Aa	13.7 ± 3.8 Bb	<.01
p-value		*<.01*	.57	.02	

Abbreviations: P_0T_0, specimens stored in artificial saliva for 24 hours, neither subjected to intrapulpal pressure (IPP) simulation nor to thermocycling; P_1T_0, specimens subjected to intrapulpal simulation (IPP) but not subjected to thermocycling; P_0T_1, specimens subjected to thermocycling but not IPP simulation; P_1T_1, specimens subjected to IPP simulation and thermocycling.

[a] *Different capital letters denote significant differences within rows, whereas different small letters denote significant differences within a column for each dentin substrate. * and ** indicate differences between ND and CAD for the same resin cement subjected to the same storage condition.*

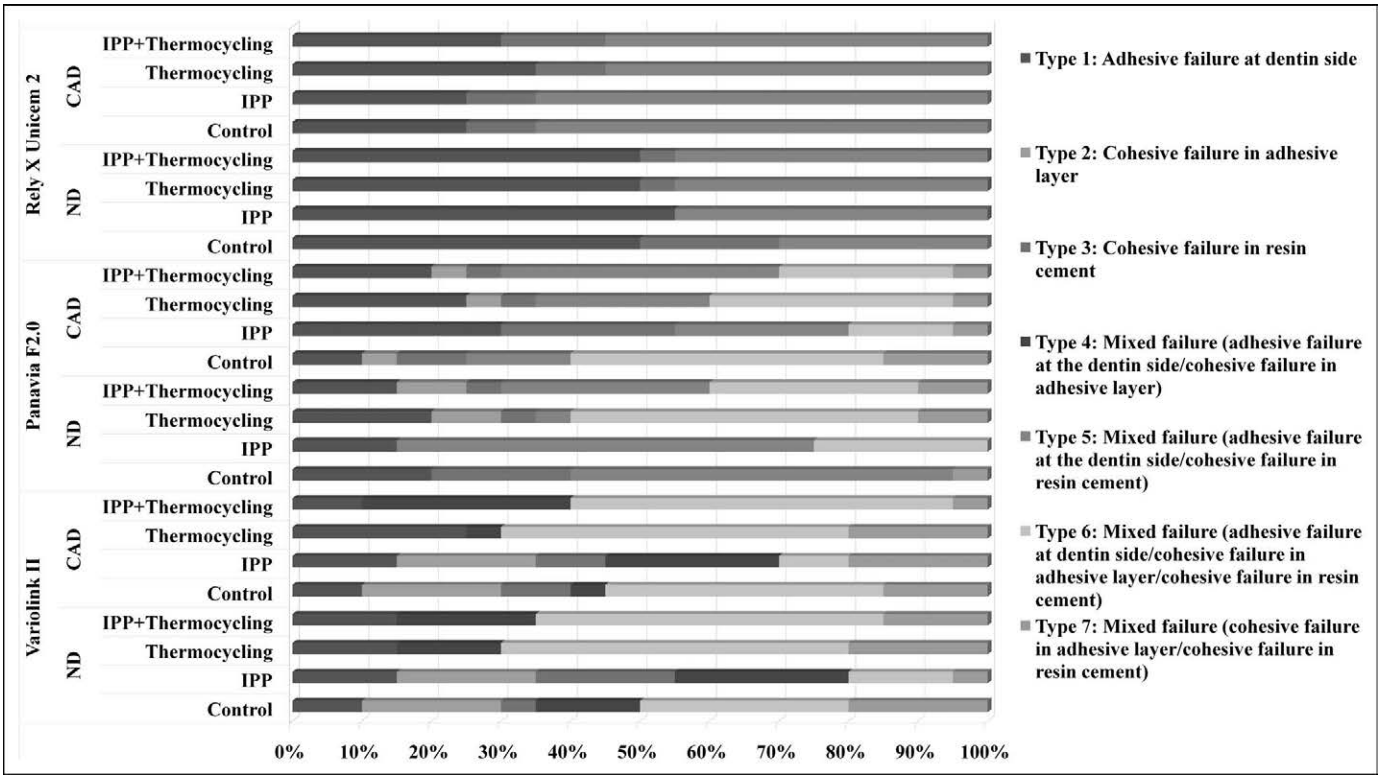

Figure 3. *The percentages of the recorded modes of failure.*

significant effect on the bond strength to different dentin substrates.

In the present study, the different resin cement systems bonded differently with the different dentin substrates (ND and CAD). The significant effect for the difference in dentin substrates on bonding was recorded by others, although they tested other adhesive systems.[19-22]

VL bonded to ND showed significantly higher bond strength values than those of PF and RX resin cement systems. The better bonding of etch-and-rinse adhesive systems over other systems has been previously reported.[23] The superiority of the etch-and-rinse resin cement system in bonding to ND might be attributed to the effect of phosphoric acid etching to dentin, which results in removal of the smear layer, thus allowing proper resin infiltration of the dentin.[24] The primer component of the etch-and-rinse adhesive Adper Scotch Bond MultiPurpose used with VL resin cement contains ethanol, which helps to remove the residual water from the collagen matrix and keeps the expanded collagen matrix stiffened, allowing for better resin infiltration.[25] Moreover, the hydrophobic bisphenol glycidyl methacrylate (Bis-GMA) and the hydrophilic 2-hydroxyethyl methacrylate (HEMA) components of VL are soluble in ethanol.[26] This allows for more resin infiltration into acid-etched matrices, forming well-hybridized resin tags that contribute to micromechanical retention.[3] On the other hand, the lower μTBS of the RX system compared with VL etch-and-rinse resin cement could be attributed to its mode of adhesion, which counts on smear layer penetration rather than its removal; thus, a weak link is expected to be formed between the resin cements and the underlying dentin.[27] Although the RX can form a chemical bond with the smear layer–covered dentin,[28] this reaction is superficial with no hybrid layer formation.[29] The nonsignificant difference between the PF and RX when bonded to ND was in accordance with De Munck and others,[4] Abo-Hamar and others,[30] and Tonial and others.[31] However, Abo-Hamar and others[30] disagreed with the results of the present study because they found that the bond strength of VL was not significantly different from those of PF and RX. This could be because they used a different etch-and-rinse adhesive system (Syntac) with the VL resin cement. Yang and others[3] showed that the bond strength of PF was higher than that of RX, which also contradicted the present study's findings. This can be attributed to the mode of cure used for the RX in their study (self-cure mode) and the dual-cure mode used in the present

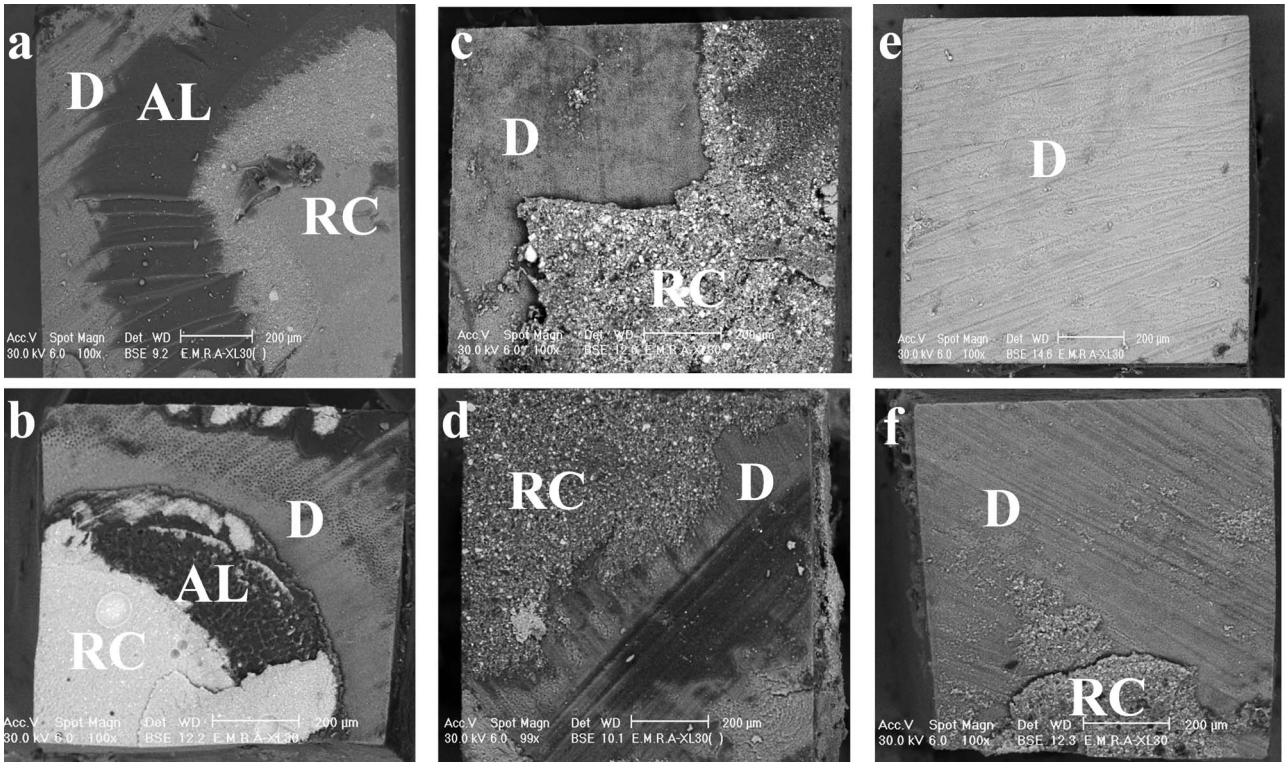

Figure 4. *SEM photomicrographs showing the predominant failure modes of Variolink II resin cement (a): bonded to ND and (b): bonded to CAD. Panavia F2.0 resin cement (c): bonded to ND and (d): bonded to CAD. RelyX Unicem 2 resin cement (e): bonded to ND and (f): bonded to CAD. AL, adhesive layer; D, dentin; RC, resin cement.*

study. It was reported that the degree of conversion of RX with the self-cure mode was lower than that with either the light- or dual-cure mode.[32]

Regarding the CAD, VL (etch-and-rinse resin cement) bond strength values were greatly affected. The decrease in bond strength of etch-and-rinse adhesives bonded to CAD has also been reported by other researchers.[12,21,22] In CAD, the intertubular dentin has been, to some extent, demineralized due to the caries process before acid etching. After the etching step with the VL resin cement system, the demineralized layer might be too deep to be efficiently infiltrated by the adhesive, including the whole demineralized substrate thickness, resulting in a defective hybrid layer.[21] In addition, it has been reported that CAD contains deposits of "β-tricalcium phosphate ("β-TCP; also called whitlockite) in the dentinal tubules. These demineralized/remineralized portions in the same carious lesion cause disproportional and nongradient infiltration of resin monomers into the CAD substrate.[33] The high hydrophilic, unsaturated, methacrylate phosphate ester functional monomer (10-MDP) content in the PF self-etch adhesive resin cement system allows an intense chemical interaction with hydroxyapatite[34] that is highly available in CAD. This interaction could promote reliable and better hybridization. As for RX, it incorporates two setting reactions: a dual-cured redox reaction for polymerization of the resinous phase and an acid-base reaction resulting in the formation of calcium phosphates. Bonding with dentin is established by the ionization of phosphoric acid methacrylates of the monomer mixture. This ionization occurs either *in situ* from the water content of the dentin or from that produced during the neutralization reaction of the phosphate monomers with the basic filler of the cement.[35] The bonding mechanism is essentially similar to glass ionomers with an intermediate interfacial layer incorporating partially dissolved smear particles and possible regional formation of a nano-hybrid layer.[36]

Regarding subjection to the different application/storage conditions, VL (etch-and-rinse) resin cement showed a drop in bond strength to ND with P_1T_0 compared with those bonded and stored with P_0T_0; whereas, PF and RX resin cements revealed no significant difference from P_1T_0. The cause of the drop in bonding of VL may be related to the removal of the smear layer through acid etching, which may

have increased the outward flow of dentinal fluid along the dentinal tubules.[37] The outward flow could counteract the adhesive monomer penetration, dilute their concentration, and prevent the optimal polymerization.[38]

On the contrary, in the PF group applied under intrapulpal pressure (P_1), the smear plugs remained in the dentinal tubules so that under simulated intrapulpal pressure, moisture contamination by dentinal fluids transudation would be minimal.[28] This helps to provide a barrier against the effect of outward flow of dentinal fluids. The RX self-adhesive resin cement can benefit from the presence of intrapulpal pressure because it can provide additional hydration to the dentin. The presence of water is believed to optimize the chemical reaction of the self-adhesive resin cement with dentin.[39] In addition, the protective effect of the smear layer may accentuate the nonsignificant effect of intrapulpal pressure on this resin cement type.[39]

Thermocycling is considered as an adjunct to assess the effect of thermal stresses and prolonged water exposure on the bond strength to dentin.[40] The protocol for thermocycling applied in this study for all resin cements, which was 10,000 cycles, was reported to correspond to one year of *in vivo* simulation.[41] The results of this study showed that thermocycling had no significant effect on the bond strength of PF and RX resin cements to ND and CAD. The chemical bond formed between hydroxyapatite and RX and the functional phosphate monomer (MDP) of PF, can account for this dentin bond stability.[42] This chemical bond was reported not to dissociate in water, according to the adhesion-decalcification concept.[43] The VL showed also a decrease in bond strength after thermocycling (T_1) compared with its bond strength without intrapulpal pressure and thermocycling (P_0T_0). The HEMA content in the Adper Scotch Bond MultiPurpose adhesive system used might be the cause of the compromised bond strength obtained. HEMA is likely to absorb large amounts of water within the adhesive and the hybrid layer during thermocycling; hence, water that remains entrapped at the resin-dentin interface jeopardizes the stability of the bond.[15]

One of the new findings of the present study was that the combination between bonding under intrapulpal pressure simulation P_1 and thermocycling T_1 dramatically decreased the bond strength of VL and RX resin cement systems. With respect to RX, the chemical bond formed between the phosphoric acid monomer and calcium of hydroxyapatite that resisted the effect of P_1 or T_1 alone was not sustained when the conditions were combined. Both conditions could have synergistic action, causing added degradation of bond strength. The recorded predominant adhesive mode of failure for RX when P_1 and T_1 were combined may justify this explanation.

The results of this study highlight that various structural components and properties of dentin, as well as biological and clinical factors of the oral cavity, could directly affect the adhesive bond. Therefore, much of our understanding of dental bonding has to be tested on normal as well as on clinically relevant caries-affected substrate faced in dental practice while simulating the *in vivo* conditions.

CONCLUSIONS

Etch-and-rinse resin cement is sensitive to dentin substrate and storage conditions. Resin cement with self-etch adhesive containing MDP revealed more reliable bonding to ND and CAD even when combining intrapulpal pressure and thermocycling. Self-adhesive resin cement bonding still cannot compete with other resin cements.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 28 April 2014)

REFERENCES

1. Heymann H, Swift EJ, Ritter AV, & Sturdevant CM (2012) *Sturdevant's Art and Science of Operative Dentistry* Elsevier/Mosby St Louis, Mo **6th edition**.
2. Hikita K, Van Meerbeek B, De Munck J, Ikeda T, Van Landuyt K, Maida T, Lambrechts P, & Peumans M (2007) Bonding effectiveness of adhesive luting agents to enamel and dentin *Dental Materials* **23(1)** 71-80.
3. Yang B, Ludwig K, Adelung R, & Kern M (2006) Micro-tensile bond strength of three luting resins to human regional dentin *Dental Materials* **22(1)** 45-56.
4. De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, & Van Meerbeek B (2004) Bonding of an auto-adhesive luting material to enamel and dentin *Dental Materials* **20(10)** 963-971.
5. Wang Y, Spencer P, & Walker MP (2007) Chemical profile of adhesive/caries-affected dentin interfaces using Raman microspectroscopy *Journal of Biomedical Materials Research Part A* **81(2)** 279-286.
6. Haj-Ali R, Walker M, Williams K, Wang Y, & Spencer P (2006) Histomorphologic characterization of noncarious

and caries-affected dentin/adhesive interfaces *Journal of Prosthodontics* **15(2)** 82-88.

7. Nakajima M, Kunawarote S, Prasansuttiporn T, & Tagami J (2011) Bonding to caries-affected dentin *Japanese Dental Science Review* **47(2)** 102-114.
8. Mount GJ, Tyas JM, Duke ES, Hume WR, Lasfargues JJ, & Kaleka R (2006) A proposal for a new classification of lesions of exposed tooth surfaces *International Dental Journal* **56(2)** 82-91.
9. Mobarak EH, El-Badrawy W, Pashley DH, & Jamjoom H (2010) Effect of pretest storage conditions of extracted teeth on their dentin bond strengths *Journal of Prosthetic Dentistry* **104(2)** 92-97.
10. El-Deeb HA, Al Sherbiney HH, & Mobarak EH (2013) Bond durability of adhesives containing modified-monomer with/without fluoride after aging in artificial saliva and under intrapulpal pressure simulation *Operative Dentistry* **38(1)** 48-56.
11. Nakajima M, Sano H, Zheng L, Tagami J, & Pashley DH (1999) Effect of moist vs dry bonding to normal vs caries-affected dentin with Scotchbond Multi-Purpose Plus *Journal of Dental Research* **78(7)** 1298-1303.
12. Erhardt MC, Toledano M, Osorio R, & Pimenta LA (2008) Histomorphologic characterization and bond strength evaluation of caries-affected dentin/resin interfaces: Effects of long-term water exposure *Dental Materials* **24(6)** 786-798.
13. Mobarak EH, & El-Badrawy WH (2012) Microshear bond strength of self-etching adhesives to caries-affected dentin identified using the dye permeability test *Journal of Adhesive Dentistry* **14(3)** 245-250.
14. Alleman DS, & Magne P (2012) A systematic approach to deep caries removal end points: The peripheral seal concept in adhesive dentistry *Quintessence International* **43(3)** 197-208.
15. Sauro S, Mannocci F, Toledano M, Osorio R, Thompson I, & Watson TF (2009) Influence of the hydrostatic pulpal pressure on droplets formation in current etch-and-rinse and self-etch adhesives: A video rate/TSM microscopy and fluid filtration study *Dental Materials* **25(11)** 1392-1402.
16. Ulker M, Ozcan M, Sengun A, Ozer F, & Belli S (2010) Effect of artificial aging regimens on the performance of self-etching adhesives *Journal of Biomedical Materials Research Part B Applied Biomaterials* **93(1)** 175-184.
17. Mobarak E, El-Deeb H, & El-Samman M (2013) The difference in microtensile-bond strength jigs influences the test outcome *Journal of Dental Research* **92(Special Issue B) (IADR Irish Division Annual Scientific Meeting)** Retrieved online 15 Nov, 2013 from: http://www.iadr.ie/wp-content/uploads/2013/11/IADR-Irish-Division-Annual-Scientific-Meeting-2013-No-headers-new.p
18. Van Landuyt KL, Mine A, De Munck J, Jaecques S, Peumans M, Lambrechts P, & Van Meerbeek B (2009) Are one-step adhesives easier to use and better performing? Multifactorial assessment of contemporary one-step self-etching adhesives *Journal of Adhesive Dentistry* **11(3)** 175-190.
19. Ceballos L, Camejo DG, Victoria Fuentes M, Osorio R, Toledano M, Carvalho RM, & Pashley DH (2003) Microtensile bond strength of total-etch and self-etching adhesives to caries-affected dentine *Journal of Dentistry* **31(7)** 469-477.
20. Omar H, El-Badrawy W, El-Mowafy O, Atta O, & Saleem B (2007) Microtensile bond strength of resin composite bonded to caries-affected dentin with three adhesives *Operative Dentistry* **32(1)** 24-30.
21. Scholtanus JD, Purwanta K, Dogan N, Kleverlaan CJ, & Feilzer AJ (2010) Microtensile bond strength of three simplified adhesive systems to caries-affected dentin *Journal of Adhesive Dentistry* **12(4)** 273-278.
22. Yoshiyama M, Urayama A, Kimochi T, Matsuo T, & Pashley DH (2000) Comparison of conventional vs self-etching adhesive bonds to caries-affected dentin *Operative Dentistry* **25(3)** 163-169.
23. Luhrs AK, Guhr S, Gunay H, & Geurtsen W (2010) Shear bond strength of self-adhesive resins compared to resin cements with etch and rinse adhesives to enamel and dentin *in vitro Clinical Oral Investigations* **14(2)** 193-199.
24. Pashley DH, Agee KA, Nakajima M, Tay FR, Carvalho RM, Terada RS, Harmon FJ, Lee WK, & Rueggeberg FA (2001) Solvent-induced dimensional changes in EDTA-demineralized dentin matrix *Journal of Biomedical Materials Research* **56(2)** 273-281.
25. Carvalho RM, Mendonca JS, Santiago SL, Silveira RR, Garcia FC, Tay FR, & Pashley DH (2003) Effects of HEMA/solvent combinations on bond strength to dentin. *Journal of Dental Research* **82(8)** 597-601.
26. Guimaraes LA, Almeida JC, Wang L, D'Alpino PH, & Garcia FC (2012) Effectiveness of immediate bonding of etch-and-rinse adhesives to simplified ethanol-saturated dentin *Brazilian Oral Research* **26(2)** 177-182.
27. Mak YF, Lai SC, Cheung GS, Chan AW, Tay FR, & Pashley DH (2002) Micro-tensile bond testing of resin cements to dentin and an indirect resin composite *Dental Materials* **18(8)** 609-621.
28. Hiraishi N, Yiu CK, King NM, & Tay FR (2009) Effect of pulpal pressure on the microtensile bond strength of luting resin cements to human dentin *Dental Materials* **25(1)** 58-66.
29. Escribano N, & de la Macorra JC (2006) Microtensile bond strength of self-adhesive luting cements to ceramic *Journal of Adhesive Dentistry* **8(5)** 337-341.
30. Abo-Hamar SE, Hiller KA, Jung H, Federlin M, Friedl KH, & Schmalz G (2005) Bond strength of a new universal self-adhesive resin luting cement to dentin and enamel *Clinical Oral Investigations* **9(3)** 161-167.
31. Tonial D, Ghiggi PC, Lise AA, Burnett LH Jr, Oshima HM, & Spohr AM (2010) Effect of conditioner on microtensile bond strength of self-adhesive resin cements to dentin. *Stomatologija* **12(3)** 73-79.
32. Tezvergil-Mutluay A, Lassila LV, & Vallittu PK (2007) Degree of conversion of dual-cure luting resins light-polymerized through various materials *Acta Odontologica Scandinavica* **65(4)** 201-205.

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

34. Fukegawa D, Hayakawa S, Yoshida Y, Suzuki K, Osaka A, & Van Meerbeek B (2006) Chemical interaction of phosphoric acid ester with hydroxyapatite *Journal of Dental Research* **85(10)** 941-944.

35. Al-Assaf K, Chakmakchi M, Palaghias G, Karanika-Kouma A, & Eliades G (2007) Interfacial characteristics of adhesive luting resins and composites with dentine *Dental Materials* **23(7)** 829-839.

36. Tay FR, Pashley EL, Huang C, Hashimoto M, Sano H, Smales RJ, & Pashley DH (2001) The glass-ionomer phase in resin-based restorative materials *Journal of Dental Research* **80(9)** 1808-1812.

37. Belli S, Unlu N, & Ozer F (2001) Bonding strength to two different surfaces of dentin under simulated pulpal pressure *Journal of Adhesive Dentistry* **3(2)** 145-152.

38. Hashimoto M, Tay FR, Svizero NR, de Gee AJ, Feilzer AJ, Sano H, Kaga M, & Pashley DH (2006) The effects of common errors on sealing ability of total-etch adhesives *Dental Materials* **22(6)** 560-568.

39. Mazzitelli C, Monticelli F, Osorio R, Casucci A, Toledano M, & Ferrari M (2008) Effect of simulated pulpal pressure on self-adhesive cements bonding to dentin *Dental Materials* **24(9)** 1156-1163.

40. Yang B, Adelung R, Ludwig K, Bossmann K, Pashley DH, & Kern M (2005) Effect of structural change of collagen fibrils on the durability of dentin bonding *Biomaterials* **26(24)** 5021-5031.

41. De Munck J, Van Landuyt K, Coutinho E, Poitevin A, Peumans M, Lambrechts P, & Van Meerbeek B (2005) Micro-tensile bond strength of adhesives bonded to Class-I cavity-bottom dentin after thermo-cycling *Dental Materials* **21(11)** 999-1007.

42. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, Inoue S, Tagawa Y, Suzuki K, De Munck J, & Van Meerbeek B (2004) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **83(6)** 454-458.

43. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, & Van Landuyt KL (2011) State of the art of self-etch adhesives *Dental Materials* **27(1)** 17-28.

©Operative Dentistry, 2015, **40-3,** 304-312

Bonding Ability of Paste-Paste Glass Ionomer Systems to Tooth Structure: *In Vitro* Studies

NB Cook • SA Feitosa • A Patel
Y Alfawaz • GJ Eckert • MC Bottino

Clinical Relevance

Bond strength and interfacial microleakage data support the continued use of traditional resin-modified glass-ionomer cement (RMGIC) with polyacrylic acid pretreatment over new paste-paste RMGIC systems conditioned with their respective nonrinse conditioner.

SUMMARY

This study investigated the effect of nonrinse conditioners (ie, Ketac Nano Primer [KNP] and GC Self Conditioner [SC]) used as substrate pretreatment and their respective paste-paste resin-modified glass-ionomer cement (RMGIC) (ie, Ketac Nano [KN] and Fuji Filling LC [FF]) on microtensile bond strength to dentin and marginal sealing when compared with traditional RMGIC (ie, Photac Fil [PF] and Fuji II LC [FII]) used in association with polyacrylic acid (ie, Ketac Cavity Conditioner [KC] and GC Cavity Conditioner [CC]). A total of 192 extracted human molars were allocated into eight groups: KNP-KN, KC-KN, KNP-PF, KC-PF, SC-FF, CC-FF, SC-FII, and CC-FII. For microtensile bond strength, the teeth were sectioned to expose occlusal dentin and restored according to the group. After 24 hours the teeth were cut to yield nine beams per tooth (±0.8 mm^2). Testing was done using a universal testing machine followed by failure mode classification. For microleakage testing, standardized cavity preparations were made on the buccal cementoenamel junction and restored according to the group. The teeth were thermocycled (500 cycles, 8°C to 48°C), sealed, immersed in methylene blue for 24 hours, and then assessed for microleakage using a stereomicroscope. Microtensile bond

N Blaine Cook, DDS, MSD, Department of Restorative Dentistry, Graduate Operative, Indiana University School of Dentistry, Indianapolis, IN, USA

Sabrina A. Feitosa, DDS, MSD, PhD student, Department of Dental Materials and Prosthodontics, São José dos Campos Dental School, São Paulo State University, São José dos Campos, SP, Brazil

Ashish Patel, BDS, MSD, Department of Restorative Dentistry, Graduate Operative, Indiana University School of Dentistry, Indianapolis, IN, USA

Yasser Alfawaz, BDS, MSD, Department of Restorative Dentistry, Graduate Operative, Indiana University School of Dentistry, Indianapolis, IN, USA

George J Eckert, MAS, Department of Biostatistics, Indiana University School of Medicine, Indianapolis, IN, USA

*Marco C Bottino, DDS, MSc, PhD, Department of Restorative Dentistry, Dental Biomaterials Division, Indiana University School of Dentistry, Indianapolis, IN, USA

*Corresponding author: 1121 W Michigan St, Indianapolis, IN 46202 USA; e-mail: mbottino@iu.edu

DOI: 10.2341/14-030-L

strengths in megapascals (mean±SE) were KNP-KN: 14.9 ± 1.6, KC-KN: 17.2 ± 1.5, KNP-PF: 31.2 ± 1.6, KC-PF: 26.2 ± 1.2, SC-FF: 23.6 ± 1.5, SC-FII: 31.2 ± 1.5, and CC-FII: 21.9 ± 1.5. Cervical margins showed more microleakage compared with occlusal margins. Overall, the use of nonrinse conditioners in association with traditional RMGICs demonstrated superior microtensile bond strengths to dentin when compared with the paste-paste RMGICs. Meanwhile, the association between polyacrylic acid (CC) and a traditional RMGIC (FII) led to the least microleakage for cervical locations when compared with all other groups.

INTRODUCTION

Restorative glass ionomer cements (GIC) were introduced commercially to dentistry in the early 1970s.[1] Though relatively fragile, they possess unique properties such as the ability to bond chemically to tooth structure, a favorable coefficient of thermal expansion, and fluoride release/recharge capability. In the 1980s, resin-modified glass ionomer cement (RMGIC) was developed. By adding a water-soluble resin monomer and a polymerization initiator to conventional GIC, the overall strength, translucency, and polishability of the material were improved without adversely affecting its aforementioned favorable properties.[2-6]

Polyacrylic acid has been widely used as a substrate conditioner to improve the adhesion of both GIC and RMGIC to tooth structure. Polyacrylic acid removes the smear layer and contains carboxyl ion groups that form hydrogen bonds that promote cleansing and wetting of the substrates.[7-9] Though laboratory studies suggest low bond strengths, closer investigation revealed primarily cohesive failure due to the relatively weak shear and tensile strengths of the GIC.[10-12] However, long-term clinical studies demonstrate durable, reliable bonding to tooth structure even in the absence of enamel.[13-17]

Two paste-paste RMGIC systems (Ketac Nano [3M ESPE, St Paul, MN, USA] and Fuji Filling LC [GC America, Alsip, IL, USA]) claim improved esthetics and bond strength. The manufacturers developed "nonrinse" dentin conditioners (Ketac Nano Primer [3M ESPE] and GC Self Conditioner [GC America]) to be used with these materials. According to the manufacturers' instructions, optimal bond strength will not be achieved using polyacrylic acid. Furthermore, the manufacturers claim that these nonrinse conditioners can be used with traditional RMGIC. We find it interesting that material safety data sheets indicate that these conditioners are primarily resin. Ketac Nano Primer has some polyacrylic acid, whereas GC Self Conditioner has none, suggesting that adhesion to tooth structure may be due to mechanisms other than GIC bonding. The aim of this study was to evaluate and compare both the microtensile bond strength and microleakage of paste-paste RMGIC systems with traditional RMGIC when the dentin substrate is conditioned with either a nonrinse conditioner or traditional polyacrylic acid.

METHODS AND MATERIALS

A total of 192 extracted nonrestored human molars were collected. The teeth were hand scaled, cleaned, and stored in distilled water at 23°C ± 2°C for a minimum of 12 hours prior to use. Half the teeth were allocated for the microtensile and half for the microleakage studies.

Microtensile Bond Strength Testing

For the microtensile test, the occlusal surfaces were ground to expose dentin using a wheel polishing machine with 180-grit silicon carbide (SiC) paper. The absence of enamel was verified using a stereomicroscope (45×). The exposed dentin was wet-finished with 400- and 600-grit SiC to produce a standardized smear layer. The teeth were stored in distilled water, then randomly allocated into eight groups (n=12). Next, the test materials (Table 1) were bonded to the teeth following manufacturer recommendations. For each group, conditioner was applied first, then the tooth was restored with the respective glass ionomer restorative. For all curing procedures, the output of the curing light was monitored using a Demetron radiometer (model 100, Demetron Research Corp, Danbury, CT, USA) to maintain a >600 mW/cm^2 light output.

Conditioner Application

- Ketac Nano groups: Ketac Nano Primer (KNP) was applied to the finished dentin surface for 15 seconds using a flexible disposable applicator (Kerr Applicators, Orange, CA, USA). The primer was air dried for 10 seconds and light cured for 10 seconds (Optilux 400 light-cure unit, Demetron Research Corp).
- Ketac Conditioner groups: Ketac Cavity Conditioner (KC) was applied to the finished dentin surface using a flexible disposable applicator, left on the tooth for 10 seconds, and then rinsed with water spray for 10 seconds. Excess moisture was blotted

Table 1: *Materials, Code, Batch Number and Chemical Composition*

GIC/Manufacturer	Batch Number	Chemical Composition
Ketac Nano (KN) (3M ESPE)	268895	Paste A: 40%-50% silane-treated glass, 20%-30% silane-treated zirconia, 5%-15% polyethylene glycol dimethacrylate (PEGDMA), 5%-15% silane-treated silica, 1%-15% 2-hydroxyethyl methacrylate (HEMA), <5% glass powder, <5% bisphenol-A diglycidyl ether dimethacrylate (BISGMA), <1% triethylene glycol dimethacrylate (TEGDMA).
	264728	Paste B: 40%-60% silane-treated ceramic, 20%-30% copolymer of acrylic and itaconic acids, 10%-20% water, 1%-10% HEMA.
Ketac Nano Primer (KNP) (3M ESPE)	N253374 N241199	40%-50% water, 35%-45% HEMA, 10%-15% copolymer of acrylic and itaconic acids.
Photac Fil (PF) (3M ESPE)	439731	L: 30%-50% polyethylenepolycarbonicacid, 25%-50% HEMA, 20%-30% water, 3%-10% diurethane dimethacrylate, 5%-10% magnesium HEMA ester.
	440857	P: >99% glass powder.
Ketac Cavity Conditioner (KC) (3M ESPE)	431890 405279	70%-80% water, 20%-30% polyacrylic acid.
Fuji Filling LC (FF) (GC America)	1010061	Paste A: 75%-85% alumino-silicate glass, 10%-12% HEMA, 2%-5% urethane dimethacrylate.
	1011251	Paste B: 20%-30% distilled water, 20%-30% polyacrylic acid, 12%-15% urethane dimethacrylate, 10%-15% silicone dioxide.
GC Self Conditioner (SC) (GC America)	1011161	28%-40% ethanol, 30%-35% distilled water, 20%-30% HEMA, 5%-10% 4-methacryloxyethyl trimellitate anhydride.
Fuji II LC (FII) (GC America)	1009221	P: 100% alumino-silicate glass. L: 20%-30% distilled water, 20%-30% polyacrylic acid, 30%-35% HEMA, <10% urethane dimethacrylate, <1% camphorquinone.
GC Cavity Conditioner (CC) (GC America)	1103151	20% polyacrylic acid, 77% distilled water, 3% aluminum chloride hydrate, <0.1% food additive blue No. 1.

dry with Kim Wipes (Kimberly Clark, Roswell, GA, USA).

- GC Self Conditioner groups: GC Self Conditioner (SC) was applied to the finished dentin surface. The conditioner was left undisturbed for 10 seconds (nonrinse conditioner).
- GC Cavity Conditioner groups: GC Cavity Conditioner (CC) (20% polyacrylic acid) was applied to the dentin surface using a flexible disposable applicator. The conditioner was left on the dentin surface for 10 seconds, rinsed away with a 10-second water spray, and then excess moisture was blotted dry with Kim Wipes.

Restoration Placement

For all groups, a clear matrix band was placed around the circumference of the tooth, and then shade A2 RMGIC material was placed on the tooth in 2-mm increments and light cured for 20 seconds per increment until a filling height of 5 mm was reached.

- Ketac Nano groups: Ketac Nano (KN) was applied to the tooth from a quick-mix capsule.
- Photac Fil groups: Photac Fil (PF) capsules were activated using a 3M ESPE capsule activator for 2 seconds, mixed for 15 seconds at a speed of 4300 cycles per minute (cpm), and applied to the tooth.
- Fuji Filling LC groups: Fuji Filling LC (FF) was extruded onto a mixing pad, hand mixed for 10 seconds, and applied to the tooth.
- Fuji II LC groups: Fuji II LC capsules (FII) were shaken, then activated per manufacturer's instructions, mixed for 10 seconds at 4300 cpm, and applied to the tooth.

Using a low-speed saw with a diamond blade (Isomet, Buehler, Lake Bluff, IL, USA), each specimen was vertically sectioned into serial slabs and the slabs sectioned further into beams with a cross-sectional area of approximately 0.8×0.8 mm^2. Nine beams were used from each specimen. Each beam was attached to a modified Bencor Multi-T testing apparatus (Danville Engineering Co, Danville, CA, USA) using a cyanoacrylate-based adhesive (Zapit, Dental Ventures of America Inc, Corona, CA, USA) and stressed to failure in tension using a universal testing machine (MTS Sintech Renew 1123, MTS

Systems Corporation, Eden Prairie, MN, USA) at a crosshead speed of 1 mm/min.

Debonded specimens were examined under a stereomicroscope (45×) to evaluate the fracture pattern. Failure modes were classified as adhesive (failure at the dentin-material interface), cohesive (failure within the dentin surface or within the material itself), or mixed (failure partially adhesive and partially cohesive). The dentin sides of representative debonded beams were assessed under a scanning electron microscope (SEM) (JSM-5310LV, Jeol Ltd, Tokyo, Japan) at 20 kV.

Microleakage

For the microleakage test, a standardized Class V cavity preparation was made on the buccal surface of each tooth with a high-speed handpiece using copious water spray and an Alpen No. 56 carbide bur (Coltène/Whaledent Inc., Cuyahoga Falls, OH, USA) changed after every two cavity preparations. The cavity dimensions were 2 mm occluso-gingivally by 3 mm mesiodistally and 2 mm in depth measured using a North Carolina periodontal probe. The preparations were centered on the cementoenamel junction, keeping the occlusal margin on enamel and the gingival margin on cementum-dentin. The cavity preparations were conditioned and restorative materials applied as previously described. The restorations were placed in a single increment and light cured following the manufacturer's instructions (refer to the procedures as described in the Microtensile Bond Strength Testing section). Immediately after curing, the restorations were contoured and polished using conventional finishing and polishing instruments (eg, Sof-Lex Finishing and Polishing System, No. 15 surgical blade) under moist conditions. Care was taken to prevent desiccation of the restoration surface.

The restored teeth were stored in 100% humidity at 37°C ± 2°C for 24 hours, then thermocycled for 500 cycles between water baths at 8°C and 48°C with a dwell time of 30 seconds and a transfer time of 10 seconds. Next, the root apex of each tooth was sealed using Loctite Super glue (Henkel Consumer Adhesives, Inc., Avon, OH, USA) and the teeth were coated with NYC long-wearing nail enamel (Coty US LLC, New York, NY, USA) to within 2 mm of the restoration margins. The teeth were then immersed in 2% methylene blue and stored at room temperature for 24 hours. After immersion, the teeth were washed with running tap water for 30 seconds and embedded in acrylic resin and sectioned with a diamond saw with water cooling (Isomet, Buehler). A 1-mm-thick longitudinal section was taken from the center of each restoration. The occlusal and gingival margins of each section were examined with a stereomicroscope (10×) to determine the microleakage. Both sides of the specimen section were examined at the occlusal and gingival margins, making a total of two occlusal and two gingival microleakage scores for each section. The highest occlusal and the highest gingival scores were used as the microleakage scores for that specimen. The following scoring system was used: 0, no leakage (no dye penetration); 1, dye penetration up to the middle half of the occlusal or cervical cavity wall; 2, dye penetration beyond the middle half of the occlusal or cervical cavity wall but not to the axial wall; and 3, penetration including the axial wall.

Statistical Analysis

Comparisons between the groups for differences in microtensile peak stress were performed using a Weibull distribution survival analysis, using the stress required for failure in place of the usual "time to event" seen in typical survival analyses. The analysis included a "frailty" term to correlate the measurements from beams fabricated from the same tooth.

Microleakage was summarized by pretreatment/material combination for occlusal and cervical surfaces. Mantel-Haenszel chi-square tests were used to compare the eight groups for differences in cervical and occlusal microleakage scores. Cochran-Mantel-Haenszel chi-square tests were used to compare the cervical and occlusal locations within each group. A multiple comparisons adjustment was used to control the overall significance level at 5% within each set of tests.[18]

RESULTS

The mean microtensile bond strength values with standard error (SE) for each group are shown in Table 2. Weibull distribution survival analysis was used to compare the differences in microtensile peak stress between the groups. Figure 1A shows the survival functions using individual observations, whereas Figure 1B shows the survival curves fitted by the Weibull models. In Figure 1B, the y-axis shows a survival probability of failure from 1 to 0, where 1 represents no failures and 0 represents total failure of all the specimens.

The mean microtensile bond strength for the Photac Fil groups (KNP-PF and KC-PF) was

Table 2: *Microtensile Bond Strength Means (MPa) and Statistical Parameters* (σ_0 and m) Obtained From the Weibull Distribution of the Initial Bond Strength

Groups	Teeth, n	Beams, n	Min	Max	Mean (SE)	Weibull Characteristic Strength, σ_0	Weibull Modulus (m)
KNP-KN	10	84	0.2	52.9	14.9 (1.6) D	16.4	1.9
KC-KN	10	89	3.1	40.6	17.2 (1.5) C,D	19.3	2.3
KNP-PF	12	106	2.2	65.8	31.2 (1.6) A	34.7	3.1
KC-PF	11	107	7.6	54.6	26.2 (1.5) A,B	29.3	3.0
SC-FF	12	98	2.6	44.9	23.6 (1.5) A,B,C	26.3	3.1
SC-FII	12	107	3.3	69.8	31.2 (1.5) A	34.7	3.5
CC-FII	12	99	1.1	56.2	21.9 (1.5) B,C	24.4	2.6

Abbreviations: Max, maximum; Min, minimum.
** No significant difference between groups with same letter (p>0.05).*

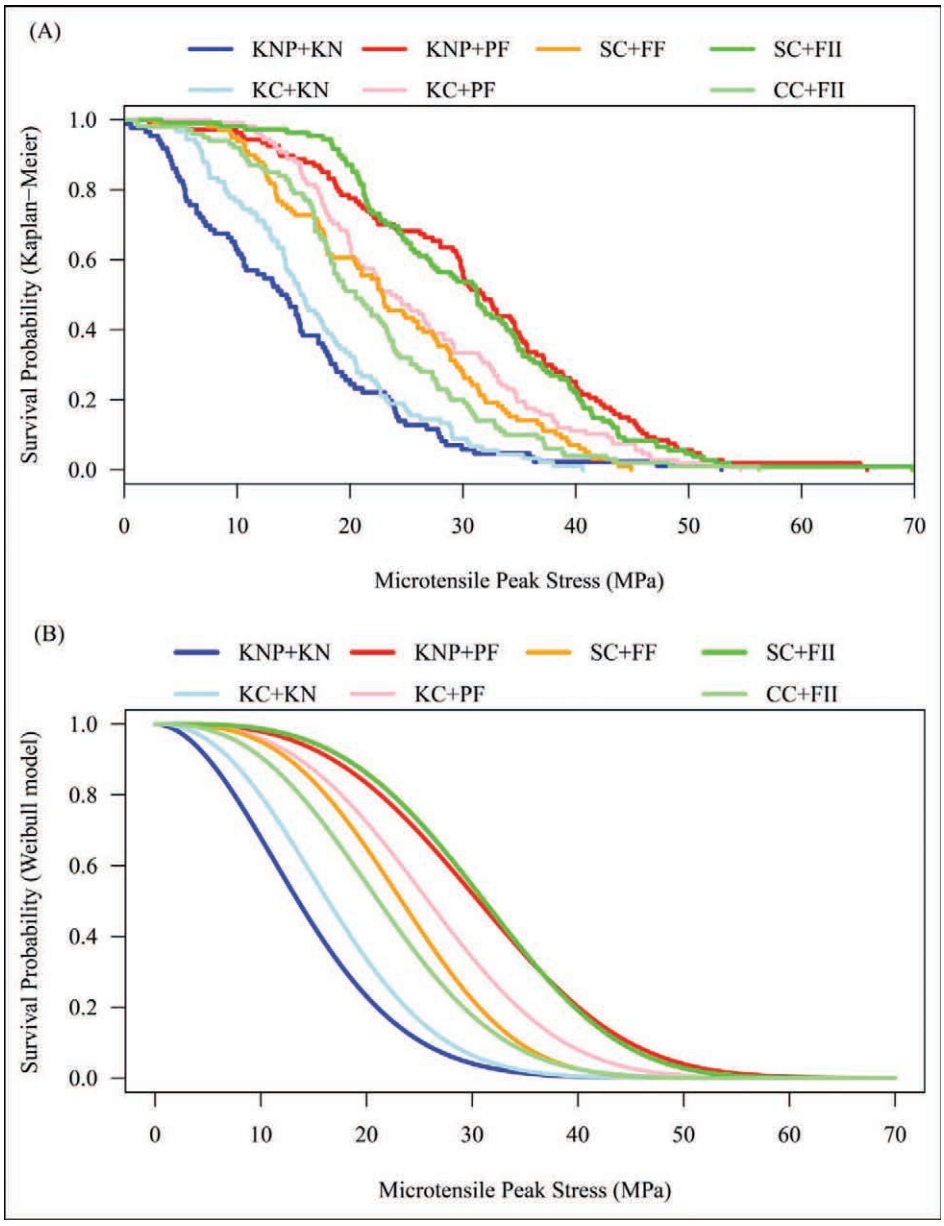

Figure 1. *(A): Survival functions using the individual observations. (B): Survival curves fitted by the Weibull models.*

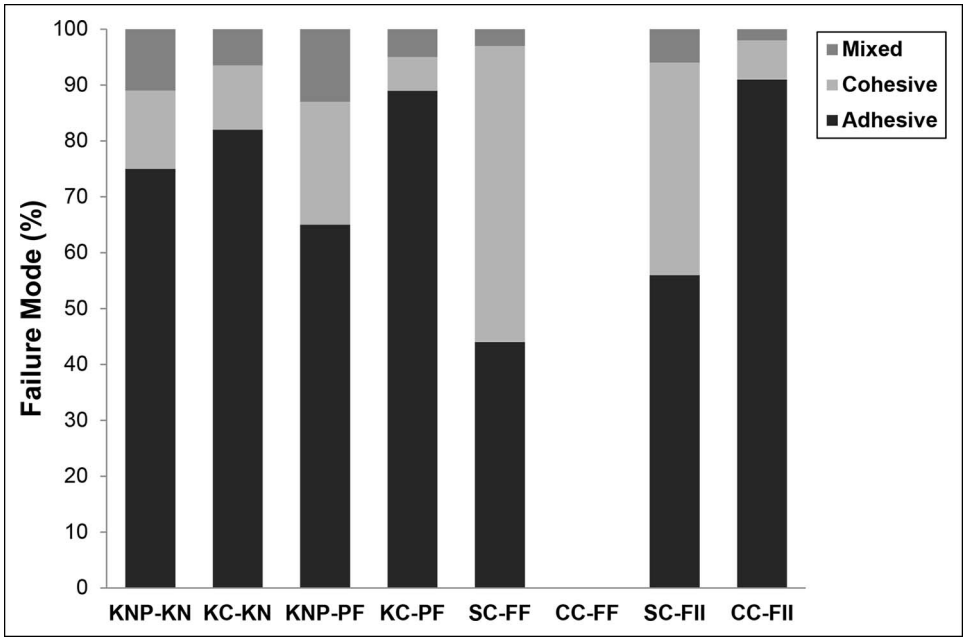

Figure 2. *Failure mode in percentage (%). CC-FF not shown because all bonds failed prior to testing.*

significantly ($p<0.05$) higher than those for the Ketac Nano groups (KNP-KN and KC-KN) when either KNP or KC were used. For GC Cavity Conditioner with Fuji Filling LC (CC-FF), no bond strengths were measured because total bond failure occurred before or during specimen preparation. Fuji II LC showed significantly ($p<0.05$) higher mean microtensile bond strength when SC-FII was used as the dentin surface conditioner compared with CC-FII. Failure mode analyses are shown in Figures 2 and 3A-F. CC-FII had the highest percentage of adhesive failures, whereas SC-FF had the lowest. SEM showed adhesive failures for groups KNP-KN, KC-KN, KNP-PF, KC-PF, and SC-FII. SC-FF and CC-FII also showed cohesive failures.

