# OPERATIVE DENTISTRY

Jan/Feb 2018
Volume 43
Number 1
1–112



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

# OPERATIVE DENTISTRY

Volume 43 / Number 1
January/February 2018

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

"Open Range" Hendricks County, Indiana, USA. Photo provided by Lois Tally of Danville, Indiana USA. Photo taken with a Nikon D800, Nikor 28-300 Lens, f/32 1/50 sec. ISO-200 © 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 317-350-4371, Fax: 317-852-3162
http://www.jopdent.org

## Editorial Staff

©Operative Dentistry, 2018, **43-1,** 1-2

**Guest Editorial**

# India Meets the Challenge and Goes for Gold

ST Grennell



**This Editorial was initially printed in the March 2017 issue of the *Indian Journal of Dental Research*. They have graciously allowed us to reprint it here.**

Citation: Grennell ST. India meets the challenge and goes for gold. *Indian Journal of Dental Research* 2017;28:119-120.

The Indian Academy of Gold Foil Operators (IAGFO) held its first World Gold Summit this past January. Seven delegates from the American Academy of Gold Foil Operators (AAGFO) came to present and participate. Invitations were sent, and many dentists came. What was the interest, why gold, and who are these academies?

Let's examine why gold is even a topic. After all, in dentistry, so many people today reject gold in favor of tooth-colored restorations. Yet these same people wear gold elsewhere on their bodies. It has become stylish for some to cover natural skin with tattooing. That renders the desire for natural coloration rather moot. As for gold, people admire it. It is a symbol of prosperity. It is beautiful. The body finds gold comfortable and does not reject it as it will other metals. Indeed, it is basically nonreactive to the body, as seen in piercings and in dental restorations.

Dental restorations made of gold are tissue compatible. They can be shaped to mimic natural tooth morphology. Cast gold wears at the same rate as enamel and does not wear the opposing dentition. It has sufficient strength to withstand masticatory forces without flexing or fracturing and often lasts the entire life of the patient. It also offers the option

Susanne T Grennell, President, American Academy of Gold Foil Operators, PO Box 153, Billings, NY 12510, USA.

DOI: 10.4103/ijdr.IJDR_147_17

of partial coverage, retaining, and supporting natural tooth structure. It can be designed to be highly esthetic without displaying gold, and it does not absorb light. Deflection of light, which gold offers, gives brilliance to the smile without glitter.

Normal oral function often wears the dentition and openings may occur in gold castings. These openings can be easily repaired with gold foil. Gold foil is also an excellent material for small lesions, particularly at the gum line. The gingiva will always shun a restoration and will remain below the margin, that is, except with gold foil, where the tissue is so comfortable that it will resume its natural sculpted position, covering the gold and resting in complete health. Gold foil is the only dental material that, if carefully placed and finished, can close the margin with no interface opening. The cast gold margin comes as close as 20 μm. No other material other than gold has such a tight marginal fit.

If conservation, preservation, and longevity are goals of the dentist and patient, then the usage of gold is the answer. In a world concerned with

toxicity, gold offers the best restoration with the least toxicity. Today, where the average life span has greatly increased, longevity of dental restorations must be a consideration, and again, gold is the best answer.

After seriously considering all the positive features of gold, one has to wonder why it is not in every dentist's armamentarium. There are two primary reasons. The first is that usage of gold, from preparation to finish, is exacting. All details should be satisfied 100%. There is no room for error. The second reason is that dental schools have dropped the teaching of gold from their curriculum because it requires exacting technique. Insufficient education is an obstacle, as proper education is essential. It is so sad that our finest and best material could become obsolete.

However, all is not lost. It would be ideal to reintroduce gold in dental schools. Moreover, it has happened in a few places. However, it is primarily three academies that maintain the "gold standard." The first is the AAGFO, which promotes the usage of direct gold. The Academy of R V Tucker Study Clubs (ARVTSC) promotes cast gold and study clubs. The Academy of Operative Dentistry (AOD) holds an annual meeting with two days of high-quality lectures, promoting excellence in all aspects of everyday dentistry. The AOD saw a need for a publication and joined forces with the AAGFO and the ARVTSC in support of *Operative Dentistry*, which is a highly esteemed and internationally acclaimed journal. The mission of these academies is to promote excellence in dentistry. In addition, camaraderie and sharing are part of this process, which holds to the highest standard.

These academies function to disseminate information and technique, encouraging the study club venue. Study club is a direct clinical application where 3 to 15 clinicians each perform a procedure on a patient, with a mentor supervising in a congenial and supportive atmosphere. This is followed by a critique that becomes a teaching and sharing discussion. Performing an actual procedure, under supervision, provides the best learning experience by far. Although the learning curve may appear to be slow at first, it is the quickest way to learn gold techniques—or any technique for that matter. Knowledge acquired in study club benefits all aspects of dentistry. The study club dentist is always improving, doing better and better dentistry.

The Academy of Gold Foil Operators began as an organization in 1952. This organization started the journal *Operative Dentistry* and then parented the AOD in 1972. In 2016, the IAGFO was recognized as an affiliate of AAGFO. IAGFO began the World Gold Summit and held its first meeting January 27-29, 2017, in Chennai, India. This is very exciting for those who embrace excellence in dentistry. Clearly, this sister group in India has done just that. Using gold adds to our abilities and expands the services we can offer our patients. Take advantage of the opportunities available and join an academy, join a study club, or be innovative and start a study club or begin an academy. Take the challenge!

©Operative Dentistry, 2018, **43-1,** 3-11

**Clinical Technique/Case Report**

# Ultrathin Monolithic Zirconia Veneers: Reality or Future? Report of a Clinical Case and One-year Follow-up

R Souza • F Barbosa • G Araújo • E Miyashita • MA Bottino • R Melo • Y Zhang

### Clinical Relevance

Translucent zirconia has become esthetic, make it a viable alternative for the manufacturing of ultrathin veneers.


## SUMMARY

**Yttria-stabilized polycrystalline zirconia ceramics have greatly advanced over the past few years. High-translucent zirconia is a newly introduced ceramic that affords high strength and esthetics and that has significantly increased the clinical indications of monolithic zirconia restorations. Thus, the purpose of this case report was to evaluate the performance of ultrathin monolithic zirconia veneers adhesively luted to enamel surfaces after minimally invasive preparations; in addition, we aimed at presenting a clinical protocol for zirconia surface treatment in order to promote bonding effectiveness to resin cement. This type of restoration presented very acceptable esthetic results and decreased the risk of fracturing the veneer during try-in and clinical use. The results were still satisfactory after one-year follow-up. However, randomized, prospective, controlled clinical trials are required to determine the long-term clinical durability of this treatment.**



*Rodrigo Othávio Assunção Souza, adjunct professor, Federal University of Rio Grande do Norte (UFRN), Department of Dentistry, Division of Prosthodontics, Natal, Brazil

Fernanda Pinheiro Barbosa, DDS, private clinical, Natal, Brazil

Gabriela Monteiro de Araújo, DDS, MSc, PhD student, Federal University of Rio Grande do Norte (UFRN), Department of Dentistry, Division of Prosthodontics, Natal, Brazil

Eduardo Miyashita, professor, Paulista University (UNIP), Department of Dentistry, São Paulo, Brazil

Marco Antonio Bottino, titular professor, Department of Dental Materials and Prosthodontics, Institute of Science and Technology, Univ Estadual Paulista–UNESP, Sao Jose dos Campos, Brazil

Renata Marques de Melo, researcher, Department of Dental Materials and Prosthodontics, Institute of Science and Technology, Univ Estadual Paulista–UNESP, Jose dos Campos, Brazil

Yu Zhang, New York University College of Dentistry, Department of Biomaterials and Biomimetics, New York, NY, USA

*Corresponding author: Av Salgado Filho, 1787, Lagoa Nova, Natal/RN, 59056-000, Brazil; e-mail: rodrigoothavio@gmail.com


DOI: 10.2341/16-350-T

## INTRODUCTION

Porcelain veneers comprise a conservative and highly esthetic treatment that also offer high predictability and good clinical performance in the long term.[1] With technological improvement and the evolution of dental restorative materials, it is currently possible to develop/produce ultrathin veneers with thicknesses of 0.1-0.3 mm, adhesively cemented on the tooth surface with minimal or no preparation, to modify color, shape, and/or positioning of the teeth.[2,3]

Several ceramic materials are currently indicated for veneers: lithium disilicate, feldspathic ceramic, feldspathic reinforced with leucite, fluorapatite, and lithium silicate reinforced with zirconia.[4-7] All of these ceramics exhibit high translucency characteristics due to the high content of glassy matrix in their composition, thus providing highly satisfactory esthetics in addition to excellent adhesion to resin cement through the conditioning with hydrofluoric acid (4%-10%) followed by silanization.[5] For these reasons, these ceramics have also been chosen for manufacturing of ultrathin veneers.[8]

On the other hand, high crystalline-content ceramics, such as tetragonal zirconia partially stabilized by yttria (Y-TZP), were originally considered only for the manufacturing of frameworks of crowns and fixed prostheses because of their high fracture resistance and ability to mask the dark substrate.[9] However, in recent years, zirconia ceramics have undergone many changes in microstructure and composition[10] to increase their translucency without significantly losing their fracture resistance,[11] thereby expanding their clinical indication. Thus, translucent zirconia has been considered as an esthetic material, as it offers indications for manufacturing crowns and anterior and posterior monolithic fixed prostheses, including veneers and ultrathin veneers.[12] The main difficulty associated with translucent zirconia involves situations with little mechanical retention of preparation, since polycrystalline zirconia is chemically inert and cannot be etched by hydrofluoric acid (4%-10%), which implies a less effective adhesion when compared to silica-based ceramics (acid-sensitive).[13]

Few clinical studies using monolithic zirconia prostheses have been reported in the literature. Rinke and Fischer[13] evaluated the clinical performance of posterior monolithic crowns and concluded that good esthetic results in the posterior region were achieved, even in cases of minimal occlusal space. In evaluating the wear of the enamel caused by monolithic translucent zirconia crowns in molars during six months of follow-up, Stober and others[14] concluded that the enamel wear of the antagonist tooth was equal to that caused by other ceramics.

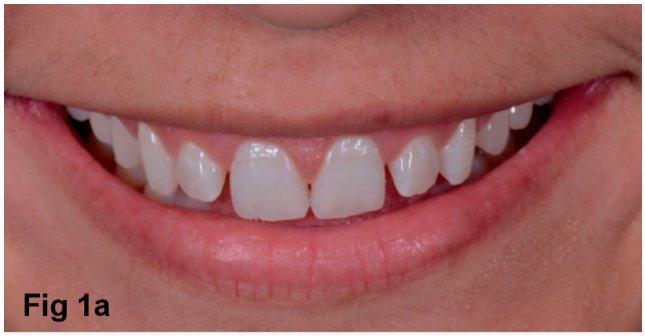
*In vitro* studies on veneers have reported a higher resistance to fracture of zirconia compared to that associated with lithium disilicate and feldspathic veneers,[16] which can be regarded as a great advantage of this material, as the proof and cementation stages of ultrathin veneers become much less critical compared to those of conventional glass ceramics. However, the same authors[16] also found that there is a possibility of zirconia veneers debonding as a result of less effective adhesion to resin cement.