The results of microleakage testing are presented in Table 3. Cervical locations had significantly more ($p<0.05$) microleakage than occlusal locations for KNP-KN, KC-KN, and SC-FF. For cervical locations, CC-FII showed the least microleakage, which was significantly less ($p<0.05$) than KNP-KN, KC-KN, and SC-FF; KNP-PF and CC-FF had significantly lower microleakage than KNP-KN and KC-KN. For occlusal locations, CC-FF had significantly higher microleakage than KC-PF ($p<0.05$), whereas none of the other comparisons reached statistical significance.

DISCUSSION

The microtensile test was used for bond strength testing due to its several advantages, including a smaller surface area resulting in better stress distribution at the bond interface; less effect of surface flaws; and less cohesive failure. A nontrimmed method was used to minimize the chance of pretest failures during specimen preparation.[19] Except for CC-FF, where all samples failed during specimen preparation, a relatively low number of pretest failures were encountered (five teeth totally debonded and 30 individual beams failed among the various groups). Ketac Nano groups (KNP-KN and KC-KN) had a higher incidence of specimen failures during beam preparation, perhaps an indication of the brittle nature of this material. Neither glue failures nor pretest failures were included in the statistical analysis.

Since being recommended by Powis and others,[7] polyacrylic acid has been widely used as a surface substrate conditioner that improves adhesion of both conventional and resin-modified GIC to tooth structure.[15,16] Overall, in the current study, the use of nonrinse conditioners (ie, KNP and SC) in association with traditional RMGIC (ie, PF and FII) demonstrated greater microtensile bond strengths to dentin when compared with the paste-paste RMGIC. With the exception of CC-FF, KN-KNP demonstrated the lowest mean microtensile bond strength among all groups tested. Coutinho and others[20] had similar findings when comparing Ketac Nano with traditional RMGIC. Furthermore, all specimens failed prior to testing when FF was placed on dentin conditioned with polyacrylic acid, suggesting that a GIC bond is not occurring. However, the

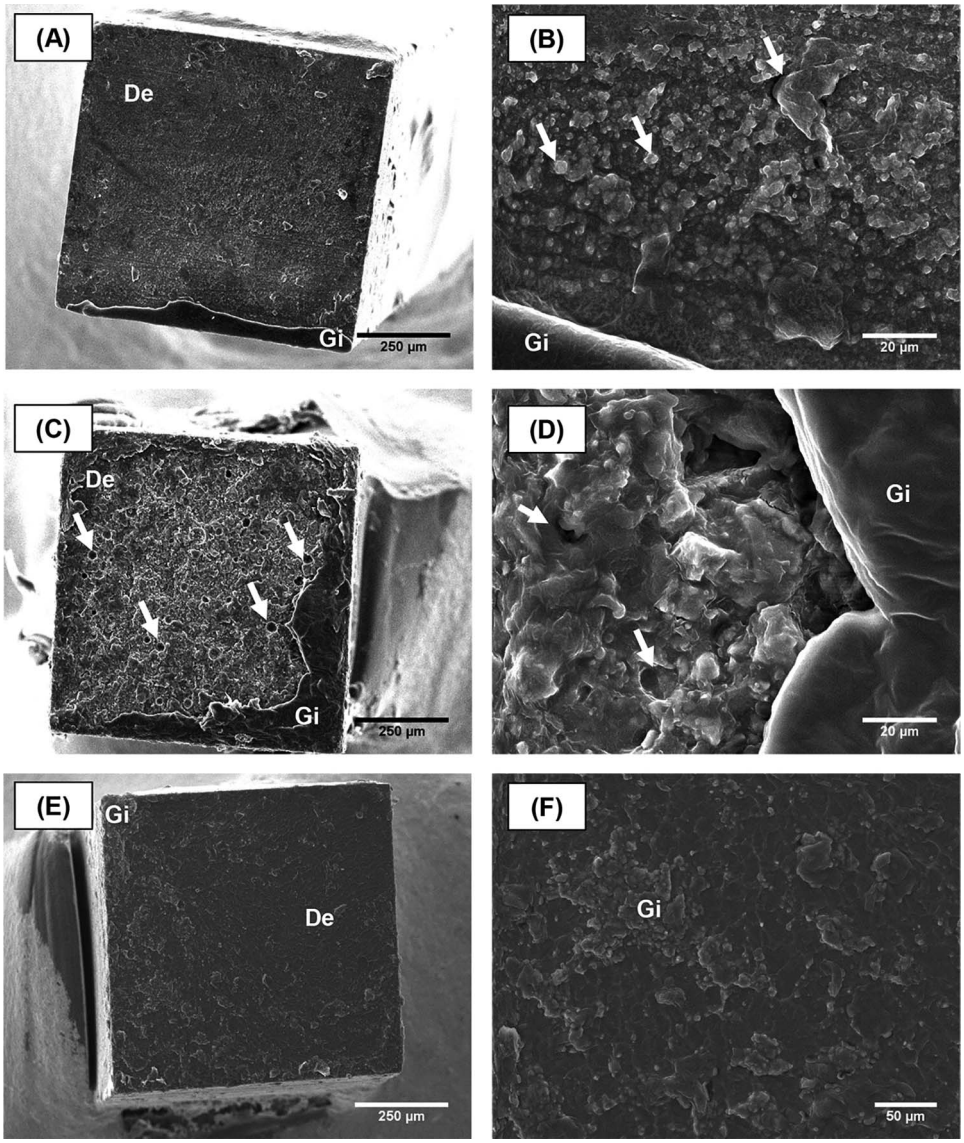

Figure 3. *Representative SEM images of the failure modes: KNP-KN (A-B): (A): SEM images show a mixed type of failure where both dentin (De) and RMGIC (Gi) can be identified on the fractured dentin surface of the beam. (B): Higher magnification SEM image clearly shows interfacial dentin surface covered by remnants of RMGIC and filler particles (arrows) occlude dentinal tubule (arrows). KNP-PF (C-D): Mixed type of failure where both dentin (De) and RMGIC (Gi) can be identified on the fractured dentin surface of the beam. (C): Several air bubbles can be seen (arrows). (D): Higher magnification SEM shows interfacial dentine surface totally covered by remnants of RMGIC (Gi) and air bubbles (arrows) no dentin surface can be identified. SC-FII (E-F): (E): Mixed type of failure where both dentin (De) and RMGIC (Gi) can be identified on the fractured dentin surface of the beam. (F): Higher magnification SEM shows interfacial dentine surface covered by RMGIC remnants (Gi).*

use of SC did enhance the bond strengths of traditional RMGIC (FII), possibly due to hydroxyethyl methacrylate present in the SC, contributing micromechanical resin bonding. The overall number of the pretest failures was very low (five teeth totally debonded and 30 individual beams failed among the various groups). The pretest failures were not included in our statistical analysis. A higher number of pretest failures during beam preparation, where beams failed or the whole restoration broke, especially on the second vertical cut, were associated with the new paste-paste RMGIC (KN). The failures may suggest that this might be related to the ceramic nanofiller content of the material that increases the material brittleness. Brittle materials are considered unsuitable for microtensile bond testing given that higher pretest failure and lower bond values are expected.[21] This may explain the low bonding value for this material when compared with other groups in this study.

SEM analysis was performed on a randomly selected, failed beam for each group. Little agreement was observed for failure-mode analysis between stereomicroscopy and SEM. For beams from groups KNP-KN, KC-KN, KNP-PF, KC-PF, and SC-FII, SEM showed mixed-type failure instead of the adhesive failure viewed with the stereomicroscope. For SC-FF, SEM and stereomicroscopy showed cohesive and adhesive type failure, respectively. The only agreement was with CC-FII, where both methods showed cohesive failure.

Table 3: *Microleakage Scores**

Groups	0, n (%)	1, n (%)	2, n (%)	3, n (%)	Mean
Cervical margin					
KNP-KN A	0 (0)	1 (8)	0 (0)	11 (92)	2.8
KC-KN A	0 (0)	0 (0)	0 (0)	12 (100)	3.0
KNP-PF B,C	6 (50)	2 (17)	0 (0)	4 (33)	1.2
KC-PF A,B,C	6 (50)	0 (0)	0 (0)	6 (50	1.5
SC-FF A,B	1 (8)	0 (0)	0 (0)	11 (92	2.8
CC-FF B,C	1 (9)	7 (64)	0 (0)	3 (27)	1.5
SC-FII A,B,C	2 (17)	1 (8)	0 (0)	9 (75)	2.3
CC-FII C	5 (45)	5 (45)	0 (0)	1 (9)	0.7
Occlusal margin					
KNP-KN A,B	3 (25)	7 (58)	0 (0)	2 (17)	1.1
KC-KN A,B	1 (8)	8 (67)	0 (0)	3 (25)	1.4
KNP-PF A,B	6 (50)	4 (33)	0 (0)	2 (17)	0.8
KC-PF A	9 (75)	3 (25)	0 (0)	0 (0)	0.3
SC-FF A,B	8 (67)	1 (8)	0 (0)	3 (25)	0.8
CC-FF B	0 (0)	9 (82)	1 (9)	1 (9)	1.3
SC-FII A,B	5 (42)	4 (42)	2 (17)	0 (0)	0.8
CC-FII A,B	3 (27)	8 (73)	0 (0)	0 (0)	0.7

** Same small-cap letters indicate statistically similar groups ($p > 0.05$) within each margin.*

In addition to bond strength, interfacial microleakage must be tested because higher bond strengths do not necessarily translate to decreased leakage. Previous studies[4] with RMGIC have shown acceptable sealing of the restoration-tooth interface. However, there is a lack of information about the sealing ability of these relatively new paste-paste RMGIC systems using nonrinse conditioners. To determine the degree of microleakage, the current study used the penetration of methylene blue dye commonly used in microleakage studies.[22-24] Here, both group and location effects were significant. As expected, less microleakage occurred at the occlusal margins where enamel is the bonding substrate. All groups performed well at the occlusal margins with the only difference being between CC-FF and KC-PF. At the cervical margins (cementum-dentin substrate), CC-FII showed the lowest microleakage scores, though not significantly different from groups KNP-PF, SC-FII, KC-PF, or CC-FF. With the exception of KNP-PF, groups conditioned with a nonrinse conditioner revealed significantly (KNP-KN, SC-FF) or marginally (SC-FII, $p=0.052$) more microleakage at the cervical margin. Except for KC-KN, when the dentin was treated with a traditional polyacrylic acid conditioner, significantly less microleakage was observed.

One might suggest that incorporation of higher amounts of resin in RMGIC can improve the physical-mechanical properties as well as bond strength; however, this can also lead to greater microleakage due to possible increases in polymerization shrinkage or differences in the coefficient of thermal expansion. Nonetheless, the quality of the bond achieved with these new conditioners is crucial in predicting the clinical longevity of these restorations. Therefore, clinical studies are needed to evaluate both short- and long-term outcomes of these newer RMGIC restorative materials.

CONCLUSION

Within the limitations of this study, the use of nonrinse conditioners in association with traditional RMGICs demonstrated superior microtensile bond strengths to dentin when compared with the paste-paste RMGICs. Meanwhile, the association between polyacrylic acid (CC) and a traditional RMGIC (FII) led to the least microleakage for cervical locations when compared with all other groups.

Acknowledgement

This study was partially supported by a grant from GC America. One of the co-authors (SAF) thanks CAPES (Grant#18922-12-0, Brazil) for the scholarship received.

Human Subject Statement

This study was conducted in accordance with all the provisions of the human subject oversight committee guidelines and policies at Indiana University School of Dentistry. The approval code for this study was 1106006167. This study was conducted at Indiana University School of Dentistry.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 21 July 2014)

REFERENCES

1. Wilson AD, & Kent BE (1972) A new translucent cement for dentistry. The glass ionomer cement *British Dental Journal* **132(4)** 133-135.
2. Mathis RS, & Ferracane JL (1989) Properties of a glass-ionomer/resin-composite hybrid material *Dental Materials* **5(5)** 355-358.
3. McCabe JF (1998) Resin-modified glass-ionomers *Biomaterials* **19(6)** 521-527.
4. Sidhu SK (2010) Clinical evaluations of resin-modified glass-ionomer restorations *Dental Materials* **26(1)** 7-12.

5. Hickel R, Dasch W, Janda R, Tyas M, & Anusavice K (1998) New direct restorative materials. FDI Commission Project *International Dental Journal* **48(1)** 3-16.
6. Tyas MJ, & Burrow MF (2004). Adhesive restorative materials: A review *Australian Dental Journal* **49(3)** 112-121.
7. Powis DR, Folleras T, Merson SA, & Wilson AD (1982) Improved adhesion of a glass ionomer cement to dentin and enamel *Journal of Dental Research* **61(12)** 1416-1422.
8. Tanumiharja M, Burrow MF, & Tyas MJ (2000) Microtensile bond strengths of glass ionomer (polyalkenoate) cements to dentine using four conditioners *Journal of Dentistry* **28(5)** 361-366.
9. 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.
10. Fritz UB, Finger WJ, & Uno S (1996) Resin-modified glass ionomer cements: Bonding to enamel and dentin *Dental Materials* **12(3)** 161-166.
11. Friedl KH, Powers JM, & Hiller KA (1995) Influence of different factors on bond strength of hybrid ionomers *Operative Dentistry* **20(2)** 74-80.
12. Xie H, Zhang F, Wu Y, Chen C, & Liu W (2008) Dentine bond strength and microleakage of flowable composite, compomer and glass ionomer cement *Australian Dental Journal* **53(4)** 325-331.
13. Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, & Van Meerbeek B (2005) Clinical effectiveness of contemporary adhesives: A systematic review of current clinical trials *Dental Materials* **21(9)** 864-881.
14. Matis BA, Cochran M, & Carlson T (1996) Longevity of glass-ionomer restorative materials: Results of a 10-year evaluation *Quintessence International* **27(6)** 373-382.
15. Loguercio AD, Reis A, Barbosa AN, & Roulet JF (2003) Five-year double-blind randomized clinical evaluation of a resin-modified glass ionomer and a polyacid-modified resin in noncarious cervical lesions *Journal of Adhesive Dentistry* **5(4)** 323-332.
16. Franco EB, Benetti AR, Ishikiriama SK, Santiago SL, Lauris JR, Jorge MF, & Navarro MF (2006) 5-year clinical performance of resin composite versus resin modified glass ionomer restorative system in non-carious cervical lesions *Operative Dentistry* **31(4)** 403-408.
17. Heintze SD, & Roulet JF (2010) Glass ionomer derivates have better retention rates in cervical restorations compared to self-etching adhesive systems *Journal of Evidence Based Dentistry Practice* **10(1)** 18-20.
18. Hommel G (1988) A comparison of two modified Bonferroni procedures *Biometrika* **75** 383-386.
19. Armstrong S, Geraldeli S, Maia R, Raposo LH, Soares CJ, & Yamagawa J (2010) Adhesion to tooth structure: A critical review of "micro" bond strength test methods *Dental Materials* **26(2)** e50-e62.
20. Coutinho E, Cardoso MV, De Munck J, Neves AA, Van Landuyt KL, Peumans M, Lambrechts P, & Van Meerbeek B (2009) Bonding effectiveness and interfacial characterization of a nano-filled resin-modified glass ionomer *Dental Materials* **25(11)** 1347-1357.
21. Sadek FT, Monticelli F, Muench A, Ferrari M, & Cardoso PE (2006) A novel method to obtain microtensile specimens minimizing cut flaws. *Journal of Biomedical Materials Research B Applied Biomaterials* **78(1)** 7-14.
22. Lott J, Fitchie J, Creasy M, & Pucket AJ (2007). Microleakage of three conventional glass ionomers using 45Ca and methylene blue *General Dentistry* **55(1)** 15-18.
23. Loguercio AD, Alessandra R, Mazzocco KC, Dias AL, Busato AL, Singer Jda M, & Rosa P (2002) Microleakage in class II composite resin restorations: Total bonding and open sandwich technique *Journal of Adhesive Dentistry* **4(2)** 137-144.
24. Abd El Halim S, & Zaki D (2011) Comparative evaluation of microleakage among three different glass ionomer types *Operative Dentistry* **36(1)** 36-42.

©Operative Dentistry, 2015, **40-3,** 313-321

The Effect of Combining Radiographs and DIAGNOdent With Visual Examination on Detection and Treatment Decisions of Noncavitated Occluso-dentinal Caries

QD Alomari • M Qudiemat • ME Khalaf
Y Al-Tarakemah

Clinical Relevance

Critical visual examination is enough to detect and decide on treatment of noncavitated occluso-dentinal caries.

SUMMARY

The aim of this laboratory study was to evaluate the effectiveness of incorporating radiographic examination and DIAGNOdent with visual examination for the detection and treatment of noncavitated occluso-dentinal caries. Four examiners examined the occlusal surfaces of 160 extracted posterior teeth. Teeth were examined three times with a one-month interval in between. The first examination was visual (V), the second examination was visual with radiograph (VR), and the third examination was visual with radiograph and DIAGNOdent (VRD). Examiners were asked to detect the presence of caries (if any) and identify the extent of caries (if present; ie, enamel or dentin). The examiners were also asked to choose a treatment for each tooth. The examined teeth were later sectioned, and the presence of caries was charted as 0 = no caries, 1 = caries confined to enamel, 2 = caries in the outer dentin, and 3 = caries in the inner dentin. Sensitivity, specificity, area under the curve (Az values), rank correlation

*Qasem D Alomari, BDS, MS, ABOD, Kuwait University, Faculty of Dentistry, Department of Restorative Sciences, Kuwait

Muawia Qudiemat, BDS, MSc, MDentSci, Kuwait University, Faculty of Dentistry, Department of Developmental and Preventive Sciences, Kuwait.

Mai E Khalaf, DDS, AEGD, MA, Kuwait University, Faculty of Dentistry, Department of General Dental Practice, Kuwait.

Yacoub Al-Tarakemah, DDS, MS, ABP, Kuwait University, Faculty of Dentistry, Department of Restorative Sciences, Kuwait.

*Corresponding author: B.O. Box 24923, Alsafat, 13110 Kuwait; e-mail: qalomari@hsc.edu.kw

DOI: 10.2341/14-138-L

coefficient, interexaminer reproducibility, and McNemar χ^2 tests were calculated. VR had statistically higher specificity and lower sensitivity than V and VRD. The means of Az values ranged from 0.58 to 0.63, with no statistical difference between the three examinations ($p>0.05$). The means of the rank correlation coefficients with histology for detection of dentinal caries were 0.48, 0.23, and 0.44 using V, VR, and VRD, respectively. Interexaminer reproducibility was best for V alone. The percentages of teeth indicated for restorative treatment were 31%, 33%, and 41% using V, VR, and VRD, respectively. The percentages of teeth correctly treated according to histology were 69.4%, 70.0%, and 67.5% using V, VR, and VRD, respectively. There was no difference in the accuracy of treatment decisions between the three examination points ($p>0.05$). The addition of radiographs and DIAGNOdent to visual examination did not have a significant effect on the improvement of the detection or treatment decisions of examiners of noncavitated occluso-dentinal carious lesions.

INTRODUCTION

Despite decreasing caries prevalence worldwide, occlusal caries remain the predominant form of dental caries at the present time.[1] This has been mainly attributed to the anatomy of the fissure system and the difficulty of plaque removal in this area, especially during the stage of tooth eruption.[2,3]

Thirty-four percent of all dental patients have been found to have questionable occlusal caries that are difficult to diagnose.[4] The early stages of occlusal caries are difficult to detect clinically, but later occlusal caries might also present as "questionable caries" where there is no frank cavitation but the surface is rough with opacities or staining.[5] Histologically, these lesions might be confined to enamel or might have spread into dentin.[6]

To improve the frequency of detection of occlusal caries lesions, combining visual examination with other diagnostic aids has been recommended.[7-10]

Although radiographic examination has limited benefits when it comes to initial or early occlusal carious lesions, it is the most widely used technique for improving caries detection and the only technique available in most dental clinics.[7,11] It has been shown that only one-third of occlusal dentinal lesions were detected clinically, while two-thirds were found clinically on bitewing radiographs as hidden caries.[12] In addition, the presence of dentinal lesions on conventional bitewing radiographs has been found to be significantly associated with infected dentin, which is soft and wet clinically.[13]

A laser-stimulated fluorescence caries detection system (DIAGNOdent) has been introduced in an attempt to quantify carious lesions and better improve clinicians' diagnostic abilities.[14] Clinically, DIAGNOdent has shown higher sensitivity and lower specificity at the D3 threshold (dentinal caries) compared with visual inspection and radiographs.[15] As a result, DIAGNOdent has been recommended for use only as an adjunct to visual examination since it has been reported to yield more false-positive results than visual and radiographic methods.[15,16]

Caries detection has a tremendous impact on treatment decisions and is directly linked to the patient's dental management and treatment outcomes.[17,18] Treatment and management of carious lesions should ideally be based on severity and activity.[6,19] Unfortunately, recent studies have shown that initial carious lesions are still managed restoratively.[19,20]

Considering the fact that previous studies have focused on the accuracy of radiographs and DIAGNOdent on detecting occlusal carious lesions separately, the aims of this study were 1) to investigate whether combining radiographs and DIAGNOdent with visual examination would improve the detection of noncavitated occlusal caries, 2) to investigate whether combining radiographs and DIAGNOdent with visual examination would influence the treatment decisions for noncavitated occlusal carious lesions, and 3) to investigate the influence of combining radiographs and DIAGNOdent with visual examination on interexaminer reproducibility for the detection of noncavitated occluso-dentinal caries.

METHODS AND MATERIALS

Teeth

After preliminary visual examination, 160 posterior teeth (premolars and molars) were selected. The included teeth were free of restorations and had no proximal caries. Teeth with dental fluorosis, tetracycline staining, and hypoplasia were also excluded. The clinical appearance of the occlusal surface of each tooth ranged from sound to discolored, with white/brown discoloration and no cavitation. The teeth were cleaned with a prophylaxis brush using pumice slurry and rinsed thoroughly with a three-way syringe and then stored in water. Later, each

tooth was embedded in an acrylic block and was randomly assigned a number from 1 to 160.

Examiners

Four examiners participated in the study. The examiners were university faculty with the following specialties: general dentistry, operative dentistry, pediatric dentistry, and prosthetic dentistry. The following clinical scenario was given to the examiners: "A healthy 18 year-old patient presents to your clinic for her/his scheduled dental appointment. Complete your clinical examination for the displayed tooth. How would you manage this tooth if the patient is at high risk for developing caries? The cost of treatment should not be a factor in your decision." A pilot study of 25 teeth (not included in the study) was carried out. Teeth were examined three times with a one-month separation period. The first examination was visual only, the second was visual with radiographs, and the third was a combination of visual, radiographs, and DIAGNOdent as described below. In each of the examination sessions, the teeth were presented randomly to the examiners.

During each examination, participants were asked to decide on the diagnosis of the tooth using the following criteria: 0 = the tooth is sound, 1 = the tooth is carious but the caries is confined to enamel, 2 = the tooth is carious and the caries is extending into dentin. The examiners were then asked to choose one of the following treatment options for the occlusal surface of the tooth: 0 = no treatment, 1 = noninvasive treatment (fluoride application or fissure sealant), 2 = preventive resin restoration (conservative cavity preparation to remove the caries, followed by resin composite restoration of the cavity and fissure sealant of the remaining fissures), 3 = resin composite/amalgam restoration.

Visual Examination

Visual examination was carried out using a dental operating light, a blunt-end periodontal probe, and a 3-in-1 syringe. No explorer was used during the examination. The examination was done by all examiners in the same setting and at the same time. The examination time was almost two hours for all of the teeth. Air drying for each tooth was allowed for up to five seconds.

Visual and Radiographic Examination

After one month from the first examination, the examiners were presented with the teeth and their corresponding radiographs and were asked to propose a diagnosis and management for each tooth using the previously mentioned criteria. The radiographs were examined on a backlit screen with no magnification. Conventional radiographic imaging was obtained for each tooth separately using the paralleling technique, with Kodak Insight film of 30.5 mm × 40.5 mm (Kodak, Rochester, MD, USA) using a Planmeca ProX Intraoral X-ray Generator Unit (Planmeca Oy, Helsinki, Finland), which was operated at 63 kVp and 8 mA and an exposure time of 0.10 seconds. The film was then developed in a Dürr Dental XR 24 Pro automatic developing machine (Dürr Dental AG, Bietigheim-Bissingen, Germany), with a pass time of seven minutes.

Visual, Radiographic, and Laser Fluorescence Examination

The third examination was conducted a month after the completion of the second examination. In this case, the teeth were presented to the examiners with their corresponding radiographs and a laser fluorescence device (DIAGNOdent, Kavo, Biberach, Germany). The devices were initially calibrated before use against the ceramic reference as recommended by the manufacturer. Each tooth was then dried for five seconds, and the probe tip designated for occlusal surface was adjusted to each tooth separately by holding the tip against a sound smooth surface and pressing the ring cuff until calibration was complete. The probe tip was positioned over the occlusal fissure without applying pressure and pivoted in all directions. The tooth was scored based on the highest reading obtained. Each examiner was provided with the manufacturer's instructions on how to use the pen and the interpretation of the resulting reading. Using the information from the visual examination, the radiograph, and the DIAGNOdent, the examiners were asked to choose a diagnosis for the occlusal surface of the tooth and to choose a management option following the previously mentioned criteria.

Histological Validation

The teeth were sectioned through a predetermined point of interest using a water-cooled 0.3-mm diamond saw at low speed (Isomet low speed saw; Buehler, Lake Bluff, IL, USA). Teeth with more than one point of interest were sectioned more than once. Computerized images were obtained for each section with a digital camera (Leica DC200; Leica Microsystems Wetzlar, GmbH, Wetzlar, Germany) linked to a stereomicroscope (Leica MZ6, Leica Microsystems Wetzlar, GmbH), using a fixed distance

Table 1: *Histological Classification of the Teeth Based on Downer's Criteria*

Score	Criteria	Number	Percentage
0	Sound	17	10.6
1	Caries in the outer enamel	51	31.9
2	Caries in the inner enamel	31	19.4
3	Caries in the outer dentin	55	34.4
4	Caries in the inner dentin	06	3.8
Total		160	100

and the same magnification (10×). The microscope was linked to a computer with a digital image analysis system (IM 500; Leica Microsystems Wetzlar, GmbH). Two examiners (the pediatric and restorative dentists) assessed the histological images using the following four-point scale (Downer): 0 = sound, 1 = caries in the outer half of the enamel, 2 = caries in the inner half of the enamel reaching up to the enamel-dentinal junction, 3 = caries in the outer half of the dentin, and 4 = caries in the inner half of the dentin.[21] The highest score from the various sections was used to define the caries status of the surface. Calibration of both examiners was undertaken by reading 25 pilot samples. In cases in which the two investigators' ratings diverged, a joint assessment was performed until consensus was reached.

Statistical Analysis

The data were analyzed statistically with SPSS software, version 17.0 (SPSS Inc, Chicago, IL, USA). Descriptive exploratory analysis was done. The histological diagnosis for each tooth was dichotomized into two categories: sound and caries limited to enamel as one group and caries extending into dentin (D3) as a second group. Sensitivity, specificity, and the area under the receiver operating characteristics curve were also calculated and reported as Az values for the three examinations and for the four observers using the histological criteria at D3 (caries into dentin). The strength of the association between each examination and the histology scores was assessed using a rank correlation coefficient (Kendall's tau b). Comparisons among the three examination points were conducted using the McNemar χ^2 test. The quality of the interexaminer repeatability was calculated using Cohen's weighted kappa test.[22] With regard to treatment decisions, only caries extending into dentin (D3) were considered in need of operative treatment. The treatment options were also regrouped into preventive (no treatment, fluoride application, or fissure sealant) and restorative (preventive resin restoration or class I cavity preparation and restoration). The level of significance was set at α=0.05.

RESULTS

Histological examination of the 160 teeth revealed that 61 teeth had caries extending into dentin, while 99 teeth were either sound or had caries confined to enamel (Table 1). The McNemar χ^2 test showed that there was a statistically significant difference between the three examination points and for the four examiners (except for the pediatric dentist between visual alone and visual combined with radiograph and DIAGNOdent). Table 2 presents the sensitivity, specificity, Az values, and the correlation coefficients for the visual examination alone, visual examination with radiographs, and visual examination with radiographs and DIAGNOdent for the four examiners for the detection of occlusal caries at the D3 diagnostic threshold. There was no statistical difference between the mean sensitivity of visual examination alone and visual examination combined with both radiograph and DIAGNOdent ($p>0.05$). The mean sensitivity of visual examination with radiograph was significantly lower than the mean sensitivity of both visual examination alone and visual examination with radiograph and DIAGNOdent ($p<0.05$). The mean specificity of visual examination with radiographs was significantly higher than that of visual examination alone or visual examination combined with radiographs and DIAGNOdent ($p<0.05$). There was no statistically significant difference between the Az values of the three examination points ($p>0.05$). However, the rank correlation with histology was lowest for visual examination combined with radiographs.

Table 3 presents the weighted Kappa values of interexaminer reproducibility for each examination point. For visual examination alone, Kappa values ranged from moderate to substantial (0.44-0.80), while for visual with radiographs and visual with radiographs and DIAGNOdent, the ranges were from fair to moderate (0.26-0.44 and 0.30-0.62, respectively).[22]

Table 4 shows the number (%) and type of treatments indicated by each examiner for management of occlusal surfaces, based on visual examination alone, visual examination with radiographs, or visual examination combined with radiographs and DIAGNOdent. The addition of radiographs to visual examination resulted in a decrease in the

Table 2: *Examiners' Performance in Detecting Occluso-dentinal Caries (D3) by Visual Examination (V), Visual Examination and Radiographs (VR), and by a Combination of Visual Examination, Radiographs, and DIAGNOdent (VRD), Presented as Sensitivity, Specificity, Area Under the Receiver Operating Characteristic Curve, and Rank Correlation Coefficient*

Method (n=160)	Sensitivity	Specificity	Az	Spearman Rank Correlation Coefficient
V				
General dentist	0.75	0.69	0.72	0.55
Restorative dentist	0.92	0.31	0.61	0.41
Prosthodontist	0.68	0.64	0.66	0.40
Pediatric dentist	0.91	0.30	0.60	0.57
Mean	0.82	0.49	0.65	0.48
VR				
General dentist	0.20	0.91	0.55	0.26
Restorative dentist	0.56	0.72	0.62	0.32
Prosthodontist	0.27	0.88	0.57	0.18
Pediatric dentist	0.56	0.51	0.53	0.15
Mean	0.40	0.76	0.57	0.23
VRD				
General dentist	0.78	0.50	0.64	0.51
Restorative dentist	0.90	0.30	0.60	0.40
Prosthodontist	0.82	0.45	0.64	0.36
Pediatric dentist	0.93	0.30	0.60	0.49
Mean	0.86	0.39	0.58	0.44

number of teeth indicated for restoration for the general dentist ($p<0.001$) and an increase in the same for the restorative dentist ($p<0.001$). There was no effect of adding the radiographs on the number of teeth indicated for restoration for the prosthodontist and the pediatric dentist ($p>0.05$). There was an increase in the number of teeth indicated for restoration when visual examination was combined with radiographs and DIAGNOdent for two of the examiners (general and restorative dentists; $p<0.05$), while there was no effect for the other two examiners (prosthodontist and pediatric dentist; $p>0.05$). The table also shows considerable variability among the four examiners in their treatment decisions regarding noncavitated occlusal caries.

Table 3: *Kappa Values of Interexaminer Reproducibility for Each Examination Point*

	V	VR	VRD
Restorative, general dentist	0.56	0.30	0.32
Restorative, prosthodontist	0.50	0.34	0.30
Restorative, pediatric dentist	0.80	0.44	0.37
General dentist, prosthodontist	0.60	0.26	0.55
General dentist, pediatric dentist	0.54	0.29	0.62
Prosthodontist, pediatric dentist	0.44	0.30	0.44
Mean	0.57	0.32	0.43

Abbreviations: V, visual; VR, visual with radiograph; VRD, visual with radiograph and DIAGNOdent.

Table 5 shows the relationship between the presence of caries, confirmed by the histological examination of the teeth, and the treatment decisions based on visual examination alone, visual examination with radiographs, and visual examination combined with both radiographs and DIAGNOdent. The number of correct treatment decisions was not different between the three examination points and for the four examiners ($p>0.05$). There was an increase in the number of overtreatment decisions when the visual examination was combined with both radiographs and DIAGNOdent for two of the examiners (general and restorative dentists; $p<0.05$).

DISCUSSION

In this study, we tested the hypothesis that the addition of radiographs and DIAGNOdent to visual examination will improve the detection and treatment decisions of noncavitated occlusal dentinal caries. The accuracy of detection and treatment of noncavitated occlusal dentinal caries was not improved by the addition of radiographs alone or the addition of radiographs with DIAGNOdent to the visual examination. Clinical visual examination can be used alone for the detection of noncavitated occlusal dentinal caries.

The study attempted to replicate a clinical setting: examiners were asked to use only visual examina-

Table 4: *Number (%) and Type of Treatment (Dichotomized Into Preventive and Restorative) Indicated by Each Examiner for Occlusal Surfaces, Based on Visual Examination (V), Visual Examination With Radiograph (VR), and Visual Examination With Radiograph and DIAGNOdent (VRD)*

Examiner	V		VR		VRD	
	Preventive	Restorative	Preventive	Restorative	Preventive	Restorative
General dentist	87 (54.4)	73 (45.6)	114 (71.2)	46 (28.8)	71 (44.4)	89 (55.6)
Restorative dentist	142 (88.8)	18 (11.2)	109 (68.1)	51 (31.9)	94 (58.8)	66 (41.2)
Prosthodontist	136 (85.0)	24 (15.0)	136 (85.0)	24 (15.0)	132 (82.5)	28 (17.5)
Pediatric dentist	80 (50.0)	80 (50.0)	69 (43.1)	91 (56.9)	80 (50.0)	80 (50.0)
Mean	111 (69.0)	49 (31.0)	107 (67.0)	53 (33.0)	94 (59.0)	66 (41.0)

tion in the first examination and to use both clinical examination and radiograph in the second examination. In the third examination, the examiners were requested to combine the visual examination with radiographs and DIAGNOdent readings; the three techniques were not used separately. Each of the four examiners was given the tooth, its radiograph, and a DIAGNOdent machine and was asked to use his or her clinical judgment to reach a diagnosis and treatment for the tooth. Previously, it has been recommended to consider the overall risk profile of the patient and site before using the cutoff points of the DIAGNOdent as the main source of information.[23] Deciding on the type of restorative treatment for a specific lesion depends on a range of variables that include patient history, fluoride and dietary status, caries risk assessment, and the status of the tooth surface. In trying to eliminate the confounding effect of these factors on the decision of the investigators, a specific clinical scenario was created with a healthy patient who is at a high risk for caries and to whom the cost of the treatment was not applicable.

Four examiners with different specialties were the participants in this study. Previously, it has been found that some specialties tend to be more aggressive when treating occlusal caries than others.[20] The results of this study also showed that the specialists of general and operative dentistry tended to overtreat occlusal caries more than the other two specialties (pediatrics and prosthodontics).

The sensitivity of visual examination alone was high, and the specificity was moderate. The area under the curve for visual examination alone was higher than the other two methods, and the rank correlation with histology was also higher than the other two methods. Previous studies have reported moderate sensitivity and substantial specificity for visual examination of dentinal occlusal caries.[8,24-26] Differences between studies can be due to several factors, such as the degree of caries in the included teeth and the experience of the examiners. In this study, the four examiners are university teachers from different specialties, and all had received training on the International Caries Detection and Assessment System.

Sensitivity values of visual examination with radiographs in the study were lower than values for visual examination alone or visual examination combined with radiographs and DIAGNOdent. This is in agreement with what has been reported in the literature.[24,27,28] Radiographs are two-dimensional images of a three-dimensional tooth, making the detection of early lesions difficult since they can be overlapped with sound tooth structure. In agreement with what has been reported previously in the literature, radiographs with visual examination had higher specificity than visual examination alone or visual examination combined with both radio-

Table 5: *Relationship Between the Presence of Dentin Caries and Treatment Decisions Based on Visual Examination Alone (V), Visual Examination With Radiographs (VR), and Visual Examination With Radiographs and DIAGNOdent (VRD)*

Examiner	V			VR			VRD		
	Under	Correct	Over	Under	Correct	Over	Under	Correct	Over
General dentist	15 (9.4)	118 (73.8)	27 (16.9)	30 (18.8)	115 (71.9)	15 (9.3)	15 (9.3)	102 (63.8)	43 (26.9)
Restorative dentist	47 (29.4)	109 (68.1)	4 (2.5)	28 (17.5)	114 (71.2)	18 (11.3)	21 (13.1)	113 (70.6)	26 (16.3)
Prosthodontist	48 (30)	103 (63.1)	11 (6.9)	44 (27.5)	109 (68.1)	7 (4.4)	45 (28.1)	103 (63.1)	12 (7.5)
Pediatric dentist	14 (8.8)	113 (70.6)	33 (20.6)	10 (6.2)	110 (68.8)	40 (25.0)	14 (8.8)	113 (70.6)	33 (20.6)
Mean	31 (19.4)	111 (69.4)	18 (11.2)	28 (17.5)	112 (70.0)	20 (12.5)	24 (15.0)	108 (67.5)	28 (17.5)

graphs and DIAGNOdent.[25,27] The mean rank correlation coefficient of the visual examination with radiographs was 0.23, which was lower than the same for both the visual examination alone (0.48) or visual examination combined with radiographs and DIAGNOdent (0.44). A study by Toraman and others[29] reported similar findings, while other studies reported higher values.[24,27] Variability between studies might be attributed to differences in the extension of the carious lesions included. In this study, we had only 3.8% of our sample with caries in the inner dentin, while the remaining teeth were either sound, containing caries confined into enamel, or caries confined to the outer third of dentin. In addition, previous studies have reported that the DIAGNOdent can have a wide range of inter- and intraexaminer variability.[10]

In a review on the performance of DIAGNOdent in the detection of dentinal caries, it has been observed that there is wide variability in the literature in the reported sensitivity and specificity of this machine.[14] In general, the DIAGNOdent usually exhibits higher sensitivity and lower specificity values than visual assessment. In our study, the same trend was observed, but the difference was not statistically significant between visual assessment alone and visual assessment combined with radiographs and DIAGNOdent. This may have been because examiners were asked not to depend on the DIAGNOdent readings alone but to use their clinical judgment as well when deciding on diagnoses and treatment. In addition, it has been reported that the performance of DIAGNOdent depends on the cutoffs used, and values obtained with the DIAGNOdent device are not the same as the values obtained with the DIAGNOdent pen.[27] In this study, it was found that visual examination combined with radiographs and DIAGNOdent had high sensitivity and low specificity values, as has been reported.[28] Furthermore, the correlation of the visual examination alone with the histology was found to be similar to that of the visual examination combined with both radiographs and DIAGNOdent, as has been reported in the literature.[29] Other studies found the correlation between DIAGNOdent and histology to be better than for visual and radiographs.[27,30] In previous reported studies, each diagnostic method was used separately, but in this study, the diagnostic methods were combined to mimic the clinical scenario, which may have had a different effect on results obtained.

There were no statistically significant differences in the Az values between the three examination points in detection of occlusal caries at the D3 level. Previous studies have reported DIAGNOdent to have higher Az values compared with visual and radiographs.[27,28] Again, the reason for this difference might be attributed to the fact that the three diagnostic methods were combined.

The interexaminer reproducibility was moderate to substantial for visual examination alone, while it was fair to moderate for the visual examination combined with radiographs and DIAGNOdent, as has been previously reported.[28] Other studies found similar or higher interexaminer reproducibility for DIAGNOdent compared with visual examination.[26,29] Such differences between studies can be due to the level of caries included in the study, in which some studies were done at the D1 level while others were done at the D3 level. The level of experience of the examiners and the number and condition of teeth included in the study can also have an impact.[23]

This laboratory study supports many previous studies in the fact that there is still a wide variability among dentists in diagnosis and treatment decisions of initial occlusal caries.[18,20,31,32] The addition of radiographs and DIAGNOdent to the visual examination did not improve the examiners' performance in detection and treatment decisions of noncavitated occluso-dentinal caries. Recently, similar results have been found in a clinical study on primary teeth, in which simultaneous combined strategies increased sensitivities but decreased specificities.[33] Furthermore, no differences were observed in the accuracy of diagnosis or treatment.

The addition of both radiographs and DIAGNOdent to the visual examination resulted in more teeth treated restoratively (mainly as preventive resin restorations). Similar results have been reported in the literature.[7] The addition of radiographs and DIAGNOdent did not increase the accuracy or the percentage of correctly treated teeth; conversely, it resulted in an increase in the percentage of overtreated teeth. The increase in the false-positive diagnosis, meaning some teeth will be treated without having the disease, has led others to conclude that DIAGNOdent should not be used as the primary source for diagnosis.[11,14,24,32]

This *in vitro* study has demonstrated that the use of radiographs and DIAGNOdent pen as adjuncts to visual examination adds little benefit in detection or treatment decisions of noncavitated occlusal carious lesions in comparison with the visual examination being carried out alone.[28,34-39]

CONCLUSIONS

1. Visual examination with radiograph had higher specificity and lower sensitivity than visual examination alone or visual examination combined with radiographs and DIAGNOdent.
2. There was no difference in the Az values between visual examination alone, visual examination with radiographs, or visual examination combined with radiographs and DIAGNOdent.
3. Visual examination with radiographs had the lowest rank correlation with histology.
4. There was an increase in the number of teeth indicated for treatment when the examiners used radiographs and DIAGNOdent with the visual examination.
5. The number of correct treatment decisions was not different between the three examination points.

Acknowledgement

This study was supported by Kuwait University research grant DR02/2013.

Human Subject Statement

This study was conducted in accordance with all the provisions of the human subject oversight committee guidelines and policies at Kuwait University. The approval code for this study was 02-2013. This study was conducted at Kuwait University.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 15 September 2014)

REFERENCES

1. Makhija SK, Gilbert GH, Funkhouser E, Bader JD, Gordan VV, Rindal DB, Bauer M, Pihlstrom DJ, Qvist V, & National Dental Practice-Based Research Network Collaborative Group (2012) The prevalence of questionable occlusal caries: findings from the Dental Practice-Based Research Network *Journal of American Dental Association* **143(12)** 1343-1350.
2. Carvalho JC (2014) Caries process on occlusal surfaces: evolving evidence and understanding *Caries Research* **48(4)** 339-346.
3. Souza JF, Boldieri T, Diniz MB, Rodrigues JA, Lussi A, & Cordeiro RC (2013) Traditional and novel methods for occlusal caries detection: performance on primary teeth *Lasers in Medical Science* **28(1)** 287-295.
4. Makhija SK, Gilbert GH, Funkhouser E, Bader JD, Gordan VV, Rindal DB, Pihlstrom DJ, & Qvist V (2014) Characteristics, detection methods and treatment of questionable occlusal carious lesions: findings from the National Dental Practice-Based Research Network *Caries Research* **48(3)** 200-207.
5. Ouellet A, Hondrum SO, & Pietz DM (2002) Detection of occlusal carious lesions *General Dentistry* **50(4)** 346-350.
6. Bjørndal L (2002) Buonocore Memorial Lecture. Dentin caries: progression and clinical management *Operative Dentistry* **27(3)** 211-217.
7. Pereira AC, Eggertsson H, Martinez-Mier EA, Mialhe FL, Eckert GJ, & Zero DT (2009) Validity of caries detection on occlusal surfaces and treatment decisions based on results from multiple caries-detection methods *European Journal of Oral Sciences* **117(1)** 51-57.
8. Heinrich-Weltzien R, Weerheijm KL, Kühnisch J, Oehme T, & Stösser L (2002) Clinical evaluation of visual, radiographic, and laser fluorescence methods for detection of occlusal caries *ASDC Journal of Dentistry for Children* **69(2)** 127-132, 123.
9. Souza-Zaroni WC, Ciccone JC, Souza-Gabriel AE, Ramos RP, Corona SA, & Palma-Dibb RG (2006) Validity and reproducibility of different combinations of methods for occlusal caries detection: an *in vitro* comparison *Caries Research* **40(3)** 194-201.
10. Kühnisch J, Ifland S, Tranaeus S, Hickel R, Stösser L, & Heinrich-Weltzien R (2007) *In vivo* detection of non-cavitated caries lesions on occlusal surfaces by visual inspection and quantitative light-induced fluorescence *Acta Odontologica Scandinavia* **65(3)** 183-188.
11. Bader JD, Shugars DA, & Bonito AJ (2002) A systematic review of the performance of methods for identifying carious lesions *Journal of Public Health Dentistry* **62(4)** 201-213.
12. Poorterman JH, Weerheijm KL, Groen HJ, & Kalsbeek H (2000) Clinical and radiographic judgment of occlusal caries in adolescents *European Journal of Oral Sciences* **108(2)** 93-98.
13. Ricketts DN, Kidd EA, & Beighton D (1995) Operative and microbiological validation of visual, radiographic and electronic diagnosis of occlusal caries in non-cavitated teeth judged to be in need of operative care *British Dental Journal* **179(6)** 214-220.
14. Bader JD, & Shugars DA (2004) A systematic review of the performance of a laser fluorescence device for detecting caries *Journal of the American Dental Association* **135(10)** 1413-1426.
15. Matos R, Novaes TF, Braga MM, Siqueira WL, Duarte DA, & Mendes FM (2011) Clinical performance of two fluorescence-based methods in detecting occlusal caries lesions in primary teeth *Caries Research* **45(3)** 294-302.
16. Twetman S, Axelsson S, Dahlén G, Espelid I, Mejàre I, Norlund A, & Tranæus S (2013) Adjunct methods for caries detection: a systematic review of literature *Acta Odontologica Scandinavia* **71(3-4)** 388-397.
17. Diniz MB, Lima LM, Eckert G, Zandona AG, Cordeiro RC, & Pinto LS (2011) *In vitro* evaluation of ICDAS and radiographic examination of occlusal surfaces and their association with treatment decisions *Operative Dentistry* **36(2)** 133-142.

18. Verdonschot EH, Angmar-Månsson B, ten Bosch JJ, Deery CH, Huysmans MC, Pitts NB, & Waller E (1999) Developments in caries diagnosis and their relationship to treatment decisions and quality of care. ORCA Saturday Afternoon Symposium 1997 *Caries Research* **33(1)** 32-40.
19. Holmgren C, Gaucher C, Decerle N, & Doméjean S (2014) Minimal intervention dentistry II: part 3. Management of non-cavitated (initial) occlusal caries lesions: non-invasive approaches through remineralization and therapeutic sealants *British Dental Journal* **216(5)** 237-243.
20. Al-Khatrash AA, Badran YM, & Alomari QD (2011) Factors affecting the detection and treatment of occlusal caries using the International Caries Detection and Assessment System *Operative Dentistry* **36(6)** 597-607.
21. Downer MC (1975) Concurrent validity of an epidemiological diagnostic system for caries with the histological appearance of extracted teeth as validating criterion *Caries Research* **9(3)** 231-246.
22. Landis JR, & Koch GG (1977) The measurement of observer agreement for categorical data. *Biometrics* **33(1)** 159-174.
23. Côrtes DF, Ellwood RP, & Ekstrand KR (2003) An *in vitro* comparison of a combined FOTI/visual examination of occlusal caries with other caries diagnostic methods and the effect of stain on their diagnostic performance *Caries Research* **37(1)** 8-16.
24. Costa AM, Paula LM, & Bezerra AC (2008) Use of Diagnodent for diagnosis of non-cavitated occlusal dentin caries *Journal of Applied Oral Sciences* **16(1)** 18-23.
25. Lussi A, Megert B, Longbottom C, Reich E, & Francescut P (2001) Clinical performance of a laser fluorescence device for detection of occlusal caries lesions *European Journal of Oral Science* **109(1)** 14-19.
26. Rocha RO, Ardenghi TM, Oliveira LB, Rodrigues CR, & Ciamponi AL (2003) *In vivo* effectiveness of laser fluorescence compared to visual inspection and radiography for the detection of occlusal caries in primary teeth *Caries Research* **37(6)** 437-441.
27. Diniz MB, Boldieri T, Rodrigues JA, Santos-Pinto L, Lussi A, & Cordeiro RC (2012) The performance of conventional and fluorescence-based methods for occlusal caries detection: an *in vivo* study with histologic validation *Journal of American Dental Association* **143(4)** 339-350.
28. Angnes V, Angnes G, Batisttella M, Grande RH, Loguercio AD, & Reis A (2005) Clinical effectiveness of laser fluorescence, visual inspection and radiography in the detection of occlusal caries *Caries Research* **39(6)** 490-495.
29. Toraman Alkurt M, Peker I, Deniz Arisu H, Bala O, & Altunkaynak B (2008) *In vivo* comparison of laser fluorescence measurements with conventional methods for occlusal caries detection *Lasers Medical Science* **23(3)** 307-312.
30. Costa AM, Bezzerra AC, & Fuks AB (2007) Assessment of the accuracy of visual examination, bite-wing radiographs and DIAGNOdent on the diagnosis of occlusal caries *European Archives of Pediatric Dentistry* **8(2)** 118-122.
31. Bader JD, & Shugars DA (2006) The evidence supporting alternative management strategies for early occlusal caries and suspected occlusal dentinal caries *Journal of Evidence Based Dental Practice* **6(1)** 91-100.
32. Ekstrand KR, Ricketts DN, Longbottom C, & Pitts NB (2005) Visual and tactile assessment of arrested initial enamel carious lesions: an *in vivo* pilot study *Caries Research* **39(3)** 173-177.
33. Mendes FM, Novaes TF, Matos R, Bittar DG, Piovesan C, Gimenez T, Imparato JC, Raggio DP, & Braga MM (2012) Radiographic and laser fluorescence methods have no benefits for detecting caries in primary teeth *Caries Research* **46(6)** 536-543.
34. Braga MM, Mendes FM, Ekstrand KR (2010) Detection activity assessment and diagnosis of dental caries lesions *Dental Clinics of North America* **54(3)** 479-493.
35. Gomez J, Zakian C, Salsone S, Pinto SC, Taylor A, Pretty IA, & Ellwood R (2013) *In vitro* performance of different methods in detecting occlusal caries lesions *Journal of Dentistry* **41(2)** 180-186.
36. Heinrich-Weltzien R, Kühnisch J, Oehme T, Ziehe A, Stösser L, & García-Godoy F (2003) Comparison of different DIAGNOdent cut-off limits for *in vivo* detection of occlusal caries *Operative Dentistry* **28(6)** 672-680.
37. Kühnisch J, Berger S, Goddon I, Senkel H, Pitts N, & Heinrich-Weltzien R (2008) Occlusal caries detection in permanent molars according to WHO basic methods, ICDAS II and laser fluorescence measurements *Community Dentistry & Oral Epidemiology* **36(6)** 475-484.
38. Rodrigues JA, Hug I, Diniz MB, & Lussi A (2008) Performance of fluorescence methods, radiographic examination and ICDAS II on occlusal surfaces *in vitro* *Caries Research* **42(4)** 297-304.
39. Seremidi K, Lagouvardos P, & Kavvadia K (2012) Comparative *in vitro* validation of VistaProof and DIAGNOdent pen for occlusal caries detection in permanent teeth *Operative Dentistry* **37(3)** 234-245.

©Operative Dentistry, 2015, **40-3,** 322-335

Localized and Generalized Simulated Wear of Resin Composites

WW Barkmeier • T Takamizawa • RL Erickson
A Tsujimoto • M Latta • M Miyazaki

Clinical Relevance

Wear is an important parameter for the selection of resin composite materials.

SUMMARY

A laboratory study was conducted to examine the wear of resin composite materials using both a localized and generalized wear simulation model. Twenty specimens each of seven resin composites (Esthet•X HD [HD], Filtek Supreme Ultra [SU], Herculite Ultra [HU], SonicFill [SF], Tetric EvoCeram Bulk Fill [TB], Venus Diamond [VD], and Z100 Restorative [Z]) were subjected to a wear challenge of 400,000 cycles for both localized and generalized wear in a Leinfelder-Suzuki wear simulator (Alabama machine). The materials were placed in custom cylinder-shaped stainless steel fixtures. A stainless steel ball bearing (r=2.387 mm) was used as the antagonist for localized wear, and a stainless steel, cylindrical antagonist with a flat tip was used for generalized wear. A water slurry of polymethylmethacrylate (PMMA) beads was used as the abrasive media. A noncontact profilometer (Proscan 2100) with Proscan software was used to digitize the surface contours of the pretest and posttest specimens. AnSur 3D software was used for wear assessment. For localized testing, maximum facet depth (μm) and volume loss (mm^3) were used to compare the materials. The mean depth of the facet surface (μm) and volume loss (mm^3) were used for comparison of the generalized wear specimens. A one-way analysis of variance (ANOVA) and Tukey post hoc test were used for data analysis of volume loss for both localized and generalized wear, maximum facet depth for localized wear, and mean depth of

*Wayne W Barkmeier, DDS, MS, professor, General Dentistry, dean emeritus, Creighton University School of Dentistry, Omaha, NE, USA

Toshiki Takamizawa, DDS, PhD, assistant professor, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Robert L Erickson, PhD, DDS, clinical professor, General Dentistry, Creighton University School of Dentistry, Omaha, NE, USA

Akimasa Tsujimoto, DDS, PhD, assistant professor, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

Mark Latta, DMD, MS, professor, General Dentistry, dean, Creighton University School of Dentistry

Masashi Miyazaki, DDS, PhD, professor, Operative Dentistry, Nihon University School of Dentistry, Tokyo, Japan

*Corresponding author: 2500 California Plaza, Omaha, NE 68178; e-mail: wbark@creighton.edu

DOI: 10.2341/13-155-L

the facet for generalized wear. The results for localized wear simulation were as follows [mean (standard deviation)]: maximum facet depth (μm)—Z, 59.5 (14.7); HU, 99.3 (16.3); SU, 102.8 (13.8); HD, 110.2 (13.3); VD, 114.0 (10.3); TB, 125.5 (12.1); SF, 195.9 (16.9); volume loss (mm^3)— Z, 0.013 (0.002); SU, 0.026 (0.006); HU, 0.043 (0.008); VD, 0.057 (0.009); HD, 0.058 (0.014); TB, 0.061 (0.010); SF, 0.135 (0.024). Generalized wear simulation results were as follows: mean depth of facet (μm)—Z, 9.3 (3.4); SU, 12.8 (3.1); HU, 15.6 (3.2); TB, 19.2 (4.8); HD, 26.8 (6.5); VD, 29.1 (5.5); SF, 35.6 (8.4); volume loss (mm^3)—Z, 0.132 (0.049); SU, 0.0179 (0.042); HU, 0.224 (0.044); TB, 0.274 (0.065); HD, 0.386 (0.101); VD, 0.417 (0.076); SF, 0.505 (0.105). The ANOVA showed a significant difference among materials ($p<0.001$) for facet depth and volume loss for both localized and generalized wear. The post hoc test revealed differences ($p<0.05$) in localized and generalized wear values among the seven resin composites examined in this study. The findings provide valuable information regarding the relative wear characteristics of the materials in this study.

INTRODUCTION

Wear of resin composite materials has been a concern of the dental profession since the materials were first advocated for the posterior dentition in the early 1970s. Early clinical trials of resin composite in the posterior region showed significant wear when compared with metallic restorations.[1] Improvements in the resin matrix and filler components of resin composites have resulted in markedly better materials for the posterior dentition than the early-generation composites.[2-11] Clinical studies over the years have shown steady improvements in the performance of later-generation resin composites.[12-23]

Due to the limited clinical information available to the profession regarding the performance of resin composite materials, laboratory wear simulation has evolved as a useful methodology for assessing relative wear resistance. Simulation models for both localized wear (occlusal contact area [OCA] wear) and generalized wear (contact-free area [CFA] wear) have been developed. Barkmeier and others[24] recently recommended that a benchmark resin composite material (Z100 Restorative, 3M ESPE, St Paul, MN, USA) with good clinical and simulated wear performance be used as a standard for laboratory wear comparison with other resin composites. The recommendation is further strengthened by the good agreement of clinical and laboratory wear rates of the Z100 Restorative material.[16,18]

Additional information regarding wear characteristics of resin composites is needed. Limited data regarding the clinical wear performance of resin composite materials are available in the literature. The purpose of this study was to expand the information base on the simulated wear of resin composite materials and compare them with a benchmark material (Z100 Restorative). The materials selected for this study are current-generation materials, and little information about the relative performance of these materials is available to the profession. In addition, this study incorporated the use of a new computer software program for wear analysis.

METHODS AND MATERIALS

Wear Simulation

Seven resin composite materials were evaluated in this study: 1) Esthet•X HD (HD) (Dentsply Caulk, Milford, DE, USA); 2) Filtek Supreme Ultra (SU) (3M ESPE); 3) Herculite Ultra (HU) (Kerr Corporation Orange, CA, USA); 4 SonicFill (SF) (Kerr Corporation), 5) Tetric EvoCeram Bulk Fill (TB) (Ivoclar Vivadent AG, Schaan, Liechtenstein); 6) Venus Diamond (VD) (Heraeus Kulzer GmbH, Hanau, Germany); and 7) Z100 Restorative (Z) (3M ESPE). The resin composite materials and their components are listed in Tables 1 and 2.

Twenty specimens of each of the seven resin composites were prepared for simulated localized wear (OCA wear) and 20 specimens of each material were prepared for generalized wear (CFA wear). Cylinder-shaped custom stainless steel fixtures used for the localized wear were machined with a cylindrical cavity 6.5 mm in diameter and 4 mm in depth. Stainless steel fixtures for generalized wear testing were machined with a cylindrical cavity 4.5 mm in diameter and 4 mm in depth. Two increments of the resin composite materials (approximately 2 mm in thickness) were cured for 40 seconds each with a Spectrum 800 curing unit (Dentsply Caulk) set at 600 mW/cm^2. A SonicFill handpiece (Kerr Corporation) was used for insertion of the SF material into the cavity in the stainless custom fixtures, and the other six materials were inserted using a condenser. Twenty-four hours later, the composite surfaces were polished

Table 1: *Resin Composite Materials*

Material	Manufacturer	Lot	Shade	Study Code
Esthet•X HD	Dentsply Caulk, Milford, DE 19963 USA	120414	A2	HD
Filtek Supreme Ultra	3M ESPE Dental Products, St. Paul, MN 55144 USA	N339152	A2 Body Shade	SU
Herculite Ultra	Kerr Corporation, Orange, CA 92867 USA	4495606	A2 Enamel Shade	HU
SonicFill[a]	Kerr Corporation, Orange, CA 92867 USA	4695352	A2	SF
Tetric EvoCeram Bulk Fill	Ivoclar Vivadent AG, Schaan, Liechtenstein	R04686	IVA	TB
Venus Diamond	Heraeus Kulzer GmbH, Hanau, Germany	010041	A2	VD
Z100 Restorative	3M ESPE Dental Products, St Paul, MN 55144 USA	N3105067	A2	Z

[a] A SonicFill handpiece 34920 was used for insertion of the SonicFill material.

flat (Figures 1 and 2) to 4000-grit using a sequence of silicon carbide papers (Struers Inc, Cleveland, OH, USA).

A Leinfelder-Suzuki wear simulation device (Alabama machine) was used for this study. The simulator has a plastic water bath, and the custom wear fixtures were mounted inside the four-station bath. A brass cylinder was then placed around each fixture in the bath to serve as a reservoir for the abrasive media (water slurry of unplasticized polymethyl methacrylate [PMMA] with an average particle size of 44 μm). The media was placed inside the brass cylinders to cover the surface of the resin composite in the custom fixtures. The water slurry of PMMA inside the brass cylinders was approximately 6 mm in height over the surface of the resin composite.

Two different wear antagonists were used in this study. For localized (OCA) wear simulation, a stainless steel ball bearing (r=2.387 mm) was mounted inside a collet assembly (Figure 3). The antagonist for the generalized (CFA) wear simulation was a stainless steel cylinder (diameter, 6.5 mm) with a flat tip (Figure 4). The antagonist tips were mounted on spring-loaded pistons to deliver the wear challenges. During the application of the load, the antagonists rotated approximately 30° as the maximum force was reached (maximum load of 78.5 N at

Table 2: *Information on Resin Composites*

Resin Composite	Matrix	Filler	Filler load
Esthet•X HD	Bis-GMA, UDMA, TEGDMA	Barium alumino-fluoro-silicate glass (BAFG) <1 μm; BAFG 0.02-2.5 μm (average 0.6-0.8); nanosized silicon dioxide (10-20 nm)	76% wt (60% vol)
Filtek Supreme Ultra (body shade)	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA	Aggregated zirconia/silica clusters (20 nm silica and 4-11nm zirconia particles), average cluster 0.6-10 μm; nonagglomerated/non-aggregated 20 nm silica and 4-11 nm zirconia	78.5% wt (63.3% vol)
Herculite Ultra (enamel shade)	Bis-GMA, TEGDMA, Bis-EMA	Barium glass (0.4 μm; silica, 20-50 nm); prepolymerized filler (barium glass and silica)	78% wt (57% vol)
SonicFill	Bis-GMA, TEGDMA, Bis-EMA	Barium glass; silica; rheological modifier	84% wt (67% vol)
Tetric EvoCeram Bulk Fill	Bis-GMA, UDMA, Bis-EMA	Barium alumino-silicate glass; prepolymerized filler (monomer, glass filler, and ytterbium fluoride); spherical mixed oxide	77% wt, including 17% prepolymerized (61% vol)
Venus Diamond	TCD-DI-HEA, UDMA	Barium alumino-fluoro-silicate glass, <20 μm; silica, 20 nm	81% wt (64% vol)
Z100 Restorative	Bis-GMA, TEGDMA	Zirconia/silica, 0.01-3.5 μm	84.5% wt (66% vol)

Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, bisphenol A glycidyl dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate; TCD-DI-HEA, 2-propenioc acid, (octahydro-4,7 methano-1H-indene-5-diyl) bis(methyleneiminocarbonyloxy-2,1-ethanediyl) ester; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Figure 1. *Stainless steel custom fixture for localized wear.*

a rate of 2 Hz), and then they counterrotated back to the original starting position as the load was relaxed to complete the cycle. Each set of specimens was exposed to 400,000 cycles in the wear simulator.

Figure 2. *Stainless steel custom fixture for generalized wear.*

Figure 3. *Stainless steel antagonist tip for simulated localized wear.*

Wear Measurements

Prior to wear simulation, each resin composite specimen was profiled using a Proscan 2100 noncontact optical profilometer (Scantron Industrial Prod-

Figure 4. *Stainless steel antagonist tip for simulated generalized wear.*

Table 3: *Analysis of Variance of Simulated Localized Wear—Facet Depth*

Source	Sum-of-Squares	*df*	Mean-Square	*F*-ratio	p-Value
Material	203,181.964	6	33,863.661	179.222	< 0.001
Error	25,130.134	133	13.293		

ucts Ltd, Taunton, England) with Proscan software. These profiles provided the pretest digitized contours (20 test specimens each for the seven resin composite materials for both localized and generalized wear testing).

Following the 400,000 wear cycles, the specimens were ultrasonically cleaned (L&R Solid State Ultrasonic T-14B, South Orange, NJ, USA) in distilled water for three minutes and then profiled again using the Proscan 2100 unit. The x-, y-, and z-coordinates of the before and after scans from the Proscan software were exported to another computer for analysis with AnSur 3D software (Minnesota Dental Research Center for Biomaterials and Biomechanics, University of Minnesota, Minneapolis, MN, USA). The x-, y-, and z-coordinates generated with the Proscan software were saved as .prn files and then imported into the AnSur 3D program.

Wear measurements were determined from differences between the before and after data sets. A computerized fit was accomplished with the before and after data sets in AnSur 3D and volume loss measurements (mm^3) were then determined for both localized and generalized wear simulation for each of the seven resin composites. Maximum depth of the wear facet (μm) was also determined for the localized wear specimens, and mean depth (μm) of the wear facet was determined for the generalized specimens. A one-way analysis of variance (ANOVA) and Tukey post hoc test were used for data analysis of volume loss for both localized and generalized wear, maximum facet depth for localized wear, and mean facet depth for generalized wear.

Scanning Electron Microscopy Observations

Specimens were prepared for argon-ion etching and scanning electron microscopy (SEM) examinations at Nihon University School of Dentistry in Tokyo, Japan. The seven resin composites examined in this manner may not have been from the same lot numbers as the resin materials subjected to the wear simulation studies at Creighton University School of Dentistry. The surfaces of the cured resin composites were polished to a high gloss with abrasive discs (Fuji Star Type DDC, Sankyo Rikagaky Co Ltd, Saitama, Japan), followed by a series of diamond pastes down to 0.25-μm particle size (DP-Paste, Struers, Ballerup, Denmark). The polished surfaces were subjected to argon-ion beam etching (IIS-200ER, Elionix, Tokyo, Japan) for 45 seconds with the ion beam directed at the polished surface (accelerating voltage of 1.0 kV, ion current density 0.4 mA/cm^2). The surfaces were then coated in a vacuum evaporator with a thin film of gold . Observations were done with a SEM (FE-8000, Elionix, Tokyo, Japan) at an operating voltage of 10 kV and magnification of 5000×.

SEM examinations were completed at the Creighton University School of Dentistry on the wear facets of the seven resin composite materials after both localized and generalized wear simulation. Following the wear analysis, representative samples of each material were sputter coated with gold and palladium (Emitech SC7620 Mini Sputter Coater, Quorum Technologies, Ashford, UK). The coated specimens were then examined with a TM3000 Tabletop Microscope (Hitachi-High Technologies Corporation, Tokyo, Japan) using an accelerating voltage of 15 kV and magnification of 5000×.

RESULTS

Wear Measurements

The one-way ANOVA tests showed a significant effect for material for both simulated localized wear and simulated generalized wear for both facet depth and volume loss ($p<0.001$). The results of the ANOVA testing are shown in Tables 3-6.

The localized wear values (maximum facet depth and volume loss) for the seven resin composite materials evaluated are presented in Tables 7 and 8. The statistical differences ($p<0.05$) in localized wear among the seven materials using the Tukey post hoc test are also shown in the same tables.

For the seven materials examined, values for the mean (standard deviation) of maximum facet depth (μm) for localized wear ranged from 59.5 (14.7) to 195.9 (16.9). The localized wear facet depth of Z was significantly less ($p<0.05$) than the other six materials in this study. The mean facet depth of SF was significantly greater ($p<0.05$) than the other six materials.

Table 4: *Analysis of Variance for Simulated Localized Wear—Volume Loss*

Source	Sum-of-Squares	*df*	Mean-Square	*F*-Ratio	*p*-Value
Material	0.184	6	0.031	197.595	< 0.001
Error	0.021	133	0.000		

The localized wear mean volumetric loss (mm^3) ranged from 0.003 (0.002) to 0.135 (0.024). The localized wear volumetric loss of Z was significantly less ($p<0.05$) than of the other six materials. The volumetric loss of SF was significantly greater ($p<0.05$) than of the other six materials.

The generalized wear values (mean facet depth and volume loss) are presented in Tables 9 and 10. The results of the Tukey test for significant differences among the materials tested are shown in the same tables.

The mean depth (μm) of the wear facets for generalized wear ranged from 9.3 (3.4) to 35.6 (8.4). The benchmark material (Z) exhibited the least facet wear depth using the generalized wear model. SU ranked second in wear resistance and was not significantly different ($p>0.05$) from Z. The generalized facet wear depth of SF was significantly greater ($p<0.05$) than of the other six materials evaluated.

The rank order of all seven materials was the same for generalized volumetric loss and generalized facet wear depth. The Z resin composite exhibited the least volumetric loss during generalized wear simulation but was not statistically different ($p>0.05$) from SU. The volume loss of SF was significantly greater ($p<0.05$) than of the other six materials in this study.

SEM Observations

SEM examinations of the seven resin composite materials evaluated by argon-ion etching are presented in Figure 5A-G. This figure demonstrates the differences in the shape and size of the filler components of the materials subjected to wear challenges in this study. The argon-ion etching shows clear differences in filler particle size, shape, and distribution. Z and SU have zirconia/silica filler particles that are more rounded or cylindrical (Figure 5B,G) than the typical ground glass particle contained in many resin composite systems. The glass filler particles of SF (Figure 5D) and VD (Figure 5F) appeared to be larger than the fillers in the other resin composite systems with ground (milled) glass filler particles (HD, Figure 5A; TB, Figure 5E; and HU, Figure 5C). The ground/milled glass particles (VD, TB, SF, HU, and HD) are more irregular in shape, when compared with the agglomerated filler particles in Z and SU.

SEM examinations of the localized wear facets (Figure 6A-6G) also demonstrated the differences in the filler systems of the resin composites in this study. SEM observations of Z and SU revealed a similar wear pattern with some fracturing of the resin matrix, resulting in micron size filler particles being fractured out on the surface. Despite this fracturing process, the localized wear of Z and SU was less than that of the other materials in this study. The SEM observations of SF (Figure 6D) showed larger glass filler particles and some fracturing of the particles resulting from the concentrated force applied with the localized antagonist. The VD SEM observations (Figure 6F) showed evidence of fatigue cracks in the resin matrix and plucking of glass filler particles from the surface. The SEM observations of HU (Figure 6C) and TB (Figure 6E) appeared to show evidence of the prepolymerized filler components of these systems.

SEM examinations (Figures 7A-G) of the surfaces of the generalized wear simulation specimens also demonstrated differences in the resin composite systems. Cracking of the glass filler component of SF (Figure 7D) was observed, as was plucking of filler particles from the surface. SU (Figure 7B), VD (Figure 7F), and Z (Figure 7G) also showed plucking of filler particles resulting from simulated generalized wear. There also was some evidence of cracks in the resin matrix of SU. The SEM examinations of TB (Figure 7E) revealed the various filler components on the worn surface of this resin composite. Prepoly-

Table 5: *Analysis of Variance for Simulated Generalized Wear—Mean Facet Depth*

Source	Sum-of-Squares	*df*	Mean-Square	*F*-ratio	*p*-Value
Material	11,237.285	6	1872.898	66.989	<0.001
Error	3718.445	133	27.958		

Table 6: *Analysis of Variance for Simulated Generalized Wear—Volume Loss*

Source	Sum-of-Squares	*df*	Mean-Square	*F*-ratio	*p*-Value
Material	2.243	6	0.374	70.195	<0.001
Error	0.708	133	0.005		

merized particles were evident on the surface of HU (Figure 7C) after simulated generalized wear.

DISCUSSION

The use of wear simulation to predict clinical performance has been a challenge for dental materials researchers for many years. Leinfelder and Suzuki[6] published much of the early work in trying to relate simulated wear with clinical wear by using a spring-loaded piston wear simulator. This device was a modification of a wear simulator developed by Roulet.[25] In their early work, Leinfelder and Suzuki[6] (1999) used a cylinder-shaped cavity in extracted human molar teeth (enamel margins) for placement of a restorative resin material and used a flat polyacetal antagonist to simulate generalized wear (Alabama method). They used 400,000 cycles for simulated, generalized wear studies. The results of their published work[6] found an excellent correlation between 400,000 cycles in the wear testing device (generalized wear simulation) and three years of clinical services for 10 resin composites. The results of their work and the reported correlation of *in vitro* and *in vivo* wear led researchers to routinely use 400,000 cycles with the Alabama testing machine.

Barkmeier and others[18] modified the testing methods for simulated generalized wear with the Alabama machine. They developed custom stainless steel fixtures to hold the test material and moved to a flat stainless steel antagonist. This improvement essentially eliminated the problem of both antagonist and tooth surface wear during the cycling procedure. In addition to simulating generalized wear, the Alabama simulator has also been used for localized wear testing using a hardened steel, cone-shaped antagonist. More recently, a collet device for holding a stainless steel ball bearing has been introduced. The ball bearings allow a new antagonist tip to be used for each specimen because of the cost differential between a chrome-plated, hardened steel antagonist and a stainless steel ball bearing. Whereas other antagonist tips, such as enamel, ceramic, or resin composite, may add additional information in wear testing, stainless steel has routinely been used to provide consistency in the wear challenges for both localized and generalized wear simulation studies. However, although a steel antagonist offers many advantages, the clinical relevance is sometimes brought into question.

Table 7: *Simulated Localized Wear—Maximum Facet Depth (μm) and SD*

Material	Depth (SD)[a]
Z	59.5 (14.7) A
HU	99.3 (16.3) B
SU	102.8 (13.8) B
HD	110.2 (13.3) B
VD	114.0 (10.3) BC
TB	125.5 (12.1) C
SF	195.9 (16.9) D

Abbreviations:Z, Z100 Restorative; HU, Herculite Ultra; SU, Filtek Supreme Ultra; HD, Esthet•X HD; VD, Venus Diamond; TB, Tetric EvoCeram Bulk Fill; SF, SonicFill
[a] *Different letters in column indicate differences at the 5% significance level.*

Much has been published* in recent years regarding the correlation of simulated wear and clinical wear. Barkmeier and others[16] using clinical data from two study sites on two resin composite materials found a good relationship between localized simulated wear in the laboratory and OCA clinical wear. A subsequent study[18] compared simulated generalized wear and CFA clinical wear of the same two resin composites and again found a good relationship between laboratory and clinical wear values. Heintze and others[11,23] have published on the relationship of simulated wear to clinical wear using multiple laboratory wear simulators. These studies have compared wear simulation data from multiple testing centers with published clinical data for the same materials. These investigators, when examining data from several sites, have found a wide range of correlations between laboratory and clinical data for both generalized and localized wear. The correlations for the Alabama method have been reported[11] as being better for generalized wear than for localized wear. However, it should be noted that various sites doing wear simulation studies with the Alabama machine use different methodologies for generating localized and generalized simulated wear. Although the absolute wear values may not be the same, depending on site methodologies and

* References 3,6,10,11,16,18,23,24,26,36-40.

Table 8: *Simulated Localized Wear—Volume Loss (mm^3) and SD*

Material	Volume (SD)[a]
Z	0.013 (0.002) A
SU	0.026 (0.006) B
HU	0.043 (0.008) C
VD	0.057 (0.009) D
HD	0.058 (0.014) D
TB	0.061 (0.010) D
SF	0.135 (0.024) E

Abbreviations:Z, Z100 Restorative; HU, Herculite Ultra; SU, Filtek Supreme Ultra; HD, Esthet•X HD; VD, Venus Diamond; TB, Tetric EvoCeram Bulk Fill; SF, SonicFill
[a] *Different letters in column indicate differences at the 5% significance level.*

Table 10: *Simulated Generalized Wear—Volume Loss (mm^3) and SD*

Material	Volume (SD)[a]
Z	0.132 (0.049) A
SU	0.179 (0.042) AB
HU	0.224 (0.044) BC
TB	0.274 (0.065) C
HD	0.386 (0.101) D
VD	0.417 (0.076) D
SF	0.505 (0.105) E

Abbreviations:Z, Z100 Restorative; HU, Herculite Ultra; SU, Filtek Supreme Ultra; HD, Esthet•X HD; VD, Venus Diamond; TB, Tetric EvoCeram Bulk Fill; SF, SonicFill
[a] *Different letters in column indicate differences at the 5% significance level.*

analysis tools, the relative wear ranking of materials from site to site should be similar.

Analysis methods for determining wear of laboratory specimens have evolved over the years. Various profiling and 3D measurement techniques have been used for wear analysis.[3,6,9,11-12,23-37] Typically, a pretest surface is compared with a posttest surface, and wear (facet depth and/or volume loss) is determined from the differences between the pretest and posttest digitized surfaces.

In the present study a Proscan 2100 noncontact profilometer with Proscan software was used to digitize the surface contours of the pretest and posttest data sets. The x-, y-, and z-coordinates of the pretest and posttest specimens were exported from the Proscan software onto a computer with AnSur 3D software. The AnSur 3D software was recently modified to be used with the digitized surface contour files generated with the Proscan software. After exporting the x-, y-, and z-coordinates, a computerized "fit" of the before and after data sets was accomplished using the AnSur 3D software. A goodness-of-fit value (root-mean-square of the difference of similar points) is also provided by the software. Four wear parameters are then generated by the software: 1) volume loss (mm^3); 2) maximum depth (μm)—lowest point on the posttest surface; 3) mean maximum depth (μm)—average of all the lowest points from the individual profiles examined; 4) mean depth (μm)—average depth of the posttest surface. The use of the Proscan 2100 noncontact profilometer and AnSur 3D analysis software produces excellent results combined with ease of use.

Table 9: *Simulated Generalized Wear—Mean Facet Depth (μm) and SD*

Material	Depth (SD)[a]
Z	9.3 (3.4) A
SU	12.8 (3.1) AB
HU	15.6 (3.2) BC
TB	19.2 (4.8) C
HD	26.8 (6.5) D
VD	29.1 (5.5) D
SF	35.6 (8.4) E

Abbreviations:Z, Z100 Restorative; HU, Herculite Ultra; SU, Filtek Supreme Ultra; HD, Esthet•X HD; VD, Venus Diamond; TB, Tetric EvoCeram Bulk Fill; SF, SonicFill
[a] *Different letters in column indicate differences at the 5% significance level.*

Barkmeier and others[24] recently proposed that Z100 Restorative (Z) be used as a benchmark material when wear simulation studies are done on resin composite materials. This recommendation was based on the performance of Z in laboratory wear simulation studies and also on excellent wear values reported from clinical studies.[16,18] In the present study, Z again produced the lowest localized and generalized wear values of the seven materials evaluated. This performance strengthens the recommendation that Z be used as a benchmark, or reference material, in future wear studies. If wear simulation studies show wear resistance in the range of Z exhibited in this study and other simulation studies,[16,18] then there should be good confidence that the material will perform well in a clinical setting.

Lambrechts and others have reported[38-39] that a restoration should ideally exhibit wear similar to enamel. They used their computerized 3D measuring technique to quantitatively measure OCA wear of human enamel. Over a four-year period, they measured enamel-to-enamel contact of premolar and molar teeth and recorded the vertical loss of enamel. Wear rates were calculated using logarith-

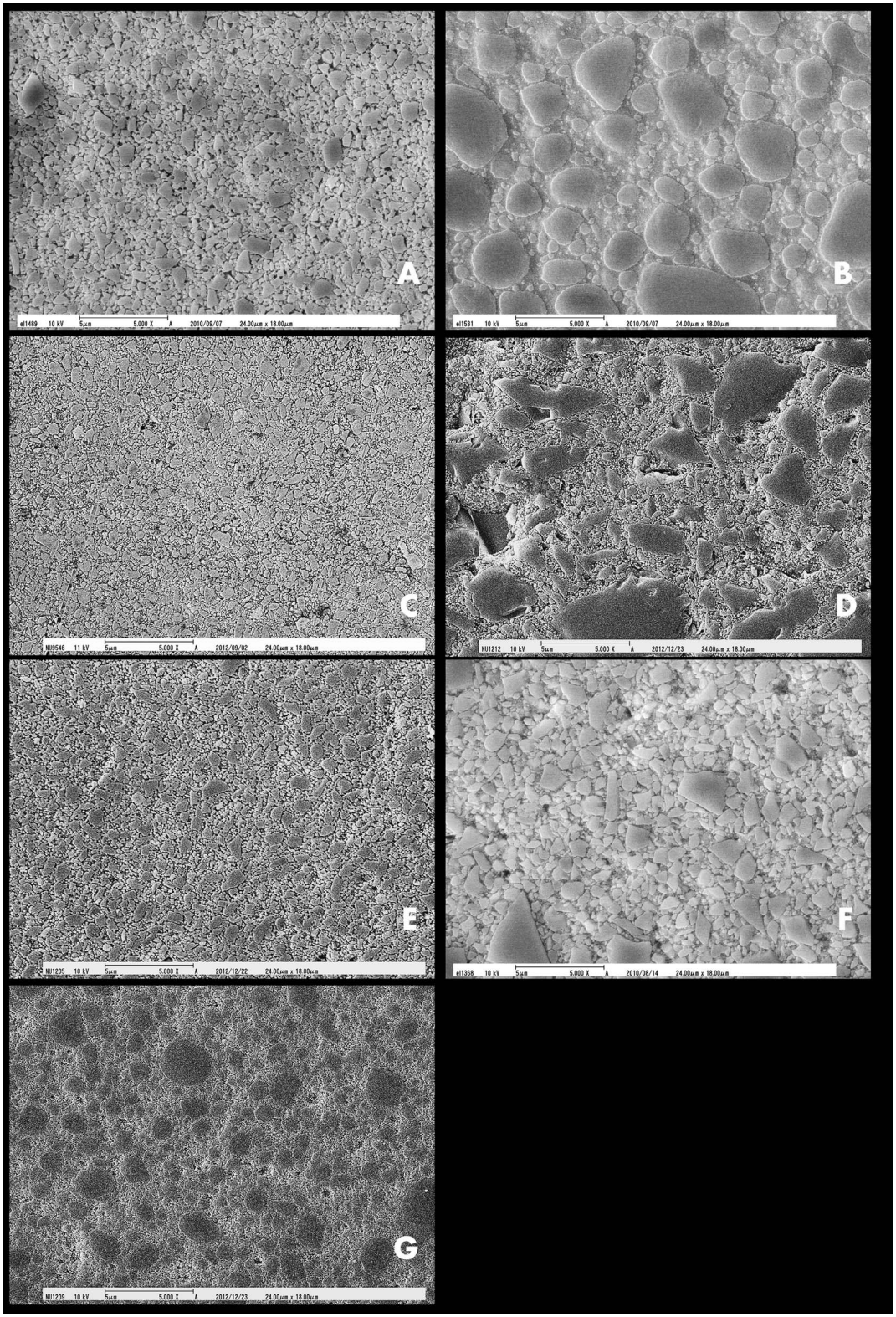

Figure 5. *(A): Esthet•X HD—argon-ion etched surface at 5000×. (B): Filtek Supreme Ultra—argon-ion etched surface at 5000×. (C): Herculite Ultra—argon-ion etched surface at 5000×. (D): SonicFill—argon-ion etched surface at 5000×. (E): Tetric EvoCeram Bulk Fill—argon-ion etched surface at 5000×. (F): Venus Diamond—argon-ion etched surface at 5000×. (G): Z100 Restorative—argon-ion etched surface at 5000×.*

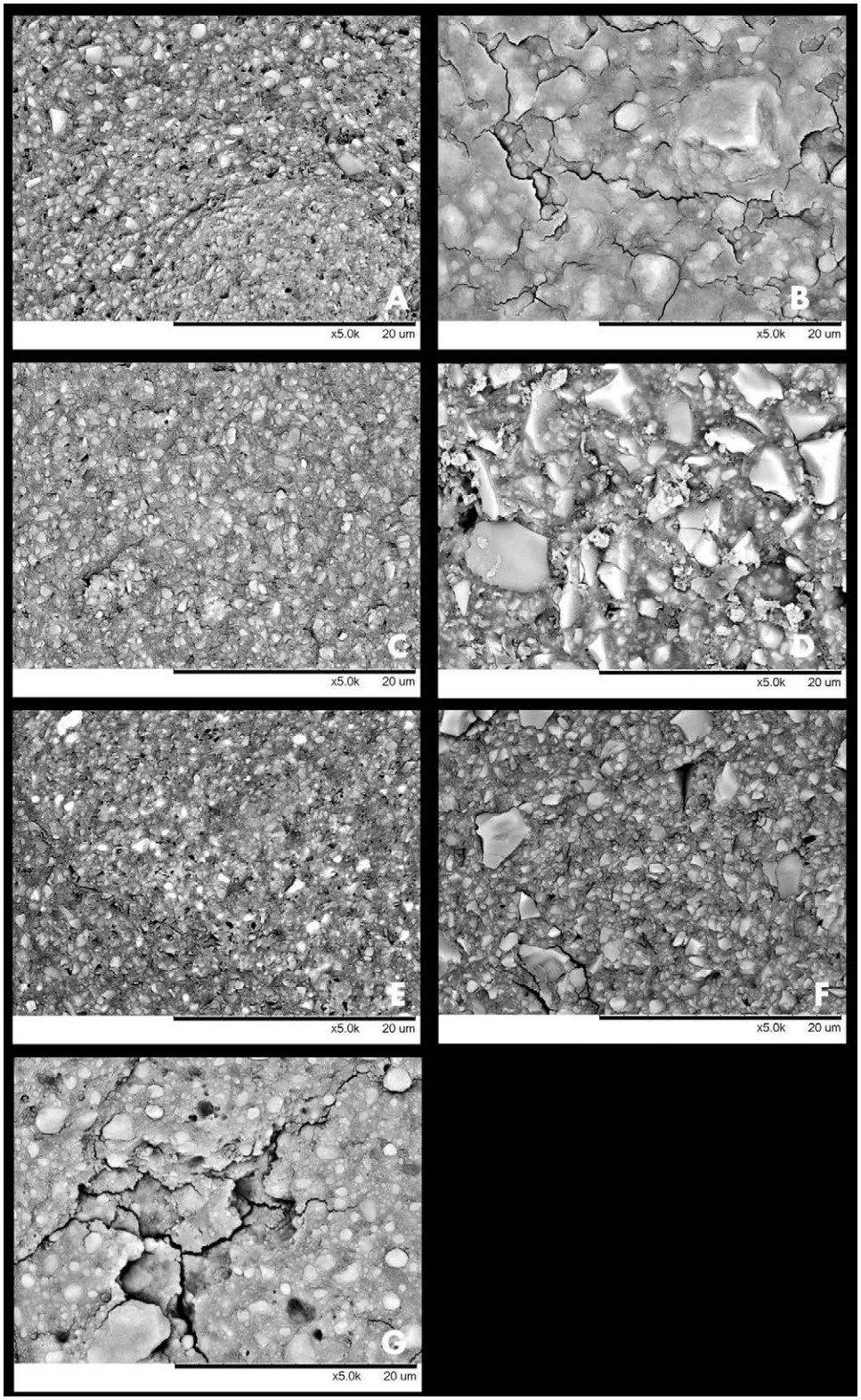

Figure 6. *(A): Esthet•X HD—localized wear near center of facet at 5000×. (B): Filtek Supreme Ultra—localized wear near center of facet at 5000×. (C): Herculite Ultra—localized wear near center of facet at 5000×. (D): SonicFill—localized wear near center of facet at 5000×. (E): Tetric EvoCeram Bulk Fill—localized wear near center of facet at 5000×. (F): Venus Diamond—localized wear near center of facet at 5000×. (G): Z100 Restorative—localized wear near center of facet at 5000×.*

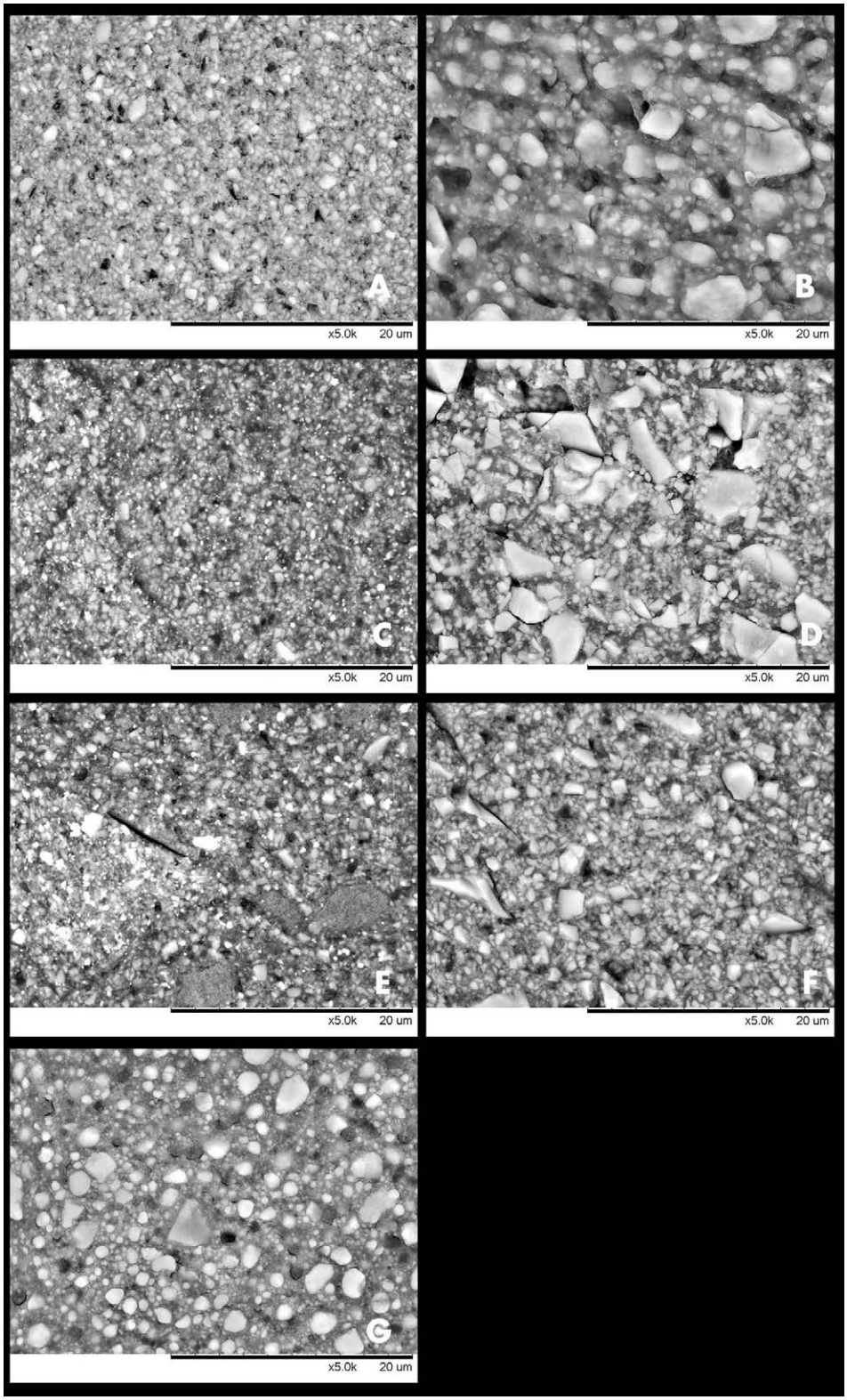

Figure 7. *(A): Esthet•X HD—generalized wear near center of facet at 5000×. (B): Filtek Supreme Ultra—generalized wear near center of facet at 5000×. (C): Herculite Ultra—generalized wear near center of facet at 5000×. (D): SonicFill—generalized wear near center of facet at 5000×. (E): Tetric EvoCeram Bulk Fill—generalized wear near center of facet at 5000×. (F): Venus Diamond—generalized wear near center of facet at 5000×. (G): Z100 Restorative—generalized wear near center of facet at 5000×.*

mic data of the polynomial fourth-degree function after regression analysis. Their *in vivo* wear rate of enamel was 29 μm per year for molar teeth and 15 μm per year for premolars. In 1997, Lambrechts[40] also reported that the wear of Z in occlusal contact areas is comparable with the OCA wear of enamel. In a previous study, Barkmeier and others[16] used regression analysis to examine localized simulated wear of Z (conical, hardened steel antagonist; r=1.575 mm) and found 9.6 μm of vertical loss per 100,000 cycles. Thus, 300,000 cycles of localized wear in the simulator (conical antagonist) would approximate one year of clinical tooth-to-tooth contact (OCA) wear of human molars, when using data from the Lambrechts studies.[39-40] In the present study, the average localized simulated wear rate of Z for 100,000 cycles was 14.9 μm using a stainless steel ball-bearing antagonist (r=2.387 mm). This value was calculated from the total wear of Z after 400,000 cycles, versus the earlier study of Z in which the slope of the regression curve was used for predicting the wear rate per 100,000 cycles. The wear rate per 100,000 cycles in the current study is greater than the previous study using regression analysis because it includes the initial rapid wear that is always present in these types of studies and is not indicative of long-term wear rates. Clinical wear studies of resin composite materials exhibit the same issues related to the initial wear-in period. Using the current study, the wear rate for localized wear simulation for 200,000 cycles would be approximately equivalent to the one-year wear rate of molar enamel (29 μm) reported in the Lambrechts studies.[39-40] The above comparisons are useful in helping to relate simulated localized wear testing of Z to natural enamel loss from tooth-to-tooth contact.