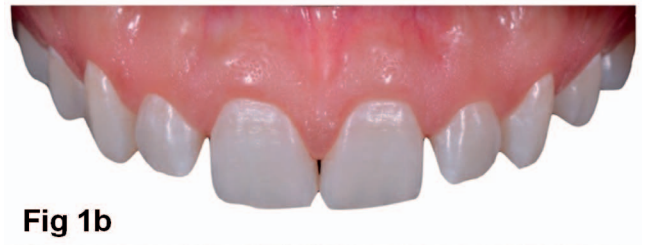
In order to optimize the adhesion between zirconia and cement, various surface treatments have been proposed: sandblasting with aluminum oxide,[17] tribochemical silica coating followed by silanization,[18] nanostructured alumina coating,[19] resin cement containing 10-methacryloxydecyl dihydrogen phosphate monomer (MDP),[20] universal primers also containing methacrylate monomers,[21] plasma processing, silica infiltration by the sol-gel method,[22] feldspathic glass infiltration,[23] selective infiltration-etching technique,[24] glaze-on technique,[25] and heating of silanes,[26] among others. Therefore, the treatment of zirconia surfaces has been the subject of much scientific research.[17,27-30] Depending on the type of treatment of the zirconia surface, it is possible to significantly improve their adherence to resin cement.[16,17,21,23] However, clinical studies with zirconia veneers and ultrathin zirconia veneers have not yet been published.

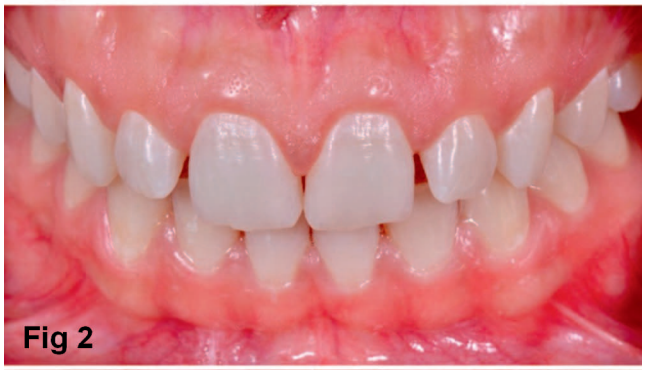
Thus, based on the promising results of surface treatments in zirconia and on the esthetic evolution of this material, the aim of this case report was to describe, through a clinical case, the manufacturing of ultrathin veneers using translucent zirconia, as well as to discuss relevant aspects for success in this type of treatment and to report a one-year follow-up of this clinical rehabilitation approach.

## CASE REPORT

A female patient, 25 years of age, sought specialized dental care, reporting small, yellowing upper front teeth with diastema as her main complaint. Upon clinical examination, the presence of a disharmonious smile with proclined lateral incisors, unfavorable dental proportions, diastema between lateral incisors and upper central incisors, and inadequate gingival contouring and zenith was observed (Fig-



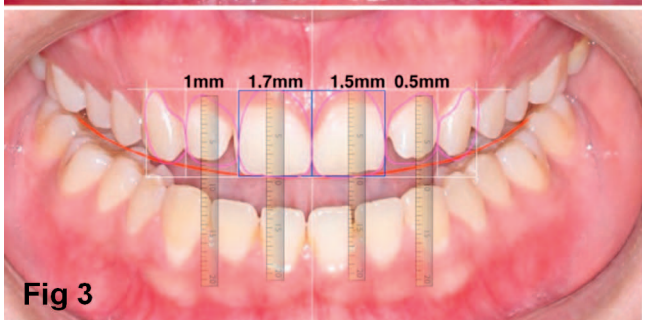




Figure 1. *(a) Initial apearance of patient smile and with (b) unfavorable dental proportions and diastema between lateral incisors and upper central incisors.*
Figure 2. *Intraoral view of teeth in occlusion. Patient with stable occlusion.*
Figure 3. *Digital smile analysis and planning for periodontal surgery before restorative procedures.*

ures 1a,b and 2). The occlusal contacts and the eccentric movements were evaluated by mounting of study models using the facial arch in a semiadjustable articulator. After digitally planning the smile, the need for correcting the contouring and gingival zenith was observed, increasing the incisal edges of the lateral incisors and vertically increasing the central incisors toward the cervical direction, setting a tooth ratio of 80% (the final width to length ratio of the centrals was 9/11.2 mm, approximately 80%). Surgery was then recommended for crown enlargement of teeth 7 through 10 (Figure 3). After 60 days, whitening therapy was initiated with the use of 16% carbamide peroxide (Whiteness Perfect/FGM, Joinville, SC, Brazil) for four weeks.

In the next session, an impression was made with vinyl polysiloxane/silicone (Express XT [commercially available in the United States as Express VPS], 3M ESPE, St Paul, MN, USA) to prepare stone models, a diagnostic wax-up, and mock-up (Systemp/Ivoclar Vivadent, Schaan, Liechtenstein) of teeth 6 to 11, according to the digital smile design (Figure 4a-c). In this stage, the occlusal contacts and the eccentric movements were evaluated again, clinically in the patient's mouth, with the assistance of articulating metallic film (Arti-fol [12 μm], Bausch, Germany). There was no need for any mock-up adjustment. Next, minimally invasive preparations were performed in teeth 6 through 11. A medium-grit diamond bur with rounded edge was used on the buccal surface of the teeth to remove a uniform thickness of 0.3 mm. Approximately 0.6 mm of the buccal surface of teeth 7 and 10 was removed because they were slightly proclined.

In order to guide all steps of tooth preparation, an index was made with a condensation silicone (Zetaplus/Zhermack, São Paulo, Brazil) (Figure 5a,b). All angles were rounded, and the cervical finish line in tilted chamfer was continuous, defined, and clear. The preparations were finished and polished with fine diamond burs, followed by multilaminated burs (Komet, Lemgo, Germany) and an Arkansas polisher (Komet) with the aid of a multiplier contra-angle (Sirona, Bensheim, Germany). In the same session, an impression of the prepared veneers was taken with addition silicone (Express XT [commercially available in the United States as Express VPS], 3M ESPE) and sent to a dental technician to manufacture the restorations in a prosthetic laboratory. Because the preparations were minimally invasive, there was no need to manufacture temporary veneers (Figure 6).

The stone models were scanned and the ultrathin veneers were fabricated with monolithic translucent zirconia (Prettau Anterior, Zirkonzahn, Gais, Switzerland) (Figure 7a,b) and milled in a Zirkonzahn CAD/CAM system. The veneers were characterized in the following way: before sintering, polishing with rubber tips followed by staining, and after sintering, new polishing and glaze. No veneering ceramics were applied.

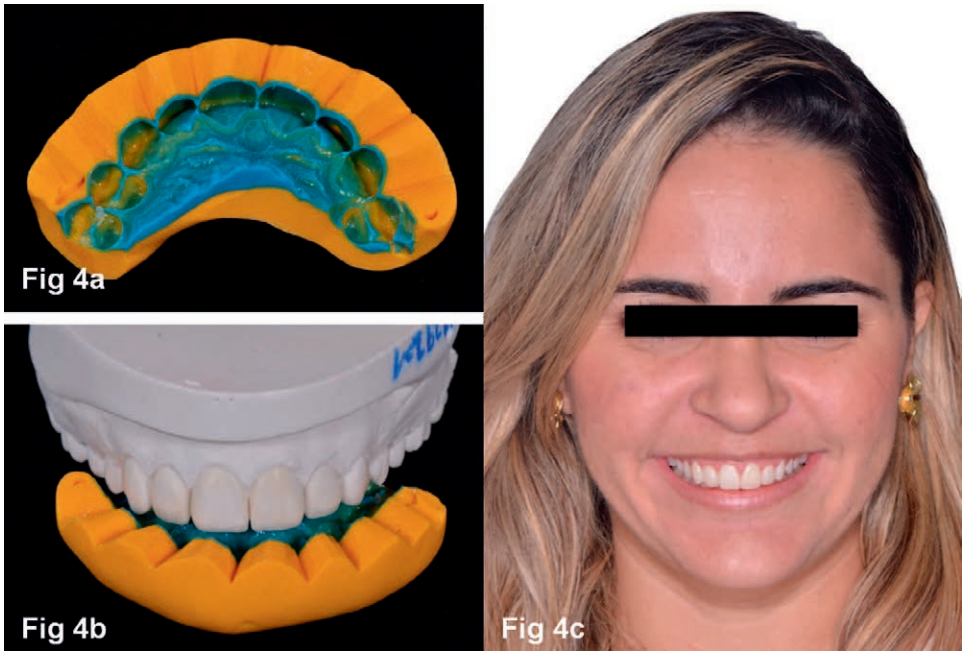



Figure 4. *(a) Silicone index for mock-up, (b) wax model, and (c) aspect of the patient after mock-up.*

After dry testing to check the marginal fit, the resin cement shade was selected using try-in pastes (Variolink Veneer Try-In, Ivoclar Vivadent, Schaan, Liechtenstein). It was possible to observe that the color was significantly affected by the shade of the try-in paste (Figure 8). Next, the veneers were washed, thoroughly dried, and the intaglio surfaces of the zirconia veneers and the tooth surfaces were treated as described below.

Prophylaxis was performed on the tooth surfaces with pumice and water, and then the surfaces were washed and thoroughly dried. These surfaces were then etched with 35% phosphoric acid (Ultra-Etch/ Ultradent) for 20 seconds, rinsed in running water, thoroughly dried, and treated with an adhesive system (Excite F/Ivoclar Vivadent). The intaglio surfaces of the veneers were abraded with particles of aluminum oxide coated with silica (CoJet, 3M ESPE) for 20 seconds (2.8 bar, 10-mm standoff distance) and dried. Silane was then applied (Monobond Plus/Ivoclar Vivadent) and left to dry for two minutes before the application of an adhesive system (Excite F/Ivoclar Vivadent) without curing.

Light-cure resin cement Variolink Veneer of "0" medium value (Ivoclar Vivadent) was inserted into the ceramic veneers, which were placed on their respective abutments. The excess resin cement was removed with a brush and floss, followed by light-curing for 40 seconds on each veneer surface (Radii Cal, SDI Limited, Victoria, Australia; 1000 mW/cm$^2$). The Radii light intensity was confirmed by a radiometer (Kondortech-Kondentech, São Paulo, Brazil). Next, glycerin gel (Liquid strip, Ivoclar Vivadent) was applied to the cervical and incisal regions of veneers and another curing cycle was performed on each face (Figure 9a-d). After curing, additional excess cement was removed with a No. 12 scalpel blade. The clinical appearance of the veneers after luting and one-year follow-up can be seen in Figures 10a,b and 11a,b, respectively. There was no need for any occlusal adjustment in the ultrathin zirconia veneers. Moreover, no protective splint was needed since the patient had no clinical signs or symptoms of bruxism.

## DISCUSSION

In the present clinical study, ultrathin veneers of monolithic cubic ultratranslucent zirconia were manufactured (Prettau Anterior, Zirkonzahn). This type of zirconia has recently been developed to provide adequate esthetic and mechanical properties for all-ceramic restorations for both anterior and posterior teeth.[31]

Several types of zirconia have been most often used in clinical dentistry, including traditional tetragonal (opaque) zirconia, with a strength range from 900 to 1200 MPa; high-translucent zirconia (900 to 1200 MPa), and cubic ultratranslucent zirconia (500 to 800 MPa).[31] This last generation of zirconia has excellent optical features compared to the other two types of zirconia described previously, and therefore we chose it for this clinical case.