The components of the materials evaluated in this study are quite different. The SEM observations revealed differences in filler particle size and shape. Studies have shown that the type of glass, shape, and size of filler components, resin matrix formulation, and degree of polymerization have a significant effect on the wear characteristics of resin composite materials.[41-42] It is interesting to note the similarity of Z and SU on the argon-etched SEM examinations (Figure 5B,G). The manufacturing process appears to result in similarly shaped filler particles, which are mostly rounded. Table 2 shows that these materials of the same manufacturer both contain zirconia/silica filler particles. The study results (Tables 7-10) indicate that these two materials are very wear resistant in both localized and generalized wear simulation. It is also interesting to observe the similarity of the worn surfaces of these two materials after localized wear simulation. Both of these materials exhibit what appear to be fatigue cracks from the aggressive pounding of the stainless steel ball-bearing antagonist. Although these cracks might initially appear to result from a rapid breakdown of the material, the resistance to wear of these two materials suggests that these cracks are a result of a very wear-resistant material that eventually develops fatigue cracks after repeated challenges from the antagonist. Materials that are not as wear resistant may lose surface area faster and not develop crack formation on the surface.

The localized and generalized wear of SF was significantly greater ($p<0.05$) than that of the other materials evaluated in this study. In examining the scanning electron micrographs of SF after both localized and generalized wear simulation, there appears to be similarities in the surface characteristics. The observations of both localized (Figure 6D) and generalized (Figure 6D) wear show a surface with large glass filler particles and plucking of glass from the surface. In addition there is fracturing of the larger glass filler particles from both localized and generalized wear simulation.

This study demonstrated differences in the resistance to wear among modern resin composites and adds useful information regarding the relative wear performance of seven commonly used materials. The study results show clear differences in the wear characteristics among the materials examined (Tables 7-10). Although concern exists that these differences may not be predictive of clinical performance, the results are useful for practicing dentists in assessing the overall performance of the materials evaluated. However, there is good evidence that the relative wear resistance of resin composite materials found in wear simulation studies may translate well to the clinical setting.[11,16,18,23] Parameters other than wear may also guide practitioners in selecting restorative materials. But, because wear is so important in the long-term clinical performance of a resin composite material, the results of this study should be carefully considered by clinicians when selecting a resin composite material for patient care.

CONCLUSIONS

Seven resin composite materials were subjected to both simulated localized and generalized wear in a Leinfelder-Suzuki (Alabama) wear machine. Z was used as a benchmark material in this study due to excellent results in past simulated wear studies and clinical trials.

The results of this study provide useful additional information on the wear characteristics of modern-day resin composite materials. The benchmark material, Z, exhibited the least amount of wear in both localized and generalized wear simulations. The testing was able to discriminate wear performance among the seven materials tested. The results of this study, coupled with the results of previous laboratory and clinical wear studies, augment the information base available to the profession and provide guidance for clinicians in the selection of a resin composite material for patient care.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 5 November 2013)

REFERENCES

1. Phillips RW, Avery DR, Mehra R, Swartz ML, & McCune RJ (1973) Observations on a composite resin for Class II restorations: Three-year report *Journal of Prosthetic Dentistry* **30(6)** 891-897.
2. Lutz F, Phillips RW, Roulet JF, & Setcos JC (1984) In vivo and in vitro wear of potential posterior composites *Journal of Dental Research* **63(6)** 914-920.
3. Sakaguchi RL, Douglas WH, Delong R, & Pintado MR (1986) The wear of a posterior composite in an artificial mouth: A clinical correlation *Dental Materials* **2(6)** 235-240.
4. Lewis G (1991) In vivo occlusal wear of posterior composite restorations *Operative Dentistry* **16(2)** 61-69.
5. Kawai K, & Leinfelder K (1995) In vitro evaluation of OCA wear resistance of posterior composites *Dental Materials* **11(4)** 246-251.
6. Leinfelder KF, & Suzuki S (1999) In vitro wear device for determining posterior composite wear *Journal of the American Dental Association* **130(9)** 1347-1353.
7. Barkmeier WW, Latta MA, Wilwerding TM, & Blake SM (2001) Wear assessment of high viscosity and conventional composite restorative materials *Operative Dentistry* **26(2)** 152-156.
8. Nagarajan VS, Jahanmir S, & Thompson VP (2004) In vitro contact wear of dental composites *Dental Materials* **20(1)** 63-71.
9. Zantner C, Kielbassa AM, Martus P, & Kunzelmann KH (2004) Sliding wear of 19 commercially available composites and compomers *Dental Materials* **20(3)** 277-285.
10. Ferracane JL (2006) Is the wear of dental composites still a clinical concern? Is there still a need for in vitro wear simulating devices? *Dental Materials* **22(8)** 689-692.
11. Heintze SD, Barkmeier WW, Latta MA, & Rousson V (2011) Round robin test: Wear of nine restorative materials in six different wear simulators—Supplement to the round robin test of 2005 *Dental Materials* **27(2)** e1-e9.
12. Braem M, Lambrechts P, Van Doren V, & Vanherle G (1985) In vivo evaluation of four posterior composites: Quantitative wear measurements and clinical behavior *Dental Materials* **2(3)** 106-113.
13. Stangel I, Barolet RY, & Robert D (1990) A five-year evaluation of two posterior composites and amalgam *Journal of Dental Research* **69(Special issue)** Abstract #1600 p 308.
14. Tyas MJ, & Wassenaar P (1991) Clinical evaluation of four composite resins in posterior teeth: Five-year results. *Australian Dental Journal* **36(5)** 369-373.
15. Barnes DM, Blank LW, Thompson VP, Holston AM, & Gingell JC (1991) A 5- and 8-year clinical evaluation of a posterior composite resin *Quintessence International* **22(2)** 143-151.
16. Barkmeier WW, Latta MA, Erickson RL, & Lambrechts P (2004) Comparison of laboratory and clinical wear rates of resin composites *Quintessence International* **35(4)** 269-274.
17. Sarrett DC, Brooks CN, & Rose JT (2006) Clinical performance evaluation of a packable posterior composite in bulk-cured restorations *Journal of the American Dental Association* **137(1)** 71-80.
18. Barkmeier WW, Erickson RL, Latta MA, & Wilwerding TM (2008) Wear simulation of resin composites and the relationship to clinical wear *Operative Dentistry* **33(2)** 177-182.
19. Palaniappan S, Bharadwaj D, Mattar DL, Peumans M, Van Meerbeek B, & Lambrechts P (2009) Three-year randomized clinical trial to evaluate the clinical performance and wear of a nanocomposite versus a hybrid composite *Dental Materials* **25(11)** 1302-1314.
20. Opdam, NJ, Bronkhorst BA, & Huysmans MC (2010) 12-year survival of composite vs. amalgam restorations *Journal of Dental Research* **89(10)** 1063-1067.
21. Palaniappan S, Bharadwaj D, Mattar DL, Peumans M, Van Meerbeek B, & Lambrechts P (2011) Nanofilled and microhybrid composite restorations: Five-year clinical wear performances *Dental Materials* **27(7)** 692-700.
22. Da Rosa Rodolpho PA, Donassollo TA, Cenci MS, Loguércio AD, Moraes RR, Bronkhorst EM, Opdam NJ, & Demarco FF (2011) 22-year clinical evaluation of the performance of two posterior composites with different filler characteristics *Dental Materials* **27(10)** 955-963.
23. Heintze SD, Faouri M, Rousson V, & Özcan M (2012) Correlation of wear in vivo and six laboratory wear methods *Dental Materials* **28(9)** 961-973.
24. Barkmeier WW, Erickson RL, Latta MA, & Wilwerding TM (2013) Wear rates of resin composites *Operative Dentistry* **38(2)** 226-233.
25. Roulet JF (1987) *Degradation of Dental Polymers* Karger Press, Basel.

26. Lambrechts P, Vuylsteke M, Vanherle G, & Davidson CL (1984) Quantitative evaluation of the wear resistance of posterior dental restorations. A new three-dimensional measuring technique *Journal of Dentistry* **12(3)** 252-256.
27. Leinfelder KF, Taylor DR, Barkmeier WW, & Goldberg AJ (1986) Quantitative wear measurements of posterior composite resins *Dental Materials* **2(5)** 198-201.
28. de Gee AJ, Pallav P, & Davidson CL (1986) Effect of abrasion medium on wear of stress-bearing composites and amalgam in vitro *Journal of Dental Research* **65(5)** 654-658.
29. Pallav P, Davidson CL, & de Gee AJ (1988) Wear rates of composites, an amalgam, and enamel under stress-bearing conditions *Journal of Prosthetic Dentistry* **59(4)** 426-429.
30. Leinfelder KF, Beaudreau RW, & Mazer RB (1989) An in vitro device for predicting wear *Quintessence International* **20(10)** 755-761.
31. de Gee AJ, & Pallav P Occlusal wear simulation with the ACTA wear machine *Journal of Dentistry* **22(Supplement 1)** S21-S27.
32. Condon JR, & Ferracane JL. (1996) Evaluation of composite wear with a new multi-mode oral wear simulator *Dental Materials* **12(4)** 218-226.
33. Perry R, Kugel G, Kunzelmann KH, Flessa HP, & Estafan D (2000) Composite restoration wear analysis: Conventional methods vs three-dimensional laser digitizer *Journal of the American Dental Association* **131(10)** 1472-1477.
34. Suzuki S, Nagai E, Taira Y, & Minesaki Y (2002) In vitro wear of indirect restoratives *Journal of Prosthetic Dentistry* **88(40)** 431-436.
35. Heintze SD (2006) How to qualify and validate wear simulation devices and methods *Dental Materials* **22(8)** 712-734.
36. Rodriguez JM, Austin RS, & Bartlett DW (2012) A methods to evaluate profilometric tooth wear measurements *Dental Materials* **28(4)** 245-251.
37. Latta MA, Barkmeier WW, Wilwerding TM, & Blake SM (2001) Localized wear of compomer restorative materials *American Journal of Dentistry* **14(4)** 238-240.
38. Lambrechts P, Debels E, Van Landuyt K, Peumans M, & Van Meerbeek B (2006) How to simulate wear? Overview of existing method *Dental Materials* **22(8**) 693-701.
39. Lambrechts P, Braem M, Vuylsteke-Wauters M, Vanherle G (1989) Quantitative in vivo wear of human enamel *Journal of Dental Research* **68(12)** 1752-1753.
40. Lambrechts P (1997) Clinical update, 3M Restorative Z100 four-year quantitative wear. Report to 3M; Retrieved online Month Date, Year from: http://multimedia.3m.com/mws/mediawebserver?mwsId=66666UgxGCuNyXTtOxTcoxz6EVtQEcuZgVs6EVs6E666666–&fn=z100 clinical 4 year wear.pdf
41. Suzuki S, Leinfelder KF, Kawai K, & Tsuchitani Y (1995) Effect of particle size variation on wear rates of posterior composites *American Journal of Dentistry* **8(4)** 173-178.
42. Oliveira GU, Mondelli RF, Charantola RM, Franco EB, Ishikiriama SK, & Wang L (2012) Impact of filler size and distribution on roughness and wear of composite resin after simulated toothbrushing *Journal of Applied Oral Science* **20(5)** 510-516.

©Operative Dentistry, 2015, **40-3,** 336-337

Departments

Faculty Positions

DENTAL SCHOOL
UT HEALTH
SCIENCE CENTER®
SAN ANTONIO

Chair, Department of Comprehensive Dentistry, School of Dentistry at the University of Texas Health Science Center at San Antonio

The University of Texas Health Science Center at San Antonio (UTHSCSA) School of Dentistry seeks an individual with an outstanding record of scientific achievement, clinical expertise, scholarly accomplishments and mentoring as its new Chair of the Department of Comprehensive Dentistry.

Comprised of over 150 faculty and as the largest Department in the School of Dentistry, the Department of Comprehensive Dentistry is made up of three divisions – Pre-doctoral, Research, and Advanced Education and Outreach. The disciplines of general dentistry, restorative dentistry, prosthodontics, oral medicine/radiology, prevention, and community outreach are coordinated to provide a comprehensive educational experience for future dental practitioners. In July 2015, the School of Dentistry's $108 million, flagship clinical facility, the Center for Oral Health Care and Research is scheduled to open, offering an environment that supports the finest in multispecialty patient care and research.

Reporting to the Dean, the Chair is the administrative leader for this large and diverse department. The Chair is appointed to a full-time, tenure-track position. The successful candidate will be a nationally recognized scholar with a strong track record of academic excellence and professional accomplishment. Candidates with an outstanding record of administration in dental education including experience in operational and financial management are encouraged to apply. Candidates must have a clinical practice background in institutional and/or private practice settings and their teaching philosophy should be consistent with the concept as taught in the UTHSCSA School of Dentistry. He/she will have a Dental Degree recognized by the Commission on Dental Accreditation or equivalent foreign BDS or DDS training and should be eligible for dental licensing in the state of Texas.

Korn Ferry is assisting with this important search. All faculty appointments are designated as security sensitive positions. Please forward nominations of appropriate candidates, or expressions of interest to:

Heather Kopecky, PhD, MBA
c/o Angie Simmons (angie.simmons@kornferry.com)
Korn Ferry
700 Louisiana Street, Suite 3900
Houston, TX 77002

The University of Texas Health Science Center is an Equal Employment Opportunity/Affirmative Action Employer including protected veterans and persons with disabilities.

Group Practice Faculty College of Dental Medicine – Illinois

The College of Dental Medicine-Illinois (CDMI) at Midwestern University is a new predoctoral dental education program located in suburban Chicago, IL. CDMI is developing an innovative, integrated and patient-centered curriculum focusing on clinical excellence, critical thinking and ethical practice with an emphasis on oral health and the prevention and management of oral diseases in an evidence-based environment.

CDMI is seeking full-time clinical faculty to serve in the Group Practice student clinics at the rank of Instructor, Assistant, or Associate Professor. Primary responsibility is for the overall well-being of the patients being treated. The Group Practice faculty member has responsibility for instruction and demonstration in one-on-one, small group and plenary settings. The Group Practice faculty will supervise student dentists and provide direct patient care as required by the degree of difficulty of certain cases or for demonstration purposes. There is an opportunity to engage in

scholarly activity, as deemed appropriate and as mutually agreed upon by the Group Practice Faculty and CDMI administration.

Candidates must possess a DMD/DDS degree, excellent clinical skills and at least 3 years experience in dental practice and/or dental education. GPR or AEGD training is desirable. This individual must be eligible for a license to practice dentistry in the state of Illinois or be eligible for a restricted faculty license. Excellent interpersonal and collegial attributes are essential along with a patient and learner-centered focus, in a humanistic environment. Experience with electronic patient records and educational software programs will be beneficial.

Appointment at the Instructor, Assistant, or Associate Professor level will be based on individual experience and credentials. Midwestern University offers competitive salaries and opportunities exist for advancement.

Interested individuals should submit a letter of application, curriculum vitae, and 3 professional references to:

Midwestern University
Dental Institute
Attn: Dr. Darryn Weinstein
3450 Lacey Rd., Suite 120
Downers Grove, IL 60515

Applications can also be made on-line at: https://www4.recruitingcenter.net/Clients/midwestern/PublicJobs/Canviewjobs.cfm?

We are proud to be an EEO/AA employer M/F/D/V. We maintain a drug-free workplace.

Chair – Department of Adult Restorative Dentistry, UNMC College of Dentistry

The University of Nebraska Medical Center College of Dentistry seeks applications for the full-time/tenure track position as Chair, Department of Adult Restorative Dentistry. The department includes biomaterials, operative dentistry, prosthodontics, priority care, TMD oral facial pain, and assessment and treatment planning. Responsibilities for the department include pre-doctoral education, service activities, clinical care and research mentorship. The Chair is expected to provide leadership in strategic planning, budgeting, department staffing and supervision, faculty development, and overall management of departmental activities. We seek a leader who will provide vision and focus to build upon a strong team and advance the mission of the department and college. The position reports to the Dean, College of Dentistry. Applicants must have a D.D.S./D.M.D. degree or foreign equivalent degree. Completion of an ADA-accredited advanced education program in Operative Dentistry, Prosthodontics, or General Dentistry is preferred. An established history of practice in dentistry, extramural research funding, and an advanced degree are highly desirable qualifications. Dental licensure in Nebraska is a requirement of appointment. Practice opportunities are available. Salary and academic rank will be commensurate with training and experience. Screening of applications will begin immediately with an expected hire date of July 1, 2015. Applications are being accepted online at: jobs.unmc.edu, position #2015-038. Individuals from diverse backgrounds are encouraged to apply.

Special Issue Introduction

As stated in Issue 1, we are celebrating our 40th year of publishing Operative Dentistry. One of the ways we have chosen to recognize this accomplishment is to present "The Best of the Best" articles published over the last 40 years. We have two reviewers, Drs. Melvin Lund and Hal Laswell, who have reviewed for us the entire forty years. We tasked them with being Guest Editors - to put together a Special Issue by selecting those superlative articles. With the help of specially invited individuals, they have completed this task and the finished compilation will be available online as part of your subscription.

This Special Issue contains published manuscripts that kept us informed or made us think about our practice of dentistry and how we could continue to improve towards excellence, to provide the best of care for our patients, and to satisfy our craving for new knowledge. The authors have shaped operative dentistry through their articles, and provided a body of work that still resonates with clinicians today.

This 48 article special issue publication comes as an attachment to your journal subscription and will be available online for all 2015 subscriptions. Printed copies will be available outside of any subscription and will cost 85.00USD. Those who are not subscribers to Operative Dentistry, the Paper version will be available for 85.00USD, the online version for 85.00USD or both for 125.00USD. Please direct any subscription inquiries to the office staff at editor@jopdent.org. Discounts will be available for orders of 10+ issues.

The Staff of Operative Dentistry

©Operative Dentistry, 2015, **40-3,** E83-E91

Inlays Made From a Hybrid Material: Adaptation and Bond Strengths

MA Bottino • F Campos • NC Ramos
MP Rippe • LF Valandro • RM Melo

Clinical Relevance

Polymer-infiltrated ceramics appear to be promising materials for inlays since they present marginal and internal adaptation results that are better than those of feldspathic ceramic. However, the bond strength values of the latter were higher than those of the former which is an important aspect for the longevity of a restoration.

SUMMARY

The aim of this study was to evaluate the internal fit, marginal adaptation, and bond strengths of inlays made of computer-aided design/computer-aided manufacturing feldspathic ceramic and polymer-infiltrated ceramic. Twenty molars were randomly selected and prepared to receive inlays that were milled from both materials. Before cementation, internal fit was achieved using the replica technique by molding the internal surface with addition silicone and measuring the cement thicknesses of the pulpal and axial walls. Marginal adaptation was measured on the occlusal and proximal margins of the replica. The inlays were then cemented using resin cement (Panavia F2.0) and subjected to two million thermomechanical cycles in water (200 N load and 3.8-Hz frequency). The restored teeth were then cut into beams, using a lathe, for microtensile testing. The contact angles, marginal integrity, and surface patterns after etching were also observed. Statistical analysis was performed using two-way repeated measures analysis of variance ($p<0.05$), the Tukey test for internal fit and marginal adaptation, and the Student *t*-test for bond strength. The failure types (adhesive or cohesive) were classified on each fractured beam. The results showed that the misfit of the pulpal walls ($p=0.0002$) and the marginal adaptation ($p=0.0001$) of the feldspathic ceramic were

Marco Antonio Bottino, DDS, MSD,PhD, professor and chair, Department of Dental Materials and Prosthodontics, São Jose dos Campos Dental School, São Paulo State University, São Jose dos Campos, Brazil

Fernanda Campos, DDS, MSD, PhD, graduate student in Prosthodontics, São Jose dos Campos Dental School, São Paulo State University, São Jose dos Campos, Brazil

Nathália Carvalho Ramos, MSD, Department of Dental Materials and Prosthodontics, São Jose dos Campos Dental School, São Paulo State University, São Jose dos Campos, Brazil

Marilia Pivetta Rippe, DDS, MSc, PhD, Federal University of Santa Maria, Restorative Dentistry (Prosthodontics), Santa Maria, Brazil

*Luiz Felipe Valandro, PhD, Federal University of Santa Maria, Restorative Dentistry (Prosthodontics), Santa Maria, Brazil

Renata Marques de Melo, DDS, MSD, PhD, researcher, Department of Dental Materials and Prosthodontics, São Jose dos Campos Dental School, São Paulo State University, São Jose dos Campos, Brazil

*Corresponding author: R. Floriano Peixoto 1184, Santa Maria, RS 97015-372, Brazil; e-mail: lfvalandro@hotmail.com

DOI: 10.2341/13-343-L

significantly higher when compared to those of the polymer-infiltrated ceramic, while the bond strength values of the former were higher when compared to those of the latter. The contact angle of the polymer-infiltrated ceramic was also higher. In the present study, the hybrid ceramic presented improved internal and marginal adaptation, but the bond strengths were higher for the feldspathic ceramic.

INTRODUCTION

An array of materials, such as ceramics, composites, and metal alloys, can be used for the indirect restoration of class I and class II cavities.[1] Although metals show good clinical results, today's patients are looking for more aesthetic materials.[2] Therefore, ceramics and composites, which have excellent aesthetic properties, are the materials of choice. Ceramic inlays are mainly made of feldspathic or lithium disilicate–based ceramics and present compressive and wear resistance higher than those of composite inlays.[3] Conversely, composite inlays are less susceptible to fracture and cause less wear to the opposing tooth.[3,4]

Therefore, manufacturers are attempting to incorporate various types of materials into the ceramic and composite matrix to address these shortcomings. The combination of polymers and ceramics results in greater strength and better load distribution, which reduces cracks and fractures.[5] The first hybrid ceramic material, which combines the characteristics of ceramics and polymers, was recently made available commercially. According to the manufacturer of that material, its composition is approximately 14% composite, which is distributed into a ceramic network, and it is indicated for inlays, onlays, veneers, and crowns (Vita Enamic, Vita Zahnfabrik, Bad Säckingen, Germany).

In addition to mechanical properties, other characteristics are desirable in these materials. For example, good marginal adaptation and bond strength to teeth are essential for the longevity of restorations.[6] Periodontal diseases, secondary caries, and endodontic problems can be caused by poor marginal adaptation through the accumulation of biofilm or the penetration of fluids from the oral cavity.[7,8] Even with the evolution of computer-aided design/computer-aided manufacturing (CAD/CAM) technology, in which restorations can be milled with fewer defects due to the homogeneity of the materials used,[9] achieving excellent marginal adaptation is still difficult.[2,10] The manufacturer claims that the new hybrid material presents improved machinability, which, in turn, results in improved marginal adaptation.

An adequate bond strength between ceramic and tooth structure is achieved with acid etching (hydrofluoric acid) and silanization.[11] This surface treatment has led to successful restorations, reducing catastrophic tooth fracture after cusp deflection.[9] Another important aspect of adequate bonding is that adequate adhesion between tooth and inlay results in less microleakage.[9] However, it is not known how the presence of the polymeric material in the ceramic matrix can affect the bond strength to resin cements.

A recent study[12] stated that the volume fraction of a polymer-infiltrated material is low; thus, its hardness was achieved mainly by the ceramic because the indenter had more chances to fall on the ceramic portion of the material. Nevertheless, taking into account that it is a new material, there are no studies in the literature showing its performance with regard to bonding and adaptation.

Thus, the aim of this study was to evaluate the adaptation of feldspathic ceramic and polymer-infiltrated ceramic inlays, fabricated using the CAD/CAM system, and to evaluate the bond strength to dentin after adhesive cementation. The hypotheses were that the type of material would not influence 1) the internal adaptation, 2) the marginal adaptation, and 3) the microtensile bond strength.

METHODS AND MATERIALS

The commercial names, types, manufacturers, and batch numbers of the materials used are listed in Table 1. Twenty human maxillary molars were selected, according to the inclusion criteria of no visible cracks or decay. The specimens were randomly divided into two groups (http://www.randomizer.org), according to the type of material (n=10): Group EN = Hybrid ceramic material (Vita Enamic), and Group VM = Feldspathic ceramic material (VitaBlock Mark II). The teeth were cleaned with chloramine 2% for one week in distilled water and stored under refrigeration.

The teeth were embedded in a cylinder (h=14 mm, Ø=25 mm) containing polyurethane resin (F16 Polyol, Axson Technologies, Saint Ouen l'Aumône, France) up to 3 mm below the cemento-enamel junction, with the occlusal surface parallel to the horizontal plane.

Table 1: *Commercial Names, Types, and Manufacturers of the Materials Used in the Study.*

Commercial Name	Type	Manufacturer	Batch Number
Vita Enamic	Hybrid ceramic	Vita Zahnfabrik, Germany	36660
VitaBlock Mark II	Feldspathic ceramic	Vita Zahnfabrik, Germany	35370
Condac	Hydrofluoric acid 10%	FGM, Brazil	060912
Monobond S	Silane	Ivoclar, Liechtenstein	P70737
ED primer	Adhesive system	Kuraray, Japan	00310A
			00184A
Panavia F2.0	Resin Cement	Kuraray, Japan	00255A
			00033A
Elite HD	Addition silicone	Zhermack, Italy	149677
			138448
			154369

Standardized cavity preparations (inlay type) were prepared in the teeth using a conical trunk diamond bur with rounded angles (KG Sorensen 3131, Barueri, São Paulo, Brazil). The burs were mounted in a high-speed hand piece fixed to a modified optical microscope. The preparations had the following dimensions: buccal-lingual width, 3 mm; occlusal box depth, 3 mm; and rounded internal line angles. Each diamond bur was used for the preparation of three teeth.

The cavities were impressed using addition silicone and a one-step impression (Elite HD + Regular Body, Zhermack, Rovigo, Badia Polesine, Italy), and the impressions were poured using type IV die stone (Durone IV, Dentsply, Petrópolis, Rio de Janeiro, Brazil). These casts were sprayed with scanning powder (Optispray CEREC, Sirona Dental Systems, Benshein, Hessen, Germany) and optically captured by scanning (inEos Blue, Sirona Dental Systems). The image was sent to the computerized unit (CAD) in numeric values, which formed a three-dimensional virtual model. The cement space in the software of the CAD/CAM system was programmed at 80 μm for all groups, according to the CEREC manufacturer. Ten restorations were made of each material in the CEREC Inlab milling machine (MC XL model, Sirona Dental Systems).

Internal Fit and Marginal Adaptation Measurements

Internal fit was measured using the replica technique and prior to cementation.[13] The tooth preparation was filled with a thin layer of light-body addition silicone, and the inlay was placed using a load of 750 g. After the impression material set, the inlay was removed, leaving a thin film of silicone adhering to the preparation, representing the space between the inlay and the tooth cavity. For the purpose of stabilization, a medium-body material was placed in the space previously occupied by the inlay, which adhered to the light-body film. With this procedure, it was possible to remove the replica of the light-body material. The replica was then cut mesio-distally, and one half-section was used to measure the thickness at the pulpal wall. Each section was then cut into three parts, and the middle section was used to measure the thickness at the axial walls (Figure 1). The measurements were performed using stereomicroscopy (Discovery V20, Carl Zeiss, Jena, Thuringia, Germany; 10-20×). The average cement thickness of the pulpal and axial walls of each tooth (two means per tooth) was used in the statistical analysis.

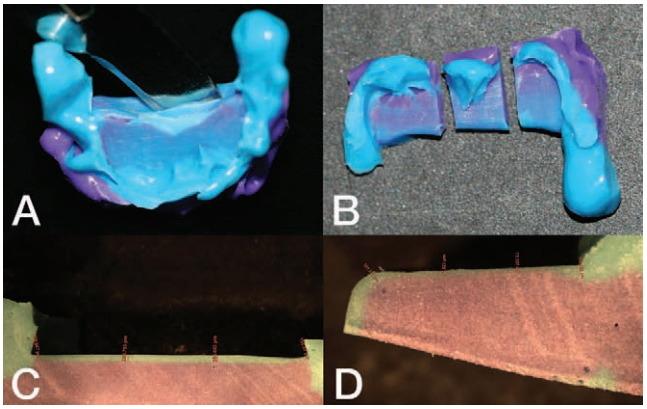

Figure 1. *Internal adaptation measurement. The replica (A) was cut in the middle (mesio-distally), and a section was used to measure the thickness at the pulpal wall (c). Each half (a) was then cut into three parts (b), and the middle section was used to measure the thickness along the axial walls (d).*

For marginal adaptation, stereomicroscopy (Discovery V20, Carl Zeiss; 10-20×) was used to measure the distance between the inlay border and the preparation margin. The marginal adaptation was measured at four sites of the occlusal region and at the proximal margins (four sites on the buccal, pulpal and lingual walls). The average of each region, occlusal and proximal, was used in the statistical analysis.

Cementation

The ceramic inlays were cemented with an adhesive. The intaglio surfaces of the inlays were etched with 10% hydrofluoric acid (IPS Ceramic Etching,

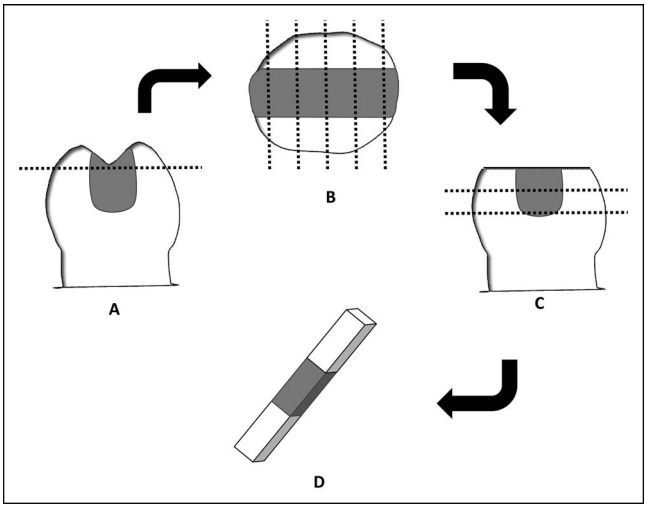

Figure 2. *Schematic representation of the restored tooth sectioning. (A): Proximal view of the restored tooth. The dashed line shows the direction of the first section, which removed the occlusal portion. (B): Occlusal view of the restored tooth. The dashed lines show the sectioning from occlusal to apical. The distance between the sections was approximately 1.0 mm. (C): Proximal view of the restored tooth. The dashed lines show the direction of the sectioning. The distance between each section was approximately 1.0 mm. (D): Stick representation, where the middle portion was the restoration.*

Ivoclar Vivadent) for one minute, washed, and gently air-dried. A silane agent (Monobond S, Ivoclar Vivadent) was then actively applied for 20 seconds, and, after one minute, an air spray was used. Next, ED Primer (Kuraray Medical Inc., Okayama, Tokyo, Japan) bond agent was applied on the enamel/dentin surfaces, the resin cement, Panavia F2.0 (Kuraray Medical Inc., Okayama, Tokyo, Japan), was mixed according to the manufacturer's instructions and applied to the intaglio surface of the inlay. The restoration was then placed, and a load of 750 g was applied over the inlay/tooth. Excess cement was removed, and photoactivation of the remaining cement was performed for 120 seconds.

The specimens were positioned in a thermomechanical cycling machine (Erios, model RE-37000), and a load was applied along the long axis of the tooth. Loading was performed for two million mechanical cycles, under a 200 N load and a frequency of 3.8 Hz, concomitantly with thermal cycles of 30-second baths at 5°C, 37°C, and 55°C, with 30-second intervals between them.

Microtensile Bond Strength Test

The specimens were fixed to a cylindrical metal base coupled to a cutting machine (Isomet, Düsseldorf, North Rhine-Westphalia, Germany) using cyanoacrylate (Super-Bonder Gel, Loctite, São Paulo, Brazil; Figure 2). The crown was sectioned in the x and y axes to produce bar specimens characterized with a nontrimmed interface and bar specimens composed of vestibular dentin, with ceramic in the middle and lingual dentin, comprising a cross-sectional bonded area of 1 mm^2.

The bar specimens were glued to the adapted device and submitted to the microtensile bond strength test (Emic DL-2000, Emic) at a speed of 0.5 mm/min until the sample fractured. The calculated bond strength of each tooth was the average bond strength of all bar specimens.

Failure Analysis

The fractured specimens were analyzed using stereomicroscopy (Discovery V20, Carl Zeiss; 25×). The types of failures were classified as adhesive (between the dentin and the cement or between the inlay and the cement), cohesive (dentin, inlay, or cement), and mixed (cohesive + adhesive failure). The most representative failures were analyzed using scanning electron microscopy (Inspect S50, FEI Company, Brno, Moravia, Czech Republic).

Contact Angle

For contact angle analysis, one disc (10 mm in diameter, 3 mm in thickness) of each material was milled in the CAD/CAM system. The contact angle was measured by means of a goniometer (Thetalite II Biolin Scientific Inc, Baltimore, MD, USA) in a controlled temperature environment, and the goniometer was connected to a computer equipped with specific software (One Attension, Biolin Scientific, Stockholm, Sweden) using the sessile drop technique. The measurements were made after surface treatment with 10% hydrofluoric acid for 60 seconds. A drop of distilled water was placed on the ceramic surface using a syringe, and, after 10 seconds, the contact angle was measured for 10 seconds (30 frames per second).

Micromorphology of the Etched Surface and Marginal Integrity

For analysis of the etched surfaces of the materials used, discs were viewed under 3000× magnification (Inspect S50, FEI Company). Further, for assessment of the marginal integrity of the restorations after milling, the inlays were analyzed under 220× magnification (Inspect S50, FEI Company).

Table 2: *Internal Adaptation (μm), Marginal Adaptation (μm), and Bond Strength (MPa) Mean Values (Standard Deviations in Parentheses)*

Groups	Internal Adaptation		Marginal Adaptation		Bond Strength
	Pulp	Axial	Occlusal	Proximal	
Enamic	124.0 (18) B	130.1 (26.7) B	163.1 (53) B	159.6 (36.5) B	5.45 (3.4) B
Vita Mark	210.6 (75.3) A	137.8 (40.2) B	222.5 (46) A	208.9 (54.9) A	10.16 (5.4) A

[a] *Different letters indicate a significant difference ($p<0.05$) between material types and tooth region in relation to internal adaptation, marginal adaptation, or bond strength.*

Statistical Analyses

The mean values of internal and marginal adaptation of the experimental groups were subjected to two-way repeated measures analysis of variance, with "material" as an independent factor and "region measured" as a dependent factor, and by the Tukey test ($\alpha=0.05$). The microtensile bond strength mean values were subjected to the Student *t*-test, with the tooth as the experimental unit (n=10).

RESULTS

The factor "material" was statistically significant in relation to internal fit ($p=0.0001$). Group EN presented the lowest values of internal gap (Table 2). The factor "region measured" was statistically significant ($p=0.003$). However, only the VM axial and VM pulpal groups presented statistically significant differences ($p=0.0002$) since the VM pulp group showed the greatest cement thickness.

The factor "material" was also statistically significant in relation to marginal adaptation ($p=0.0001$). The group EN presented the lowest values of marginal gap (Table 2). In this case, the factor "region measured" was not statistically significant ($p=0.359$).

The mean values and standard deviations for the bond strengths of the experimental groups are listed in Table 2. Statistical analysis showed that the bond strengths were statistically significantly different ($p=0.03$).

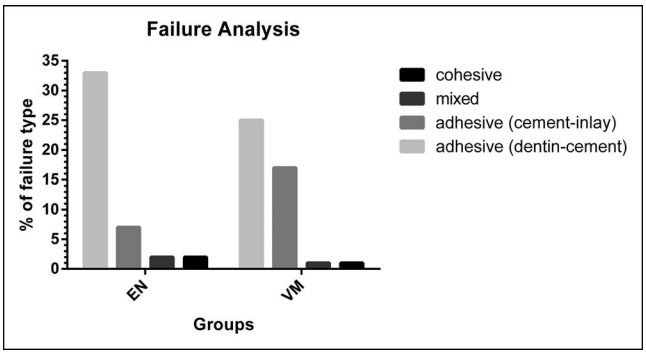

Figure 3. *Graphic representation of the failure types.*

The types of failures were similarly distributed in the groups (Figure 3). Representative micrographs are shown in Figure 4.

The contact angles (EN=61.91° and VM=12.68°) were different for the materials (Figure 5A,B).

The micrographs of the etched surfaces are displayed in Figure 5C,D. The surface patterns of the materials were noticeably different.

The marginal integrity after milling (Figure 5E,F) differed slightly, as VM seemed to have more irregular borders than EN.

DISCUSSION

Adaptation is critical to the success of a restoration. Large gaps at the interface between the cement and inlay may cause dehydration shrinkage, increased water sorption, plasticity, or hygroscopic expansion of the cement. Therefore, the lack of marginal integrity at the inlay–cement interface can lead to restoration failure.[14]

The aim of this present study was to evaluate the marginal adaptation of inlays made from feldspathic ceramic and a polymer-infiltrated ceramic machined in a CAD/CAM system and to evaluate the bond strength to dentin after adhesive cementation. The first hypothesis—that there was no difference between the materials for internal fit of the restorations—was rejected. The hybrid material showed better internal adaptation, confirming the characteristics given by the manufacturer. According to Coldea and others[12] and Dirxen and others,[15] feldspathic ceramic has an average hardness greater than 6 GPa, while the polymer-infiltrated-ceramic-network material, or hybrid material, has an average hardness value of 2.5 GPa. The modulus of elasticity of the hybrid ceramic is similar to that of dentin, which makes the stress distribution very different from that of a feldspathic ceramic (more brittle). This lower hardness value and modulus of elasticity may

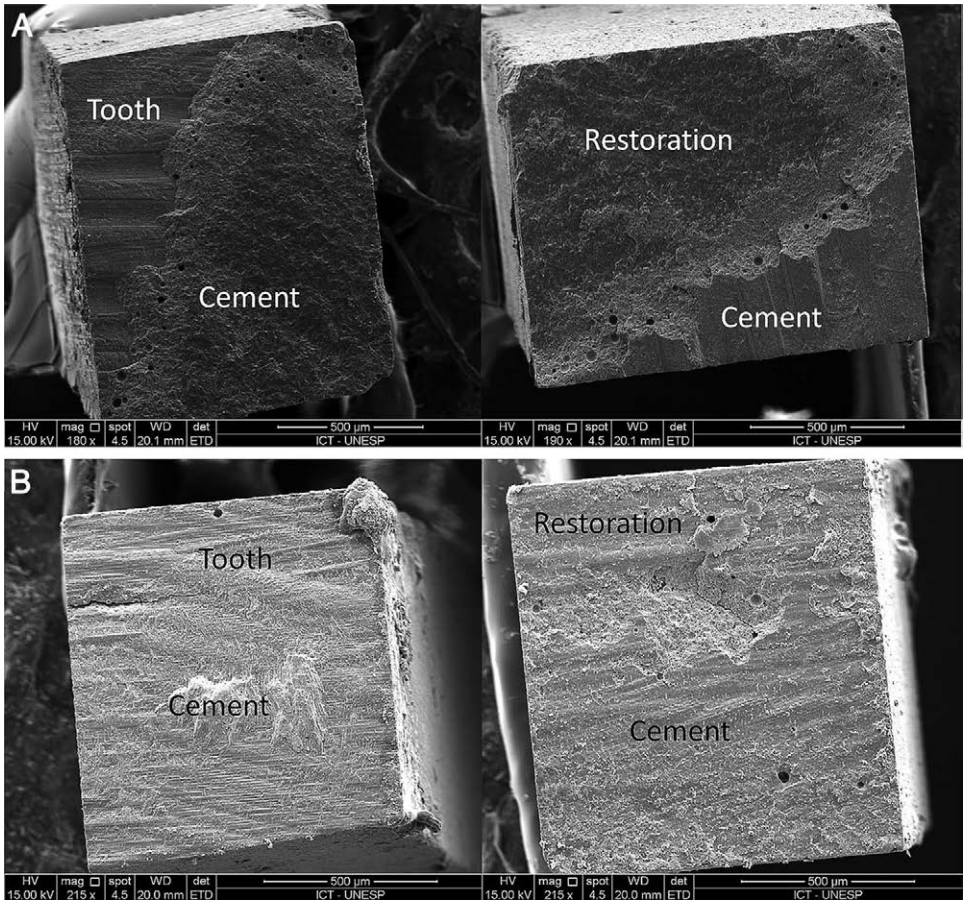

Figure 4. *Micrographs of the failure types. Opposite sides of the same beam: Vita Mark II (A) and Enamic (B). (A) Adhesive failure predominantly between restoration and cement. (B) Adhesive failure predominantly between dentin and cement.*

represent better machinability, leading to more accurate internal adaptation of the hybrid material when compared with the ceramic.

Ideally, the cement thickness must be lower than 100 μm to ensure satisfactory clinical results.[13] If the cement thickness exceeds this value, the shrinkage increases, which may cause deflection of the cusps due to shrinkage stress. Vanlioglu and others[16] used a cement thickness of 120 μm to compare adaptation of onlays made of lithium disilicate. In the present study, the cement thickness used was 80 μm; with this amount of relief, restorations needed no adjustment for complete seating. This space was recommended by the CEREC machine manufacturer (MC XL model, Sirona Dental Systems). Furthermore, the powder used to capture the image by the digital scanner may have contributed to the increased thickness.[17] However, the application of the powder and the cement space were the same for all groups; therefore, the comparison was not impaired.

The second hypothesis of the present study was also rejected because the type of material influenced marginal adaptation. The EN group presented statistically lower values of marginal gap when compared to the VM group. According to Enamic's manufacturer (Vita Zahnfabrik), this material delivers significantly better marginal precision after milling, creating thinner margins. This better marginal accuracy can be seen in Figure 5E, which shows that the restoration's margin is smoother than that shown in Figure 5F, which represents the feldspathic ceramic.