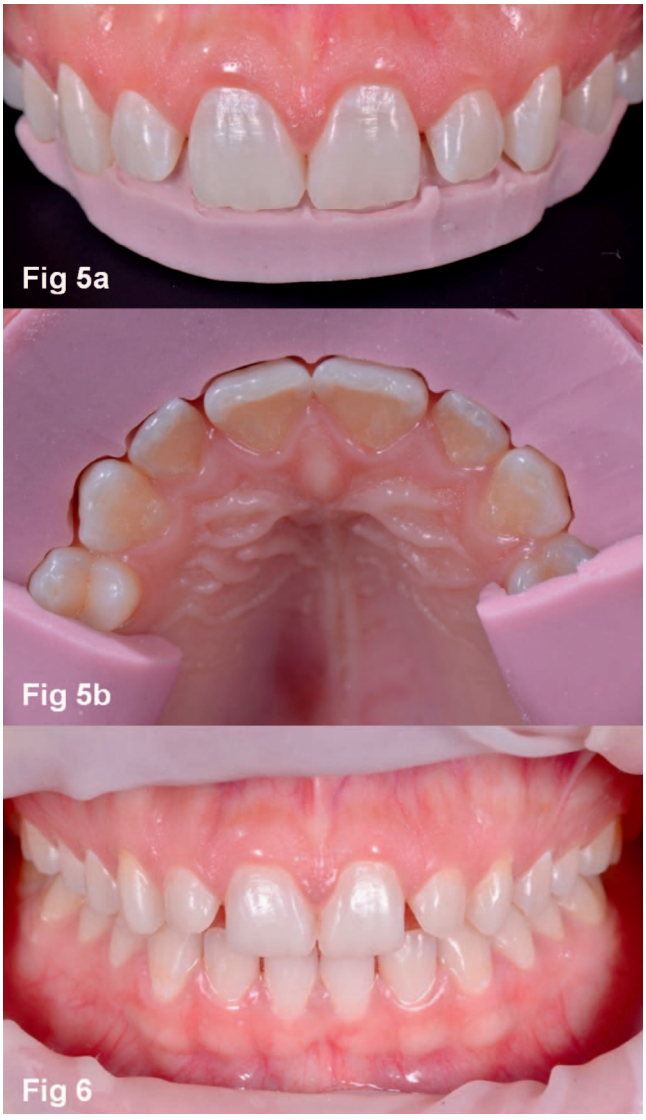



Figure 5. *Index positioned on the teeth before preparations: (a) buccal and (b) incisal views.*
Figure 6. *Clinical aspect after final preparation of the anterior teeth.*

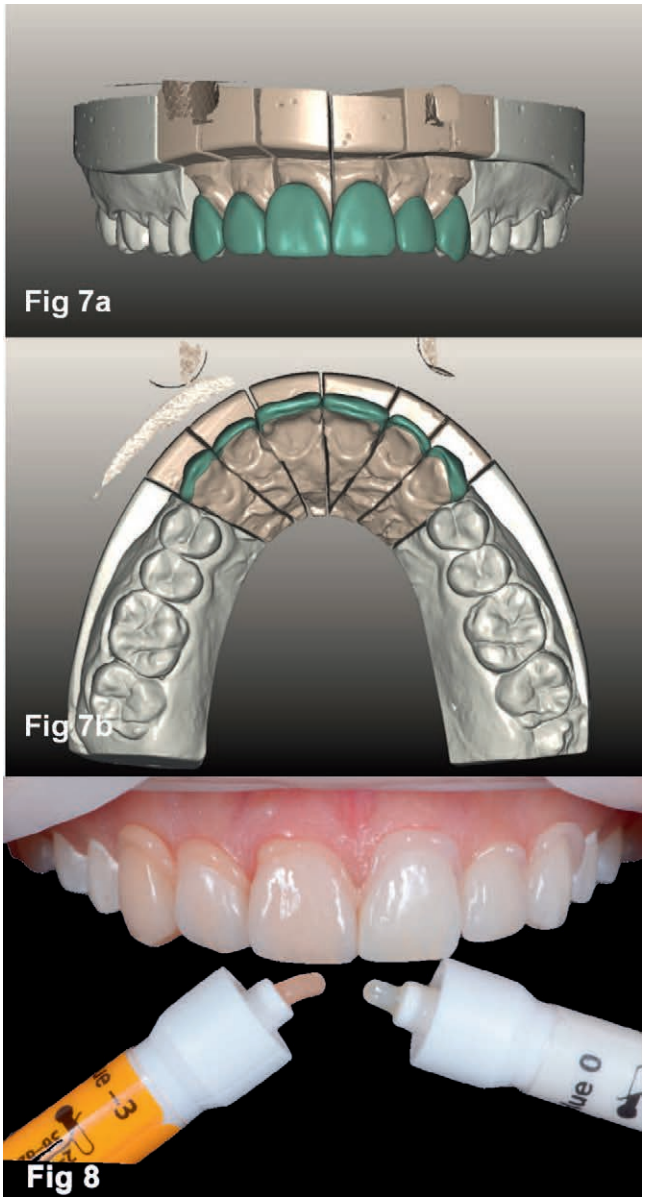



Figure 7. *Three-dimensional images of the veneers from 13 to 23 on the scanned model of the prepared teeth: (a) buccal and (b) incisal views.*
Figure 8. *Resin cement color test of ceramic veneers with try-in pastes (Variolink Veneer, Ivoclar Vivadent) of medium value 0 (left) and low value −3 (right). The cement of medium value 0 was selected.*

In order to achieve adequate translucency, the microstructure of zirconia was modified. It is known that zirconia can exist in three crystallographic forms depending on the temperature at ambient pressure: monoclinic, tetragonal, and cubic phases.[32] In conventional zirconia, 0.5%-1.0% of its weight is alumina and 3%-6% is yttrium oxide. On the other hand, translucent zirconia has 0.11% to 0.26% alumina[33] and a yttria concentration close to 12%.[34]

Alumina acts as a light-scattering center in zirconia as a result of its different refractive index, reducing the translucency of zirconia.[33] Furthermore, the amount of zirconia in the tetragonal phase was reduced and a larger amount of cubic zirconia was incorporated, thus enabling a more uniform transmission of light through zirconia.[9] Factors such as porosity[35] and grain size[36] also affect the translucency of zirconia. It was evident that zirconia is less translucent than glass ceramics, and the translucency decreased more slowly with material thickness.[37]

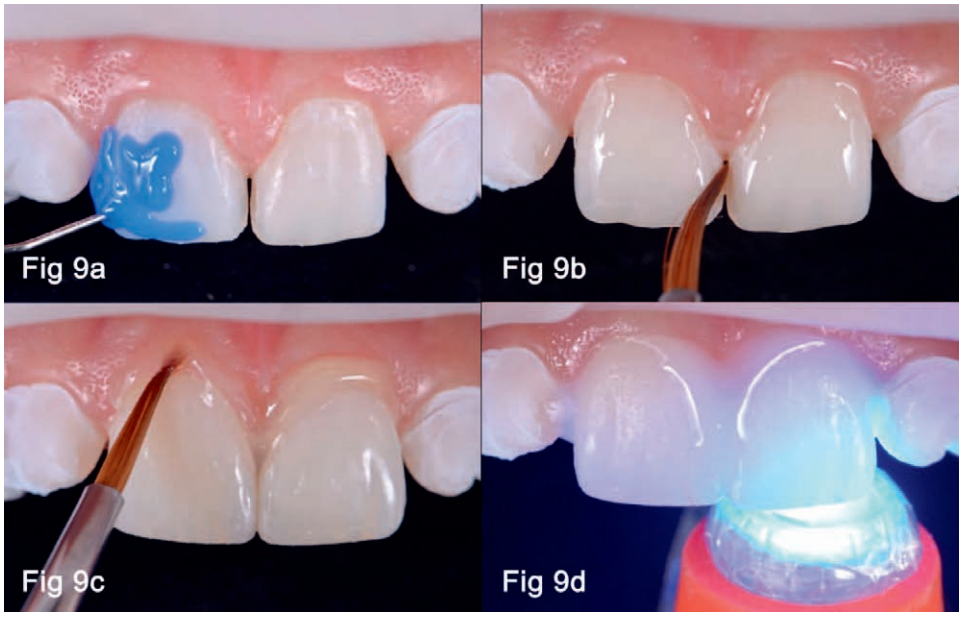



Figure 9. *Teeth surface treatment—(a) Etching with 35% phosphoric acid/15 seconds; (b) Application of the adhesive on the the tooth surface; (c) Removal of excess cement with a thin brush and dental floss; (d) Cement photopolymerization with glycerine gel application on the adhesive interface.*

However, high translucency can be observed for thinner translucent zirconia, specifically that which measures about 0.3 mm.[38] This was confirmed by the pleasant appearance achieved in this clinical case, in which 0.3 mm translucent zirconia veneers were used.

Few studies on zirconia veneers have been reported,[15,39] and none of them have used translucent zirconia. Alghazzawi and others[38] evaluated the influence of cement color on the final color of ceramic veneers and observed that conventional zirconia was

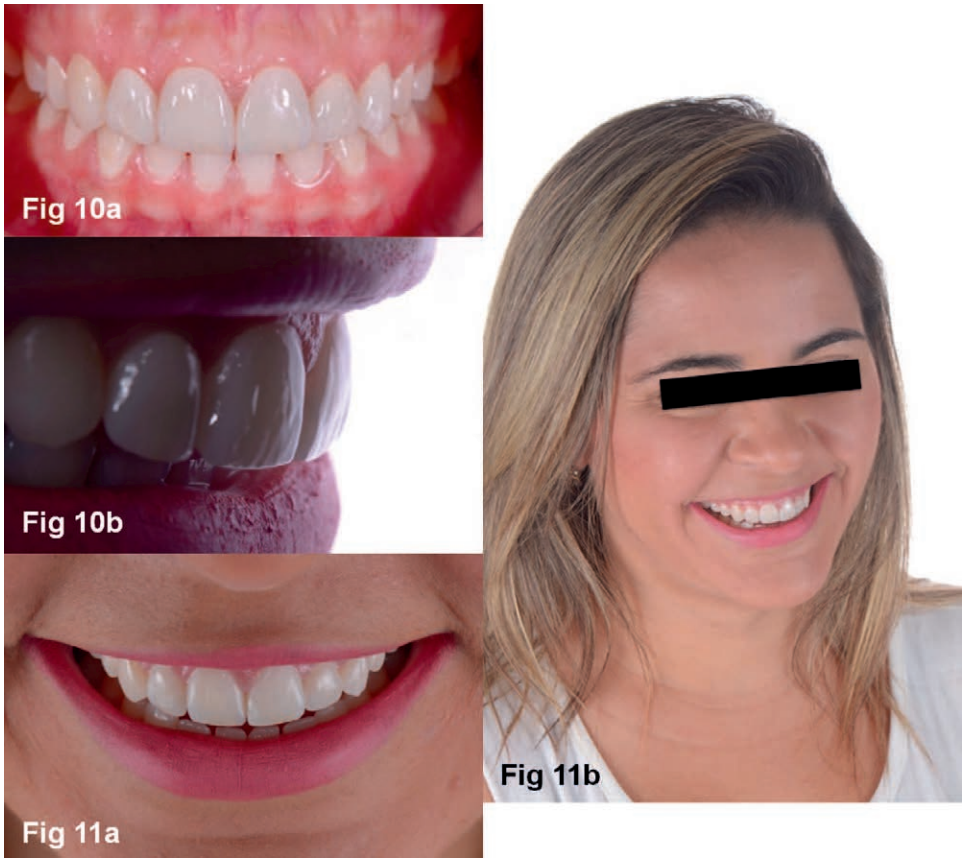



Figure 10. *(a) Final intraoral view in occlusion; (b) Close-up view of the central incisor characterization.*
Figure 11. *Final (a) smile and (b) facial aspect after one-year follow-up.*

neither influenced by the color of the resin cement nor by the color of the substrate, even at a minimum thickness. Currently, it is possible to manufacture veneers/ultrathin veneers made entirely from translucent zirconia.[40] According to our observations, and contrary to what was found for conventional zirconia, there was an influence of cement shade on the final color of the veneers during the selection of cement with the try-in test pastes.

With regard to the mechanical properties of translucent zirconia, the zirconia used in this clinical case exhibits a flexural strength of 670 MPa, according to the manufacturer's data sheet. This high-strength ultratranslucent zirconia offers great advantages over feldspathic ceramics or lithium disilicate ultrathin veneers, considering that these restorations are difficult to manipulate before cementation as a result of their brittleness, which may lead to fracture during the try-in stage.

Unarguably, the greatest difficulty in performing a treatment with zirconia veneers lies in its low adherence to resin cement, compared to that of the ceramics that can be conditioned by hydrofluoric acid (4%-10%) followed by silanization.[12] The loss of retention (debonding) of zirconia restorations has been reported in clinical studies.[41] For this reason, many surface treatments have been proposed to modify the surface of zirconia and to optimize adhesion to resin cements.[16,17,21,23,26,42] Among these treatments, silica coating has presented some of the best bonding results.

The tribochemical silica coating process (Cojet and Rocatec, 3M ESPE) consists of air-abrasion of the zirconia surface with alumina particles coated by silica, which promotes adhesion between the 3-methacryloxypropyltrimethoxysilane silane coupling agent and the silica adhered on the zirconia surface because of the impact.[16] The silicatization associated with a 10-MDP primer seems to be the most common form of surface treatment through which to provide long-lasting bonding to zirconia.[17,26] The phosphate ester groups in this silane bond directly to the surface oxides of zirconia, and the methacrylate group makes covalent bonds with the resin matrix of the cement.[43] Therefore, in the present clinical case, the monolithic zirconia veneers were air-abraded with 30 μm of alumina coated by silica (Rocatec Soft, 3M ESPE), followed by 10-MDP silane agent. We know that the one-year follow-up does not offer enough time with which to validate this type of treatment, but up until the writing of this article, none of the veneers had debonded. However, further *in vitro* and long-term clinical trials should be conducted to predict the clinical performance of high-translucent monolithic zirconia veneers.

## CONCLUSIONS

Based on the clinical case presented herein and scientific evidence, we can conclude that the use of translucent zirconia ultrathin veneers provides satisfactory esthetics; however, further longitudinal studies are necessary to confirm this type of treatment.

#### Acknowledgement

The authors are grateful to Vagner Laboratory (São Paulo, Brazil) for manufacturing the monolithic zirconia ultrathin veneers.

#### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Rio Grande do Norte, Brazil.

#### 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 April 2017)*

#### REFERENCES

1. Peumans M, Meerbeek BV, Lambrechts P, & Vanherke G (2000) Porcelain veneers: A review of the literature *Journal of Dentistry* **28(3)** 163-177.
2. Strassler HE (2007) Minimally invasive porcelain veneers: Indications for a conservative esthetic dentistry treatment modality *General Dentistry* **55(7)** 686-694.
3. Carpena G, Ballarin A, & Aguiar JA (2015) New ceramics approach for contact lens *Odovtos International Journal of Dental Sciences* **17(1)** 12-18.
4. Soares PV, Spini PH, Carvalho VF, Souza PG, Gonzaga RC, Tolentino AB, & Machado AC (2014) Esthetic rehabilitation with laminated ceramic veneers reinforced by lithium disilicate *Quintessence International* **45(2)** 129-133.
5. Conrad HJ, Seong W-J, & Pesun IJ (2007) Current ceramic materials and systems with clinical recommendations: A systematic review *Journal of Prosthetic Dentistry* **98(5)** 389-404.
6. Trinkner TF, & Roberts M (2001) Fluorapatite-leucite glass ceramic veneers for aesthetic anterior restorations *Practical Procedures & Aesthetic Dentistry* **13(1)** 37-41.
7. Manicone PF, Lammetti RP, & Raffaelli L (2007) An overview of zircônia ceramics: Basic properties and clinical applications *Journal of Dentistry* **35(11)** 819-826.
8. Spear F, & Holloway J (2008) Which all-ceramic system is optimal for anterior esthetics? *Journal of the American Dental Association* **139(9)** 19S-24S.

9. Zhang Y (2014) Making yttria-stabilized tetragonal zircônia *Dental Materials* **30(10)** 1195-1203.

10. Matsuzaki F, Sekine H, Honma S, Takanashi T, Furuya K, Yajima Y, & Yoshinari M (2015) Translucency and flexural strength of monolithic translucent zirconia and porcelain-layered zircônia *Dental Materials Journal* **34(6)** 910-917.

11. Koutayas SO, Vagkopoulou T, Pelekanos S, Koidis P, & Strub JR (2009) Zirconia in dentistry: Part 2. Evidence-based clinical breakthrough *European Journal of Esthetic Dentistry* **4(4)** 348-380.

12. Thompson JY, Stoner BR, Piascik JR, & Smith R (2011) Adhesion/cementation to zircônia and other non-silicate ceramics: Where are we now? *Dental Materials* **27(1)** 71-82.

13. Rinke S, & Fischer C (2013) Range of indications for translucent zirconia modifications: Clinical and technical aspects *Quintessence International* **44(8)** 557-566.

14. Stober T, Bermejo JL, Rammelsberg P, & Schmitter M (2014) Enamel wear caused by monolithic zirconia crowns after 6 months of clinical use *Journal of Oral Rehabilitation* **41(4)** 314-322.

15. Alghazzawi TF, Lemons J, Liu P-R, Essig ME, & Janowski GM (2012) The failure load of CAD/CAM generated zirconia and glass-ceramic laminate veneers with different preparation designs *Journal Prosthetic Dentistry* **108(6)** 386-393.

16. Sarmento HR, Campos F, Sousa RS, Machado JPB, Souza ROA, Bottino MA, & Özcan M (2013) Influence of air-particle deposition protocols on the surface topography and adhesion of resin cement to zirconia *Acta Odontologica Scandinavica* **72(5)** 346-353.

17. Alves MLL, Campos F, Bergoli CD, Botino MA, Özcan M, & Souza ROA (2016) Effect of adhesive cementation strategies on the bonding of Y-TZP to human dentin *Operative Dentistry* **41(1)** 1-8.

18. May LG, Passos SP, Capelli DB, Özcan M, Bottino MA, & Valandro LF (2010) Effect of silica coating combined to a MDP-based primer on the resin bond to Y-TZP ceramic *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **95(1)** 69-74.

19. Srikanth R, Kosmac T, Della Bona A, Yin L, & Zhang Y (2015) Effects of cementation surface modifications on fracture resistance of zirconia *Dental Materials* **31(4)** 435-442.

20. Queiroz JRC, Duarte DA, Souza ROA, Fissmer SF, Massi M, & Bottino MA (2011) Deposition of $SiO_x$ thin films on Y-TZP by reactive magnetron sputtering: Influence of plasma parameters on the adhesion properties between Y-TZP and resin cement for application in dental prosthesis *Materials Research* **14(2)** 212-216.

21. Pereira LL, Campos F, Dal Piva AM, Gondim LD, Souza RO, & Özcan M (2015) Can application of universal primers alone be a substitute for airborne-particle abrasion to improve adhesion of resin cement to zirconia? *Journal of Adhesive Dentistry* **17(2)** 169-174.

22. Campos TMB, Ramos NC, Machado JPB, Bottino MA, Souza ROA, & Melo RM (2016) A new silica-infiltrated Y-TZP obtained by the sol-gel method *Journal of Dentistry* **48** 55-61.

23. Chai H, Kaizer M, Chughtai A, Tong H, Tanaka C, & Zhang Y (2015) On the interfacial fracture resistance of resin-bonded zirconia and glass-infiltrated graded zirconia *Dental Materials* **31(11)** 1304-1311.

24. Aboushelib MN1 Kleverlaan CJ, & Feilzer AJ (2007) Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials *Journal of Prosthetic Dentistry* **98(5)** 379-388.

25. Ha JY, Son JS, Kim YK, Kim KH, & Kwon TY (2013) Effect of heat treatment of dental zirconia ceramic treated with three different primers on the bonding of resin cement *Macromolecular Research* **21(1)** 71-77.

26. Melo RM, Souza RO, Dursun E, Monteiro EB, Valandro LF, & Bottino MA (2015) Surface treatments of zirconia to enhance bonding durability *Operative Dentistry* **40(6)** 636-643.

27. Queiroza JRC, Duarteb DA, Souza ROA, Fissmerb SF, Massib M, & Bottino MA (2011) Deposition of SiOx thin films on Y-TZP by reactive magnetron sputtering: Influence of plasma parameters on the adhesion properties between Y-TZP and resin cement for application in dental prosthesis *Materials Research* **14(2)** 212-216.

28. Lung CY, Botelho MG, Heinonen M, & Matinlinna JP (2012) Resin zircônia bonding promotion with some novel coupling agents *Dental Materials* **28(8)** 863-872.

29. Castro HL, Corazza PH, & Paes-Júnior TA (2012) Influence of Y-TZP ceramic treatment and different resin cements on bond strength to dentin *Dental Materials* **28(11)** 1191-1197.

30. Inokoshi M, Kameyama A, De Munck J, Minakuchi & Van Meerbeek B (2013) Durable bonding to mechanically and/or chemically pre-treated dental zirconia *Journal of Dentistry* **41(2)** 170-179.

31. Rondoni D (2016) Zirconia: Some practical aspects from the technologist's point of view *International Journal of Esthetic Dentistry* **11(2)** 2-6.

32. Harada K, Raigrodski AJ, Chung KH, Flinn BD, Dogan S, & Mancl LA (2016) A comparative evaluation of the translucency of zirconias and lithium disilicate for monolithic restorations *Journal of Prosthetic Dentistry* **116(2)** 257-263.

33. Sulaiman TA, Abdulmajeed AA, Donovan TE, Ritter AV, Lassila LV, Vallittu PK, & Närhi TO (2015) Degree of conversion of dual-polymerizing cements light polymerized through monolithic zirconia of different thicknesses and types *Journal of Prosthetic Dentistry* **114(1)** 103-108.

34. Vichi A, Louca C, Corciolani G, & Ferrari M (2011) Color related to ceramic and zirconia restorations: A review *Dental Materials* **27(1)** 97-108.