The materials did not perform the same in the microtensile experiment since higher mean values were obtained in the VM group, leading to the rejection of the third hypothesis. The differences in microstructure of the materials could explain this result. The ceramic material used in the VM group has a microstructure based on fine-structure feldspar ceramic, while EN is a "hybrid" material containing a silicate ceramic matrix filled with polymer, which includes triethylene glycol dimethacrylate (TEGDMA) and urethane dimethacrylate (UDMA). Although there are clear microstructural differences, the similar clinical indications for these materials enable the statistical comparison of bond

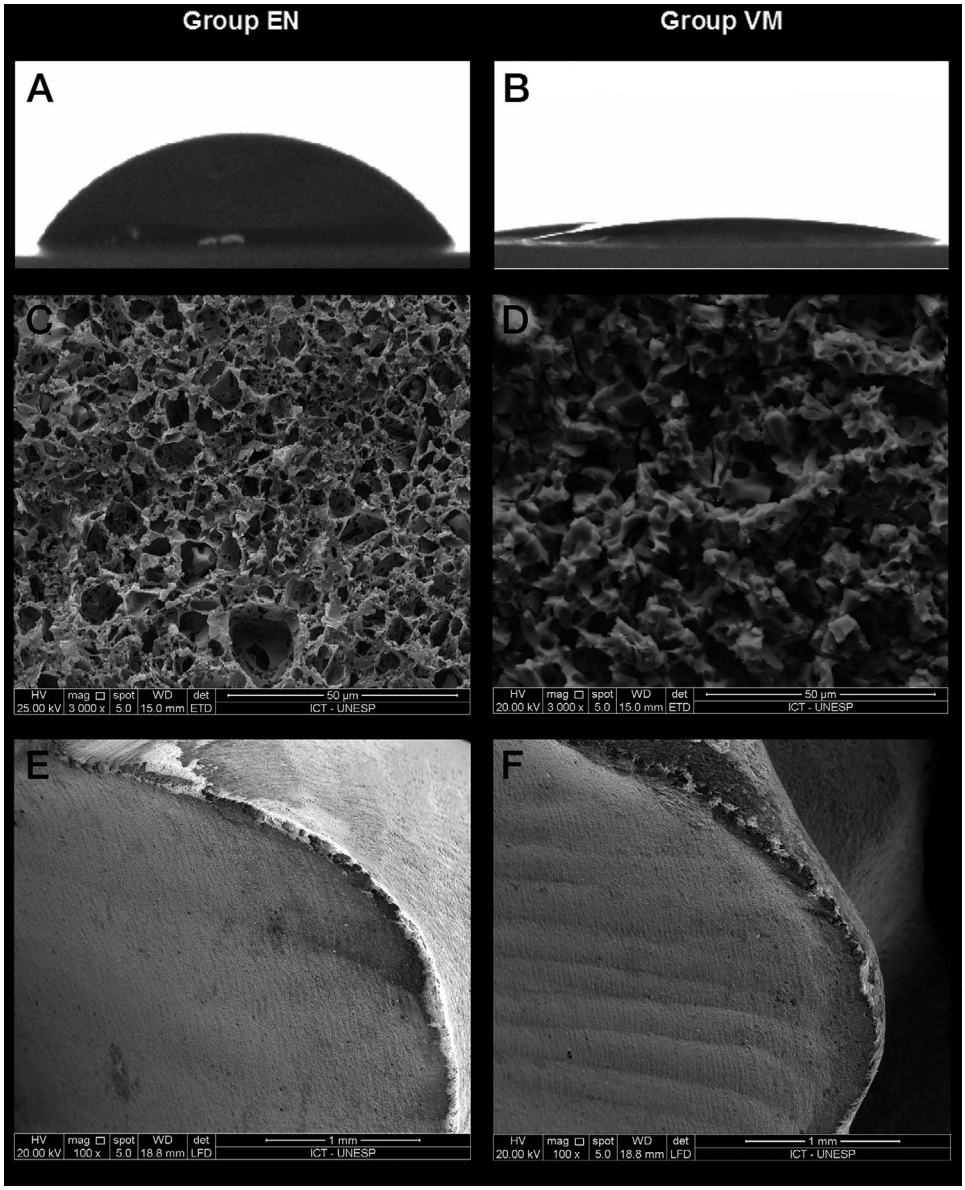

Figure 5. *Contact angles on EN (A) and VM (B). (C): Micrograph of the etched surfaces of EN (C) and VM (D); micrographs of the inlay border after milling of EN (E) and VM (F).*

strengths. However, it is possible that the bonding performance of EN is closer to that presented by polymeric materials; this requires further investigation.

The inlay model was preferred for the microtensile model used in the current study, even knowing that the adhesion interface has a slightly inclined plane due to the expulsive characteristics of the cavities. The current model simulates the C-factor cavity configuration present in the clinical situation, which is often disregarded in studies that use flat tooth surfaces. The bond strength values obtained in this present study were similar to those reported in other studies that used the same scenario, with bar specimens derived from inlay restorations, having a bond strength range of 5-10 MPa.[18,19]

The mechanism of adhesion between feldspar ceramic and resin cement has been established in the literature.[11,18,20] Surface treatment with hydrofluoric acid etching increases surface roughness by selective conditioning of the glassy phase contained in the ceramic.[11] This was observed in the etched ceramic micrograph. In addition, this procedure improves the wettability and the surface-free energy.[21] This was also demonstrated by contact angle analysis since the etched ceramic attained a small angle. On the contrary, the contact angle values of EN were high and were even worse after etching. This indicates that, after surface conditioning, the

resin net was even more dominant because of glass dissolution, making the surface more hydrophobic.

It would be expected that adhesion between the hybrid material and the resin cement was higher due to the chemical interaction between the polymers present in both materials. The etched surface topography contributes to this belief. Thus, it is believed that the highly polymerized resin matrix (UDMA and TEGDMA) did not present many reactive bonds that could interact with the resin cement, thereby diminishing bond strength.[22]

Therefore, from the evaluation of failure modes, it appears that the weakest link was the cement–dentin interface due to the highest percentage of failures and since all failure types occurred with practically the same frequency for both materials. Thus, the evaluation of failure modes helped elucidate questions about the performance of the adhesive interface, meaning that if total adhesive failures occurred, poor adhesion quality was achieved.[23,24] It is interesting that the adhesive failures encountered in the present study were not purely adhesive but were only "predominantly" adhesive. This can be explained by the test geometry.[25]

Further studies should be conducted with other test geometries and other types of surface treatments for the hybrid ceramic, which can improve the wettability and surface energy of this material. Improving bond strength values, or at least approximating this value to that of feldsphatic ceramics, will promote stronger bonding and produce less degradable interfaces.[2]

CONCLUSION

Within the limitations of this study, the internal and marginal accuracy of the hybrid ceramic was better than the feldspathic ceramic. However, the bond strength after fatiguing the specimens was higher for the feldspathic ceramic, which can have a greater impact in the success of the restoration if longer periods of time are considered.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 9 June 2014)

REFERENCES

1. Fron Chabouis H, Smail Faugeron V, & Attal JP (2013) Clinical efficacy of composite versus ceramic inlays and onlays: A systematic review *Dental Materials* **29(12)** 1209-1218.
2. Hopp CD, & Land MF (2013) Considerations for ceramic inlays in posterior teeth: A review *Clinical, Cosmetic and Investigational Dentistry* **5** 21-32.
3. Magne P, & Belser UC (2003) Porcelain versus composite inlays/onlays: Effects of mechanical loads on stress distribution, adhesion, and crown flexure *International Journal of Periodontics and Restorative Dentistry* **23(6)** 543-555.
4. Mormann WH, Stawarczyk B, Ender A, Sener B, Attin T, & Mehl A (2013) Wear characteristics of current aesthetic dental restorative CAD/CAM materials: Two-body wear, gloss retention, roughness and Martens hardness *Journal of the Mechanical Behavior of Biomedical Materials* **20(April)** 113-125.
5. Mark JE (1996) Ceramic-reinforced polymers and polymer-modified ceramics *Polymer Engineering and Science* **36(24)** 2905-2920.
6. Pera P, Gilodi S, Bassi F, & Carossa S (1994) In vitro marginal adaptation of alumina porcelain ceramic crowns *Journal of Prosthetic Dentistry* **72(6)** 585-590.
7. Bindl A, & Mormann WH (2005) Marginal and internal fit of all-ceramic CAD/CAM crown-copings on chamfer preparations *Journal of Oral Rehabilitation* **32(6)** 441-447.
8. Kokubo Y, Ohkubo C, Tsumita M, Miyashita A, Vult von Steyern P, & Fukushima S (2005) Clinical marginal and internal gaps of Procera AllCeram crowns *Journal of Oral Rehabilitation* **32(7)** 526-530.
9. El Zohairy AA, De Gee AJ, Mohsen MM, & Feilzer AJ (2003) Microtensile bond strength testing of luting cements to prefabricated CAD/CAM ceramic and composite blocks *Dental Materials* **19(7)** 575-583.
10. Zaruba M, Kasper R, Kazama R, Wegehaupt FJ, Ender A, Attin T, & Mehl A (2013) Marginal adaptation of ceramic and composite inlays in minimally invasive mod cavities *Clinical Oral Investigations* **18(2)** 579-587.
11. Brentel AS, Ozcan M, Valandro LF, Alarca LG, Amaral R, & Bottino MA (2007) Microtensile bond strength of a resin cement to feldspathic ceramic after different etching and silanization regimens in dry and aged conditions *Dental Materials* **23(11)** 1323-1331.
12. Coldea A, Swain MV, & Thiel N (2013) Mechanical properties of polymer-infiltrated-ceramic-network materials *Dental Materials* **29(4)** 419-426.
13. Souza RO, Ozcan M, Pavanelli CA, Buso L, Lombardo GH, Michida SM, Mesquita AM, & Bottino MA (2012) Marginal and internal discrepancies related to margin design of ceramic crowns fabricated by a CAD/CAM system *Journal of Prosthodontics* **21(2)** 94-100.
14. Rosentritt M, Behr M, Lang R, & Handel G (2004) Influence of cement type on the marginal adaptation of all-ceramic MOD inlays *Dental Materials* **20(5)** 463-469.
15. Dirxen C, Blunck U, & Preissner S (2013) Clinical performance of a new biomimetic double network material *The Open Dentistry Journal* **7** 118-122.

16. Vanlioglu BA, Evren B, Yildiz C, Uludamar A, & Ozkan YK (2012) Internal and marginal adaptation of pressable and computer-aided design/computer-assisted manufacture onlay restorations *International Journal of Prosthodontics* **25(3)** 262-264.
17. Hembree JH Jr (1995) Comparisons of fit of CAD-CAM restorations using three imaging surfaces *Quintessence International* **26(2)** 145-147.
18. Feitosa SA, Corazza PH, Cesar PF, Bottino MA, & Valandro LF (2013) Pressable feldspathic inlays in premolars: Effect of cementation strategy and mechanical cycling on the adhesive bond between dentin and restoration *Journal of Adhesive Dentistry* **16(2)** 147-154.
19. Saavedra G, Ariki EK, Federico CD, Galhano G, Zamboni S, Baldissara P, Bottino MA, & Valandro LF (2009) Effect of acid neutralization and mechanical cycling on the microtensile bond strength of glass-ceramic inlays *Operative Dentistry* **34(2)** 211-216.
20. Souza RO, Castilho AA, Fernandes VV, Bottino MA, & Valandro LF (2011) Durability of microtensile bond to nonetched and etched feldspar ceramic: Self-adhesive resin cements vs conventional resin *Journal of Adhesive Dentistry* **13(2)** 155-162.
21. Phoenix RD, & Shen C (1995) Characterization of treated porcelain surfaces via dynamic contact angle analysis *International Journal of Prosthodontics* **8(2)** 187-194.
22. Rinastiti M, Ozcan M, Siswomihardjo W, & Busscher HJ (2010) Immediate repair bond strengths of microhybrid, nanohybrid and nanofilled composites after different surface treatments *Journal of Dentistry* **38(1)** 29-38.
23. Nikolaus F, Wolkewitz M, & Hahn P (2013) Bond strength of composite resin to glass ceramic after saliva contamination *Clinical Oral Investigations* **17(3)** 751-755.
24. Klosa K, Wolfart S, Lehmann F, Wenz HJ, & Kern M (2009) The effect of storage conditions, contamination modes and cleaning procedures on the resin bond strength to lithium disilicate ceramic *Journal of Adhesive Dentistry* **11(2)** 127-135.
25. Della Bona A, Anusavice KJ, & Shen C (2000) Microtensile strength of composite bonded to hot-pressed ceramics *Journal of Adhesive Dentistry* **2(4)** 305-313.

©Operative Dentistry, 2015, **40-3,** E92-E101

Raman Spectroscopic Assessment of Degree of Conversion of Bulk-Fill Resin Composites — Changes at 24 Hours Post Cure

M Par • O Gamulin • D Marovic
E Klaric • Z Tarle

Clinical Relevance

Degree of conversion (DC) affects various physical properties and biocompatibility of a composite restoration. Adequate DC is especially important for bulk-fill materials, which are designed for placement in thick layers.

SUMMARY

Objective: The aim of this study was to determine degree of conversion (DC) of solid and flowable bulk-fill composites immediately and after 24 hours and investigate the variations of DC at surface and depths up to 4 mm.

Materials and Methods: Eight bulk-fill composites (Tetric EvoCeram Bulk Fill [shades IVA and IVB], Quixfil, X-tra fil, Venus Bulk Fill, X-tra Base, SDR, Filtek Bulk Fill) were investigated, and two conventional composites (GrandioSO, X-Flow) were used as controls. The samples (n = 5) were cured for 20 seconds with irradiance of 1090 mW/cm^2. Raman spectroscopic measurements were made immediately after curing on sample surfaces and after 24 hours of dark storage at surface and at incremental depths up to 4 mm. Mean DC values were compared using repeated measures analysis of variance (ANOVA) and *t*-test for dependent samples.

Results: Surface DC values immediately after curing ranged from 59.1%-71.8%, while the 24-hour postcure values ranged from 71.3%-86.1%. A significant increase of DC was observed 24 hours post cure for all bulk-fill composites, which amounted from 11.3% to 16.9%. Decrease of DC through depths up to 4 mm varied widely among bulk-fill composites and ranged from 2.9% to 19.7%.

Conclusions: All bulk-fill composites presented a considerable 24-hour postcure DC increase and clinically acceptable DC at depths up to 4

*Matej Par, DMD, private dental practice, Zagreb, Croatia

Ozren Gamulin, PhD, Department of Physics and Biophysics, School of Medicine, University of Zagreb, Zagreb, Croatia

Danijela Marovic, PhD, DMD, Department of Endodontics and Restorative Dentistry, School of Dental Medicine, University of Zagreb, Zagreb, Croatia

Eva Klaric, PhD, DMD, Department of Endodontics and Restorative Dentistry, School of Dental Medicine, University of Zagreb, Zagreb, Croatia

Zrinka Tarle, PhD, DMD, professor, Department of Endodontics and Restorative Dentistry, School of Dental Medicine, Zagreb, Croatia

*Corresponding author: Dr Par, Guduliceva 5, Zagreb 10000 Croatia; e-mail: mpar@inet.hr

DOI: 10.2341/14-091-L

mm. Conventional control composites were sufficiently cured only up to 2 mm, despite significant postcure polymerization.

INTRODUCTION

A continuing evolution of dental resin composites has led to the development of bulk-fill materials. In a wide variety of commercial materials, bulk-fill composites have drawn particular attention due to proposed placement of thick layers up to 4 mm. Apart from the clinically relevant time-savings, bulk placement can prevent void incorporation and contamination between layers, resulting in a more compact filling. Less air entrapment might also improve the degree of conversion (DC) due to decreased oxygen inhibition.[1]

On the other hand, bulk placement raises concerns regarding the depth of cure as well as the effect of a high volume of contracting material on polymerization shrinkage. It has been demonstrated that placement in incremental oblique layers produces less shrinkage stress because individual layers have higher unbonded surface, thus virtually reducing the Configuration-factor of an individual layer.[2] Also, the DC of conventional composites decreases rapidly at depths over 2-3 mm due to light attenuation and insufficient activation of the photoinitiator system.[3] Manufacturers of the bulk-fill composites claim that these materials have sufficiently reduced polymerization shrinkage stress and improved depth of cure, so that they are suitable for placement in layers up to 4 mm. This is attained by various strategies, eg, by increasing material translucency by using macrofillers,[4] introducing particles with low elastic modulus,[5] modifying resin composition,[6,7] or by using alternative photoinitiator systems.[4,5] Several studies have supported favorable properties of bulk-filling composite materials. Adequate depth of cure,[8,9] reduced cuspal deflection in comparison to conventional composites,[10] and a good marginal integrity[11] have been demonstrated for some bulk-fill composites. Although these individual composites performed well, the properties of the whole group of bulk-fill materials should be investigated further.

Adequate DC is clinically significant because it affects virtually every physical property—strength, hardness, elastic modulus, dimensional stability, solubility, water sorption, and color stability[12]—as well as biocompatibility.[13] Higher DC implies larger polymeric molecules that yield better physical properties and less free monomer that may leach from the restoration and potentially exert cytotoxic and genotoxic effects.

The efficiency of curing decreases progressively with increasing depth. As the curing light is attenuated by the absorption and scattering at increasing depths,[14] fewer free radicals are formed to initiate the polymerization. This problem has been addressed under the term depth of cure, which refers to a layer of thickness that is adequately cured, while it remains unclear how to define the "adequate" cure. For convenience, it has been determined as a depth at which the microhardness value equals the surface value multiplied by 0.8. This definition of "adequate" cure is being questioned by some investigators who propose different methods for determination of depth of cure.[15] Another classical method for determining the depth of cure, the ISO 4049 method, is still used but has been demonstrated to overestimate the depth of cure of bulk-fill composites.[16]

Conventional composite materials usually reach a DC of about 50%-75%.[17,18] For bulk-fill materials, DC values ranging from 50%-79% have been reported.[7,19] Some authors suggest a DC of 55% as a minimal value for clinical success.[19] Although the exact threshold DC value required for clinical success of a restoration cannot be determined, DC is a useful predictor of a restoration's physical and mechanical properties, as well as biocompatibility. Furthermore, comparing the DC at various depths may be helpful for assessment of curing efficiency, which decreases with increasing depth.[20]

To assess the DC, direct and indirect methods can be used. Direct methods based on vibrational spectroscopy are considered more accurate because they directly quantify the amount of unreacted C=C bonds.[21] Indirect methods of DC determination correlate the DC and microhardness values and may prove inaccurate.[22] Although Fourier transform infrared (FTIR) spectroscopy has been traditionally used for DC assessment, Raman spectroscopy provides an alternative method that may, in some experimental designs, prove simpler and more adaptive than FTIR.[23] Sample preparation for Raman spectroscopy has no specific requirements, and the sample can be used as-made, ie, no additional sample processing is required.[23] In addition to easier sample handling, Raman also enables multiple measurements on the same sample due to the nondestructive sample preparation. While FTIR spectroscopy measures the absorption of incident radiation, Raman is based on the inelastic scattering phenomenon. Molecules in the sample are excited to a virtual energy state by laser light and then relaxed to a molecular vibrational state followed by emission of photons with energy different from energy of the

incident photon. A Raman photon is emitted if a molecule undergoes a transition to a higher vibrational energy state than its original state (Stokes-Raman), or to a lower energy vibrational state (Anti-Stokes Raman). The energy spectrum of emitted photons is determined by vibrational energy states that are characteristic for specific functional groups and chemical bonds. As in FTIR spectroscopy, the band at 1640 cm^{-1} is assigned to the vibrations of C=C bonds and its relative change in intensity before and after curing is used to calculate the DC. When used for the DC assessment of dental composites, Raman and FTIR spectroscopy yield similar results.[23]

The aim of this study was to determine the DC of solid and flowable bulk-fill composites and investigate the variations of DC at the surface and at four clinically relevant depths, using conventional composites as controls. DC was determined using Fourier transform-Raman (FT-Raman) spectroscopy immediately after curing and after 24 hours of dark storage at 37°C. The null hypotheses were: 1) there is no difference between the DC values immediately after curing and 24 hours after curing, 2) there is no difference in the DC values between various depths for a given material, and 3) there is no difference in the DC values between various materials for a given depth.

METHODS AND MATERIALS

The composite materials used are listed in Table 1. Conventional composites GrandioSO (VOCO, Cuxhaven, Germany) and X-Flow (Dentsply, York, PA, USA) were used as controls for solid and flowable bulk-fill composites, respectively.

Five samples were made for each composite material (n=5). For sample preparation, a custom-made cylindrical stainless steel split-mold with an aperture diameter of 3 mm and depth of 6 mm was used. Uncured composite material was applied into the mold, the mold aperture was covered with a polyethylene terephthalate (PET) film, and curing was performed with a LED curing unit, Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein), for 20 seconds with irradiance of 1090 mW/cm^2 ("high" mode). The irradiance of the curing unit was tested with a Cure Rite radiometer (Caulk Dentsply, Konstanz, Germany) before each composite material. The variations were under 10%, and the value of 1090 mW/cm^2 represents the arithmetic mean of 10 measurements. The curing time of 20 seconds corresponds to or exceeds the manufacturer's recommendations for all tested materials. The curing unit tip was positioned at the angle of 90°, immediately adjacent to the mold aperture, contacting the PET film covering the sample. Sample curing was done at 21°C ± 1°C and 60% ± 15% relative humidity. Immediately after curing, Raman spectra were collected from the sample surfaces. The samples were then placed in an incubator (Cultura, Ivoclar Vivadent) at 37°C ± 1°C and 90% ± 10% relative humidity. After a dark storage period of 24 hours, Raman spectra were collected from five depths: 0 mm (surface), 1 mm, 2 mm, 3 mm, and 4 mm.

FT-Raman spectroscopy measurements were performed using a Spectrum GX spectrometer (Perkin-Elmer, Waltham, MA, USA). The excitation was a Nd:YAG laser at 1064 nm wavelength, with laser power of 800 mW and resolution of 4 cm^{-1}. The samples were mounted on a universal holder that enabled translation along the cylindrical sample, thereby exposing different depths to the excitation laser light. During the measurement, the exposed sample surface was about 0.5 mm in diameter. For each spectrum, 100 scans were recorded. Spectra of the uncured composites (n=5) were recorded in the same manner. The spectra were processed with the Kinetics add-on for Matlab (Mathworks, Natick, MA, USA).

DC calculation was performed by comparing the relative change of the band at 1640 cm^{-1}, representing the C=C stretching mode to a reference band, before and after the polymerization. For GrandioSO, Tetric EvoCeram Bulk Fill, Quixfil, X-tra fil, Venus Bulk Fill, X-tra Base, and Filtek Bulk Fill, the aromatic C=C band at 1610 cm^{-1} was used as a reference. Due to the lack of the aromatic C=C stretching mode in the case of SDR and X-Flow, reference bands at 1600 cm^{-1} and 1458 cm^{-1} (C-H stretching mode) were used, respectively.[19,24] Integrated intensities of C=C and reference bands were used for DC calculation by the following equation: $DC = 1 - R_{polymerized}/R_{unpolymerized}$, where R = (C=C band area)/(reference band area).[21] The normality of distribution of residuals was verified using the Shapiro-Wilk test. DC values obtained from multiple depths 24 hours post cure were compared using repeated measures analysis of variance (ANOVA) and Benjamini-Hochberg adjustment for multiple comparisons. Surface DC values obtained immediately after curing were compared to the surface values obtained 24 hours post cure using a dependent samples *t*-test. Statistical analysis was made in SAS (SAS Institute, Cary, NC, USA); *p*-values lower than 0.05 were considered statistically significant.

Table 1: ***Manufacturers' Information About the Composite Materials Used***

Type	Composite Material (Manufacturer)	Shade/Lot (expiration date)	Composition	Filler Amount, wt%/vol%
Conventional solid	GrandioSO (VOCO, Cuxhaven, Germany)	A2/1222126 (2014/11)	Inorganic fillers in a methacrylate matrix (Bis-GMA, TEGDMA)	89/73
Conventional flowable	X-Flow (Dentsply, York, PA, USA)	A2/1206001145 (2014/05)	Sr-Al-Na-F-P silicate glass, difunctional and multifunctional acrylate and methacrylate resins, diethylene glycol dimethacrylate, highly dispersed silicon dioxide, ultraviolet (UV) stabilizer, ethyl-4-dimethylaminobenzoate, camphorquinone, butylated hydroxy toluene, iron pigments, titanium dioxide	60/38
Bulk-fill solid	Tetric EvoCeram Bulk Fill IVA (Ivoclar Vivadent, Schaan, Liechtenstein)	IVA/P82299 (2015/12)	Dimethacrylates: Bis-GMA, Bis-EMA, UDMA, barium glass, ytterbium trifluoride, mixed oxide and prepolymer; additives, catalysts, stabilizers, pigments	81/61
	Tetric EvoCeram Bulk Fill IVB (Ivoclar Vivadent, Schaan, Liechtenstein)	IVB/R77065 (2016/10)		
	Quixfil (Dentsply, York, PA, USA)	Universal/ 121200233 (2014/05)	UDMA, TEGDMA, dimethacrylate and trimethacrylate resins, carboxylic acid modified dimethacrylate resin, butylated hydroxy toluene (BHT), UV stabilizer, camphorquinone, ethyl-4-dimethylaminobenzoate, silanated strontium aluminum sodium fluoride phosphate silicate glass	86/66
	X-tra fil (VOCO, Cuxhaven, Germany)	U/1311472 (2015/03)	Inorganic filler in a methacrylate matrix (Bis-GMA, UDMA, TEGDMA)	86/70
Bulk-fill flowable	Venus Bulk Fill (Heraeus Kulzer, Hanau, Germany)	Universal/010030 (2014/07)	Multifunctional methacrylate monomers (UDMA, EBADMA), Ba-Al-F silicate glass, YbF_3, SiO_2	65/38
	X-tra Base (VOCO, Cuxhaven, Germany)	Universal/1310503 (2015/06)	Inorganic fillers in a methacrylate matrix (aliphatic dimethacrylate)	75/61
	SDR (Dentsply, York, PA, USA)	Universal/ 1301001101 (2014/12)	Ba-Al-F-B silicate glass, Sr-Al-F silicate glass, modified UDMA, ethoxylated bisphenol A dimethacrylate (EBPADMA), TEGDMA, camphorquinone, photoaccelerator, BHT, UV stabilizer, titanium dioxide, iron oxide pigments, fluorescing agent	68/45
	Filtek Bulk Fill (3M ESPE, Saint Paul, MN, USA)	A3/N502066 (2016/02)	Bis-GMA, UDMA, Bis-EMA, Procrylat resin, ytterbium trifluoride, zirconia/silica	65/43

Abbreviations: Bis-EMA/EBADMA: ethoxylated bisphenol-A-dimethacrylate; Bis-GMA: bisphenol-A-glycidylmethacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate.

RESULTS

Mean DC values are presented in Figure 1 and Table 2. Decrease of DC through depths is presented as a given-depth/top ratio in Figure 2.

Statistical analysis revealed a significant 24-hour post cure increase in DC for all composites. Within the same material, statistical analysis showed significant differences between the surface/4 mm and 1 mm/4 mm DC values.

Conventional composites GrandioSO and X-Flow demonstrated a considerable decrease of DC at 3-mm and 4-mm depth, while DC decrease of bulk-fill composites was notably lower. Among bulk-fill composites, Tetric EvoCeram Bulk Fill IVA and IVB demonstrated the most pronounced DC decrease through depths, while Venus Bulk Fill and SDR showed the lowest DC decrease through depths.

The influence of different shades on DC was assessed by comparing the IVA and IVB shades of Tetric EvoCeram Bulk Fill. Significantly lower DC of shade IVB was found only at 4-mm depth, while differences at other depths were not significant.

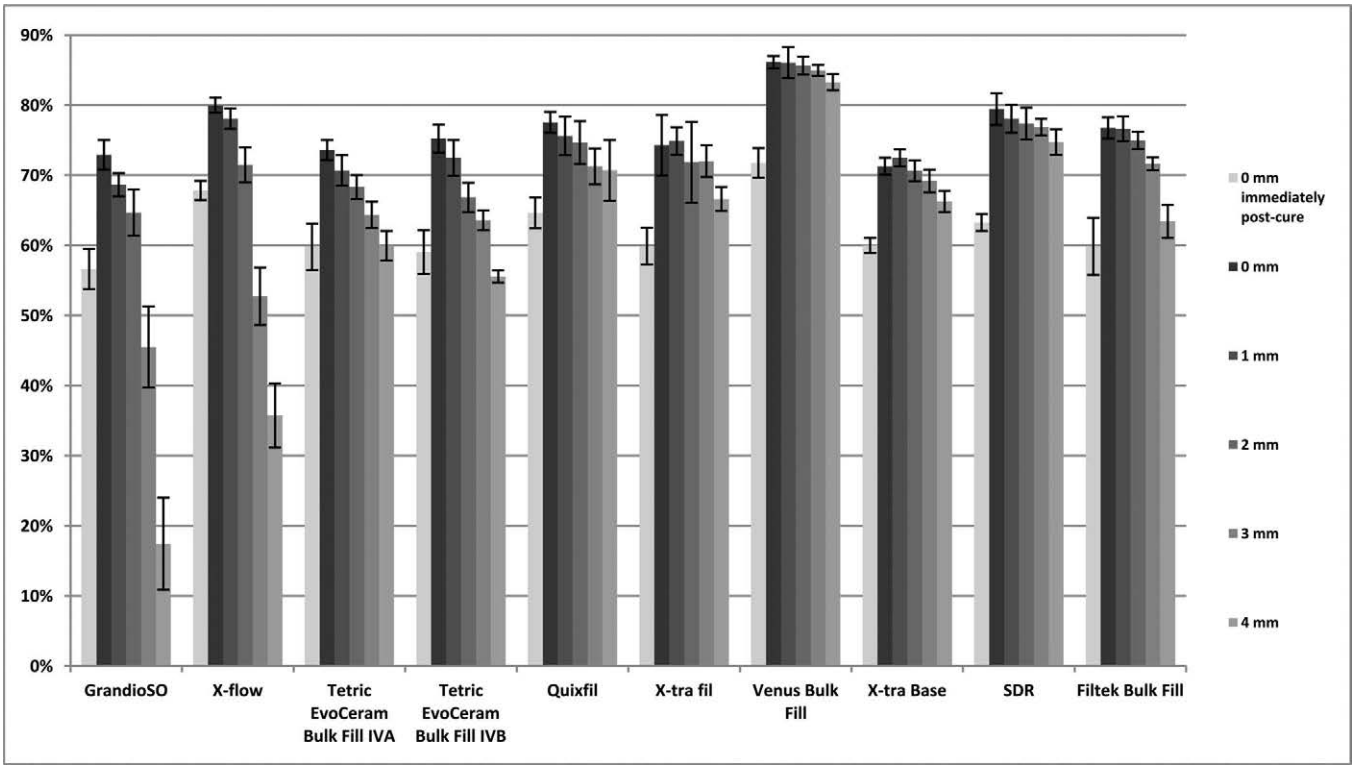

Figure 1. *Mean degree of conversion values ±SD.*

DISCUSSION

The present study was performed to determine the DC of the majority of currently available commercial bulk-fill composites by means of Raman spectroscopy. Additionally, the development of DC measured immediately and 24 hours post cure, decrease in DC through different depths up to 4 mm, between-material differences at individual depths, and differences in DC between two composite shades were observed.

Methodology

Raman spectroscopy is often used as an alternative to FTIR for determination of the DC of composite materials. An important advantage of Raman spectroscopy is the nondestructive sample preparation,

Table 2: ***Mean Degree of Conversion Values Immediately After Curing and 24 Hours Post Cure at Five Measuring Depths (n=5)***

Composite material	Immediately After Curing		24 Hours Post Cure											
	0 mm (Surface)		0 mm (Surface)		1 mm		2 mm		3 mm		4 mm			
	Mean, %	SD	Mean, %	SD	Mean, %	SD	Mean, %	SD	Mean, %	SD	Mean, %	SD		
GrandioSO	56.6*	2.9	72.9*	Aac 2.1	68.6	ABacg 1.7	64.7	Ba 3.3	45.5	Ca 5.8	17.4	Da 6.6		
X-Flow	67.8*	1.4	80.0*	Ab 1.1	78.1	Ab 1.5	71.5	Bbe 2.5	52.7	Cb 4.1	35.7	Db 4.6		
Tetric EvoCeram Bulk Fill IVA	59.8*	3.3	73.6*	Aa 1.4	70.7	Bacd 2.2	68.3	Bcd 1.7	64.4	Cc 1.9	60.0	Dc 2.1		
Tetric EvoCeram Bulk Fill IVB	59.1*	3.1	75.2*	Aad 2	72.5	Bdef 2.5	66.9	Cad 2.1	63.6	Dc 1.4	55.6	Ed 0.9		
Quixfil	64.6*	2.2	77.6*	Abd 1.5	75.6	Abe 2.7	74.7	ABefg 3.0	71.3	Bdf 2.6	70.7	Be 4.3		
X-tra fil	59.9*	2.6	74.3*	Aace 4.3	74.9	Abf 2	71.9	Abcf 5.8	72.0	Adf 2.3	66.6	Bei 1.7		
Venus Bulk Fill	71.8*	2.1	86.1*	Af 0.9	86.1	Ag 2.2	85.6	Ah 1.3	85.0	ABe 0.8	83.3	Bf 1.2		
X-tra Base	60.0*	1.1	71.3*	Ac 1.2	72.5	Adfg 1.2	70.7	ABb 1.5	69.2	Bf 1.6	66.3	Cg 1.5		
SDR	63.3*	1.2	79.5*	Ab 2.3	78.1	Ab 2	77.4	ABg 2.3	76.9	ABg 1.2	74.7	Bh 1.8		
Filtek Bulk Fill	59.9*	4.1	76.8*	Ade 1.5	76.6	Ab 1.8	75.0	Af 1.2	71.6	Bd 0.9	63.4	Ci 2.3		

** Statistically significant differences between surface DC values immediately after curing and 24 hours post cure. For DC values obtained 24 hours post cure, same uppercase letters indicate statistically similar groups in rows and same lowercase letters indicate statistically similar groups in columns.*

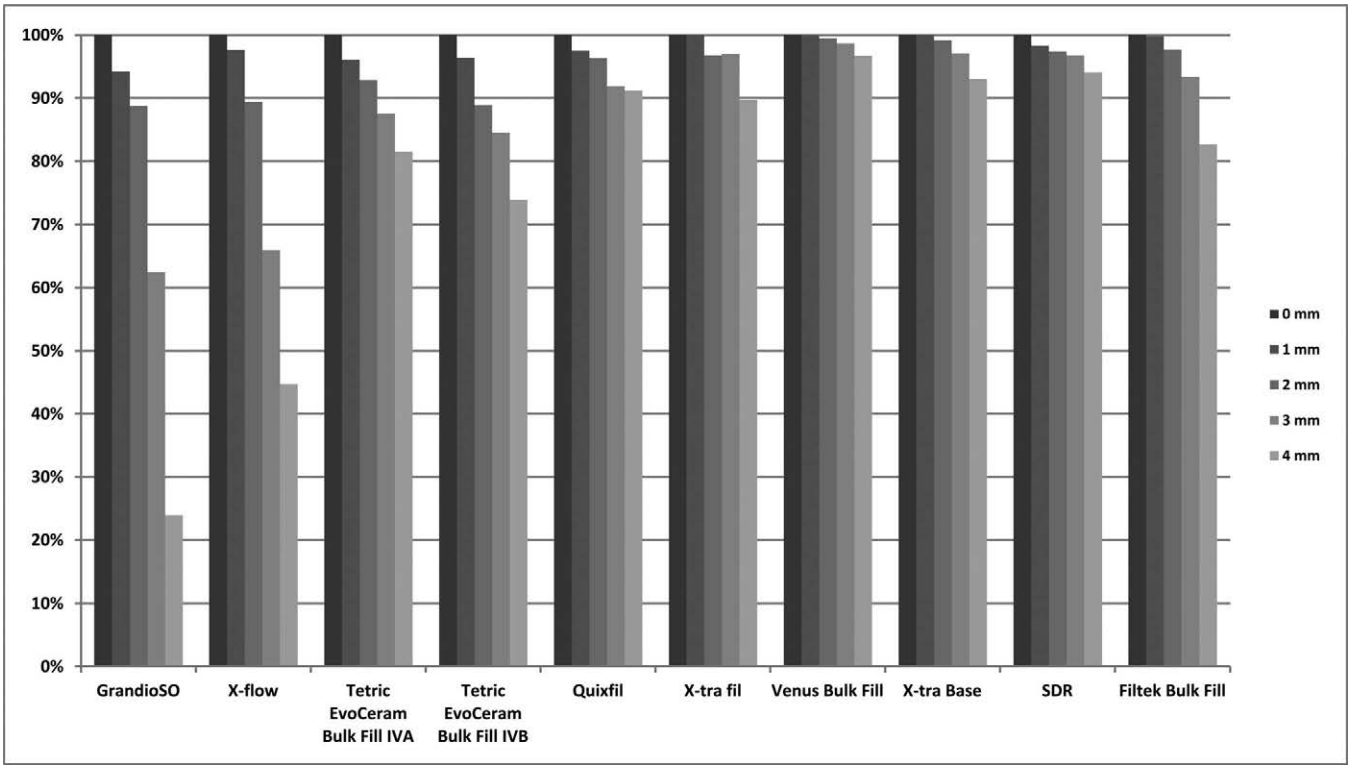

Figure 2. *Decrease of degree of conversion through depths presented as a given-depth/top ratio.*

which enabled multiple measurements at various depths on the same sample. Unlike FTIR, Raman is insensitive to eventual water contamination, which allowed for the samples to be kept in an incubator at 37°C ± 1°C and 90% ± 10% relative humidity during the 24-hour storage period in an attempt to better reproduce conditions in the oral cavity.

FTIR spectroscopy was used in a recent study examining the DC of bulk-fill composites.[19] In that study, recorded surface DC values for SDR, Venus Bulk Fill, X-tra Base and GrandioSO were consistently lower than in the present work. The difference can be ascribed to lower curing light irradiance (halogen curing unit of 600 mW/cm^2 vs polywave LED curing unit of 1090 mW/cm^2). However, the DC rankings obtained were concurrent, with the DC surface values decreasing in the following order: Venus Bulk Fill > SDR > GrandioSO > X-tra Base. This demonstrates the comparability of the results despite the differences in curing conditions and methodology used (Raman vs FTIR).

Surface DC Immediately Post Cure and 24 Hours Post Cure

The first hypothesis was rejected, since a substantial postcure increase in the DC values at the surface of the samples was noted. This effect is already well known in conventional resin composites,[25-27] but only one study showed it in flowable bulk-fill materials.[19] According to Burtscher,[28] even a small increase in the extent of DC near the end of the polymerization process can largely affect the density of cross-linking in the polymer network, and thus the mechanical properties (hardness and elastic modulus) of resin composites.[26] Postpolymerization was found to be more pronounced in samples with initially lower DC. Namely, in highly polymerized samples, reactive sites are immobilized in the polymer network, whereas in samples with initially lower DC, a higher amount of unreacted radicals allows for the increased mobility to make contact with other reactive species in the polymer network.[26]

DC increase after 24 hours of dark storage at 37°C amounted to 11.3% (X-tra Base) to 16.9% (Filtek Bulk Fill) and was statistically significant for both bulk-fill and conventional composites. Some of the previous studies were inconclusive regarding the extent of postcure polymerization,[29,30] but one study[19] has reported a considerable increase in DC of bulk-fill composites. The continuation of polymerization after curing may affect the comparability of DC values reported in various studies, since DC

measurements can be performed at different postcure times.[7,19]

A previous study has assessed surface DC values for GrandioSO, Venus Bulk Fill, X-tra Base, SDR, and Filtek Bulk Fill immediately after polymerization and 24 hours post cure.[19] Both the DC values recorded immediately after curing and 24 hours post cure were slightly lower than those obtained in our study. This can be attributed to lower curing radiant exposure (20 seconds at 600 mW/cm^2). Moreover, the previous study[19] reported more extensive postcure DC increase for GrandioSO, Venus Bulk Fill, and SDR than recorded in our study, which can be explained by the finding that the amount of postcure polymerization is higher in samples that received lower radiation doses.[25]

X-tra Base demonstrated the lowest postcure DC increase and the lowest DC values up to 3 mm depth of all flowable materials. Filler load of X-tra Base amounts for 61 vol%, which is in the range of some solid bulk-fill composites, namely Tetric EvoCeram Bulk Fill (Table 1). High filler amounts may impair the mobility of reactive sites and in turn decrease the DC.[31,32] This might also be one of the reasons for somewhat lower postcure increases in DC. Also, high filler load may additionally contribute to lower DC at depth by decreasing a composite's translucency.[4] However, a conclusive answer cannot be given at this time since the exact resin composition is not provided.

The highest postcure DC increase was observed for SDR and Filtek Bulk Fill. SDR contains a patented modified UDMA, which is claimed to reduce polymerization shrinkage and shrinkage stress.[6] Similarly, Filtek Bulk Fill contains a proprietary monomer analogous to Bis-GMA and patented as Procrylat resin.[33] It is possible that these modified monomers have altered polymerization kinetics and delayed the monomer conversion, which could explain higher postcure DC increase.

From a clinical standpoint, a rather high postcure DC increase implies that restorations do not develop their final mechanical properties immediately after curing. Upon continuing postcure polymerization, mechanical properties gradually increase up to (at least) 24 hours post cure. The results of this study stress the importance of timing as a consequential factor, which should be taken into account in scientific investigations of bulk-fill composites. Assessment of DC, as well as other properties affected by DC, should be made at standardized postcure times (eg, after 24 hours) to ensure comparability to other studies.

DC at Various Depths 24 Hours Post Cure

Surface DC values of bulk-fill composites measured 24 hours post cure ranged from 71.3% (X-tra Base) to 86.1% (Venus Bulk Fill), which fits or slightly exceeds the previously reported DC range of conventional[17] and bulk-fill composites.[7,19]

Successive measurements at various depths were made to assess the influence of increasing depth of cure efficiency, since the decrease of DC at depth is inevitable due to light attenuation.[15] For tested bulk-fill composites, surface/4 mm DC decrease ranged from 2.9% (Venus Bulk Fill) to 19.7% (Tetric EvoCeram Bulk Fill IVB), and for most bulk-fill composites (Quixfil, X-tra fil, Venus Bulk Fill, X-tra Base, SDR) amounted to less than 10%. In comparison, DC of conventional composites GrandioSO and X-Flow decreased at 4 mm depth for 55.5% and 44.3%, respectively. Thus, bulk-fill composites presented improved curing efficiency at depth, which supports manufacturers' recommendations for placement of 4-mm layers.

Within the same material, DC values at 4 mm were significantly lower than surface and 1 mm values for all tested composites. Hence, the second hypothesis was rejected. However, for most of the bulk-fill composites, the surface/4 mm differences were well below 10%, which can be considered clinically acceptable. Statistical significance that was noted for some of the clinically negligible differences (amounting to 2%-4%) was due to low standard deviations. The statistical heterogeneity between individual depths was most pronounced in Tetric EvoCeram Bulk Fill IVA and IVB. In the group of solid bulk-fill composites, Tetric EvoCeram Bulk Fill also demonstrated the highest DC decrease at depth, which may be due to its higher opacity in comparison to other bulk-fill composites.[4] Among the flowable bulk-fill composites, the highest DC decrease at depth was observed for Filtek Bulk Fill. Filtek Bulk Fill differs from other materials in this study by containing zirconia filler, which was shown to decrease translucency due to resin/filler refractive index mismatch.[34] Lower translucency of Filtek Bulk Fill in comparison to most available bulk-fill materials was also reported by Bucuta and Ilie.[4]

Differences in light penetration through composite material may be attributed to the light scattering at the filler-resin interfaces and absorbance by photoinitiators and pigments.[35] Thus, the curing efficiency at increasing depths is affected by the filler composition, which determines the light attenuation. Some of the tested bulk-fill materials (Tetric EvoCeram

Bulk Fill IVA and IVB, Filtek Bulk Fill) are notably less translucent than the other bulk-fill materials tested.[4] Differences in translucence affected the DC decrease at depth, as can be seen by comparing the 4-mm/surface DC ratios.[20] Three composites with higher opacity (Tetric EvoCeram Bulk Fill IVA and IVB, Filtek Bulk Fill) show 4-mm/surface ratios of 81%, 74%, and 83%, respectively, while the other bulk-fill composites show ratios equal to or higher than 90% (Figure 2). On the other hand, their opacity gives them advantage over other bulk-fill composites in terms of acceptable esthetics for placement in the visible zone, eg, mesial class II restorations.

The third hypothesis was also rejected; in multiple comparisons of the DC values at the same depth, statistical analysis revealed significant differences between some of the tested materials. Generally, more statistical heterogeneity was observed at greater depths, ie, at 3 mm and 4 mm. Since the DC of a composite material depends on multiple intrinsic factors,[12] the observed between-material differences are expected. Also, the differences became more apparent at greater depths due to differences in light attenuation and efficiency of photoinitiator system in various bulk-fill composites.

A study by Bucuta and Ilie[4] assessed the light transmittance and microhardness of bulk-fill composites. The transmittance values at 4 mm depth were decreasing in the following order: Venus Bulk Fill > SDR > X-tra Base > Filtek Bulk Fill. In our study, the same ranking for DC values at 4 mm depth 24 hours post cure was obtained, with statistically significant differences among all four materials. Thus, DC of flowable bulk-fill composites at a maximal recommended depth of 4 mm appears to be highly dependent on the material's translucency.

Venus Bulk Fill showed the highest DC values and the lowest DC decrease at depth of all tested bulk-fill composites. This can be the result of relatively low filler load (38 vol%) and very high translucency.[5] Although Venus Bulk Fill attains very high DC, low filler loading may impair mechanical properties.[8,16] The load-bearing capability of the restoration could be improved by placing an additional 2-mm layer of universal/posterior composite over the Venus Bulk Fill core, as recommended by the manufacturer.

A possible influence of different shades of Tetric EvoCeram Bulk Fill (IVA and IVB) on DC was investigated. For a given irradiation dose, DC of a composite is determined by resin formulation, photoinitiator system, filler load, and filler morphology.[36] Additionally, composite translucency plays an important role in DC decrease at depth.[4] Based on the manufacturer's information, it can be assumed that both shades of Tetric EvoCeram Bulk Fill have the same photoinitiator system as well as resin and filler composition, differing only by a low number of pigmented particles that are used to adjust the shade. These particles are responsible for the curing light attenuation and might influence the DC at depth. The differences in attenuation between two shades are expected to become more pronounced as the distance of light path increases. IVA shade showed significantly higher DC than IVB shade at 4 mm depth, while no significant difference was observed at other depths. It appears that the light path of 4 mm is required for differences in light attenuation due to different shades to reflect as a difference in DC.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following can be concluded:

1) All tested bulk-fill composites presented a considerable 24-hour postcure DC increase, up to 16.9%.
2) All tested bulk-fill composites presented clinically acceptable DC at depths up to 4 mm.
3) Differences between surface DC and 4-mm DC values varied widely among bulk-fill composites (2.9% for Venus Bulk Fill to 19.7% for Tetric EvoCeram Bulk Fill IVB). For most of the bulk fill composites, the difference was under 10%.