35. Klimke J, Trunec M, & Krell A (2011) Transparent tetragonal yttria-stabilized zirconia ceramics: Influence of scattering caused by birefringence *Journal of the American Ceramic Society* **94(6)** 1850-1858.

36. Jiang L, Liao YM, Wan QB, & Li W (2011) Effects of sintering temperature and particle size on the translucency

of zirconium dioxide dental ceramic *Journal of Materials Science: Materials in Medicine* **22(11)** 2429-2435.

37. Tong H, Tanaka CB, Kaizer MR, & Zhang Y (2016) Characterization of three commercial Y-TZP ceramics produced for their high-translucency, high-strength and high-surface area *Ceramics International* **42(1)** 1077-1085.
38. Alghazzawi TF, Lemons J, Liu P-R, Essig ME, & Janowski GM (2012) Evaluation of the optical properties of CAD-CAM generated yttria-stabilized zirconia and glass-ceramic laminate veneers *Journal of Prosthetic Dentistry* **107(5)** 300-308.
39. Kelly JR, Nishimura I, & Campbell SD (1996) Ceramics in dentistry: Historical roots and current perspectives *Journal of Prosthetic Dentistry* **75(1)** 18-32.
40. Al-Amleh B, Lyons K, & Swain M (2010) Clinical trials in zirconia: A systematic review *Journal of Oral Rehabilitation* **37(8)** 641-652.
41. Vanderlei AD, Queiroz JR, Bottino MA, & Valandro LF (2014) Improved adhesion of Y-TZP ceramics: A novel approach for surface modification *General Dentistry* **62(1)** 22-27.
42. Ha JY, Son JS, Kim YK, Kim KH, & Kwon TY (2013) Effect of heat treatment of dental zirconia ceramic treated with three different primers on the bonding of resin cement *Macromolecular Research* **21(1)** 71-77.
43. Kern M (2009) Resin bonding to oxide ceramics for dental restorations *Journal of Adhesion Science and Technology* **23(7-8)** 1097-1111.

©Operative Dentistry, 2018, **43-1,** 12-21

## Clinical Research

# 12 Years of Repair of Amalgam and Composite Resins: A Clinical Study

J Estay • J Martín • V Viera • J Valdivieso • C Bersezio • P Vildosola • IA Mjor *In Memoriam* MF Andrade • RR Moraes • G Moncada • VV Gordan • E Fernández



### Clinical Relevance

Repairing defective composite resin and amalgam restorations is a safe and effective treatment that might increase restoration longevity.


### SUMMARY

**Objective: The objective of this study was to clinically evaluate repaired posterior amalgam and composite restorations over a 12 year period, investigate the influence of repair in the survival of restorations, and compare their behavior with respect to controls.**

**Methods: Thirty-four patients, 18 to 80 years of age with 167 restorations, 67 composite resin (RC), and 100 amalgam (AM) restorations, participated. Restorations with localized, marginal, anatomical deficiencies and/or secondary caries, and "clinically judged" suitable for repair or replacement according to US Public Health Service (USPHS) criteria, were randomly assigned to four groups: repair (n=35, 20 AM, 15 RC), replacement (n=43, 21 AM, 22 RC), positive control (n=71, 49 AM, 22 RC), or negative control (n=18, 10 AM, 8 RC). The quality of the restorations was blind scored according to the modified USPHS criteria. Two examiners scored them at initial status (κ=0.74) and after one to five, 10, and 12 years (κ=0.88). Wilcoxon and Mann-Whitney tests provided for comparisons within the same group and between years, respectively.**

**Results: After 12 years, all groups behaved similarly in marginal adaptation, marginal stain, teeth sensitivity, anatomic form, and**



Juan Estay, PhD, DS, DDS, Restorative Dentistry, University of Chile, Santiago, Chile

Javier Martín, PhD, DS, DDS, Restorative dentistry, University of Chile, Santiago, Chile

Valentina Viera, Restorative Dentistry, University of Chile, Santiago, Chile

Josefa Valdivieso, Restorative Dentistry, University of Chile, Santiago, Chile

Cristian Bersezio, PhD, DDS, Restorative Dentistry, University of Chile, Santiago, Chile

Patricio Vildósola, PhD, DDS, MsC, DDS, Restorative Dentistry, University of Chile and Universidad Andres Bello, Santiago, Chile

Ivar A Mjor *In Memoriam*, BDS, MSD, MS, Dr.Odont, Department of Restorative Dental Sciences, Division of Operative Dentistry, University of Florida College of Dentistry, Gainesville, FL, USA

Marcelo Ferrarezi Andrade, MSc, DDS, School of Dentistry at Araraquara, Restorative Dentistry, Universidad Estadual Paulista, São Paulo, Brazil

Rafael R Moraes, DDS, MS, PhD, professor, Restorative Dentistry, School of Dentistry, Federal University of Pelotas, Pelotas, Brazil

Gustavo Moncada, PhD, DDS, Restorative Dentistry, University of Chile, Santiago, Chile

Valeria V Gordan, DDS, MS, MSCI, Department of Restorative Dental Sciences, University of Florida, Gainesville, FL, USA

*Eduardo Fernández, DDS, DS, PhD, professor, Restorative Dentistry, University of Chile, Santiago, Chile and Instituto de Ciencias Biomédicas, Universidad Autónoma de Chile.

*Corresponding author: 11 de Septiembre 1881 of 2108, Santiago 7500505, Chile; e-mail: edofdez@yahoo.com.


DOI: 10.2341/16-313-C

**luster ($p \geq 0.05$). Better behavior in roughness was observed in replaced RC ($p = 0.049$).**

**Conclusions: Given that most clinical parameters investigated were similar between all groups during the follow-up, the repair of RC and AM restorations is a good clinical option because it is minimally invasive and can consistently increase the longevity of restorations.**

## INTRODUCTION

When a partial restoration of amalgam or composite resin fails, by secondary caries, fracture, or other causes, the simplest treatment, when possible, would be to repair the localized defect instead of full restoration replacement. However, clinicians commonly prefer to replace the restoration[1] despite the fact that current evidence suggests that secondary caries corresponds to a primary lesion with identical biofilm composition that does not compromise further the locally affected area.[2]

Studies indicate that there is no relationship between marginal staining[3] or marginal gap size and the appearance of new caries lesions adjacent to restorations.[4] Radiolucent halos beneath composite restorations may also not justify restoration replacement.[5] Previous studies[6-9] indicated that repaired and replaced restorations showed similar survival outcomes regarding marginal defects and secondary caries in patients with low and medium caries risk, and most of the restorations were considered clinically acceptable after five, seven, and 10 years of clinical service.[7,10,11] This could be a reliable alternative to increase the longevity of restorations, thereby avoiding an increase in cavity size and postponing the indication of more invasive treatments, such as crown restorations and root canal treatments.

The objective of this study was to clinically evaluate repaired posterior amalgam and composite restorations over a period of 12 years. The principal goals were 1) to investigate the failures of restorations that were repaired and replaced and 2) to compare their clinical condition to control groups. The hypothesis was that repair of amalgam and composite restorations with occlusal marginal defects would improve their clinical conditions, with performance similar to replacement after 12 years of clinical service.

## METHODS AND MATERIALS

### Study Design

A total of 34 patients from 18 to 80 years of age (mean 26.4 years), comprising both females (58%) and males (42%) who had a total of 67 posterior composite resin and 100 posterior amalgam restorations, were recruited at the Operative Dentistry Clinic at the Dental School of the University of Chile. The restorations presented localized, marginal, anatomical deficiencies and/or secondary caries adjacent to restorations that deviated from the ideal and thus were rated Bravo or Charlie according to the modified US Public Health Service (USPHS) criteria. The restorations were assigned to experimental groups (repair and replacement) and a control group, where the restoration was controlled without treatment. The study protocol was approved by the Institutional Research Ethics Committee of the Dental School at the University of Chile (Project PRI-ODO-0207/NCT02043873). All patients signed informed consent forms, completed registration forms, and agreed to participate in the study independent of the treatment received. Patients whose restorations failed were removed from the study and retreated but were still included in the final analytical statistics according to the intention to treat "CONSORT" protocol[12] (Figure 1).

### Inclusion and Exclusion Criteria

*Inclusion*

- Amalgam and composite restorations placed in posterior teeth with localized, marginal, anatomical deficiencies and/or secondary caries that were clinically judged to be suitable for repair or replacement according to the modified USPHS/Ryge criteria (Table 1)
- Patients with more than 20 teeth
- Posterior restorations in functional occlusion with an opposing natural tooth
- Asymptomatic restored tooth
- At least one proximal contact area with an adjacent tooth
- Patients older than 18 years
- Patients who agreed to and signed the consent form for participating in the study
- Remaining tooth structures in good condition

*Exclusion*

- Patients with contraindications for regular dental treatment based on their medical history
- Patients with special esthetic requirements that could not be solved by repair treatments
- Patients with xerostomia or taking medication that significantly decreased salivary flow
- Patients with high caries risk
- Patients with psychiatric or physical diseases that interfered with oral hygiene
- Composite and amalgam restorations with localized defects by secondary caries or marginal