In this study, all tested bulk-fill composites performed adequately with respect to DC. Further study investigating other properties is needed to advocate their clinical use.

Acknowledgments

This investigation was supported by Croatian Science Foundation (Project 08/31 Evaluation of new bioactive materials and procedures in restorative dental medicine). Dental companies Ivoclar Vivadent (Schaan, Liechtenstein) and Dentsply DeTrey (Konstanz, Germany) are gratefully acknowledged for the generous donation of the resin composite materials used in this study. The authors also thank Marin Kosovic for technical assistance and Ivan Sever for statistical analysis.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 27 May 2014)

REFERENCES

1. Gauthier MA, Stangel I, Ellis TH, Zhu XX (2005) Oxygen inhibition in dental resins *Journal of Dental Research* **84**(**8**) 725-729.
2. Park J, Chang J, Ferracane J, Lee IB (2008) How should composite be layered to reduce shrinkage stress: Incremental or bulk filling? *Dental Materials* **24**(**11**) 1501-1505.
3. Knezevic A, Ristic M, Demoli N, Tarle Z, Music S, Negovetic Mandic V (2007) Composite photopolymerization with diode laser *Operative Dentistry* **32**(**3**) 279-284.
4. Bucuta S, Ilie N (2014) Light transmittance and micromechanical properties of bulk fill vs. conventional resin based composites *Clinical Oral Investigations* Jan 11 [Epub ahead of print] doi: 10.1007/s00784-013-1177-y.
5. Ivoclar Vivadent (2013) Tetric EvoCeram Bulk Fill Scientific Documentation. Retrieved online February 1, 2014 from: http://www.ivoclarvivadent.com/zoolu-website/media/document/17021/Tetric+EvoCeram+Bulk+Fill.
6. Dentsply (2011) SDR Scientific Compendium. Retrieved online February 1, 2014 from: http://www.dentsplymea.com/sites/default/files/Scientific%20compendium%20SDR%20-%202011.pdf.
7. Czasch P, Ilie N (2013) *In vitro* comparison of mechanical properties and degree of cure of bulk fill composites *Clinical Oral Investigations* **17**(**1**) 227-235.
8. Alrahlah A, Silikas N, Watts DC (2014) Post-cure depth of cure of bulk fill dental resin-composites *Dental Materials* **30**(**2**) 149-154.
9. Fleming GJ, Awan M, Cooper PR, Sloan AJ (2008) The potential of a resin-composite to be cured to a 4 mm depth *Dental Materials* **24**(**4**) 522-529.
10. Moorthy A, Hogg CH, Dowling AH, Grufferty BF, Benetti AR, Fleming GJ (2012) Cuspal deflection and microleakage in premolar teeth restored with bulk-fill flowable resin-based composite base materials *Journal of Dentistry* **40**(**6**) 500-505.
11. Roggendorf MJ, Kramer N, Appelt A, Naumann M, Frankenberger R (2011) Marginal quality of flowable 4-mm base vs. conventionally layered resin composite *Journal of Dentistry* **39**(**10**) 643-647.
12. Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G (2013) Progress in dimethacrylate-based dental composite technology and curing efficiency *Dental Materials* **29**(**2**) 139-156.
13. Krifka S, Seidenader C, Hiller KA, Schmalz G, Schweikl H (2012) Oxidative stress and cytotoxicity generated by dental composites in human pulp cells *Clinical Oral Investigations* **16**(**1**) 215-224.
14. Lee S, Kim T, Son S, Park J, Kim J, Kim H, Kwon YH (2013) Influence of light-curing units on the polymerization of low-shrinkage composite resins *Dental Materials Journal* **32**(**5**) 688-694.
15. Leprince JG, Leveque P, Nysten B, Gallez B, Devaux J, Leloup G (2012) New insight into the "depth of cure" of dimethacrylate-based dental composites *Dental Materials* **28**(**5**) 512-520.
16. Flury S, Hayoz S, Peutzfeldt A, Husler J, Lussi A (2012) Depth of cure of resin composites: Is the ISO 4049 method suitable for bulk fill materials? *Dental Materials* **28**(**5**) 521-528.
17. Vasudeva G (2009) Monomer systems for dental composites and their future: A review *Journal of the California Dental Association* **37(6)** 389-398.
18. Tarle Z, Knezevic A, Demoli N, Meniga A, Sutalo J, Unterbrink G, Ristic M, Pichler G (2006) Comparison of composite curing parameters: Effects of light source and curing mode on conversion, temperature rise and polymerization shrinkage *Operative Dentistry* **31**(**2**) 219-226.
19. Alshali RZ, Silikas N, Satterthwaite JD (2013) Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals *Dental Materials* **29**(**9**) e213-217.
20. Bouschlicher M, Rueggeberg F, Wilson B (2004) Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions *Operative Dentistry* **29(6)** 698-704.
21. Shin WS, Li XF, Schwartz B, Wunder SL, Baran GR (1993) Determination of the degree of cure of dental resins using Raman and FT-Raman spectroscopy *Dental Materials* **9(5)** 317-324.
22. Marovic D, Panduric V, Tarle Z, Ristic M, Sariri K, Demoli N, Klaric E, Jankovic B, Prskalo K (2013) Degree of conversion and microhardness of dental composite resin materials *Journal of Molecular Structure* **1044** 299-302.
23. Pianelli C, Devaux J, Bebelman S, Leloup G (1999) The micro-Raman spectroscopy, a useful tool to determine the degree of conversion of light-activated composite resins *Journal of Biomedical Materials Research* **48(5)** 675-681.
24. Zhang Y, Wang Y (2012) Improved degree of conversion of model self-etching adhesives through their interaction with dentine *Journal of Dentistry* **40**(**1**) 57-63.
25. Leung RL, Fan PL, Johnston WM (1983) Post-irradiation polymerization of visible light-activated composite resin *Journal of Dental Research* **62(3)** 363-365.
26. Mohamad D, Young RJ, Mann AB, Watts DC (2007) Post-polymerization of dental resin composite evaluated with nanoindentation and micro-Raman spectroscopy *Archives of Orofacial Sciences* **2** 26-31.
27. Schneider LF, Consani S, Ogliari F, Correr AB, Sobrinho LC, Sinhoreti MA (2006) Effect of time and polymerization cycle on the degree of conversion of a resin composite *Operative Dentistry* **31**(**4**) 489-495.
28. Burtscher P (1993) Stability of radicals in cured composite materials *Dental Materials* **9(4)** 218-221.
29. Truffier-Boutry D, Demoustier-Champagne S, Devaux J, Biebuyck JJ, Mestdagh M, Larbanois P, Leloup G (2006) A physico-chemical explanation of the post-polymerization shrinkage in dental resins *Dental Materials* **22**(**5**) 405-412.
30. Tarumi H, Imazato S, Ehara A, Kato S, Ebi N, Ebisu S (1999) Post-irradiation polymerization of composites containing bis-GMA and TEGDMA *Dental Materials* **15(4)** 238-242.

31. Halvorson RH, Erickson RL, Davidson CL (2003) The effect of filler and silane content on conversion of resin-based composite *Dental Materials* **19**(**4**) 327-333.

32. Eliades GC, Vougiouklakis GJ, Caputo AA (1987) Degree of double bond conversion in light-cured composites *Dental Materials* **3**(**1**) 19-25.

33. 3M ESPE (2012) Filtek Bulk Fill Technical Product Profile. Retrieved online February 1, 2014 from: http://multimedia.3m.com/mws/mediawebserver?mwsId=SSSSSufSevTsZxtUoY_vmx2UevUqevTSevTSevTSeSSSSSS--&fn=Filtek_bulk_fill_flowable_tpp_R2.pdf.

34. Guo G, Fan Y, Zhang JF, Hagan JL, Xu X (2012) Novel dental composites reinforced with zirconia-silica ceramic nanofibers *Dental Materials* **28**(**4**) 360-368.

35. Musanje L, Darvell BW (2006) Curing-light attenuation in filled-resin restorative materials *Dental Materials* **22**(**9**) 804-817.

36. Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G (2013) Progress in dimethacrylate-based dental composite technology and curing efficiency *Dental Materials* **29**(**2**) 139-156.

©Operative Dentistry, 2015, **40-3,** E102-E111

Bonding of Adhesive Luting Agents to Caries-affected Dentin Induced by a Microcosm Biofilm Model

AC Peixoto • AA Bicalho • CP Isolan
TT Maske • RR Moraes • MS Cenci
CJ Soares • AL Faria-e-Silva

Clinical Relevance

Remaining caries-affected dentin after the cavity preparation for indirect restorations results in reduced bond strength of adhesive luting agents.

SUMMARY

Objectives: To evaluate the bond strength of adhesive luting agents applied to caries-affected dentin (CAD).

Methods: Thirty-six noncarious human third molars were abraded to expose an occlusal dentin surface. Caries lesions were induced in half of the samples using a microcosm biofilm model. Biofilm was cultivated under an anaerobic atmosphere for 14 days in a medium enriched with mucin. The same medium containing 1% sucrose was alternated for 4 hours per day. Cylinders of resin cement (RelyX ARC, RelyX U200, or BisCem) were built up over the dentin substrate and submitted to shear bond load. The samples were then longitudinally sectioned. The hardness and elastic modulus of dentin were measured at different depths from the occlusal surface. A three-dimensional finite element simulation was performed to analyze the residual stress distribution during the shear bond strength test. Bond strength data were analyzed by two-way analysis of variance (ANOVA) and hardness and elastic modulus by split-plot ANOVA. Multiple comparisons were performed with the SNK test (α=0.05).

Results: For all cements, the highest bond strengths were observed in sound dentin. Re-

Aline C Peixoto, DDS, Federal University of Sergipe, Department of Dentistry, Aracaju, Brazil

Aline A Bicalho, DDS, MS, PhD student, School of Dentistry, Federal University of Uberlândia, Operative Dentistry and Dental Materials, Uberlandia, Brazil

Cristina P Isolan, DDS, MSc, PhD student, Graduate Program in Dentistry, Federal University of Pelotas, Pelotas, Brazil

Tamires T Maske, DDS, MSc student, Graduate Program in Dentistry, Federal University of Pelotas, Pelotas, Brazil

Rafael R Moraes, DDS, MS, PhD, School of Dentistry, Graduate Program in Dentistry, Federal University of Pelotas, Restorative Dentistry, Pelotas Brazil

Maximiliano S Cenci, DDS, MSc, PhD, Graduate Program in Dentistry, Federal University of Pelotas, Department of Operative Dentistry, Pelotas, Brazil

Carlos J Soares, DDS, MSc, PhD, School of Dentistry, Federal University of Uberlândia, Operative Dentistry and Dental Materials, Uberlandia, Brazil

*Andre L Faria-e-Silva, DDS, MSc, PhD, Federal University of Sergipe, Department of Dentistry, Aracaju, Brazil

*Corresponding author: Rua Claudio Batista s/n, Bairro Santório, Aracaju, SE 49060-100
Brazil; e-mail: andrelfsilva@hotmail.com

DOI: 10.2341/14-169-L

lyx ARC bond strength was similar to that of RelyX U200 for both substrates; BisCem had the lowest values. CAD had lower hardness (above a depth of 100 μm) and elastic modulus (above a depth of 150 μm) values than sound dentin. Stress distribution during the bond strength test was similar under all experimental conditions.

Conclusion: Impairment of the mechanical properties of dentin promoted by carious lesions reduced the bond strength of adhesive luting agents.

INTRODUCTION

Preparations for adhesively bonded dental restorations are conservative, with tissue removal almost entirely limited to carious structures.[1,2] In some cases, caries-infected dentin is removed and the underlying remineralizable dentin (caries-affected dentin, CAD) is preserved to avoid pulpal complications.[3,4] In clinical practice, it is possible that CAD may be left unintentionally in some areas of the cavity. The performance of composite indirect restorations is related to their adhesion to tooth structures, and proper bonding to CAD is required for their longevity.[5,6]

In the past, luting procedures for indirect restorations were routinely performed using resin cements with bonding agents. More recently, self-adhesive resin cements (SARCs) were introduced to facilitate these procedures.[7-9] The bonding mechanism of SARCs is attributed to both a chemical reaction between phosphate methacrylates and hydroxyapatite and the infiltration of these materials into tooth tissues.[10-13] Several studies have demonstrated reduced bonding ability of dental adhesives to CAD compared with sound tissue.[14-18] By contrast, few studies have evaluated the bond strength of SARCs applied to CAD, and their results have been conflicting.[19,20] A recent study found no difference in SARC bond strength between natural CAD and sound dentin,[20] whereas another study using a pH-cycling model to produce CAD found reduced bond strength to this substrate.[19]

A difficulty of evaluating the bond strength of adhesive materials applied to CAD is standardizing the dentin substrate. Use of natural CAD is difficult because different areas of the substrate can exhibit significant differences in mineral content.[17] The technique used to remove caries and to determine the extent and location of CAD is also controversial and might influence the results.[14,21] Several studies have used the pH-cycling method to artificially induce CAD formation.[19,22,23] The use of a microcosm biofilm model is another approach for *in vitro* formation of CAD.[24] Thus, the aim of this study was to evaluate the bond strength of adhesive luting agents applied to CAD induced by a microcosm biofilm model. The study hypothesis was that the luting agents would exhibit reduced bond strength when applied to CAD.

METHODS AND MATERIALS

Study Design

This *in vitro* investigation was conducted using a 2 × 3 factorial study design to evaluate the factors "substrate" in two levels (sound dentin or CAD) and "adhesive luting agents" in three levels (three different materials). The luting agents evaluated were the regular resin cement RelyX ARC (3M ESPE, St Paul, MN, USA) and the self-adhesive resin cements RelyX U-200 (3M ESPE) and BisCem (Bisco, Schaumburg, IL, USA). CAD was produced using a microcosm biofilm model cultivated for 14 days over the sound dentin. The hardness and elastic modulus of the sound dentin and CAD were evaluated at various depths from the occlusal surface of the specimens (n=5). The bond strength of luting agents applied to sound dentin and CAD was evaluated by the shear bond strength test followed by failure-mode analysis (n=6). Three-dimensional (3D) finite element analysis (FEA) was used to calculate the corresponding residual stress in the tooth during the shear bond strength test.

Sample Preparation

For this study, 36 noncarious human third molars stored in 0.05% thymol saline solution at 4°C for no more than three months were used in this study. The occlusal surfaces were ground flat with a plaster trimmer under running water, followed by the use of 100 grit SiC papers to remove the enamel and expose a flat medium dentin surface. Samples were sectioned parallel to the occlusal surface 2 mm below the cemento-enamel junction using a water-cooled slow-speed diamond saw (#7020, KG Sorensen, Barueri, Brazil); the roots were discarded.

The specimens were ultrasonically cleaned with distilled water, and the dentin surfaces were wet-polished with 600 grit SiC paper for one minute to standardize the smear layer. Surfaces were inspected with an optical stereomicroscope at 40× magnification to ensure the absence of enamel. The specimens were placed in Falcon tubes immersed

Table 1: *Description of Cements Used in the Study and Their Application Protocols*

Cement	Classification	Application Protocol[a]
RelyX ARC	Regular resin cement	The dentin was etched with phosphoric acid for 15 seconds followed by rinsing for the same time. Excess water was removed using absorbent paper. The primer of Scotchbond Multipurpose Plus (3M ESPE) adhesive system was applied and the solvent volatilized with a gently air-stream. The bonding agent was applied in a single layer and light-cured for 10 seconds. The resin cement RelyX ARC was mixed and inserted into the orifices of the elastomer mold.
RelyX U200	Self-adhesive resin cement	The dentin surface was rinsed, and excess water was removed with absorbent paper. The cement was mixed and inserted into the orifices of the elastomer mold.
BisCem	Self-adhesive resin cement	The dentin substrate was rinsed, and all surface water was removed with a strong air stream for five seconds. The cement was mixed and inserted into the orifices of the elastomer mold.

[a] *According to manufacturers' instructions.*

in distilled water and sterilized with gamma radiation at 15 kGy for 13.3 seconds. After sterilization, the specimens were stored at 4°C.

Induction of Caries

Half of the specimens were randomly assigned to caries induction using a microcosm biofilm model. The surfaces were covered with two coats of an acid-resistant, fast-drying nail varnish leaving only the occlusal surface exposed. For the cariogenic challenge, 20 mL of fresh saliva stimulated by paraffin film was collected from a healthy volunteer (a 48-year-old woman) who had not taken any antibiotics for at least six months previously. The volunteer abstained from oral hygiene for 24 hours and from food ingestion for two hours before collection. Then, 400 μL of the collected saliva was inoculated over the exposed occlusal surfaces in Falcon tubes. After one hour, 4 mL of defined medium enriched with mucin (DMM)[25] containing 1% sucrose was added, and the tubes were incubated at 37°C under an anaerobic atmosphere (80% N_2, 10% CO_2, 10% H_2) for four hours. The samples were then rinsed with 10 mL of sterile saline solution, inserted into a Falcon tube containing DMM without sucrose and incubated for 20 hours under an anaerobic atmosphere. The biofilms were cultivated for 14 days in this alternating sucrose regimen, and the medium was replaced twice per day. The period needed for CAD to be produced was determined in a pilot experiment.

Bond Strength Evaluation

All samples, including sound dentin specimens, were embedded in acrylic resin cylinders to facilitate handling. Elastomer molds with four cylindrical orifices (1.5 mm diameter × 0.5 mm thickness) were placed onto the dentin substrate. The adhesive resin luting agents were used to fill the orifices. The classification of and application protocol for the luting agents are described in Table 1. After filling the orifices, a polyester strip and a polymerized composite resin disc (Filtek Z350 XT, shade CT, 5 mm diameter × 2 mm thickness) were placed over the filled mold. A load of 750 gf was applied over the composite disc for 5 minutes. The load was then removed, and the resin cement cylinders were light-cured for 40 seconds through the composite disc, simulating the clinical situation in which the resin cement is indirectly light-activated. Photoactivation was performed using a light-emitting diode curing unit (Radii, SDI, Bayswater, Australia) with an irradiance of 1400 mW/cm^2.

After cement polymerization, the elastomer molds were removed, and the samples were stored in distilled water at 37°C for 24 hours. For the shear test, a stainless steel wire (0.2 mm diameter) was looped around each cylinder and aligned with the bonded interface. The test was conducted on a mechanical testing machine (DL500, EMIC, São José dos Pinhais, Brazil) at a crosshead speed of 0.5 mm/min until failure. Fractured specimens were observed under magnification of up to 500× on an optical microscope to classify the failure mode: type I = adhesive failure, or type II = mixed failure.

Mechanical Properties of Sound Dentin and CAD

After the shear test, the tooth samples were longitudinally sectioned through their center with a water-cooled diamond saw. Half of the specimens were embedded in polymethyl methacrylate and wet-polished with 600, 1200, 1500, and 2000 grit SiC abrasive papers, followed by a final polishing with a 1 μm polycrystalline diamond suspension. Cross-sectional hardness measurements were performed with a Vickers indenter (CSM Micro-Hardness Tester; CSM Instruments, Peseux, Switzerland). Indentations were made in the dentin at depths of

Table 2: *Means (95% Confidence Intervals) for Bond Strength, MPa (n=6)*[a]

Cement	Substrate		Pooled Average
	Sound Dentin	Caries-affected Dentin	
RelyX ARC	4.8 (4.3 – 5.3)	3.1 (2.4 – 3.8)	4.0 (3.5 – 4.5)[y]
RelyX U200	5.9 (5.3 – 6.5)	3.4 (2.7 – 4.1)	4.7 (4.1 – 5.3)[y]
BisCem	3.2 (2.2 – 4.2)	2.3 (2.1 – 2.5)	2.8 (2.3 – 3.3)[z]
Pooled average	4.6 (4.1 – 5.1)[y]	3.0 (2.7 – 3.3)[Z]	

[a] *For pooled averages, distinct letters indicate significantly statistical differences ($\alpha = 0.05$).*

Table 3: *Ratios Between Type I and Type II Failure Modes for All Groups*[a]

Cement	Substrate		*P* Value
	Sound Dentin	Caries-affected Dentin	
RelyX ARC	11	3	0.245
RelyX U200	3.8	11	0.416
BisCem	2	5	0.318
P value	0.103	0.301	

[a] *Type I indicates adhesive failure; Type II indicates mixed (failure partially adhesive and partially cohesive within dentin).*

10, 20, 30, 40, 50, 100, 150, and 200 μm from the occlusal surface of each specimen. Indentation was performed with a controlled force, the test load being increased or decreased at a constant speed and ranging between 0 and 500 mN at 60-second intervals; the maximal load was maintained for 15 seconds. The load and penetration depth of the indenter were measured continuously during the load-unload hysteresis. Hardness was defined as the applied force divided by the apparent area of the indentation at maximal force. Elastic modulus was calculated from the slope of the tangent to the indentation depth curve at maximal force.

Residual Stress Calculation by 3D FEA

To calculate the corresponding residual stress in the tooth, a 3D finite element simulation was conducted using a resin cement cylinder of the same dimensions as that used in the bond strength test bonded to a dentin disc of 5-mm diameter. Boundary conditions were achieved by fixing the dentin cylinder at all external lateral and bottom surfaces. The elastic modulus of sound dentin and CAD was simulated as calculated from the indentation test and Poisson's ratio was obtained from the literature.[26] The elastic modulus of the resin cement was calculated using a Knoop indentation method as described previously, and Poisson's ratio for the resin cement was obtained from a previous study.[27] The model was meshed using linear, eight node, isoparametric, and arbitrary hexahedral elements. A 100 N load was applied through the simulated steel wire as a rigid body, using a dynamic loading process with the same parameters as the experimental test.

FEA was performed using MSC.Mentat (pre- and post-processor) and MSC.Marc (solver) software (MSC Software Corporation, Santa Ana, CA, USA). Then, von Mises equivalent stress and maximal principal stress were used to express the stress conditions. Stress distribution was analyzed using a model with three-quarters of the geometry showing the external and internal view of the stress distribution. Stress values were obtained for 10 nodes located at the center of the dentin structure where the areas of greatest stress concentration were observed.

Statistical Analysis

Data analysis was performed using the SigmaStat v.3.5 statistical software package (Systat Software Inc. Chicago, IL, USA). Bond-strength data were subjected to two-way analysis of variance (ANOVA). All pairwise multiple comparison procedures were performed by the Student-Newman-Keuls (SNK) method. Hardness and elastic modulus data were separately submitted to split-plot design ANOVA and SNK as a *post hoc* test. Nonlinear regression analysis was used to investigate the relationship between dentin depth and hardness/elastic modulus. Data on failure mode ratio were submitted to χ^2 and Fisher's exact tests. The significance level was set at $\alpha = 0.05$ for all analyses.

RESULTS

Bond Strength and Failure Mode

Regarding bond strength (Table 2), a significant effect was detected for the factors "substrate" ($p<0.001$) and "cement" ($p=0.002$), but not for the interaction between the factors ($p=0.42$). The power of performed tests was 0.98 and 0.87 for the factors "substrate" and "cement," respectively. Irrespective of the cement applied, significantly higher bond strength was observed for sound dentin than for CAD. RelyX U200 and RelyX ARC exhibited similar results for the two substrates, with significantly

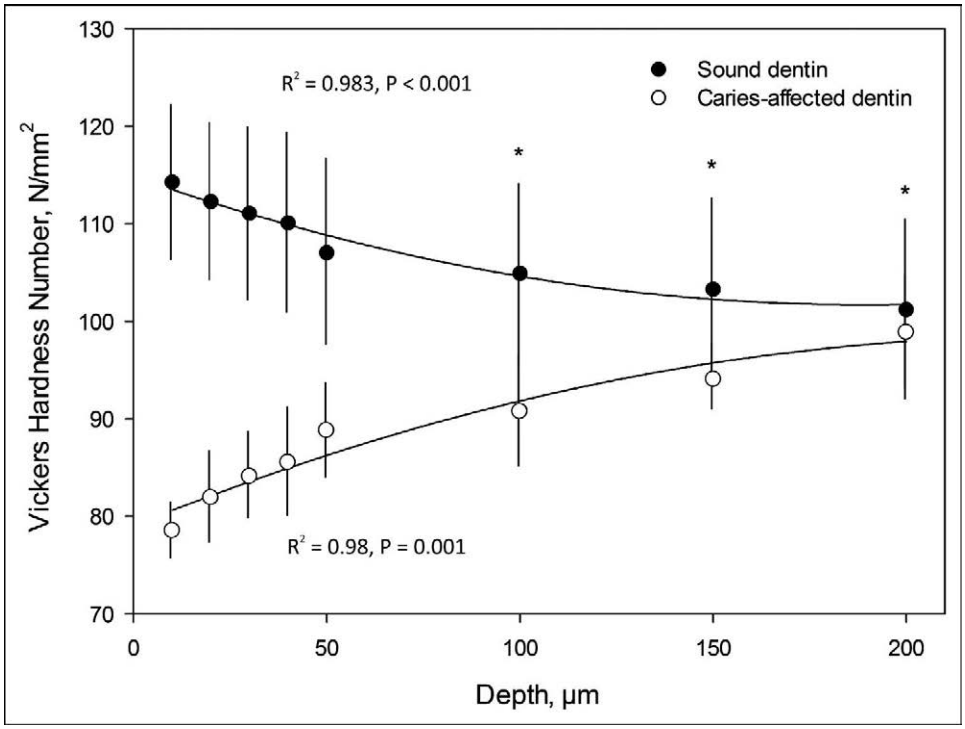

Figure 1. *Results and nonlinear regression plots for hardness (* indicates absence of significant difference between dentin types at the same depth; error bars indicate standard deviation). A logarithmic reduction in hardness was associated with increased depth in sound dentin, whereas a logarithmic increase in hardness was associated with increased depth in caries-affected dentin.*

higher bond strengths than BisCem. Neither cement type nor substrate affected failure mode (Table 3).

Hardness and Elastic Modulus

Regarding Vickers hardness (Figure 1), a significant effect was observed for the factor "dentin type" (p=0.019), but not for the subplot "depth" (p=0.266). The interaction between the factor and the subplot was significant (p<0.001). The power of performed tests was 1.00. Except for the depths of 100, 150, and 200 μm, sound dentin was significantly harder than CAD. A significant logarithmic reduction in hardness was associated with increased depth in sound dentin (R^2=0.983; p<0.0001), whereas a significant logarithmic increase in hardness was associated with increased depth in CAD (R^2=0.98; p<0.0001).

Regarding elastic modulus (Figure 2), significant effects were observed for the factor "dentin type" (p=0.005), the subplot "depth" (p<0.001), and their interaction (p<0.001). The power of performed tests was 0.75. Sound dentin had a significantly higher elastic modulus than CAD, except at depths of 150 and 200 μm. A significant linear increase in elastic modulus was associated with increased depth in both sound dentin (R^2=0.788; p=0.014) and CAD (R^2=0.967; p<0.001).

Residual Stress on FEA

Figures 3 and 4 illustrate the high stress concentration in the inferior region of the resin cement cylinders, corresponding to the contact loading application of the stainless steel wire used to loop the cylinders for the shear test. The peak in stress concentration (expressed in red color) into the dentin substrate near the bonded interface was verified in all groups.

Figure 3 shows the von Mises stress distribution for all experimental conditions tested. No appreciable differences were found between the resin cements or substrates. The stress concentration in the dentin substrate and at the bottom of the resin cement cylinder where the steel wire applied the load was consistent with the most frequent failure mode observed in the experimental tests, irrespective of the resin cement and substrate.

Figure 4 shows the stress distribution inside the bonded dentin area at the interface with the bottom of the resin cement cylinder. No appreciable differences were found between the resin cements or substrates tested. The Von Mises stress and maximal principal stress values obtained at the 10 nodes that coincided with the highest stress levels recorded inside the dentin substrate are shown in Table 4 and are coincident with most of the failure modes observed in the shear test. Similar values were found for both study factors.

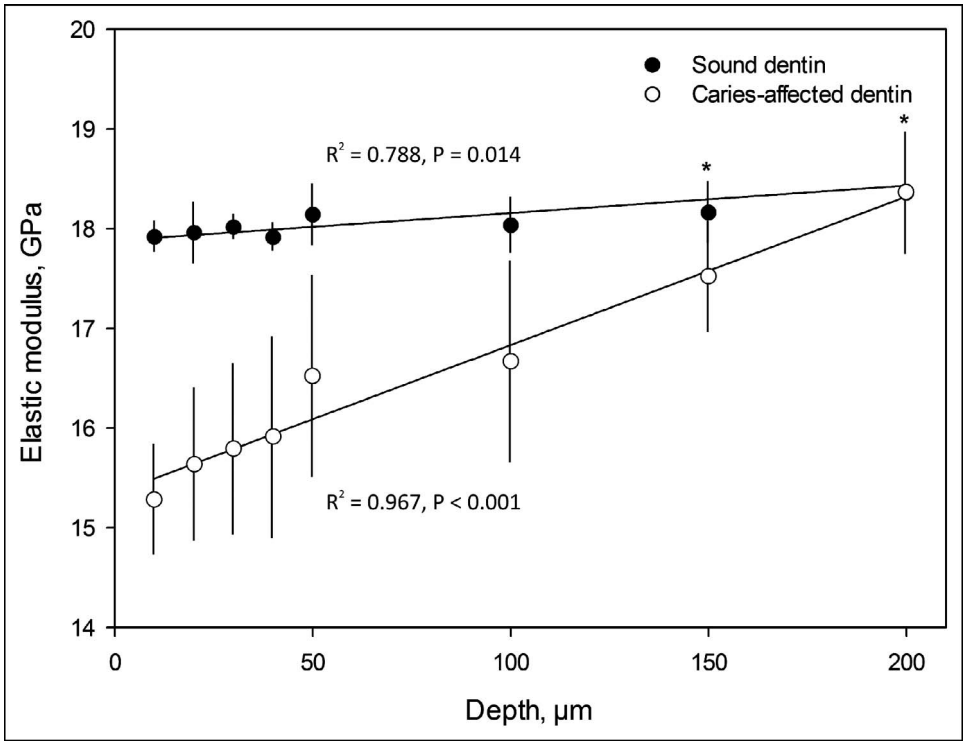

Figure 2. *Results and linear regression plots for elastic modulus (* indicates absence of significant difference between dentin types at the same depth; error bars indicate standard deviation. A linear increase in elastic modulus was associated with increased depth in sound and caries-affected dentin.*

DISCUSSION

The possible presence of CAD after conservative preparation requires bonding protocols that are effective when applied to this substrate. Studies on the bond strength of SARCs applied to CAD are lacking. Several studies have used extracted teeth with carious lesions for this purpose,[2,14,17,20,21,23] but it is difficult to standardize caries removal and to identify regions of dentin that are sufficiently similar for bond strength studies. Therefore, the results of studies using natural caries lesions as substrates for adhesion can be highly variable and may even impair the proper evaluation of adhesive

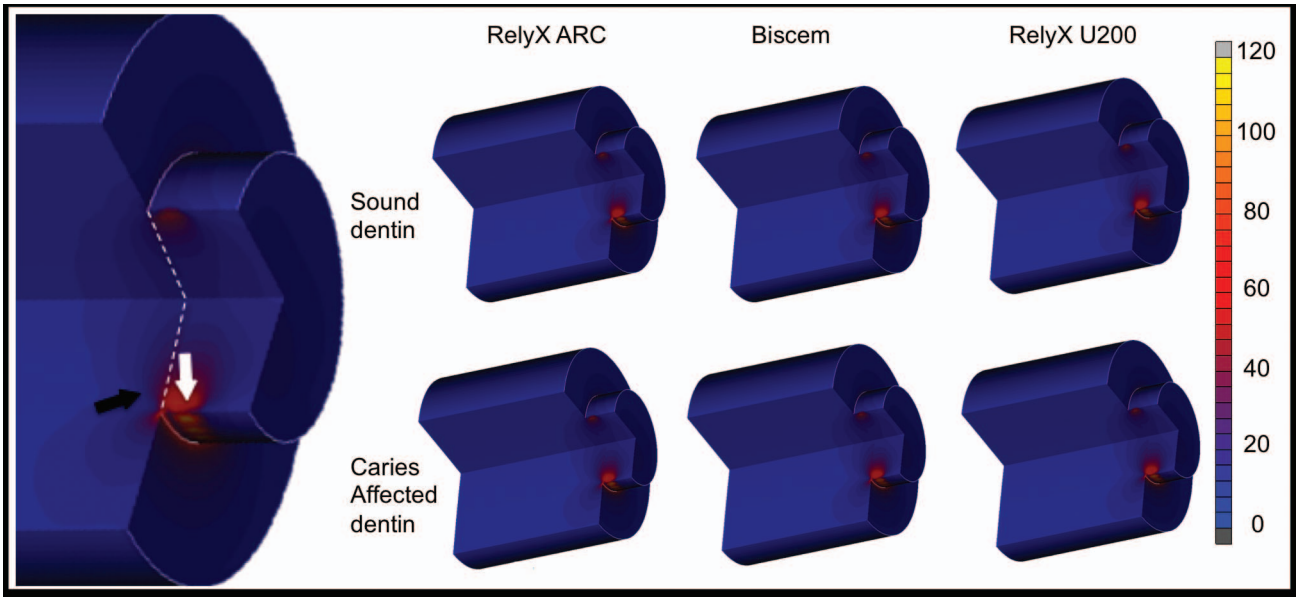

Figure 3. *Equivalent von Mises stress distribution (MPa) for resin cements bonded to sound and caries-affected dentin. The white arrow indicates the stress concentration in the resin cement cylinder; the black arrow indicates the peak in stress concentration (shown in red) into the dentin substrate near the bonded interface.*

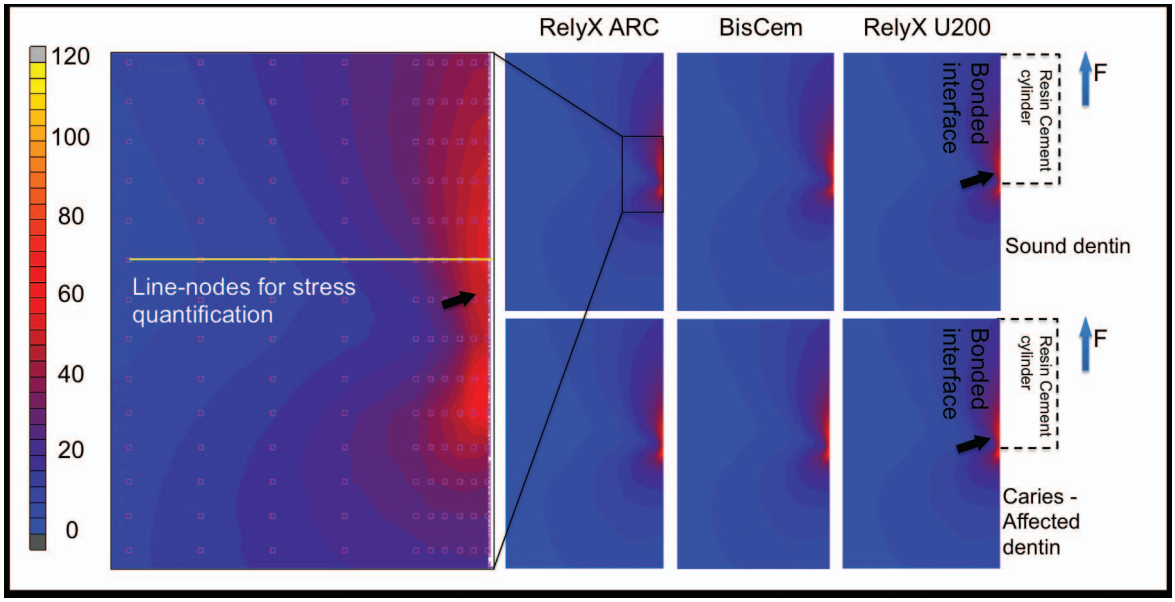

Figure 4. *Equivalent von Mises stress distribution (MPa) extracted into the cut dentin structure where the resin cement was bonded. Line nodes represent the nodes where stress values were extracted for quantitative analysis. Black arrows indicate the peak in stress concentration (shown in red) in the dentin substrate near the bonded interface.*

materials applied to CAD.[14,21] The present study used a microcosm biofilm model to induce carious lesions in dentin. This model simulates the complex interaction that occurs in the tooth substrate during the caries process and thus might be considered a more clinically relevant method than other artificial caries-induction methods, such as pH-cycling.

Evaluation of the mechanical properties of the substrates showed that the protocol used for caries induction resulted in lesions 100 μm deep. Above this depth, CAD exhibited reduced hardness compared with sound dentin. A similar trend was observed for elastic modulus, except that the substrates differed above a depth of 150 μm. The impaired mechanical properties of CAD near the surface where the biofilm was cultivated are due to a reduction of mineral content promoted by acids produced by bacterial metabolism, as occurs *in vivo*.[28,29] An important observation was the reduction of the hardness of sound dentin toward its deeper regions. This finding can be explained by the increased number and diameter of dentinal tubules toward the pulpal chamber, which reduces the area of inter- and peritubular dentin and consequently makes the substrate softer.[30]

Table 4: *Results of Maximum Principal Stress (σmax) and Von Misses Stress (vm) in MPa Versus Dentin Depth*

Distance to the Bonded Surface (mm)	RelyX ARC				BisCem				RelyX U200			
	Caries-affected Dentin		Sound Dentin		Caries-affected Dentin		Sound Dentin		Caries-affected Dentin		Sound Dentin	
	σmax	vm	σmax	vm	σmax	vm	σmax	vm	σmax	vm	σmax	vm
0.00	32.5	52.9	33.5	53.9	32.6	53.5	33.7	54.9	32.4	52.8	33.4	54.3
0.01	33.1	48.6	35.9	51.2	33.3	49.2	36.2	52.2	32.6	48.3	35.5	51.2
0.02	31.1	42.6	33.0	44.3	31.2	43.2	33.3	45.1	30.7	42.5	32.7	44.4
0.03	29.0	37.1	29.8	37.6	29.2	37.5	30.0	38.2	28.8	37.0	29.6	37.7
0.04	26.6	31.9	27.1	32.1	26.7	32.2	27.2	32.6	26.4	31.8	26.9	32.2
0.05	22.6	24.9	22.8	24.9	22.7	25.2	22.9	25.3	22.5	25.0	22.7	25.1
0.10	16.9	16.4	16.7	16.2	17.0	16.6	16.8	16.4	16.9	16.4	16.7	16.3
0.15	12.2	10.8	11.8	10.5	12.2	10.8	11.8	10.5	12.2	10.8	11.9	10.5
0.20	9.6	8.4	9.2	8.0	9.6	8.4	9.2	8.0	9.7	8.4	9.3	8.0
0.25	7.6	6.5	7.2	6.2	7.6	6.5	7.2	6.2	7.7	6.5	7.3	6.2

The bond strength of all of the evaluated resin luting agents was lower on CAD than on sound dentin. Thus, the study hypothesis was accepted. It could be expected that the reduced elastic modulus of CAD would result in a different stress distribution during the shear test compared with sound dentin. In this study, the difference in bond strength between the substrates could be partially explained by stress distribution during testing. The high stress concentration into dentin verified in all groups (Figures 3 and 4) suggests that the dentin pullout may be due to the biomechanics of the shear bond test.[31] The stress concentration into dentin located near the load application was similar for all groups (Figure 4). The peak in stress verified in the dentin may also contribute to failure of the bonded interface. Although the stress concentration was similar for sound and CAD, the strength of the CAD is lower; thus, the adhesive interface is more affected. Furthermore, the differences observed between the substrates might be explained by the dentin bonding mechanism.

The regular resin cement RelyX ARC is used in a three-step, etch-and-rinse adhesive system. This system forms a homogeneous and continuous hybrid layer in sound dentin, resulting in higher bond strength.[32] In CAD, possible collagen degradation promoted by carious lesions could result in the formation of a more heterogeneous hybrid layer with reduced cohesive strength.[22,23] Additionally, the reduction in mineral content allows deeper acid etching, increasing the extent of noninfiltrated exposed collagen.[23] A larger area of noninfiltrated collagen may act to concentrate stress during bond strength testing.[33]

The main bonding mechanism of SARCs, in contrast to regular resin cements, is chemical chelation between functional acid methacrylates and calcium from tooth tissues.[11,12] Thus, the reduced mineral content of CAD results in a less effective interaction with SARCs. In addition, a thicker and more organic smear layer is produced in CAD than in sound dentin, impairing the infiltration ability of SARCs.[34] However, it is important to emphasize that RelyX U200 showed higher bond strengths than BisCem. Other studies evaluating these two SARCs applied to sound dentin have had similar findings.[19,35,36] Differences in the composition of these two materials may explain these results. BisCem contains the highly hydrophilic monomer HEMA. Despite the absence of pulpal pressure simulation in the present study, this monomer is able to attract the intrinsic water of dentin, which may impair cement polymerization.[37] Impaired mechanical properties have been demonstrated for HEMA-based materials when stored in water for 24 hours.[38] In addition, greater interlocking within the underlying dentin was demonstrated for RelyX Unicem (which has a composition similar to that of U200) compared with BisCem using a Masson trichrome technique.[39]

The microcosm biofilm model used in the present study is a method that produces reproducible CAD. Despite the limitations of *in vitro* studies, our results show that the impaired mechanical properties (ie hardness and elastic modulus) caused by caries affect the bond strength of adhesive luting agents applied to dentin. However, clinical trials are necessary to confirm whether bonding to CAD during the cementation of indirect restorations may affect the survival rates of these restorations.

CONCLUSIONS

The mechanical properties alteration produced by the biofilm model reduced the bond strength of adhesive luting agents to caries-affected dentin.

Acknowledgments

A.C.P is grateful to Capes for her scholarship. This study was supported by Fapitec/Capes, grant # 23038.009072/2012-32.

Human Subjects Statement

This study was conducted at the Federal University of Sergipe in Brazil and was approved by the local research ethics committee (protocol #128.227/2012).