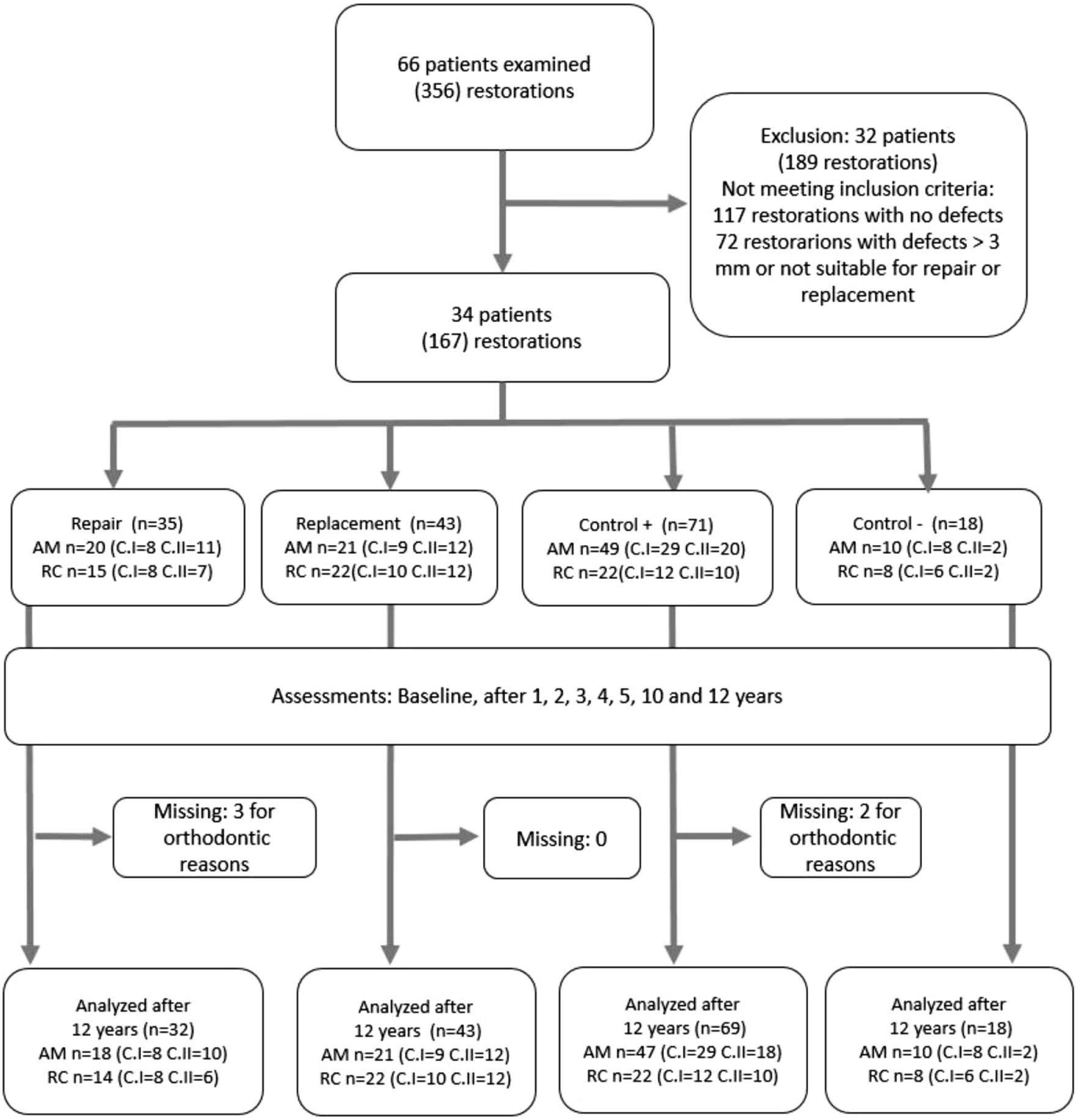



Figure 1. *Flow chart of clinical trial.*

defects greater than 3 mm and located and/or in the proximal surfaces

- Clinical judgment that repair was not indicated in resin or amalgam restorations, for example, when defective area covered most of the restoration.

Due to local ethics committee requirements at the time the trial was formulated, it was not recommended to include high-caries-risk patients and defects greater than 3 mm because repair was classified as an experimental treatment 12 years ago.

Table 1: *USPHS/Ryge Modified Clinical Criteria*

| Clinical Characteristics | Alpha | Bravo | Charlie |
|---|---|---|---|
| Marginal adaptation | Explorer does not catch when drawn across the restoration–tooth interface | Explorer falls into crevice or has one-way catch when drawn across the restoration–tooth interface | Dentin or base is exposed |
| Surface roughness | Surface of restoration has no defects | The surface of restoration has minimal surface defects | Surface of restoration has severe surface defects |
| Secondary caries | No clinical diagnosis of caries | Not applicable | Clinical diagnosis of caries |
| Marginal stain | No discoloration between the restorations and tooth | Discoloration on less than half of the circumferential margin | Discoloration on more than half of the circumferential margin |
| Teeth sensitivity | No sensitivity when an air syringe is activated for two seconds at a distance of half an inch from the restoration with the facial surface of the proximal tooth covered with gauze | Sensitivity is present when an air syringe is activated for two seconds at a distance of half an inch from the restoration with the facial surface of the proximal tooth covered with gauze | Sensitivity is present when an air syringe is activated for two seconds at a distance of half an inch from the restoration with the facial surface of the proximal tooth covered with gauze, and sensitivity does not cease when the stimulus is removed |
| Anatomic form | General contour of the restorations follows the contour of the tooth | General contour of the restoration does not follow the contour of the tooth | The restoration has an overhang |
| Luster | The restoration surface is shiny and has an enamel-like, translucent surface | Restoration surface is dull and somewhat opaque | The restoration surface is distinctly dull and opaque and is esthetically displeasing |

### Sample Size Determination and Randomization

Sample size was determined *a priori* using G*Power 2.22[13] with an error probability of $\alpha = 0.05$, effect size of 0.3, and power ($\beta - 1$ error probability) of 0.80. The restorations with marginal defects (Bravo) were randomly assigned (performed with PASS 2004 software, NCSS, Kaysville, UT, USA) to one of two groups of treatment: repair (n=35) and replacement (n=43). Two control groups were constituted: a positive control without marginal defects (Alpha) (n=71) and a negative control with clinically acceptable marginal defects (Bravo) (n=18). Only faculty members were allowed to provide the restorative treatment.

### Caries Risk Assessment

Computer software (Cariogram) was used to assess individual patients' caries risk; the software weighted the interaction between the following 10 caries-related factors: caries experience, related general disease, diet contents, diet frequency, plaque amount by the Silness Loe Index, semiquantitative detection of mutans streptococci and lactobacilli in saliva by caries risk test (CRT) bacteria (Ivoclar Vivadent AG, Schaan, Liechtenstein), fluoride program, amount of saliva stimulated secretion by CRT buffer (Ivoclar), saliva buffer capacity, and clinical judgment. Patients were classified in the following three Cariogram caries risk categories: high = 0% to 40% chance to avoid caries, intermediate = 41% to 60% chance to avoid caries, and low = 61% to 100% chance to avoid caries. In addition, the results indicated where targeted actions to improve the situation would have the best effect.

### Restoration Assessment

Two examiners underwent calibration exercises each year (J.M. and E.F.). The Cohen kappa interexaminer coefficient was 0.74 at the initial status and 0.88 at 12 years. The quality of the restorations was evaluated using the modified USPHS/Ryge criteria (Table 1). The two examiners assessed the restorations independently by visual (mouth mirror #5, Hu Friedy Manufacturing Co Inc, Chicago, IL, USA) and tactile examination using an explorer (N8 23, Hu Friedy) and indirectly by radiographic (Sirona Heliodent Vario, Charlotte, NC, USA) examination (Bite Wing, DF57, Kodak Dental System Healthcare, Rochester, NY, USA). The assessment of this initial status was considered as data immediately prior to the intervention for further statistical analysis.

If any difference was recorded between the two examiners and agreement could not be reached, a third clinician (G.M.) was called to assist with the

decision process. If the three clinicians did not reach an agreement, the lowest score was recorded.

### Treatment Groups

*Repair*—The clinicians (P.V. and G.M.) used carbide burs (330-010 Komet, Brasseler GmbH Co, Lemgo, Germany) to explore the defective margins of the restorations, beginning with the removal of part of the restorative material adjacent to the defect to act as an exploratory cavity. This allowed a proper diagnosis and evaluation of the extent of the defect. Provided that the defect was limited and localized, the clinician then removed any defective, demineralized, and soft tooth tissue. For composite restorations, a one-step, self-etch adhesive was used (Adper Prompt L-Pop, 3M ESPE, St Paul, MN, USA) according to the manufacturer's instructions, followed by a restoration with nanofill composite resin restorative material (Filtek Supreme, 3M ESPE). For amalgam restorations, mechanical retention was used inside the existing amalgam restoration followed by use of a dispersed-phase amalgam alloy (Original D, Wyckle Research Inc, Carson City, NV, USA) following the manufacturer's intructions. Rubber dam isolation was used during the entire procedure.

*Replacement*—The clinicians totally removed and replaced the defective restorations. After completing the cavity preparations, restoration was carried out with new composite resin or amalgam, according to the material of the previous restoration. Elimination of the soft, infected carious dental tissue was made using carbide burs at high speed under full water irrigation. During cavity preparation, no preventive extension or undercut area was created, and all cavity angles were rounded. In deep dentin, a glass-ionomer liner (Vitrebond, 3M ESPE) was applied. Adper Prompt L-Pop (3M ESPE) was applied and the composite (Filtek Supreme, 3M ESPE) inserted using an incremental technique. For new amalgam restorations (Tytin, Kerr Corp, Orange, CA, USA), bonding agent and/or liner were not used. Rubber dam isolation was used during the entire procedure.

All treatments were performed by the same clinicians (P.V. and G.M), who did not serve as examiners. The occlusion was checked, and the restorations were finished and polished following the restorative material manufacturer's instructions.

*Positive Control*—The restorations were made at the start of the study and did not receive any treatment.

*Negative Control*—The defective restorations did not receive any treatment.

### Restoration Assessment and Follow-Up

Patients were recalled each year in the first five years of clinical service, then after 10 and 12 years for clinical evaluation by the same examiners using the USPHS/Ryge modified clinical criteria as used at initial assessment (baseline). Failed restorations were removed from the study and treated according to their diagnosed needs. Digital photographs and bitewing radiographs were taken for all the restorations before and after treatment and every year prior to the examination.

### Statistical Analysis

The Mann-Whitney test was performed for comparisons between groups at 12-year recall. The Wilcoxon test was performed for comparisons between the initial state and after one-year and 12-year recall in the same group with a significance level of 0.05. The statistical analysis was performed using SPSS 21.0 (IBM, New York, NY, USA) The "intention to treat" CONSORT protocol was used to analyze data on restorations that were evaluated in year 12 and lacked data from a previous evaluation. Restorations that could not be assessed in year 12 were considered absent and were not entered into the analysis.

## RESULTS

The recall of this cohort of patients at 12 years was 100%. The distribution according to caries-risk patients was 80% medium risk (n=133) and 20% low risk (n=34); five missing restorations (2.99%) were lost by orthodontic treatment. The clinical condition of restorations evaluated at the initial state, after one year, and after 12 years is shown in Table 2.

### Between-Group Comparisons: Mann-Whitney Test

*Amalgam Restorations*—When comparing values of the 12th year in all Ryge parameters of repair vs the positive control group, statistically significant differences were observed for marginal adaptation ($p$=0.021), roughness ($p$=0.041), and secondary caries ($p$=0.026), with better performance in the positive control group.

When comparing values of the 12th year in all Ryge parameters of repair vs the negative control group, no statistically significant differences were observed.

Table 2: *Frequency of Alpha Scores for Amalgam Restorations and Resin Composite Over Time Expressed as Percentage*

| | | Repair | | | Replacement | | | Positive Control | | | Negative Control | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | IA | 1 | 12 | IA | 1 | 12 | IA | 1 | 12 | IA | 1 | 12 |
| Amalgam | MA | 20% | 60% | 0% | 10% | 91% | 5% | 100% | 82% | 16% | 0% | 0% | 0% |
| | A | 20% | 60% | 10% | 19% | 76% | 10% | 82% | 80% | 18% | 60% | 50% | 10% |
| | R | 50% | 60% | 10% | 29% | 67% | 5% | 92% | 82% | 31% | 90% | 80% | 0% |
| | MS | 80% | 95% | 50% | 52% | 95% | 24% | 100% | 94% | 41% | 70% | 60% | 20% |
| | S | 100% | 100% | 95% | 91% | 100% | 91% | 100% | 100% | 100% | 100% | 100% | 100% |
| | SC | 80% | 100% | 90% | 62% | 100% | 100% | 100% | 100% | 100% | 100% | 100% | 100% |
| | L | 35% | 80% | 5% | 43% | 91% | 0% | 76% | 61% | 14% | 40% | 40% | 0% |
| Resin composite | MA | 20% | 60% | 0% | 27% | 91% | 23% | 100% | 96% | 23% | 0% | 0% | 0% |
| | A | 27% | 93% | 27% | 18% | 91% | 41% | 86% | 73% | 23% | 50% | 50% | 13% |
| | R | 53% | 93% | 7% | 59% | 82% | 46% | 96% | 96% | 55% | 75% | 75% | 38% |
| | MS | 40% | 87% | 13% | 64% | 100% | 55% | 91% | 91% | 55% | 88% | 63% | 38% |
| | S | 100% | 100% | 100% | 91% | 100% | 100% | 100% | 100% | 100% | 100% | 100% | 100% |
| | SC | 100% | 100% | 80% | 46% | 100% | 96% | 100% | 100% | 100% | 100% | 100% | 100% |
| | L | 60% | 87% | 13% | 36% | 96% | 41% | 96% | 73% | 23% | 88% | 63% | 13% |

*Abbreviations: MA, marginal adaptation; IA, initial assessment (baseline); A, anatomy; R, roughness; MS, marginal staining; S, sensitivity; SC, secondary caries; L, luster.*

When comparing values of the 12th year in all Ryge parameters of replacement vs the positive control group, statistically significant differences were observed for roughness ($p$=0.043), with better performance in the positive control group.

When comparing values of the 12th year in all Ryge parameters of replacement vs the negative control group, no statistically significant differences were observed.

When comparing values of the 12th year in all Ryge parameters of replacement vs the repair group, no statistically significant differences were observed.

*Composite Resin Restorations*—When comparing values of the 12th year in all Ryge parameters of repair vs the positive control group, statistically significant differences were observed for roughness ($p$=0.014), with better performance in the positive control group.

When comparing values of the 12th year in all Ryge parameters of repair vs the negative control group, no statistically significant differences were observed.

When comparing values of the 12th year in all Ryge parameters of replacement vs the positive control group, no statistically significant differences were observed.

When comparing values of the 12th year in all Ryge parameters of replacement vs the negative control group, no statistically significant differences were observed.

When comparing values of the 12th year in all Ryge parameters of replacement vs the repair group, statistically significant differences were observed for roughness ($p$=0.049), with better performance in the replacement group.

### Within-Group Comparisons: Wilcoxon Test

*Amalgam Restorations*—In the repair group, roughness ($p$=0.013), marginal staining ($p$=0.007), and luster ($p$=0.007) presented statistically significant differences between baseline and 12-year follow-up, whereas the remaining clinical parameters had similar outcomes ($p \geq 0.05$). When comparing first-year follow-up with 12-year follow-up, significant differences were observed for marginal adaptation, anatomy, roughness, marginal staining, and luster ($p < 0.007$).

When comparing the inital assessment and 12-year exam for the replacement group, marginal adaptation ($p$=0.021), marginal staining ($p$=0.014), secondary caries ($p$=0.005), and luster ($p$=0.011) showed statistically significant differences, and the remaining clinical parameters showed similar outcomes ($p > 0.05$). When comparing first-year follow-up with 12-year follow-up, significant differences were observed for marginal adaptation, anatomy, roughness, marginal staining, and luster ($p < 0.00$).

In the positive control group, all parameters showed statistically significant differences ($p \leq 0.001$) except in sensitivity and secondary caries.

Table 3: *Characteristics of Failed Restorations*

| Group | Material | Class | Reason for Failure | Year of Recall at Time of Failure |
|---|---|---|---|---|
| Repair | Amalgam | I | Secondary caries | 10 |
| | Amalgam | II | Marginal adaptation | 10 |
| | Amalgam | I | Secondary caries | 4 |
| | Composite | I | Marginal adaptation | 3 |
| | Composite | I | Secondary caries | 10 |
| | Composite | I | Secondary caries | 3 |
| | Composite | I | Secondary caries | 10 |
| Replacement | Composite | I | Secondary caries | 4 |
| Positive control | Amalgam | II | Marginal adaptation | 1 |
| Negative control | Amalgam | II | Secondary caries | 1 |
| | Amalgam | II | Secondary caries | 5 |
| | Amalgam | II | Secondary caries | 5 |

In the negative control group, all parameters showed statistically significant differences ($p \leq 0.046$) except in marginal adaptation, sensitivity, and secondary caries.

*Composite Resin Restorations*—When comparing the assessments at baseline and 12-year exams for the repair group, roughness ($p=0.008$) and luster ($p=0.020$) showed statistically significant differences; the other clinical caracteristics had similar results ($p>0.05$). When comparing first-year follow-up with 12-year follow-up, significant differences were observed for marginal adaptation, anatomy, roughness, marginal staining, and luster ($p<0.004$).

In the replacement group, anatomy ($p=0.029$) and secondary caries ($p=0.001$) presented statistically significant differences; the remaining clinical parameters showed similar outcomes ($p>0.05$). When comparing first-year follow-up with 12-year follow-up, significant differences were observed for marginal adaptation, anatomy, roughness, marginal staining, and luster ($p<0.002$).

In the positive control group, all parameters showed statistically significant differences ($p \leq 0.003$) except in sensitivity and secondary caries.

In the negative control group, luster presented significant differences when comparing 12-year vs initial assessment ($p=0.014$) and vs one-year ($p=0.046$); the remaining clinical parameters showed similar outcomes ($p>0.05$).

The observed failures in the study groups are shown in Table 3.

## DISCUSSION

This clinical, prospective study assessed patients who received repair in localized defects of amalgam and composite restorations for a 12-year observation period compared to full restoration replacement. In order to evaluate the influence of repair on the longevity of restorations, a comparison year after year with the initial state of the restorations at the beginning of this study was necessary. Following this logic, it was assumed that a restoration recently replaced (replacement group as a positive control) was an Alpha score restoration because it was performed by an experienced clinician and under all the ideal conditions, unlike other designs where the effectiveness of a treatment is itself compared from its initial state (baseline).

Over time, continuing deterioration in all parameters was observed for all groups. This finding is related to the deterioration of the restorative material properties with time, particularly the surface properties in this study. Tooth sensitivity, interestingly, remained at levels close to zero, which means that the so-called bonding degradation ascribed to dentin adhesives over time[14,15] was not able to clinically influence the restorations, at least in terms of sensitivity. Secondary caries was also a low-occurrence event that was observed in a few cases since the third year of follow-up, showing that both treatments are equally well tolerated. A similar observation has been shown in reports with five- and 10-year follow-up.[6,8,16] However, in the current study, surface roughness had significantly greater deterioration in repaired composite resin restorations than repaired amalgam restorations at the 12-year observation period. This finding is explained by the different ways that the surface qualities of composites and amalgams might react to oral conditions with time. Whereas biofilm, dietary media, and toothbrushing, for example, are virtually unable to affect the roughness of metal alloys,

composite resins may present surface alteration because of those effects. *In situ* studies[17-20] have shown that composite resin experience structural alteration caused by oral biofilms, with decreased surface hardness and higher roughness when compared with amalgam in particular, mainly due to amalgam's antibacterial effect over dental biofilms.

Interestingly, when comparing the performance of amalgam repair vs positive control, that is, restorations without marginal defects (alpha), the latter group had better behavior in marginal adaptation, secondary caries, and roughness. It is important to note that it is not possible to determine if the failures of the restorations were in the repaired area or the old portion of restoration or even if a restoration that has defects has a tendency to deteriorate again.

As stated in the inclusion criteria, restorations had some marginal/anatomic defects or secondary caries in order to be included in the study. For composite resin restorations, the main cause of inclusion was wide marginal defects, which might be considered material- and technique-dependent clinical issues. In contrast, the main reason for repair in amalgams was secondary caries, which is explained by the fact that the antibacterial potential of corrosion products from amalgam is not fully in place in old restorations. The same initial failure conditions were not observed in the subsequent follow-ups since no differences in the reasons for failure were observed between materials. In contrast, Opdam and others,[21] in a retrospective study using a multivariant Cox regression, found that composite resin restorations showed better performance and better prognosis if caries was the repair indication compared to fracture as the reason for failure.

Despite the low number of Alpha score restorations for marginal adaptation and marginal staining, these parameters apparently have no direct relationship with the incidence of caries, confirming the review of Demarco and others[22] indicating that other factors, such as patient cariogenic risk, operator characteristics, and socioeconomic conditions, are key factors for the longevity of posterior composite restorations. Amalgam restorations presented an increased number of Bravo scores for marginal staining at 12 years, although this clinical feature seems to have no relationship with the development of secondary caries, nor is it a risk factor by itself. This finding has been documented previously in an *in vitro* study.[23]

When observing the behavior of the replacement group, amalgam showed a decrease of several clinical parameters over time, related mainly to marginal stability, unlike the group of composites. Composite restorations have the advantage of being bonded to the tooth by an adhesive interface; on the other hand, anatomy was the parameter that deteriorated statistically over time. This finding could be explained mainly by the mechanical behavior of the restorative material placed in a previously defective zone that probably had mechanical failures by overextension, excessive masticatory forces, or habits that could accelerate the deterioration of the restoration surface; this original situation was not modified.

When the presence of secondary caries, tooth sensitivity, or wide marginal gaps is detected, it makes sense to carry out an intervention urgently; in fact, the criteria used in the current study state that these situations constitute the failure of the restoration. However, not all Charlie scores observed here indicate failures. Some restorations with parameters such as marginal staining, luster, or superficial roughness were detected from the first year of evaluation, and these restorations remained in a functional state without being associated with secondary caries, restoration fracture, or marginal gaps for more than 12 years. Therefore, it seems that these clinical conditions have questionable predictive value in the longevity of the restoration, and are mainly related to the demand for esthetics by patients and dentists.

The sample was made up of restorations of different ages that were equally assigned to study groups, showing similar outcomes and failure rates after 12 years, so this factor apparently does not have a relationship in the decision-making process for treatment of either repair or replacement. One study[24] suggested that patient caries risk, the clinical setting of the study, and the socioeconomic characteristics of patients would be determinants of the reasons for failure when compared to the clinical age of the evaluated restorations.

In the present study, after 12 years for both repaired and replaced restorations, composite resin restorations showed an incidence rate of secondary caries of 5% to 10%, and no new caries lesions were observed on restorations. These results were obtained from a sample of patients of medium and low cariogenic risk determined using Cariogram. Our findings are consistent with the results of the group of low cariogenic risk following 12 years by Opdam and others,[24] where the cariogenic risk was assigned retrospectively using clinical records data on the incidence of new caries in the observation period,

indirectly confirming the congruence between the two estimates of risk. Also, the study of Opdam and others noted that there were differences in results between amalgam and composite resin restorations in patients with a high cariogenic risk.

A limitation of this study could be the current number of restorations in the observation; this situation can overstate findings such as the failure rate of the repair group, where each failed restoration (three failures in amalgam restorations and four failures in composite resin restoration over time) represents a high percentage of failures. However, the replaced restorations have values similar to the 12-year follow-up of restorations by Opdam and others.[24]

Another limitation of this study was the fact that the blinding of the evaluators made it difficult to ensure that the failures or Charlie values corresponded in 100% of the cases to the repaired area or areas belonging to old restorations. Adding to the difficulty was that all restorations show a degree of deterioration over time, that intervening restorations have considerable clinical service, and that, despite this, the replaced restorations were in a similar state as the repaired. Hickel[25] recommended overcoming this problem by using the SQUACE (semiquantitative clinical evaluation) method, which consists of a diagram of the restoration, for taking into consideration the extent of a clinical defect or observation in relation to the entire restoration or to record the exact location of the defect using the FDI World Dental Federation clinical criteria. However, the Ryge/USPHS and FDI World Dental Federation criteria do not consider the evaluation of the restoration–repair interface; this could be an interesting point to analyze because this could be the cause of the Charlie values in parameters such as surface roughness and luster.