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 1 October 2014)

REFERENCES

1. Cardoso MV, de Almeida Neves A, Mine A, Coutinho E, Van Landuyt K, De Munck J, & Van Meerbeek B (2011) Current aspects on bonding effectiveness and stability in adhesive dentistry *Australian Dental Journal* **56(Supplement 1)** 31-44. http://dx.doi.org/10.1111/j.1834-7819.2011.01294.x.
2. de Almeida Neves A, Coutinho E, Cardoso MV, Lambrechts P, & Van Meerbeek B (2011) Current concepts and techniques for caries excavation and adhesion to residual dentin. *Journal of Adhesive Dentistry* **13(1)** 7-22. http://dx.doi.org/10.3290/j.jad.a18443
3. Hevinga MA, Opdam NJ, Frencken JE, Truin GJ, & Huysmans MC (2010) Does incomplete caries removal reduce strength of restored teeth? *Journal of Dental*

Research **89(11)** 1270-1275. http://dx.doi.org/10.1177/0022034510377790

4. Maltz M, & Alves LS (2013) Incomplete caries removal significantly reduces the risk of pulp exposure and post-operative pulpal symptoms *Journal of Evidence-Based Dental Practice* **13(3)** 120-122. http://dx.doi.org/10.1016/j.jebdp.2013.07.014
5. Edelhoff D, & Ozcan M (2007) To what extent does the longevity of fixed dental prostheses depend on the function of the cement? Working Group 4 materials: Cementation *Clinical Oral Implants Research* **18(Suppl 3)** 193-204. http://dx.doi.org/10.1111/j.1600-0501.2007.01442.x
6. Hickel R, & Manhart J (2001) Longevity of restorations in posterior teeth and reasons for failure. *Journal of Adhesive Dentistry* **31(1)** 45-64.
7. Abo-Hamar SE, Hiller KA, Jung H, Federlin M, Friedl KH, & Schmalz G (2005) Bond strength of a new universal self-adhesive resin luting cement to dentin and enamel *Clinical Oral Investigations* **9(3)** 161-167. http://dx.doi.org/10.1007/s00784-005-0308-5
8. Burke FJ, Fleming GJ, Abbas G, & Richter B (2006) Effectiveness of a self-adhesive resin luting system on fracture resistance of teeth restored with dentin-bonded crowns *European Journal of Prosthodontics and Restorative Dentistry* **14(4)** 185-188.
9. Ibarra G, Johnson GH, Geurtsen W, & Vargas MA (2007) Microleakage of porcelain veneer restorations bonded to enamel and dentin with a new self-adhesive resin-based dental cement *Dental Materials* **23(2)** 218-225. http://dx.doi.org/10.1016/j.dental.2006.01.013
10. Al-Assaf K, Chakmakchi M, Palaghias G, Karanika-Kouma A, & Eliades G (2007) Interfacial characteristics of adhesive luting resins and composites with dentine *Dental Materials* **23(7)** 829-839. http://dx.doi.org/10.1016/j.dental.2006.06.023
11. Ferracane JL, Stansbury JW, & Burke FJ (2011) Self-adhesive resin cements—Chemistry, properties and clinical considerations *Journal of Oral Rehabilitation* **38(4)** 295-314. http://dx.doi.org/10.1111/j.1365-2842.2010.02148.x
12. Gerth HU, Dammaschke T, Zuchner H, & Schafer E (2006) Chemical analysis and bonding reaction of RelyX Unicem and Bifix composites—A comparative study *Dental Materials* **22(10)** 934-941. http://dx.doi.org/10.1016/j.dental.2005.10.004
13. Monticelli F, Osorio R, Mazzitelli C, Ferrari M, & Toledano M (2008) Limited decalcification/diffusion of self-adhesive cements into dentin *Journal of Dental Research* **87(10)** 974-979. http://dx.doi.org/10.1177/154405910808701012
14. Sirin Karaarslan E, Yildiz E, Cebe MA, Yegin Z, & Ozturk B (2012) Evaluation of micro-tensile bond strength of caries-affected human dentine after three different caries removal techniques *Journal of Dentistry* **40(10)** 793-801. http://dx.doi.org/10.1016/j.jdent.2012.05.013
15. Yoshiyama M, Doi J, Nishitani Y, Itota T, Tay FR, Carvalho RM, & Pashley DH (2004) Bonding ability of adhesive resins to caries-affected and caries-infected dentin *Journal of Applied Oral Science* **12(3)** 171-176. http://dx.doi.org/10.1590/S1678-77572004000300002
16. Zanchia CH, D'Avila OP, Rodrigues-Junior SA, Burnett LH Jr, Demarco FF, & Pinto MB (2010) Effect of additional acid etching on bond strength and structural reliability of adhesive systems applied to caries-affected dentin *Journal of Adhesive Dentistry* **12(2)** 109-115. http://dx.doi.org/10.3290/j.jad.a17529
17. Joves GJ, Inoue G, Nakashima S, Sadr A, Nikaido T, & Tagami J (2013) Mineral density, morphology and bond strength of natural versus artificial caries-affected dentin *Dental Materials Journal* **32(1)** 138-143. http://dx.doi.org/10.4012/dmj.2012-243
18. Mobarak EH, El-Korashy DI, & Pashley DH (2010) Effect of chlorhexidine concentrations on micro-shear bond strength of self-etch adhesive to normal and caries-affected dentin *American Journal of Dentistry* **23(4)** 217-222.
19. Rocha C, Faria-E-Silva A, & Peixoto A (2014). Bond strength of adhesive luting agents to caries-affected dentin *Operative Dentistry* **39(4):** 383-388. http://dx.doi.org/10.2341/13-062-L
20. Suzuki TY, Godas AG, Guedes AP, Catelan A, Pavan S, Briso AL, & Dos Santos PH (2013) Microtensile bond strength of resin cements to caries-affected dentin *Journal of Prosthetic Dentistry* **110(1)** 47-55. http://dx.doi.org/10.1016/S0022-3913(13)60339-X
21. Mobarak EH, & El-Badrawy WH (2012) Microshear bond strength of self-etching adhesives to caries-affected dentin identified using the dye permeability test *Journal of Adhesive Dentistry* **14(3)** 245-250. http://dx.doi.org/10.3290/j.jad.a22420
22. Lenzi TL, Braga MM, & Raggio DP (2014) Shortening the etching time for etch-and-rinse adhesives increases the bond stability to simulated caries-affected primary dentin *Journal of Adhesive Dentistry* **6(3)** 235-241 http://dx.doi.org/10.3290/j.jad.a31103
23. Lenzi TL, Tedesco TK, Calvo AF, Ricci HA, Hebling J, & Raggio DP (2014) Does the method of caries induction influence the bond strength to dentin of primary teeth? *Journal of Adhesive Dentistry* **16(4)** 333-338, http://dx.doi.org/10.3290/j.jad.a31799
24. Filoche SK, Soma KJ, & Sissons CH (2007) Caries-related plaque microcosm biofilms developed in microplates *Oral Microbiology Immunology* **22(2)** 73-79, http://dx.doi.org/10.1111/j.1399-302X.2007.00323.x
25. Wong L, & Sissons C (2001) A comparison of human dental plaque microcosm biofilms grown in an undefined medium and a chemically defined artificial saliva *Archives of Oral Biology* **46(6)** 477-486, http://dx.doi.org/10.1016/S0003-9969(01)00016-4
26. Kinney JH, Gladden JR, Marshall GW, Marshall SJ, So JH, & Maynard JD (2004) Resonant ultrasound spectroscopy measurements of the elastic constants of human dentin *Journal of Biomechanics* **37(4)** 437-441, http://dx.doi.org/10.1016/j.jbiomech.2003.09.028
27. Pereira RD, Vadivia AD, Bicalho AA, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2014) Effect of photo-activation protocol on mechanical properties of

resin cements and bond strength of fiberglass posts to root dentin *Operative Dentistry* In press, http://dx.doi.org/10.2341/14-115-L.

28. Joves GJ, Inoue G, Sadr A, Nikaido T, & Tagami J (2014) Nanoindentation hardness of intertubular dentin in sound, demineralized and natural caries-affected dentin *Journal of the Mechanical Behavior Biomedical Materials* **32** 39-45, http://dx.doi.org/10.1016/j.jmbbm.2013.12.017
29. Almahdy A, Downey FC, Sauro S, Cook RJ, Sherriff M, Richards D, Watson TF, Banerjee A, & Festy F (2012) Microbiochemical analysis of carious dentine using Raman and fluorescence spectroscopy *Caries Research* **46(5)** 432-440, http://dx.doi.org/10.1159/000339487
30. Schilke R, Lisson JA, Bauss O, & Geurtsen W (2000) Comparison of the number and diameter of dentinal tubules in human and bovine dentine by scanning electron microscopic investigation *Archives of Oral Biology* **45(5)** 355-361, http://dx.doi.org/10.1016/S0003-9969(00)00006-6
31. Versluis A, Tantbirojn D, & Douglas WH (1997) Why do shear bond tests pull out dentin? *Journal of Dental Research* **76(6)** 1298-1307, http://dx.doi.org/10.1177/00220345970760061001
32. Sarr M, Kane AW, Vreven J, Mine A, Van Landuyt KL, Peumans M, Lambrechts O, Van Meerbeek B, & De Munck J (2010) Microtensile bond strength and interfacial characterization of 11 contemporary adhesives bonded to bur-cut dentin. *Operative Dentistry* **35(1)** 94-104, http://dx.doi.org/10.2341/09-076-L
33. Hashimoto M, Ohno H, Kaga M, Sano H, Tay FR, Oguchi H, Araki Y, & Kubota M (2002) Over-etching effects on micro-tensile bond strength and failure patterns for two dentin bonding systems *Journal of Dentistry* **30(2-3)** 99-105, http://dx.doi.org/10.1016/S0300-5712(02)00004-0
34. Kunawarote S, Nakajima M, Foxton RM, & Tagami J (2011) Effect of pretreatment with mildly acidic hypochlorous acid on adhesion to caries-affected dentin using a self-etch adhesive *European Journal of Oral Sciences* **119(1)** 86-92, http://dx.doi.org/10.1111/j.1600-0722.2010.00788.x
35. Faria-e-Silva AL, Menezes MS, Silva FP, Reis GR, & Moraes RR (2013) Intra-radicular dentin treatments and retention of fiber posts with self-adhesive resin cements *Brazilian Oral Research* **27(1)** 14-19, http://dx.doi.org/10.1590/S1806-83242013000100003
36. Lisboa DS, Santos SV, Griza S, Rodrigues JL, & Faria-e-Silva AL (2013) Dentin deproteinization effect on bond strength of self-adhesive resin cements *Brazilian Oral Research* **27(1)** 73-75, http://dx.doi.org/10.1590/S1806-83242013000100013
37. Collares FM, Ogliari FA, Zanchi CH, Petzhold CL, Piva E, & Samuel SM (2011) Influence of 2-hydroxyethyl methacrylate concentration on polymer network of adhesive resin *Journal of Adhesive Dentistry* **13(2)** 125-129, http://dx.doi.org/10.3290/j.jad.a18781
38. Papadogiannis D, Lakes RS, Papadogiannis Y, & Tolidis K (2013) Mechanical viscoelastic behavior of dental adhesives *Dental Materials* **29(6)** 693-701, http://dx.doi.org/10.1016/j.dental.2013.03.017
39. Mazzitelli C, Monticelli F, Toledano M, Ferrari M, & Osorio R (2010) Dentin treatment effects on the bonding performance of self-adhesive resin cements *European Journal of Oral Sciences* **118(1)** 80-86, http://dx.doi.org/10.1111/j.1600-0722.2009.00703.x

©Operative Dentistry, 2015, **40-3,** E112-E121

Shear Bond Strength of Porcelain Veneers Rebonded to Enamel

HA St Germain Jr • TH St Germain

Clinical Relevance

Adhesive debonding of porcelain veneer restorations occurs infrequently; however, an intact veneer with residual resin cement can be rebonded. The use of a noninvasive technique to remove the resin cement facilitates attaining an enamel bond strength similar to a newly cemented porcelain veneer.

SUMMARY

In this laboratory research, shear bond strength (SBS) and mode of failure of veneers rebonded to enamel in shear compression were determined. Three groups (A, B, and C; n=10 each) of mounted molar teeth were finished flat using wet 600-grit silicon carbide paper, and 30 leucite-reinforced porcelain veneers (5.0 × 0.75 mm) were air abraded on the internal surface with 50 μm aluminum oxide, etched with 9.5% hydrofluoric acid, and silanated. The control group (A) veneer specimens were bonded to enamel after etching with 37% phosphoric acid using bonding resin and a dual cure resin composite cement. Groups B and C were prepared similarly to group A with the exception that a release agent was placed before the veneer was positioned on the prepared enamel surface and the resin cement was subsequently light activated. The debonded veneers from groups B and C were placed in a casting burnout oven and heated to 454°C/850°F for 10 minutes to completely carbonize the resin cement and stay below the glass transition temperature (T_g) of the leucite-reinforced porcelain. The recovered veneers were then prepared for bonding. The previously bonded enamel surfaces in group B were air abraded using 50 μm aluminum oxide followed by 37% phosphoric acid etching, while group C enamel specimens were acid etched only. All specimens were thermocycled between 5°C and 55°C for 2000 cycles using a 30-second dwell time and stored in 37°C deionized water for 2 weeks. SBS was determined at a crosshead speed of 1.0 mm/min. SBS results in MPa for the groups were (A) = 20.6±5.1, (B) = 18.1±5.5, and (C) = 17.2±6.1. One-way analysis of variance indicated that there were no significant interactions (α=0.05), and Tukey-Kramer *post hoc* comparisons (α=0.05) detected no significant pairwise differences. An adhesive mode of failure at the enamel interface was observed to occur more often in the experimental groups (B = 40%, C = 50%). Rebonding the veneers produced SBS values that were not significantly different from the control group. Also, no significant difference in SBS values were

*Henry A St Germain Jr, DMD, MSD, MAEd, Section of Operative Dentistry, Department of Adult Restorative Dentistry, University of Nebraska Medical Center College of Dentistry, Lincoln, NE, USA

Thomas H St Germain, DDS, MBA, private practice, Omaha, NE, USA

*Corresponding author: University of Nebraska Medical Center College of Dentistry, Adult Restorative Dentistry, 4000 East Campus Loop South, Lincoln, NE 68583-0740, USA; e-mail: hstgerma@unmc.edu

DOI: 10.2341/14-123-L

observed whether the debonded enamel surface was air abraded and acid etched or acid etched only.

INTRODUCTION

Porcelain veneer restorations were introduced in the early 1980s as a conservative and reliable esthetic restorative service.[1-3] These restorations can be used to modify tooth color and shape, close diastemata, and improve minor alignment problems. Significant changes in clinical procedures and porcelain materials have occurred during the past 25 years.[4-9] Low-fusing feldspathic porcelain was the first veneer restoration material used with a high degree of clinical success, and, more recently, leucite-reinforced and lithium-disilicate porcelains are now routinely used for porcelain veneer restorations. Clinically, the long-term success of porcelain veneer restorations is dependent on a preparation substrate in enamel, favorable occlusal relationships, and the ability of the porcelain to be etched and adhesively bonded to resin composite cements.[10,11] Although porcelain veneers are generally successful under the appropriate clinical conditions, it is recommended that informed consent be provided to patients, including the possibilities of postoperative sensitivity, marginal discoloration, fracture, debonding, and wear of opposing teeth.[12]

Fractures of porcelain veneers represent the most common mode of failure.[13-15] A static fracture occurs when a veneer segment fractures and the remainder of the veneer still remains intact on the tooth. A cohesive fracture is categorized by a loss of a segment of porcelain and typically occurs when there is excessive functional or parafunctional loading.[10,13] Cohesive fracture of a veneer restoration can potentially be repaired with resin composite as a short-term treatment option; however, a new veneer or full-coverage crown is often necessary.[16] When a porcelain veneer completely debonds from the tooth in an adhesive failure mode and is still intact, an assessment is necessary to determine whether there is resin cement remaining on the tooth surface or on the internal surface of the veneer. If resin cement remains on the tooth, then the failure is likely due to a lack of adequate etching and silanation of the intaglio (internal) porcelain surface. On the other hand, if resin cement remains inside the veneer, then a poor-quality bond to the enamel substrate or perhaps a lack of adequate enamel with exposed dentin for adhesive bonding can be identified as the problem.[9,17,18]

Contributing factors leading to the occurrence of fractures and/or debonding include exposure of dentin during tooth preparation, placing finish lines on large composites, bonding to endodontically treated teeth, and heavy parafunctional occlusal loading.[5] A prospective, however relatively short-term clinical study published in 2009, evaluated the survival of 200 porcelain veneer restorations up to a period of 72 weeks.[19] The most frequent failure type noted was debonding, occurring with 11 units , with nine veneer restorations being able to be rebonded, while two required modification of the preparation design and new veneer restorations. Porcelain veneers that are bonded to dentin at the margins or internally are more likely to debond than veneers bonded entirely within enamel.[17] When confronted clinically with a significant amount of exposed dentin substrate, rebonding a porcelain veneer restoration will not be as successful as bonding to enamel entirely. Typically, when the porcelain veneer restoration fails and the tooth preparation has exposed dentin, the restoration will likely need to be changed to a full-coverage crown in order to gain the advantage of mechanical retention. The authors of a 16-year prospective study in 2007 reported that the overall survival rate of porcelain veneer restorations was 73%. Failed restorations were associated with unacceptable esthetics and mechanical complications that included occlusal trauma, veneer fracture, and loss of retention. Although loss of retention accounted for only 12.5% of failures, this clinical problem may not require restoration remakes and potentially can be managed by rebonding the veneer(s).[20]

In cases where adhesive bond failures with porcelain veneer restorations occur, resin cement is usually retained on the internal surface of the porcelain veneer, and it is difficult to distinguish cement from the porcelain, especially when the resin cement is a similar shade. A predictable and noninvasive method for removing resin cement from the veneer is essential in order to rebond the restoration. References available in the literature to remove luting composite from inside a debonded intact veneer have recommended using a casting burnout oven or porcelain ovens.[12,21] The porcelain veneer with bonded resin cement is placed in the oven, and the temperature is increased to 600°C/1112°F and held at that temperature for 5 to 30 minutes. The porcelain veneer can then be removed, cooled to room temperature, cleaned with acetone, re-etched with 9.5% hydrofluoric acid, rinsed with water, dried, and coated with silane in preparation

for rebonding. It is important to note that the type of porcelain (feldspathic, LR, or lithium disilicate) and the glass transition temperature (T_g) is not identified in these studies. The clinician needs to be fully aware of the type of porcelain and its T_g before subjecting the restoration to such high temperatures and risking deformation of the veneer restoration.

In addition to removing the resin composite cement from inside the porcelain veneer, successful rebonding of veneer restorations also requires attention to preparing the previously bonded tooth enamel substrate for subsequent bonding. Although resin cements can be bonded effectively to mineralized tooth surfaces, bonding to a previously resin-polymerized enamel surface requires additional consideration. Research investigating the repair of resin composite restorations suggests using a carbide or diamond bur with or without 50 μm aluminum oxide (Al_2O_3) air abrasion and/or 30 μm silica coating air abrasion for preparing a resin composite surface for rebonding.[23-25] Acid etching or the use of Al_2O_3 or silica-coating air abrasive techniques are not likely to cause significant changes in morphology of the prepared enamel surface; however, modification of the tooth preparation by roughening with a bur could alter the original enamel preparation, including finish lines, compromising a rebonding procedure. .

The purpose of this laboratory research was to investigate the efficacy of rebonding a debonded porcelain veneer restoration after determining a noninvasive procedure to remove resin composite cement from the internal surface of a leucite-reinforced (LR) porcelain veneer restoration.

METHODS AND MATERIALS

Thirty freshly extracted human molar teeth (UNMC-IRB#258-08NH) stored in 0.1% thymol were randomly divided into three groups of 10 and thoroughly rinsed with deionized water. The teeth were mounted in a 1 × ¾-inch phenolic ring form using Epoxide resin (Buehler, Lake Bluff, IL, USA) and finished to a flat surface using wet 600-grit silicon carbide (SiC) paper to produce a surface area of enamel 8.0 mm in diameter. .

Thirty OPC (Jeneric/Pentron, Wallingford, CT, USA) LR pressed porcelain veneer (5.0 × 0.75 mm) specimens were air abraded on the inner surface with 50 μm Al_2O_3 at 30 psi for 5 seconds (Micro-Etcher, Danville Engineering Co, Danville, CA, USA), rinsed for 15 seconds with deionized water, etched with 9.5% hydrofluoric acid for 1 minute (Ultradent Porcelain Etch, Ultradent Products, Inc, South Jordan, UT, USA), rinsed with deionized water for 15 seconds, air-dried, and coated with silane (Kerr Silane Primer, Kerr Corp, Orange, CA, USA) for 2 minutes followed by air drying to remove excess silane. The control group (A) specimens (n=10) were prepared and bonded to an etched (37% phosphoric acid for 15 seconds, followed by a 15-second deionized water rinse, followed by air drying) enamel surface using Optibond Solo Plus adhesive resin and NX3 Nexus dual-cure resin composite cement according to the manufacturer's directions (Kerr Corp). A 200-kg perpendicular load using a dental surveyor was placed on the external surface of the veneer after cement application on the internal surface of the veneer for 30 seconds, excess resin cement was removed with a brush, and the external surface of the veneer was light activated for 40 seconds using a Model 101 SmartLite PS (Dentsply/Caulk, Milford, DE, USA) LED curing light with an 11-mm-diameter tip positioned 1.0 mm from the veneer surface.

According to the manufacturer, the LED curing light was fully compatible with the initiator chemistry of the bonding resin and resin composite cement used, and the light irradiance (intensity) of the LED curing light was confirmed with a Model 100 Curing Radiometer (Demetron Research Corp, Danbury, CT, USA) before light activating each specimen to be ≥800 mW/cm^2. Group A served as the control specimens and were stored in 37°C deionized water prior to further testing. Groups B (n=10) and C (n=10) were prepared similarly to group A with the exception that a 12-μm film thickness release agent (Rubber-Sep, George Taub Products, Inc, Jersey City, NJ, USA) was placed on the enamel surface immediately before the conditioned veneer with resin composite cement was seated on the enamel tooth substrate in order to simulate an adhesive debonding at the enamel–resin composite cement interface of the veneer specimen. After two trials with temperatures at 315°C/600°F and 398°C/750°F for 10 minutes each without successfully removing the resin composite, the debonded veneers from groups B and C were placed in a calibrated Radiance Multi-stage casting burnout oven (Radiance Co, St. Louis, MO, USA) and heated to 454°C/850°F for 10 minutes in a clean ceramic crucible in order to carbonize the resin composite cement well below the T_g of 590°C/1094°F[26] of the LR porcelain material. The internal surfaces of the veneers were air abraded with 50 μm Al_2O_3 for 5 seconds, which easily cleaned the residual carbonized resin compos-

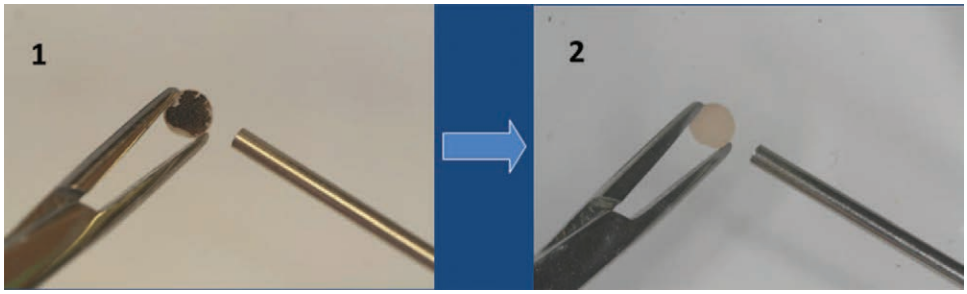

Figure 1. *Porcelain veneer specimen after carbonization at 454°C/850°F in a casting oven (1) and prepared for rebonding by cleaning with 50 μm aluminum oxide (2).*

ite cement, and were inspected using a 20× light microscope to verify removal of all visible residual resin cement, as depicted in Figure 1.

The recovered veneer specimens and enamel surfaces were then prepared for rebonding. Preparation of the previously bonded enamel surfaces for group B specimens included air abrasion using 50 μm Al_2O_3 for 5 seconds followed by rinsing with deionized water, air drying, and an application of 37% phosphoric acid etching for 15 seconds followed by rinsing with deionized water and air drying. Group C previously bonded enamel surfaces were etched only with 37% phosphoric acid for 15 seconds, rinsed with deionized water, and air-dried. After the group B and C specimens were rebonded, all of the bonded veneer specimens were thermocycled between 5°C and 55°C for 2000 cycles using a 30-second dwell time. All specimens were then stored in 37°C deionized water for 2 weeks.

Group A, B, and C specimens were subsequently loaded in shear compression until fracture occurred using an Instron Universal testing machine Model 1123 (Instron Corp, Canton, MA, USA) with Bluehill software at a crosshead speed of 1.0 mm/min. The Instron testing configuration is illustrated in Figure 2. The bond strength at failure/fracture in megapascals (MPa) was recorded for all specimens. Observation of the mode of failure was also performed with a binocular light microscope at 20× magnification to categorize failure as adhesive or cohesive. Representative samples from the control group and each experimental group were sputter coated with a 20-μm layer of gold-palladium and examined with a JEOL JSM-6100 Scanning Electron Microscope (JEOL USA, Inc, Peabody, MA, USA) at 200× using an acceleration voltage of 25 kV to assist in visualization of the debonded or fractured specimens.

Using a one-way analysis of variance (ANOVA), shear bond strength (SBS) values of the control and experimental groups were compared (α=0.05). Tukey-Kramer *post hoc* tests were conducted to determine pairwise differences between the groups (α=0.05).

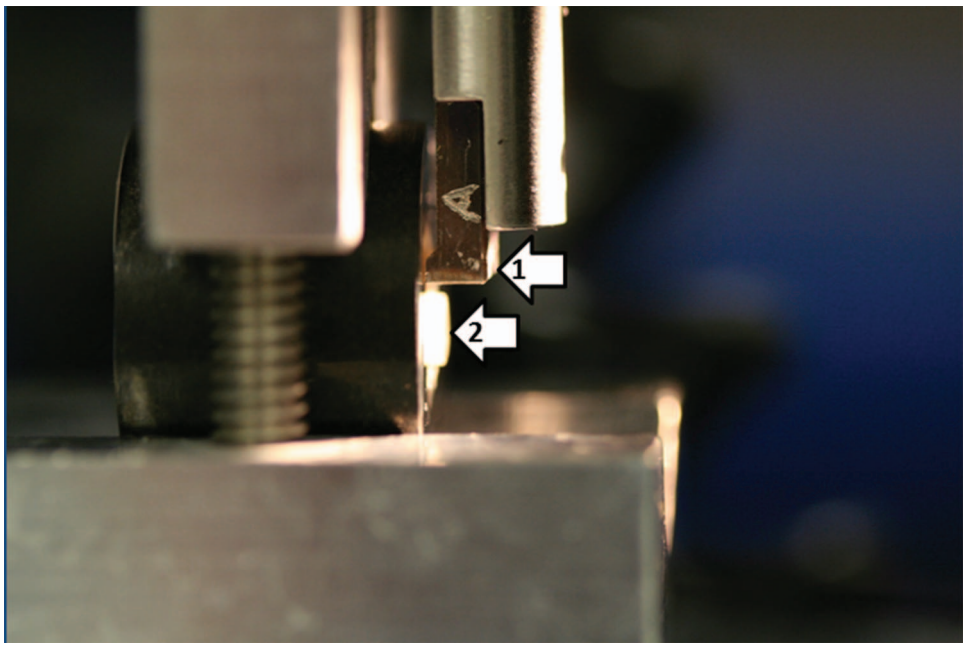

Figure 2. *Orientation of the chisel tip (1) and porcelain veneer specimen positioned on the Instron for shear bond strength testing.*

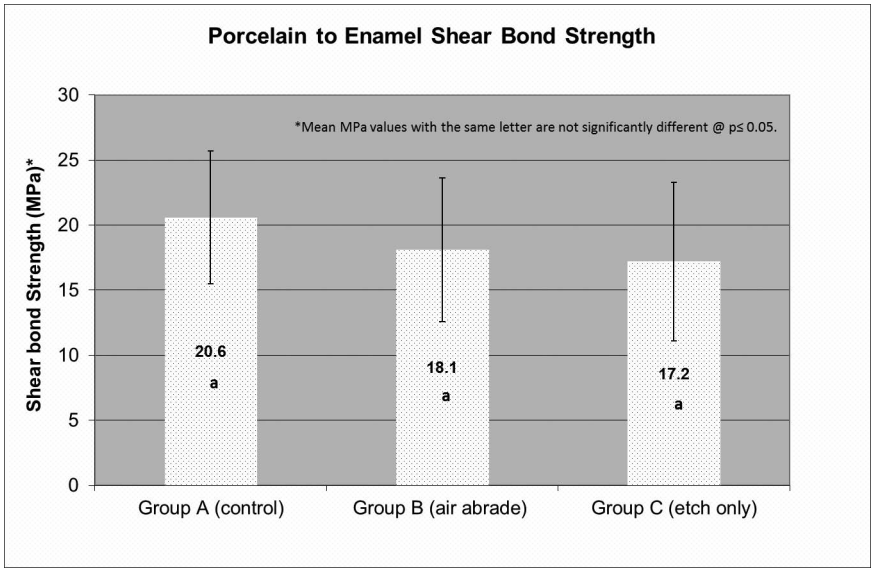

Figure 3. *Porcelain to enamel shear bond strength (SBS) results. Group A had the highest mean SBS values but was not statistically different from groups B (α=0.05) and C (α=0.05.*

The null hypothesis tested was that the SBS of veneer restorations after the experimental rebonding procedure would not be significantly different from the control bond strength.

RESULTS

SBS results in MPa (mean±SD) for the specimen groups were (A) 20.6 ± 5.1, (B) 18.1 ± 5.5, and (C) 17.2 ± 6.1, as depicted in Figure 3. One-way ANOVA indicated that there were no significant interactions. Tukey-Kramer *post hoc* comparisons detected no significant pairwise differences, and the null hypothesis was accepted. Group A (control) specimens had the highest mean SBS; however, this was not significantly different at α=0.05 from the experimental groups (B and C).

The mode of shear bond failure observed was either adhesive or cohesive. Adhesively debonded specimens appeared to have minimal residual adhesive resin on the enamel surface. No adhesive failures were observed at the veneer–resin cement interface. Cohesively fractured specimens confirmed a mode of failure within the LR ceramic material itself. No cohesive failures were observed with the enamel tooth structure. An 80% incidence of cohesive fracture was observed in group A (control group), and rebonded groups B and C had 60% and 50% cohesive fracture, respectively. Adhesive debonding was observed to occur 20% of the time for control group A, 40% for experimental group B, and 50% for experimental group C, as illustrated in Figure 4. No damage to the prepared enamel surface was noted

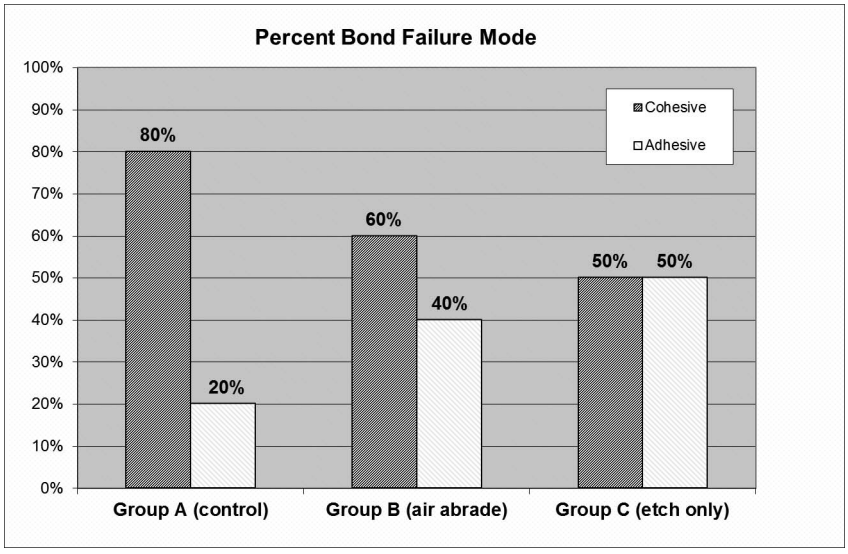

Figure 4. *Percentage bond failure mode for groups A, B, and C. Group A had 80% cohesive fracture, and groups B and C had higher percentages (40% and 50%) of adhesive debonding.*

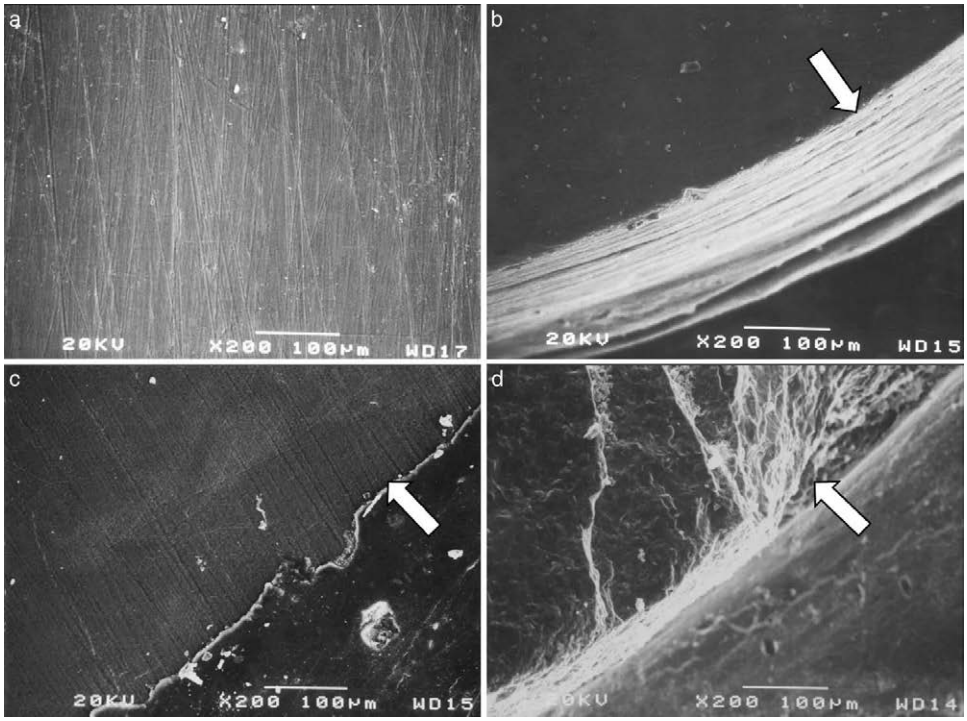

Figure 5a. *Scanning electron microscopic image (200×) illustrating the 600-grit carborundum paper finishing of the unbonded enamel surface.*

Figure 5b. *Scanning electron microscopic image (200×) depicting an intact porcelain veneer specimen edge prior to shear bond testing.*

Figure 5c. *Scanning electron microscopic image (200×) showing an adhesively debonded enamel surface interface after shear bond testing.*

Figure 5d. *Scanning electron microscopic image (200×) illustrating a cohesively fractured porcelain veneer interface after shear bond testing.*

during the SBS testing since the 600-grit SiC surface striations from the enamel surface preparation were visible at 20× magnification and appeared undisturbed.

Although significant differences were not present between the SBS mean values, the experimental groups (B and C) had lower SBS mean values and a higher incidence of adhesive failure compared with group A. Scanning electron micrographic (SEM) photomicrographs of the prepared enamel surface finished wet with 600-grit SiC paper, intact veneer interface, adhesive debonding interface, and cohesively fractured porcelain material at a magnification of 200× are depicted in Figures 5 (a-d) .

DISCUSSION

In situations where sufficient enamel remains for successful adhesive bonding, an attempt to rebond an intact porcelain veneer restoration may be an appropriate treatment option. The results of this study suggest that conservative clinical procedures can be used to predictably remove resin composite cement from the internal surface of an adhesively debonded LR porcelain veneer restoration and to rebond the veneer restoration to the previously prepared and bonded enamel tooth surface. The null hypothesis was accepted whereby the mean SBS values of the control and experimental (rebonded) specimen groups were not significantly different.

The mean SBS value range of 17.2 to 20.6 MPa in our study is consistent with other research investigating the SBS of LR porcelain veneer materials bonded to enamel.[27,28] The OPC LR porcelain material (Jeneric/Pentron) used in this study has been shown to be similar to the Empress (Ivoclar/Vivadent AG, Schaan, Liechtenstein) LR porcelain material. Empress is more familiar to clinicians than OPC since it has been marketed for a longer period of time and continues to be available. Although Empress is "precerammed" and OPC is not, after heat processing, the two materials show no differences in mechanical properties.[29]

A clinical procedure for rebonding an adhesively debonded and intact porcelain veneer recommends the removal of the resin composite cement from the inside of the veneer with carbide burs and/or diamond burs using magnification followed by sandblasting, re-etching with hydrofluoric acid, resilanating, and recementation.[9] Although this technique seems reasonable, there is a risk of damaging the veneer, and a noninvasive means of resin cement removal from the porcelain veneer would more favorably minimize risk. In 1990 , it was reported that successfully burning out the residual resin cement from the internal surface of an adhesively debonded acid-etched fixed partial denture would require placing the prosthesis in a casting burnout oven at 700°C/1292°F for 10 to 15 minutes, followed by ultrasonic cleaning for 5

minutes.[22] This protocol was developed for use with a base metal alloy, and if used with an LR porcelain, permanent deformation of the restoration would occur due to the burnout temperature exceeding the T_g (590°C/1094°F) of the LR porcelain.

Since it was known that previously published temperature recommendations for resin cement burnout were established for use with feldspathic porcelains (T_g range of 518°C/950°F to 945°C/1733°F), these high temperatures would have closely approached or exceeded the T_g for the OPC LR porcelain, and it was decided to initially expose the resin-bonded porcelain specimen to 315°C/600°F for 30 minutes.[12,21] This temperature did not result in any carbonization of the resin composite cement. It was subsequently decided to run a trial for 10 minutes at 398°C/750°F, and although the resin composite did exhibit a carbonized, or "charred," surface, Al_2O_3 air abrasion was not able to easily remove the resin composite cement, and the use of rotary instrumentation to remove residual cement was considered potentially damaging.. We determined that the best results for complete carbonization of the resin composite cement were achieved at a temperature of 454°C/850°F held for 10 minutes, and the residual resin cement was very easily removed using 50 μm Al_2O_3 air abrasion. This temperature was below the OPC porcelain T_g of 590°C/1094°F, so deformational damage to the veneer was not anticipated. It is possible that an increase in the cement carbonization temperature we used for a shorter period of time might also be effective in removing resin cement from LR veneers, and future research is needed to identify the best temperature and time combination for both LR and newer lithium disilicate porcelain veneer materials. The T_g of a resin composite material 75% filled by weight is 107°C/225°F[30]; however, the disintegration temperature values of resin composite materials used for cementation have not been reported in the literature. Newer resin composite cements typically have higher filler levels than previously available resin cements and may likely require higher burnout temperatures for adequate carbonization to occur.

It was preferable to use a casting burnout oven instead of a porcelain oven for this procedure since contamination of the porcelain oven "muffle" due to the carbonization of the resin composite material may cause discoloration of subsequently fired porcelain.[31] Although 454°C/850°F for 10 minutes was optimally effective for the removal of the particular brand of resin composite cement used in our research and well below the T_g of the LR porcelain material, it is suggested that future research be done to quantify both the T_g and the carbonization temperatures of currently available resin composite materials.

The results of the SBS tests for the bonded control veneer specimens had higher mean values; however, as mentioned previously, these values were not significantly different from the rebonded (experimental) veneer specimens. The adhesively debonded enamel interface illustrated in Figure 5c shows the striations produced by the 600-grit SiC wet paper finishing of the enamel surface prior to bonding (Figure 5a). It is likely that this adhesively debonded enamel surface has residual resin tags in the tooth structure that would favor rebonding, as suggested in previous research.[32] An intact veneer specimen top edge is illustrated in Figure 5b, providing a frame of reference for the cohesively fractured porcelain material illustrated in Figure 5d. The veneer specimen is still bonded to enamel, and it is evident that the edge of the veneer in Figure 5d has been damaged and that fragments of porcelain material have been lost.

Our research simulated a "clean" adhesive failure of a porcelain veneer between the acid-etched and bonding resin-coated enamel surface and the resin composite cement inside the veneer specimen. It is important to mention that the rebonded veneer specimens that were either acid-etched only (group C) or acid-etched after Al_2O_3 air abrasion (group B) had lower mean SBS values than the control veneer group, and although this was not significant, the rebonded veneer specimens did have a higher incidence of adhesive failure at the enamel interface compared to the bonded control veneer specimens. This may not be problematic clinically, however, since if the rebonded porcelain veneer would subsequently fail again in an adhesive mode, it could be rebonded a second time; however, at this point, it may be advisable to consider other, less conservative restorative options. These results are in agreement with previous research findings that when the SBS values are lower with a variety of porcelain veneer, cement, and tooth surface interfaces, there is a higher incidence of adhesive debonding.[5,17,33] Clinical research is needed to validate the significance of these laboratory research findings.

Suggested clinical procedures for repairing resin composite restorations have included the use of rotary instrumentation to remove partially bonded composite, air abrasion with Al_2O_3 or silica, phosphoric acid etching, and placement of a bonding resin.[23-25] Pit and fissure sealants often have to be reapplied during recall examination, and, similar to

the recommendations for repairing composite resin, it is suggested that visibly remaining sealant be removed with a bur or Al_2O_3 air abrasion; however, cleaning of the enamel surface with nonfluoridated pumice is also recommended before phosphoric acid etching.[34] In our research, roughening the enamel surface with rotary instrumentation was not desirable since we did not want to alter the tooth preparation and increase the cement film thickness of the rebonded veneer. It has been reported that if the fit of the porcelain veneer has a resin cement thickness of ≤ 50 μm, there will be a lower incidence of adhesive bond failure.[35]

A laboratory study published in 1980 revealed that when a composite restoration breaks cleanly at the enamel–composite interface, the best procedure for rebonding to that surface is by simply etching the debonded enamel surface with 37% phosphoric acid to avoid rotary instrumentation removal of rebondable resin tags remaining on the enamel surface.[32] In addition, other research investigating Al_2O_3 air abrasion as an enamel surface treatment prior to acid etching for rebonding metallic orthodontic brackets did not result in significantly higher SBS compared to acid etching alone.[36] Although these research studies used different resin composite materials than our current study, acid-etching procedures, the use of bonding resins, and the enamel substrate can be considered similar, and the results of our current study are in agreement with the findings of these prior studies that acid etching alone is sufficient for surface preparation of the previously bonded enamel surface. It is realistic to assume, however, that if minor fragments of resin composite cement adhering to the enamel surface interfere with the complete seating of the porcelain veneer, judicious removal of the fragment with a finishing carbide or fine diamond bur may be required.

CONCLUSIONS

The long-term success of veneer restorations is dependent on an adequate enamel surface for bonding with a conservative preparation design, favorable occlusal considerations, and effective adhesive bonding systems. The results of this research suggest a conservative clinical technique when attempting to rebond a porcelain veneer restoration. The use of a casting burnout oven to selectively carbonize the resin composite cement at 454°C/850°F for 10 minutes, which was below the T_g of the LR porcelain veneer material, facilitated rebonding of an adhesively debonded veneer to a previously bonded and prepared tooth enamel surface.

No significant differences in SBS (MPa) were observed in the rebonded veneer groups regardless of whether the previously bonded enamel surface was air abraded with 50 μm Al_2O_3. The mean SBS values of the control group (A) were higher than the experimental groups (B and C), and 80% of these specimens fractured cohesively; however, these values were not significantly different from the mean SBS of the experimental groups. The incidence of adhesive shear bond failure at the resin composite cement–enamel surface interface was noted to be higher in the rebonded experimental groups than in the control group.

SEM photomicrographs at a magnification of 200× suggest that only bonding resin remained on the enamel substrate for the adhesively debonded veneer specimens. Also, failure within the ceramic material itself was evident for cohesively fractured veneer specimens. Future research is needed to evaluate the efficacy of rebonding veneer restorations fabricated from lithium disilicate porcelain bonded with the newly available adhesive resin composite cements.

Acknowledgements

Appreciation is extended to the following individuals: Dr Mark Beatty, professor and director, Section of Dental Biomaterials, Department of Adult Restorative Dentistry, UNMC College of Dentistry, Lincoln, Nebraska, for assistance with the statistical analyses; Mr Bobby Simetich, dental biomaterials research technician, Section of Dental Biomaterials, Department of Adult Restorative Dentistry, UNMC College of Dentistry, Lincoln, Nebraska, for assistance with the shear bond tests; Dr Dennis Feely, associate professor, Department of Oral Biology, UNMC College of Dentistry, Lincoln, Nebraska, for assistance with the SEM photomicrographs; and Mr Aaron Gardner of Precision Aesthetics, Lincoln, Nebraska for assistance with the fabrication of the porcelain veneer specimens. This research was supported by a UNMC College of Dentistry Student Summer Research Fellowship awarded to Dr Thomas St Germain.

Human Subjects Statement

This study was conducted in accordance with all the provisions of the institutional review board guidelines and policies of the Universityof Nebraska Medical Center College of Dentistry. The approval code for this study is #258-08NH.

Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 12 September 2014)

REFERENCES

1. Calamia JR (1983) Etched porcelain facial veneers: A new treatment modality *New York Journal of Dentistry* **53** 255-259.

2. Horn HR (1983) Porcelain laminate veneers bonded to etched enamel *Dental Clinics of North America* **27** 671.
3. Simonsen RJ, & Calamia JR (1983) Tensile bond strength of etched porcelain *Journal of Dental Research* **62** Abstract #1154 p 297.
4. Dumfahrt H, & Shaffer H (2000) Porcelain laminate veneers: A retrospective evaluation after 1 to 10 years of service: Part II—Clinical results *International Journal of Prosthodontics* **13(1)** 9-18.
5. Peumans M, De Munck J, Fieuws S, Lambrechts P, Vanherle G, & Van Meerbeek B (2004) A prospective ten-year clinical trial of porcelain veneers *Journal of Adhesive Dentistry* **6(1)** 65-76.
6. Strassler HE (2007) Minimally invasive porcelain veneers: Indications for a conservative esthetic dentistry treatment modality *General Dentistry* **55(7)** 686-694.
7. Gurel G, Sesma N, Calamita MA, Coachman C, & Morimoto S (2013) Influence of enamel preservation on failure rates of porcelain laminate veneers *International Journal of Periodontics and Restorative Dentistry* **33(1)** 31-39.
8. Swift EJ Jr, & Friedman MJ (2006) Critical appraisal: Porcelain veneer outcomes, part 1 *Journal of Esthetic Restorative Dentistry* **18(1)** 54-57.
9. Calamia JR, & Calamia CS (2007) Porcelain laminate veneers: Reasons for 25 years of success *Dental Clinics of North America* **51(2)** 399-417.
10. Magne P, & Douglas W (1999) Porcelain veneers: Dentin bonding optimization and biomimetic recovery of the crown *International Journal of Prosthodontics* **12(2)** 111-121.
11. Friedman MJ (1998) A 15-year review of porcelain veneer failure—A clinician's observations *Compendium of Continuing Education in Dentistry* **19(6)** 625-630.
12. Summitt JB, Robbins JW, Hilton TJ, & Schwartz RS (2006) *Fundamentals of Operative Dentistry: A Contemporary Approach* 3rd ed Quintessence Publishing, Hanover Park, Ill.
13. Seymour KG, Cherukara GP, & Samarawickrama DYD (2001) Stresses within porcelain veneers and the composite lute using different preparation designs *Journal of Prosthodontics* **10(1)** 16-21.
14. Kihn PW, & Barnes DM (1998) The clinical longevity of porcelain veneers: a 48-month clinical evaluation *Journal of the American Dental Association* **129(6)** 747-752.
15. Gurel G, Morimoto S, Calamita MA, Coachman C, & Sesma N (2012) Clinical performance of porcelain laminate veneers: Outcomes of the aesthetic pre-evaluative temporary (APT) technique *International Journal of Periodontics and Restorative Dentistry* **32(6)** 625-635.
16. Kimmich M, & Stappert CF (2013) Intraoral treatment of veneering porcelain chipping of fixed dental restorations: A review and clinical application *Journal of the American Dental Association* **144(1)** 31-44.
17. Ozturk E, Bolay S, Hickel R, & Ilie N (2013) Shear bond strength of porcelain laminate veneers to enamel, dentine and enamel-dentine complex bonded with different adhesive luting systems *Journal of Dentistry* **41(2)** 97-105.
18. Troedson M, & Derand T (1998) Shear stresses in the adhesive layer under porcelain veneers—A finite analysis study *Acta Odontologica Scandinavica* **56(5)** 257-262.
19. Cotert HS, Dundar M, & Ozturk B (2009) The effect of various preparation designs on the survival of porcelain laminate veneers *Journal of Adhesive Dentistry* **11(5)** 405-411.
20. Layton D, & Walton T (2007) An up to 16-year prospective study of 304 porcelain veneers *International Journal of Prosthodontics* **20(4)** 389-396.
21. Della Bona A, & Barghi N (1993) Removal of partially or fully polymerized resin from porcelain veneers *Journal of Prosthetic Dentistry* **69(4)** 443-444.
22. Haywood VB, Kanoy BE Jr, Bruggers KJ, & Andreaus SB (1990) Thermal removal of composite resin: Effect on rebonding etched metal *Journal of Prosthetic Dentistry* **63(3)** 289-291.
23. da Costa TR, Serrano AM, Altman AP, Loguercio AD, & Reis A (2012) Durability of composite repair using different surface treatments *Journal of Dentistry* **40(6)** 513-521.
24. Palasuk J, Platt JA, Cho SD, Levon JA, Brown DT, & Hovijitra ST (2013) Effect of surface treatments on microtensile bond strength of repaired aged silorane resin composite *Operative Dentistry* **38(1)** 91-99.
25. Ozcan M, & Pekkan G (2013) Effect of different adhesion strategies on bond strength of resin composite to composite-dentin complex *Operative Dentistry* **38(1)** 63-72.
26. Fischer H, Hemelik M, Telle R, & Marx R (2005) Influence of annealing temperature on the strength of dental glass ceramic materials *Dental Materials* **21(7)** 671-677.
27. Ayad MF, Fahmy NZ, & Rosenstiel SF (2008) Effect of surface treatment on roughness and bond strength of a heat-pressed ceramic *Journal of Prosthetic Dentistry* **99(2)** 123-130
28. Kalender A, Onal B, Turkun M, Dalgar H, & Kaytan B (2005) Shear bond strengths of three different resin luting cements to bovine teeth *General Dentistry* **53(1)** 38-42.
29. Gorman CM, McDevitt WE, & Hill RG (2000) Comparison of two heat-pressed all-ceramic dental materials *Dental Materials* **16(6)** 389-395.
30. Simonsen R, Thompson V, & Barrack G (1983) *Etched Cast Restorations: Clinical and Laboratory Techniques* Quintessence Publishing, Chicago.
31. Moraes JCS, & Grandini CR (2011) The glass transition temperature in dental composites In: Cuppoletti J (ed) *Metal, Ceramic and Polymeric Composites for Various Uses* InTech Europe, Rijeka, Croatia pp 307-353.
32. Argen Corp (2007) *Argen® (2007) Troubleshooting Guide for Alloys, Soldering, Investment, and Porcelain* Argen Corp, San Diego, Calif.

33. Boyer DB, & Hormati AA (1980) Rebonding composite resin to enamel at sites of fracture *Operative Dentistry* **5(3)** 102-106.

34. Peumans M, Hikita K, De Munck J, Van Landuyt K, Poitevin A, Lambrechts P, & Van Meerbeek B (2007) Bond durability of composite luting agents to ceramic when exposed to long-term thermocycling *Operative Dentistry* **32(4)** 372-379.

35. Srinavasan V, Deery C, & Nugent, Z (2005) In-vitro microleakage of repaired dental sealants: A randomized, controlled trial *International Journal of Paediatric Dentistry* **15(1)** 57-60.

36. Liu HL, Lin CL, Sun MT, & Chang YH (2009) Numerical investigation of macro- and micro-mechanics of a ceramic veneer bonded with various cement thicknesses using the typical and submodeling finite element approaches *Journal of Dentistry* **37(2)** 141-148.

37. Pakshir HR, Zarif Najafi H, & Hajipour S (2012) Effect of enamel surface treatment on the bond strength of metallic brackets in rebonding process *European Journal of Orthodontics* **34(6)** 773-777.

©Operative Dentistry, 2015, **40-3,** E122-E131

Longitudinal Evaluation of Bond Strength to Enamel of Dental Adhesive Systems Associated with Nd:YAG Laser

DC Barcellos • GR Batista • CR Pucci
ES Persici • AB Borges • CRG Torres
BM Fonseca • SEP Gonçalves

Clinical Relevance

The photothermal mechanism of action of the Nd:YAG laser for the parameters used in this study likely promoted alterations at the bond interfaces of enamel, negatively influencing the bond strength and, consequently, the durability of resin composite restorations.

SUMMARY

Objectives: This study evaluated the durability of bond strength to enamel using total-etch (Single Bond/SB) and self-etch (Clearfil SE Bond/CSEB) adhesives associated with neodymium:yttrium-aluminu- garnet (Nd:YAG) laser irradiation through the uncured adhesives.

Methods: Bovine incisors were worn to expose an area of enamel and were divided into four groups: group 1 (control) SB + polymerization; group 2 (control) CSEB + polymerization; group 3 (laser) – SB + Nd:YAG laser (174.16 J/

Daphne Camara Barcellos, DDS, MS, PhD, postdoctoral student, Department of Restorative Dentistry, Institute of Science and technology, UNESP - Univ Estadual Paulista, São José dos Campos, Brazil

Graziela Ribeiro Batista, DDS, MS, PhD Student, Department of Restorative Dentistry, Institute of Science and technology, UNESP - Univ Estadual Paulista, São José dos Campos, Brazil

Cesar Rogério Pucci, assistant professor, DDS, MS, PhD, Department of Restorative Dentistry, Institute of Science and technology, UNESP - Univ Estadual Paulista, São José dos Campos, Brazil

Erasmo de Souza Persici, MS, electrical engineer, Aerospace Engineering Department, Technological Institute of Aeronautics, São José dos Campos, Brazil

Alessandra Bühler Borges, Assistant Professor, DDS, MS, PhD, Department of Restorative Dentistry, Institute of Science and technology, UNESP - Univ Estadual Paulista, São José dos Campos, São Paulo, Brazil

Carlos Rocha Gomes Torres, associate professor, DDS, PhD, Department of Restorative Dentistry, Institute of Science and technology, UNESP - Univ Estadual Paulista, São José dos Campos, Brazil

Beatriz Maria Fonseca, DDS, MS, PhD Student, Department of Restorative Dentistry, Institute of Science and technology, UNESP - Univ Estadual Paulista, Saõ José dos Campos, Brazil

*Sergio Eduardo de Paiva Gonçalves, associate professor, DDS, MS, PhD, Department of Restorative Dentistry, Institute of Science and technology, UNESP - Univ Estadual Paulista, São José dos Campos, Brazil

*Corresponding author: Avenida Engenheiro Francisco José Longo, 777, Jardim São Dimas, São José dos Campos, SP, Brazil; e-mail: sergio@fosjc.unesp.br

DOI: 10.2341/13-181-L

cm^2) + polymerization; and group 4 (laser) CSEB + Nd:YAG (174.16 J/cm^2) + polymerization. Blocks of composite were fabricated and stored for 24 hours or 12 months, sectioned into beams, and submitted to microtensile tests. Results were analyzed by three-way analysis of variance (ANOVA) (adhesive, technique, and storage time) and Tukey tests.

Results: ANOVA revealed significant differences for adhesive × technique and technique × storage time ($p<0.05$). The mean values (MPa) for interaction adhesive × technique (standard deviation) were as follows: SB/control = 35.78 (6.04)a; SB/laser = 26.40 (7.25)b, CSEB/control = 26.32 (5.71)b, CSEB/laser = 23.90 (7.49)b. For interaction technique × storage time the mean values were as follows: control/24 hours = 32.58 (6.49)a; control/12 months = 29.52 (8.38)a; laser/24 hours = 29.37 (5.71)a; laser/12 months = 20.92 (6.5)b. Groups with the same letters showed no statistically significant differences.

Conclusion: Scanning electron microscope analysis showed evident areas of micromorphological alterations in lased samples after 12 months of water storage. Nd:YAG laser irradiation of enamel through unpolymerized total-etch adhesive significantly reduced bond strength compared with the control. Bond strength decreased when enamel samples irradiated with Nd:YAG laser through unpolymerized adhesives were stored in water for 12 months.

INTRODUCTION

The post-Buonocore,[1] 1955 era, known as the "adhesive era," has shown that the total acid etch adhesive systems present excellent performance in this substrate and maintain the longitudinal stability of the bond interface. The enamel acid-etching technique is based on selective demineralization of the hydroxyapatite crystals present in tooth enamel, resulting in an extremely roughened surface with high energy. These characteristics provide high wetting capacity of the resinous monomers, which, on polymerizing, results in the formation of prolongations called tags that "anchor" the resin to the tooth.[2]

On the other hand, following the modern trend of simplifying the clinical steps and saving operating time, new bonding strategies were developed. In 1994, Watanabe and others[3] introduced the self-etching adhesives to the market with the proposal of optimizing the bond process, reducing the clinical steps, eliminating the acid-etching and washing steps, and providing better interaction with the dentinal substrate (because these adhesives are less aggressive). However, their etching capacity has been shown to be more restricted, as they present low reactivity with the mineral component, lower availability of H^+ ions, and high molecular weight, compared with phosphoric acid, promoting etching that is not as deep and retentive on dental enamel.[4,5] The highly hydrophilic nature of their composition may also contribute to the reduction in longevity of restorations in areas of extensive availability of enamel,[6,7] since this characteristic favors the sorption of liquids, nanoleakage, and consequent degradation of the bond. Therefore, total acid etch adhesive systems are considered the "gold standard" when compared with the self-etching adhesives existent in the market.

New alternatives for perfecting the bonding pattern have been exhaustively studied. Gonçalves and others[8] developed an irradiation technique with Nd:yttrium-lithium-fluoride (YLF) laser on dentin impregnated with unpolymerized adhesive. They obtained significantly higher shear bond strength values compared with unradiated dentin, leading them to believe that irradiation with laser could lead to the formation of a more resistant substrate with more chemical affinity for the bonding/adhesive process. After this, other authors[9,10] observed that this technique now used on enamel also improved the bond strength and could consequently optimize the longevity of the restoration.

Although the laser irradiation technique developed by Gonçalves and others[8] had been promising in improving the bond strength to both dentin and enamel,[9,10] only the immediate results were observed. No longitudinal studies were found. Therefore, the longitudinal evaluation of bond strength achieved when using the laser irradiation technique on the unpolymerized adhesive is shown to be relevant. The aim of this study was to evaluate *in vitro* the influence of neodymium:yttrium-aluminu-garnet (Nd:YAG) laser on the microtensile bond strength to enamel of a two-step total-etch and a two-step self-etch adhesive when the laser was applied on the unpolymerized adhesives at time intervals of 24 hours and after a one-year period of storage in water at 37°C. In this study, three null hypotheses were tested: 1) The different adhesive systems do not affect the bond strength to enamel; 2) Nd:YAG laser irradiation through unpolymerized adhesives does not affect bond strength to enamel; and 3) The storage period does not affect the bonding effectiveness of etch-and-rinse and self-etch adhesives to enamel.

METHODS AND MATERIALS

One hundred and twenty freshly extracted bovine incisor teeth were used in this study. The roots were sectioned with a steel diamond disc (KG Sorensen, Rio de Janeiro, Brazil) at the cement-enamel junction. The buccal surfaces were worn using abrasive papers (600 grit) coupled to a circular polishing machine (PA-10, Panambra, São Paulo, Brazil) under water cooling to obtain a 5-mm^2 area of flat enamel.

The teeth were divided into four groups (n=30) according to the surface treatment performed, as follows:

- Group 1 (control): The surfaces were etched for 15 seconds with 37% phosphoric acid gel, rinsed, and dried with air spray for 10 seconds. Two layers of Single Bond/SB total-etch adhesive (3M ESPE, St Paul, MN, USA) were actively applied on the surface for 15 seconds and gently air-dried for 10 seconds. The adhesive was light-activated for 10 seconds with a LED light unit (Emitter A, Schuster, Santa Maria, RS, Brazil) with a power density of 600 mW/cm^2.
- Group 2 (control): The surfaces received the application of Clearfil SE Bond/CSEB self-etch adhesive (Kuraray Medical Inc, Tokyo, Japan). One layer of primer agent was applied actively for 20 seconds and gently air-dried for 10 seconds. One layer of bonding agent was applied actively for 20 seconds and gently air-dried for 10 seconds. The adhesive was light-activated for 10 seconds.
- Group 3 (experimental/laser): The surfaces received the application of SB total-etch adhesive (3M ESPE), following the same protocol used for group 1. Before light polymerization, the surfaces were irradiated with Nd:YAG laser in noncontact mode, scanning for 60 seconds. The adhesive was light-activated for 10 seconds.
- Group 4 (experimental/laser): The surfaces received the application of CSEB self-etch adhesive (Kuraray), following the same protocol used for group 2. Before light polymerization, the surfaces were irradiated with Nd:YAG laser in noncontact mode, scanning for 60 seconds. The adhesive was light-activated for 10 seconds.

Treatment with Nd:YAG Laser

The Nd:YAG laser equipment used in this study was the Laser Pulse Master 600 iQ (American Dental Technologies Inc, Corpus Christi, TX, USA) at a wavelength of 1.064 μm. The output energy of this laser device was 140 mJ per pulse, with a pulse repetition rate of 10 pulses/s (10 Hz) for 60 seconds. The laser was fitted with a noncontact tip 320 μm in diameter and was applied freehand by one calibrated operator in noncontact mode scanning over a 5-mm × 5-mm area of flat enamel. The energy density was 174.16 J/cm^2. During laser application the laser tip was at a 90° angle, perpendicular to the surface, and at a distance of 1 mm from the surface.[9-11]

Restoration Placement

Nanocomposite resin blocks (Filtek Z350, 3M ESPE), approximately 4 mm high, were built on the treated surfaces using a two-piece split Teflon mold. Each 2-mm portion was light-activated for 40 seconds.

The bonded teeth were stored in distilled water (pH=7.0) at 37°C for 24 hours or 12 months.[12-16] The water was changed every week during the course of one year.[15]

The test specimens were cut into parallel sections measuring approximately 1 mm, made from the mesial to the distal and from the cervical to the occlusal surface, using a diamond disc attached to a Labcut 1010 (Extec Technologies Inc, Enfield, CT, USA) cutting machine to obtain sticks, producing a minimum of seven sticks per tooth. The sections were made at low speed under water cooling to prevent stress induction at the bond interface.

The sticks were attached to a microtensile device in a universal testing machine (DL-1000, EMIC, São José dos Pinhais, PR, Brazil) with a 10-kg load cell at a crosshead speed of 0.5 mm/min, in accordance with the ISO 11405 Standard. The bond strength data were expressed in megapascals (MPa).

Statistical Analysis

To the pretest failures (PTFs) and debonded resin blocks, the lowest measured value was assigned.[17] To the cohesive failures (enamel or composite), the specimens were discarded.[17] The mean value for the sticks originating from each tooth was calculated and used for the statistical analysis.

Data (expressed in MPa) were analyzed by three-way analysis of variance (ANOVA; adhesive, technique, and storage time) followed by Tukey test (α=5%).

Scanning Electron Microscopy (SEM) Examination

Two teeth from each group were prepared for SEM analysis. The specimens were sectioned perpendicu-

Table 1: *Descriptive Analysis for the Different Groups*

Adhesive	Technique	Storage Time	Mean[a]	SD
SB	Control	24 h	36.44 ^{A}	6.72
SB	Control	12 mo	35.79 ^{AB}	5.50
SB	Laser	24 h	30.19 ^{ABC}	5.83
CSEB	Control	24 h	29.38 ^{BCD}	3.93
CSEB	Laser	24 h	28.56 ^{CDE}	5.67
CSEB	Control	12 mo	23.26 ^{DEF}	5.66
SB	Laser	12 mo	22.61 ^{EF}	6.63
CSEB	Laser	12 mo	19.24 ^{F}	6.13

Abbreviation: SD, standard deviation.
[a] *Means followed by the same letters do not differ statistically (*p*>0.05).*

larly to the bond interface. The sections were polished with 2000 and 4000 mesh sheets. Phosphoric acid etchant was applied for 15 seconds and then rinsed off with water for 10 seconds. Specimens were dehydrated, sputter-coated with gold-palladium (according to Marimoto and others[10]), and examined by SEM.

RESULTS

The mean bond strength values in all groups are presented in Table 1.

ANOVA revealed that total-etch adhesive presented higher bond strength values compared with self-etch adhesive (p=0.000); the unlased surface treatment presented higher bond strength values compared with the lased surface treatment (p=0.000); and storage in water for 24 hours presented higher bond strength values compared with storage in water for 12 months (p=0.0000).

Table 2 shows the results of the Tukey test for the interaction between the independent variables of adhesive and technique (p=0.0014). The adhesive SB, associated with the control technique, presented significantly higher mean bond strength values when compared with SB associated with the laser technique or with CSEB for both techniques used.

Table 3 shows the results of the Tukey test for the interaction between the independent variables of technique and storage time (p=0.0123). The technique of irradiating the tissue with Nd:YAG laser in the longitudinal time interval (12 months of storage) presented significantly lower mean bond strength values when compared with the technique of irradiating the tissue with Nd:YAG laser in the time interval of 24 hours and the control technique, regardless of the storage time.

For the fracture type, an increase in the occurrence of adhesive fractures in the time interval of 12 months was observed, regardless of the technique used.

Figures 1 through 3 show SEM images obtained of the interfaces created in all of the groups.

DISCUSSION

In this study we used the indirect storage technique (storage of restored teeth) for evaluating the longitudinal bond strength, as in the studies conducted by De Munck and others,[12] Toledano and others,[13] Osorio and others,[14,15] and Abdalla.[16] According to Osorio and others[15] and Pashley and Tay,[18] the water sorption phenomenon in resin-enamel bond occurs over time, inducing resin swelling and weakening of the adhesive joint.

The first null hypothesis was rejected, because the results of the present research showed the superiority of SB compared with CSEB. The 10-methacryl-

Table 2: *Results of Tukey Test (5%) for Interaction Between Factors Adhesive × Technique*

Adhesive	Technique	Mean (SD)	Homogeneous Groups[a]	
Single Bond	Control	35.78 (6.04)	A	
Single Bond	Laser	26.40 (7.25)		B
Clearfil SE Bond	Control	26.32 (5.71)		B
Clearfil SE Bond	Laser	23.90 (7.49)		B

Abbreviation: SD, standard deviation.
[a] *Means followed by the same letters do not differ statistically (*p*>0.05).*

Table 3: *Results of Tukey Test (5%) for Interaction Between Factors Technique × Storage Time*

Technique	Storage Time	Mean (SD)	Homogeneous Groups[a]	
Control	24 h	32.58 (6.49)	A	
Control	12 mo	29.52 (8.38)	A	
Laser	24 h	29.37 (5.71)	A	
Laser	12 mo	20.92 (6.50)		B

Abbreviation: SD, standard deviation.
[a] *Means followed by the same letters do not differ statistically (p>0.05).*

oyloxydecyl dihydrogen phosphate (MDP) acidic monomer results in a lower number of ionizable radicals in an aqueous solution, determining a pH ≈ 2.0. However, the phosphoric acid (35-37%) exhibits a large number of ionizable radicals in an aqueous solution, resulting in a pH ≈ 0.6. Thus, the etching capacity of self-etch CSEB to demineralize the substrate is more restricted when compared with that of total-etch SB.[19] CSEB is considered a self-etching adhesive of mild or weak aggressiveness[20] and presents low reactivity with the mineral component. As enamel has a high mineral content, and as a result of the lower availability of H^+ ions to the acidic monomers, these ions may be practically or completely neutralized by the minerals dissolved from enamel before they perform an adequate etching pattern.[21]

In addition, the higher the ionization constant (Ka) value, the greater the force of the acid. Consequently, the greater the acidity, the lower the pKa value of the acid (negative logarithm of the ionization constant). MDP has a pKa = 2.2, with a capacity to dissolve 1.6 g of hydroxyapatite for each gram of MDP acidic monomer. Phosphoric acid has a pKa = 2.0, with a capacity to dissolve 5.1 g of hydroxyapatite for each gram of phosphoric acid. Salz and others[22] explained that the acidic monomers have a high molecular weight in comparison with phosphoric acid, having a negative influence on the capacity of hydroxyapatite dissolution. There-

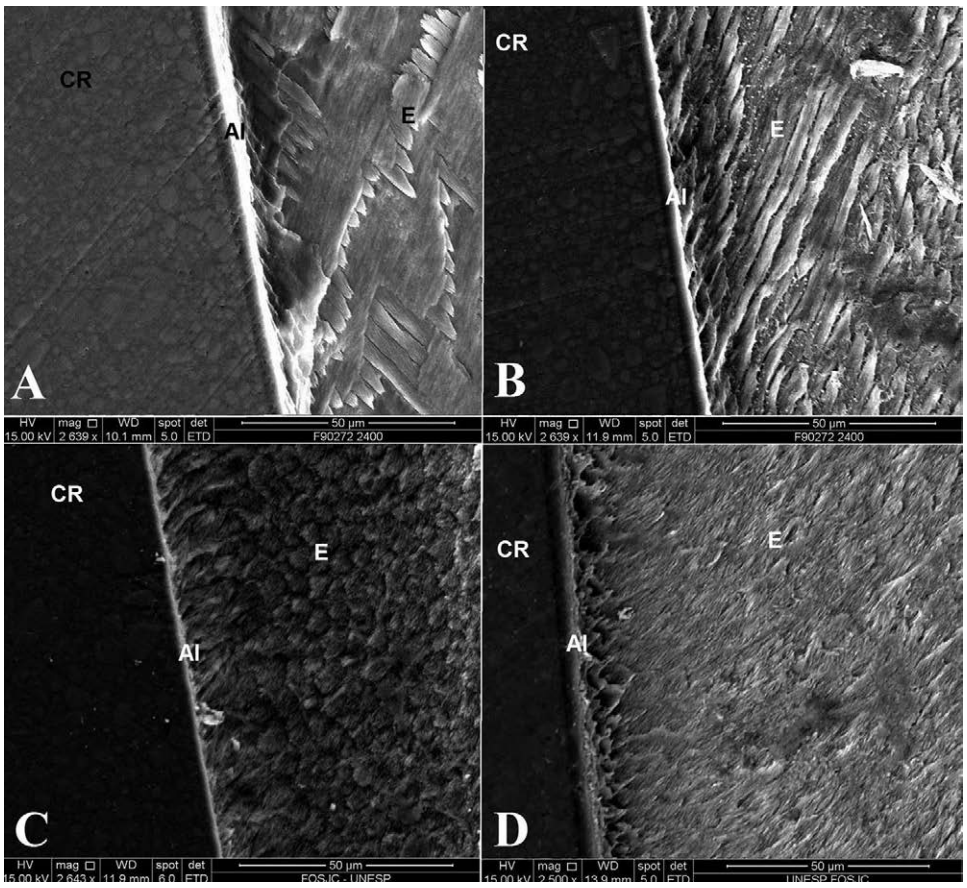

Figure 1. *(A, B) Bond interface that received, respectively, SB and CSEB by control technique and storage for 24 hours. The formation of a thicker bond interface may be observed for the group that received SB; (C, D) Bond interface that received, respectively, SB and CSEB by control technique and storage for 12 months. No areas of degradation of adhesive layer were observed over time. (Legend: CR, composite resin; AI, adhesive interface; E, enamel.)*

fore, the higher pH and pKa values may explain the lower bond strength values for CSEB compared with SB observed in this study. Total-etch adhesives in enamel are considered the "gold standard," because phosphoric acid is capable of producing a satisfactory enamel etching pattern, resulting in excellent micromechanical entanglement by the tags formed.[23]

When acid etching is deeper and more retentive on enamel, greater penetration of the adhesive into the substrate occurs, promoting a thicker bond interface.[24] As can be observed in Figure 1A, the interface formed by SB is thicker when compared with that formed by CSEB (Figure 1B), which is thinner. This phenomenon likely occurred as a result of the etching attaining the interprismatic, peripheral, and central layers of the enamel prisms,[24] confirming the greater power of action and dissolution of hydroxyapatite by the phosphoric acid in SB.

The second null hypothesis was rejected, because Nd:YAG laser irradiation significantly decreased the bond strength in comparison with the control technique. According to Castro and others,[25] SB is not capable of absorbing wavelengths between 950 and 1100 nm, which includes the wavelength of 1064 nm emitted by the Nd:YAG laser. As the Nd:YAG laser causes a change only when absorbed, this adhesive likely did not undergo any change from the direct action of laser. Moreover, Arrais and others[26] observed that SB and CSEB present absorption spectra of a similar spectral band. It may therefore be speculated that these adhesives presented a similar behavior with regard to the nonabsorption of the wavelength emitted by the Nd:YAG laser.

The results of this study are in contrast to those of Matos and others[9] and Marimoto and others,[10] who observed a significant improvement in the bond strength of enamel impregnated with nonpolymerized adhesives and irradiated with Nd:YAG laser, using 49.76 J/cm^2 and 174.16 J/cm^2, respectively. They believe that this technique would promote the fusion and recrystallization of this tissue in the presence of the adhesive,[8,10] resulting in the formation of a new substrate formed of melted hydroxyapatite and adhesive, and this would be mechanically interlocked and have greater affinity for bonding.[8,10]

However, lower parameters than 174.16 J/cm^2 of Nd:YAG laser irradiation promote chemical and morphological alterations of the enamel surface, such as formation of small bubbles, similar to craters, irregular elevations and fine cracks, and melted and solidified enamel with craters at the surface[27,28]; and generated the formation of areas of decalcification of 15 μm and the formation of craters, with the enamel surface fractured, melted/fused, and recrystallized with a glazelike aspect.[28,29] Ariyaratnam and others[30] believed that the formation of craters, fissures, and fractured enamel could occur as a result of the rapid thermal cycle on the enamel surface during irradiation with Nd:YAG laser. The formation of craters similar to bubbles would be the result of overheating of the enamel surface submitted to subsequent cooling to ambient temperature, and the higher the energy density (ED), the greater are the photothermal effects on the tissues.[30] Fowler and Kuroda[31] explained that even low EDs (between 9 and 120 J/cm^2) could promote slight melting of the enamel surface, which indicates that temperatures >1400°C could be attained on the enamel surface. In addition, high EDs promote chemical and structural alterations on the tooth surface, making the irradiated enamel surface more fragile[32] and significantly reducing its surface microhardness.[33]

Therefore, according to the results of this study, it is believed that the photothermal effects promoted by the high ED of the Nd:YAG laser (174.16 J/cm^2) chemically and morphologically changed the enamel surface impregnated with the adhesive systems, with the formation of microcracks, fractured areas, and fissures on the irradiated enamel surface[27-30] and reduction in the microhardness of the enamel surface,[32,33] which have a negative influence on the bonding process, when compared with the control technique.

The third hypothesis was rejected, because significant reductions in resin-enamel bond strengths were observed after 12 months of water storage. The phenomenon of water sorption occurs with the passage of time, inducing an increase in the volume of resin and rupturing the adhesive bonds at the tooth-restoration interface.[15] Both CSEB and SB have water and alcohol as solvents.[34] Poor solvent evaporation during the application of adhesive systems may determine separation of the phases and a lower rate of polymerization, resulting in the formation of weakened interfaces, with increasing susceptibility of the adhesive layer to degradation over the course of time.[15,35]

According to the results of this study, significant differences were observed for the interaction between factors adhesive × technique. Unlased SB presented significantly higher bond strength values when compared with lased SB and compared with lased and unlased CSEB. As previously explained, the higher pH and pKa values of MDP, when

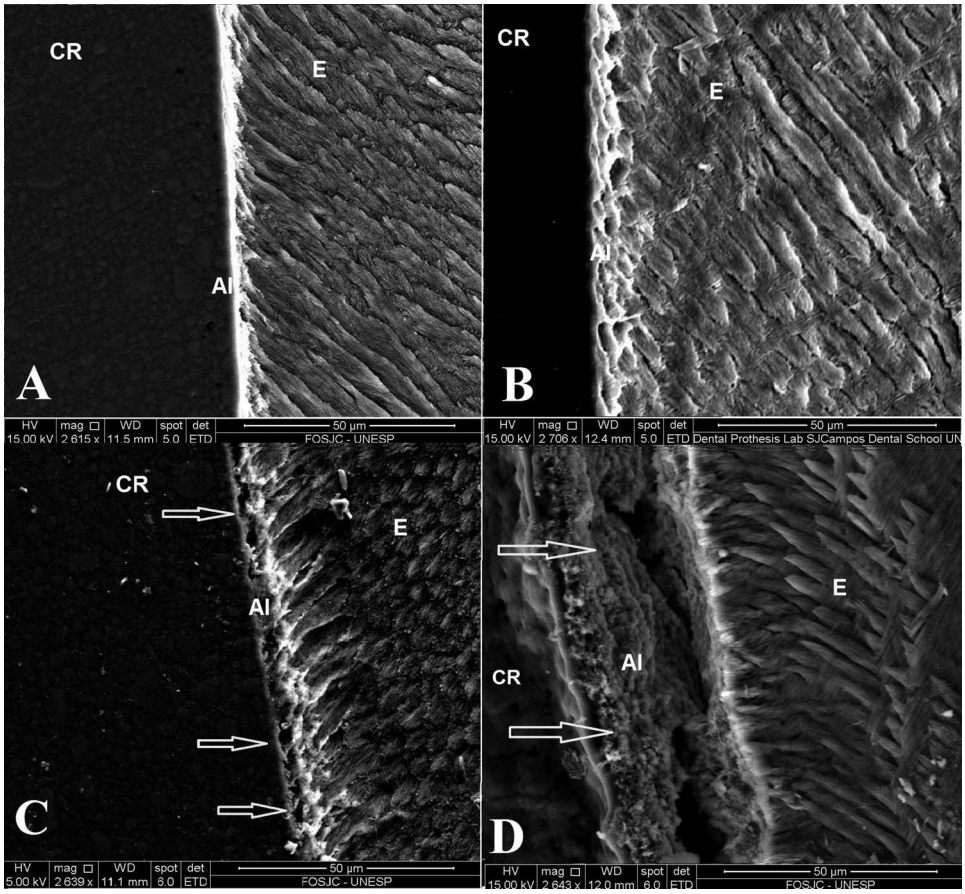

Figure 2. *(A, B) Bond interface that received, respectively, SB and CSEB adhesives by experimental/laser technique and storage for 24 hours; (C, D) Bond interface that received, respectively, SB and CSEB by experimental/laser technique and storage for 12 months. For SB, the presence of gaps in the bond interface (A). For CSEB, we observed the presence of a melted mass in the irradiated substrate (B) (arrows). (Legend: CR, composite resin; AI, adhesive interface; E, enamel.)*

compared with those of phosphoric acid, may explain the lower bond strength values for unlased CSEB compared with unlased SB observed in this study.

Furthermore, regardless of the adhesive used, the lased enamel negatively influenced the bond strength. It is believed that the high ED of Nd:YAG laser on the enamel impregnated with the non-polymerized adhesives may have promoted changes in the morphology of the enamel surface, creating areas with microcracks, fractures, and fissures[27-29] and a reduction in the microhardness of the irradiated enamel surface,[32,33] harming the bond strength as a result of the formation of a more debilitated enamel-resin interface (in comparison to that associated with the control technique).

For the interaction between the variables technique and storage time, lased enamel stored for 12 months presented significantly lower bond strength when compared with lased enamel stored for 24 hours or with unlased enamel stored for 24 hours or 12 months. Oho and Morioka[28] observed that EDs between 67 and 160 J/cm^2 on enamel caused a reduction in the following components: water, carbonate, and organic substances. Theoretically, the reduction of water and organic substances should improve the bond strength longitudinally. The smaller the quantity of organic components (proteins) in the tissue, the less the degradation of the bond interface with the passage of time.[15] Moreover, the smaller the quantity of water in the tissue, the greater the possibility of preventing the action of water on the monomers when the adhesive is light-activated, making the bond interface more stable with the passage of time.[36] However, one of the hypotheses proposed was that the high ED of Nd:YAG laser on the surface of the could have promoted deleterious changes in the morphology[27-29] and reduction in microhardness of the enamel surface,[32,33] which may have contributed to accelerating the degradation, in comparison with the control technique.

As was observed in the SEM images (Figures 2A,B and 3B), there was formation of a bond interface with the characteristics of melting and fusion, with an aspect of "melted lava." These images could indicate that temperatures >1400°C were attained on the enamel surface.[31] According to Lin and

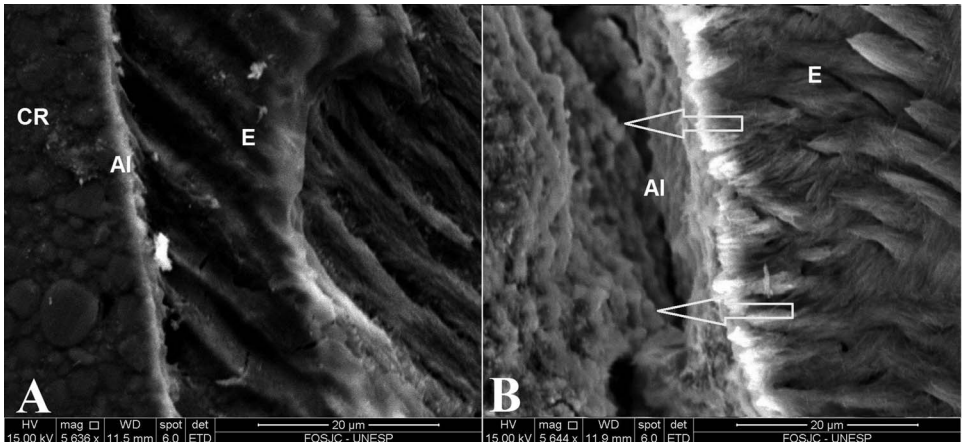

Figure 3. *(A, B) Bond interface that received CSEB by experimental/laser technique and storage for, respectively, 24 hours and 12 months (5700× magnification). For 12 months of storage, complete loss of the bond interface may be observed, as is the presence of a melted mass formed by adhesive and tissue, with formation of a large gap between the enamel and resin (arrows) (B). (Legend: CR, composite resin; AI, adhesive interface; E, enamel.)*

others,[37] temperatures above 1125°C promote the formation of a new crystalline phase, tricalcium phosphate-α [Ca3(PO4)2-α]. Furthermore, Kawasaki and others[38] observed that an ED, as from 100 J/cm^2 of Nd:YAG laser, could produce Ca3(PO4)2-α formation. This new crystalline phase has a higher degree of solubility and degradability than hydroxyapatite, which could reduce the chemical stability of the irradiated tissue[37] and consequently reduce the longitudinal bond strength.

The SEM images clearly illustrate the bond interface modified by Nd:YAG laser in the specimens stored for 12 months. When the bond interface formed by SB (Figure 2C) was exposed to the water for 12 months, we observed a bond interface with various areas of degradation (empty spaces that separated the resin from the tooth enamel) and the presence of subjacent enamel modified by the laser irradiation. At the interface formed by CSEB (Figures 2D and 3A) we observed a complete loss of characterization of the bond interface, with the presence of a melted, hydrolyzed mass and gaps, clearly demonstrating the advanced degradation of this interface.

With regard to the fracture type analysis, Leloup and others[39] explained that adhesive systems with high microtensile bond strength values presented higher rates of cohesive failure, whereas adhesive systems showing low bond strength values after the microtensile test presented higher rates of adhesive failures[39] and, consequently, high rates of pretest failure.[18] The SB/control/24 hours presented the highest bond strength values (36.44±6.72) and the highest number of cohesive failures, which was higher than the number of adhesive fractures, without the occurrence of pretest failure. However, the CSEB/laser/12 months presented the lowest bond strength values (19.24±6.13) and the highest values of adhesive failure and premature failure.

Although the results obtained in this study cannot be directly extrapolated to a clinical situation, it may be suggested that the photothermal mechanism of action of Nd:YAG laser for the high ED used in this study likely promoted alterations at the bond interfaces of the enamel, negatively influencing the bond strength and, consequently, the durability of resin composite restorations.

Further *in vitro* studies are necessary to observe the longitudinal behavior of the bond interface using the irradiation technique with Nd:YAG laser with different parameters from those evaluated in the present study. Different power, energy density, frequency, and scanning time parameters of Nd:YAG laser may modify dental tissues without promoting harmful photothermal effects on the formation of the bond interface.

CONCLUSIONS

According to the methodology used, and based on the results obtained, we may conclude that

- Self-etch adhesive showed reduced bond strength to enamel compared with total-etch adhesive;
- Nd:YAG laser irradiation through unpolymerized adhesives affected bond strength to enamel;
- The 12-month water storage period affected the bonding effectiveness of adhesives to enamel;
- Nd: YAG laser irradiation of enamel through the unpolymerized total-etch adhesive significantly reduced bond strength; and
- Bond strength decreased when enamel irradiated with Nd:YAG laser through the unpolymerized adhesives was stored in water for 12 months.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 8 November 2013)

REFERENCES

1. Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34(6)** 849-853.
2. Gwinnett AJ (1981) Acid etching for composite resins *Dental Clinics of North America* **25(2)** 271-289.
3. Watanabe I, Nakabayashi N, & Pashley DH (1994) Bonding to ground dentin by a phenyl-P self-etching primer *Journal of Dental Research* **73(6)** 1212-1220.
4. Moura SK, Pelizzaro A, Dal Bianco K, de Goes MF, Loguercio AD, Reis A, & Grande RH (2006) Does the acidity of self-etching primers affect bond strength and surface morphology of enamel? *Journal of Adhesive Dentistry* **8(2)** 75-83.
5. Rotta M, Bresciani P, Moura SK, Grande RH, Hilgert LA, Baratieri LN, Loguercio AD, & Reis A (2007) Effects of phosphoric acid pretreatment and substitution of bonding resin on bonding effectiveness of self-etching systems to enamel *Journal of Adhesive Dentistry* **9(6)** 537-545.
6. Spencer P, Ye Q, Park J, Misra A, Bohaty BS, Singh V, Parthasarathy R, Sene F, Gonçalves SEP, & Laurence J (2012) Durable bonds at the adhesive/dentin interface: an impossible mission or simply a moving target? *Brazilian Dental Science* **15(1)** 4-18.
7. Reis A, Moura K, Pellizzaro A, Dal-Bianco K, de Andrade AM, & Loguercio AD (2009) Durability of enamel bonding using one-step self-etch systems on ground and unground enamel *Operative Dentistry* **34(2)** 181-191.
8. Gonçalves SE, de Araujo MA, & Damião AJ (1999) Dentin bond strength: Influence of laser irradiation, acid etching, and hypermineralization *Journal of Clinical Laser Medicine and Surgery* **17(2)** 77-85.
9. Matos AB, Oliveira DC, Navarro RS, de Eduardo CP, & Matson E (2000) Nd:YAG laser influence on tensile bond strength of self-etching adhesive systems *Journal of Clinical Laser Medicine and Surgery* **18(5)** 253-257.
10. Marimoto AK, Cunha LA, Yiu KCK, Huhtala MFRL, Barcellos DC, Prakki A, & Gonçalves SEP (2013) Influence of Nd:YAG laser on the bond strength of self-etching and conventional adhesive systems to dentin and enamel(dental hard tissues) *Operative Dentistry* **8(4)** 447-455.
11. Ribeiro CF, Gonçalves SEP, Yiu KCK, Borges AB, Barcellos DC, & Brayner R (2013) Dentin bond strength: Influence of Er:YAG and Nd:YAG lasers *International Journal of Periodontics and Restorative Dentistry* **3(3)** 373-377.
12. De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, Lambrechts P, & Vanherle G (2003) Four-year water degradation of total-etch adhesives bonded to dentin *Journal of Dental Research* **82(2)** 136-140.
13. Toledano M, Osorio R, Osorio E, Aguilera FS, Yamauti M, Pashley DH, & Tay F (2007) Effect of bacterial collagenase on resin-dentin bonds degradation *Journal of Materials Science: Materials in Medicine* **18(12)** 2355-2361.
14. Osorio R, Pisani-Proenca J, Erhardt MC, Osorio E, Aguilera FS, Tay FR, & Toledano M (2008) Resistance of ten contemporary adhesives to resin-dentine bond degradation *Journal of Dentistry* **36(2)** 163-169.
15. Osorio R, Monticelli F, Moreira MA, Osorio E, & Toledano M (2009) Enamel-resin bond durability of self-etch and etch & rinse adhesives *American Journal of Dentistry* **22(6)** 371-375.
16. Abdalla AI (2010) Effect of long-term water aging on microtensile bond strength of self-etch adhesives to dentin *American Journal of Dentistry* **23(1)** 29-33.
17. Roulet JF, & Van Meerbeek B (2007) Statistics: A nuisance, a tool, or a must? *Journal of Adhesive Dentistry* **9(3)** 287-288.
18. Pashley DH, & Tay FR (2001) Aggressiveness of contemporary self-etching adhesives. Part II: Etching effects on unground enamel *Dental Materials* **17(5)** 430-444.
19. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, & Vanherle G (2003) Adhesion to enamel and dentin: Current status and future challenges *Operative Dentistry* **28(3)** 215-235.
20. Tay FR, & Pashley DH (2001) Aggressiveness of contemporary self-etching systems. I: Depth of penetration beyond dentin smear layers *Dental Materials* **17(4)** 296-308.
21. Miyazaki M, Hinoura K, Honjo G, & Onose H (2002) Effect of self-etching primer application method on enamel bond strength *American Journal of Dentistry* **15(6)** 412-416.
22. Salz U, Mücke A, Zimmermann J, Tay FR, & Pashley DH (2006) pKa value and buffering capacity of acidic monomers commonly used in self-etching primers *Journal of Adhesive Dentistry* **8(3)** 143-150.
23. Torres CR, Barcellos DC, Pucci CR, Lima Gde M, Rodrigues CM, & Siviero M (2009) Influence of methods of application of self-etching adhesive systems on adhesive bond strength to enamel *Journal of Adhesive Dentistry* **11(4)** 279-286.
24. Torii Y, Itou K, Nishitani Y, Ishikawa K, & Suzuki K (2002) Effect of phosphoric acid etching prior to self-etching primer application on adhesion of resin composite to enamel and dentin *American Journal of Dentistry* **15(5)** 305-308.
25. Castro FL, Andrade MF, Hebling J, & Lizarelli RF (2012) Nd:YAG laser irradiation of etched/unetched dentin through an uncured two-step etch-and-rinse adhesive and its effect on microtensile bond strength *Journal of Adhesive Dentistry* **14(2)** 137-145.
26. Arrais CA, Pontes FM, Santos LP, Leite ER, & Giannini M (2007) Degree of conversion of adhesive systems light-

cured by LED and halogen light *Brazilian Dental Journal* **18(1)** 54-59.

27. Hess JA (1990) Scanning electron microscopic study of laser-induced morphologic changes of a coated enamel surface *Lasers in Surgery and Medicine* **10(5)** 458-462.
28. Oho T, & Morioka T (1990) A possible mechanism of acquired acid resistance of human dental enamel by laser irradiation *Caries Research* **24(2)** 86-92.
29. Eduardo CP, Cecchini RCM, Marques JLL, & Matsumoto K (1995) Scanning electron microscopy study on enamel etching with Nd:YAG laser and phosphoric acid *Journal of Clinical Laser Medicine and Surgery* **13(2)** 81-85.
30. Ariyaratnam MT, Wilson MA, Mackie IC, & Blinkhorn AS (1997) A comparison of surface roughness and composite/enamel bond strength of human enamel following the application of the Nd:YAG laser and etching with phosphoric acid *Dental Materials* **13(1)** 51-55.
31. Fowler BO, & Kuroda S (1986) Changes in heated and in laser-irradiated human tooth enamel and their probable effects on solubility *Calcified Tissue International* **38(4)** 197-208.
32. Tagomori S, & Iwase T (1995) Ultrastructural change of enamel exposed to a normal pulsed Nd-YAG laser *Caries Research* **29(6)** 513-520.
33. Jennett E, Motamedi M, Rastegar S, Frederickson C, Arcoria C, & Powers JM (1994) Dye-enhanced ablation of enamel by pulsed lasers *Journal of Dental Research* **73(12)** 1841-1847.
34. Toledano M, Osorio R, de Leonardi G, Rosales-Leal JI, Ceballos L, & Cabrerizo-Vilchez MA (2001) Influence of self-etching primer on the resin adhesion to enamel and dentin *American Journal of Dentistry* **14(4)** 205-210.
35. Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, & Van Meerbeek B (2005) Clinical effectiveness of contemporary adhesives: A systematic review of current clinical trials *Dental Materials* **21(9)** 864-881.
36. Gamborgi GP, Loguercio AD, & Reis A (2007) Influence of enamel border and regional variability on durability of resin-dentin bonds *Journal of Dentistry* **35(5)** 371-376.
37. Lin CP, Lee BS, Lin FH, Kok SH, & Lan WH (2001) Phase, compositional, and morphological changes of human dentin after Nd:YAG laser treatment *Journal of Endodontics* **27(6)** 389-393.
38. Kawasaki K, Tanaka Y, & Takagi O (2000) Crystallographic analysis of demineralized human enamel treated by laser-irradiation or remineralization *Archives of Oral Biology* **45(9)** 797-804.
39. Leloup G, D'Hoore W, Bouter D, Degrange M, & Vreven J (2001) Meta-analytical review of factors involved in dentin adherence *Journal of Dental Research* **80(7)** 1605-1614.

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.

DENTSPLY CAULK

ivoclar vivadent

GC America Inc.

BISCO

ULTRADENT

SDI Your Smile. Our Vision.

Kerr

OPERATIVE DENTISTRY

Volume 40 / Number 3
May/June 2015

www.jopdent.org
227–338

Clinical Technique/Case Report

Laboratory Research

Departments

Online Only