Our findings are consistent with previous reports of five-, seven-, and 10-year observation periods,[6-9] showing the same trend that the repair of restorations is as effective as the total restoration replacement, with the advantages of preserving healthy tooth structure, consuming less clinical time, being more tolerated by patients, presenting lower economic costs, and increasing the longevity of existing restorations.

## CONCLUSIONS

Repairing amalgam and composite resin restorations is a treatment as effective as replacing restorations. Considering the many benefits of the repair treatment, this treatment modality should be indicated more often in patients with low to medium cariogenic risk and failures due to secondary caries or marginal defects.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Institutional Research Ethics Committee of the Dental School at the University of Chile. The approval code for this study is Project PRI-ODO-0207/ NCT02043873.

### 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 presented in this article.

*(Accepted 21 February 2017)*

### REFERENCES

1. Mjor IA, Moorhead JE, & Dahl JE. (2000) Reasons for replacement of restorations in permanent teeth in general dental practice *International Dental Journal* **50(6)** 361-366.
2. Kidd EA, Toffenetti F, &Mjor IA (1992) Secondary caries *International Dental Journal* **42(3)** 127-138.
3. Kidd EA, Joyston-Bechal S, & Beighton D (1995) Marginal ditching and staining as a predictor of secondary caries around amalgam restorations: A clinical and microbiological study *Journal of Dental Research* **74(5)** 1206-1211.
4. Kuper NK, Opdam NJ, Ruben JL, de Soet JJ, Cenci MS, Bronkhorst EM, & Huysmans, MC (2014) Gap size and wall lesion development next to composite *Journal of Dental Research* **93(Supplement 7)** 108s-113s.
5. Mua B, Barbachan ESB, Fontanella VR, Giongo FC, & Maltz M (2015) Radiolucent halos beneath composite restorations do not justify restoration replacement *American Journal of Dentistry* **28(4)** 209-213.
6. Martin J, Fernandez E, Estay J, Gordan VV, Mjor IA, & Moncada G (2013) Management of class I and class II amalgam restorations with localized defects: Five-year results *International Journal of Dentistry* **2013** 450260.
7. Gordan VV, Riley JL III, Blaser PK, Mondragon E, Garvan CW, & Mjor IA (2011) Alternative treatments to replacement of defective amalgam restorations: Results of a seven-year clinical study *Journal of the American Dental Association* **142(7)** 842-849.
8. Fernandez E, Martin J, Vildosola P, Oliveira OB Jr, Gordan V, Mjor I, Bersezio C, Estay J, de Andrade MF, & Moncada G (2015) Can repair increase the longevity of composite resins? Results of a 10-year clinical trial *Journal of Dentistry* **43(2)** 279-286.
9. Moncada G, Vildosola P, Fernandez E, Estay J, de Oliveira OB Jr, de Andrade MF, Martin J, Mjor IA, & Gordan VV (2015) Longitudinal results of a 10-year

clinical trial of repair of amalgam restorations *Operative Dentistry* **40(1)** 34-43.

10. Fernandez E, Martin J, Vildosola P, Estay J, de Oliveira O Jr, Gordan V, Mjor I, Gonzalez J, Loguercio A, & Moncada G (2015) Sealing composite with defective margins: Good care or over treatment? Results of a 10-year clinical trial *Operative Dentistry* **40(2)** 144-152.
11. Martin J, Fernandez E, Estay E, Gordan VV, Mjor IA, & Moncada G (2013) Minimal invasive treatment for defective restorations: Five-year results using sealants *Operative Dentistry* **38(2)** 125-133.
12. Sarkis-Onofre S, Cenci MS, Demarco FF, Lynch CD, Fleming PS, Pereira-Cenci T, & Moher D (2015) Use of guidelines to improve the quality and transparency of reporting oral health research *Journal of Dentistry* **43(4)** 397-404.
13. Faul F, Erdfelder E, Lang AG, & Buchner A (2007) G*Power 3: A flexible statistical power analysis program for the social, behavioral, and biomedical sciences *Behavior Research Methods* **39(2)** 175-191.
14. 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.
15. Perdigao J, Reis A, & Loguercio AD (2013) Dentin adhesion and MMPs: A comprehensive review *Journal of Esthetic and Restorative Dentistry* **25(4)** 219-241.
16. Moncada G, Fernandez E, Mena K, Martin J, Vildosola P, De Oliveira OB Jr, Estay J, Mjor IA, & Gordan VV (2015) Seal, replacement or monitoring amalgam restorations with occlusal marginal defects? Results of a 10-year clinical trial *Journal of Dentistry* **43(11)** 1371-1378.
17. Padovani G, Fucio S, Ambrosano G, Sinhoreti M, & Puppin-Rontani R (2014) In situ surface biodegradation of restorative materials *Operative Dentistry* **39(4)** 349-360.
18. Barbosa RP, Pereira-Cenci T, Silva WM, Coelho-de-Souza FH, Demarco FF, & Cenci MS (2012) Effect of cariogenic biofilm challenge on the surface hardness of direct restorative materials in situ *Journal of Dentistry* **40(5)** 359-363.
19. Busscher HJ, Rinastiti M, Siswomihardjo W, & van der Mei HC (2010) Biofilm formation on dental restorative and implant materials *Journal of Dental Research* **89(7)** 657-665.
20. Auschill TM, Arweiler NB, Brecx M, Reich E, Sculean A, & Netuschil L (2002) The effect of dental restorative materials on dental biofilm *European Journal of Oral Science* **110(1)** 48-53.
21. Opdam NJ, Bronkhorst EM, Loomans BA, & Huysmans MC (2012) Longevity of repaired restorations: A practice based study *Journal of Dentistry* **40(10)** 829-835.
22. Demarco FF, Correa MB, Cenci MS, Moraes RR, & Opdam NJ (2012) Longevity of posterior composite restorations: not only a matter of materials *Dental Materials* **28(1)** 87-101.
23. Magalhaes CS, Freitas AB, Moreira AN, & Ferreira EF (2009) Validity of staining and marginal ditching as criteria for diagnosis of secondary caries around occlusal amalgam restorations: An in vitro study *Brazilian Dental Journal* **20(4)** 307-313.
24. Opdam NJ, Bronkhorst EM, Loomans BA, & Huysmans MC (2010) 12-year survival of composite vs. amalgam restorations *Journal of Dental Research* **89(10)** 1063-1067.
25. Hickel R, Peschke A, Tyas M, Mjor I, Bayne S, Peters M, Hiller KA, Randall R, Vanherle G, & Heintze SD (2010) FDI World Dental Federation: Clinical criteria for the evaluation of direct and indirect restorations-update and clinical examples *Clinical Oral Investigations* **14(4)** 349-366.

©Operative Dentistry, 2018, **43-1,** 22-30

# A Clinical Study on the Effect of Injection Sites on Efficacy of Anesthesia and Pulpal Blood Flow in Carious Teeth

QH Zheng • QC Hong • L Zhang • L Ye • DM Huang

### Clinical Relevance

Infiltration anesthesia over the midpoint of the line connecting the root apexes of two adjacent teeth causes less reduction of pulpal blood flow.


## SUMMARY

**This randomized clinical trial evaluated the efficiency of maxillary infiltration anesthesia in carious teeth at two different injection sites and their impact on the laser Doppler recordings of pulpal blood flow (PBF) during a caries excavation procedure. The null hypothesis tested was that there are no differences in the efficiency of anesthesia and PBF reduction between maxillary infiltrations at the two injection sites. One hundred twenty patients were divided into three groups according to the degree of carious lesion of their maxillary left central incisors (moderate caries, deep caries, or no caries). Forty patients in each group randomly received infiltrations over the root apex of maxillary left central incisors (site X) or over the midpoint of the line connecting the root apexes of both maxillary left central and lateral incisors (site Y) using 0.9 mL 2% lidocaine with 1:100,000 adrenaline. Teeth were pulp tested at five-minute intervals after injection except for the period of cavity cutting, which was done 12 minutes after injection. The PBF changes after injection were monitored by laser Doppler flowmetry. The observation period in this study was 60 minutes. Success of anesthesia was defined as no or mild pain on cavity cutting by visual**


Qinghua Zheng, DDS, PhD, State Key Laboratory of Oral Diseases, National Clinical Research Center for Oral Diseases, West China School of Stomatology, Sichuan University, Chengdu, Sichuan Province, China

Qingchun Hong, State Key Laboratory of Oral Diseases, National Clinical Research Center for Oral Diseases, West China School of Stomatology, Sichuan University, Chengdu, Sichuan Province, China

Lan Zhang, State Key Laboratory of Oral Diseases, National Clinical Research Center for Oral Diseases, West China School of Stomatology, Sichuan University, Chengdu, Sichuan Province, China

Ling Ye, State Key Laboratory of Oral Diseases, National Clinical Research Center for Oral Diseases, West China School of Stomatology, Sichuan University, Chengdu, Sichuan Province, China

*Dingming Huang, PhD, State Key Laboratory of Oral Diseases, National Clinical Research Center for Oral Diseases, West China Hospital of Stomatology, Sichuan University, Chengdu, Sichuan Province, China

*Corresponding author: 14# 3rd Section, Renmin South Road, Chengdu 86610041, Sichuan Province, China; e-mail: dingminghuang@163.com

DOI: 10.2341/16-371-C

**analog scale recordings. Deep caries group showed significantly higher baseline PBF ($p<0.05$). All groups showed 100% success of anesthesia and similar duration time ($p>0.05$). Subgroups that had the injection at site Y showed significantly less reduction of PBF ($p<0.05$). Cavity-cutting procedures increased the amplitude of the PBF around the lowest value after injection. Independent of the cavity depth, carious anterior teeth anesthetized by infiltration further from the apex had significantly less reduction on the pulpal blood flow compared with teeth anesthetized by infiltration at the apex.**

## INTRODUCTION

An intact pulpal blood flow (PBF) is critical for maintaining the health of the dental pulp.[1] Because the dental pulp is enclosed in a low-compliance environment surrounded by enamel and dentin and lacks of collateral circulation, it is more vulnerable and does not easily tolerate injury.[1] Under the long-term accumulation of noxious stimuli, even a small rise of pulpal pressure can significantly affect the local circulation.[2,3] The reduction of PBF ensuing from an increase in pulpal pressure fails to provide sufficient oxygen and nutrients for pulpal cells and has the compounding effect of reducing the clearance of large molecular weight toxins or waste products, thus causing irreversible pulpal pathosis.[4,5]

Maxillary infiltration is a common method for pain control in dentistry, which provides effective pulpal anesthesia in more than 60% of the cases.[6-13] Epinephrine added to anesthetics benefits the duration and quality of pulp anesthesia, which mainly depends on its vasoconstrictor properties on blood vessels in close proximity to the injection site.[14] Despite its positive effect on anesthesia, the vasoconstriction effect was considered to have adverse effects on the pulp status of the teeth, particularly if the pulp is inflamed preoperatively. [14] Many studies have assessed the effects of local anesthetics with vasoconstrictors on pulpal circulation.[14-16] They confirmed that epinephrine in local anesthetics does reduce PBF, regardless of whether it is administered by infiltration or nerve block. However, it remains unclear how to minimize the side effect on PBF reduction and simultaneously provide promising effects of anesthesia.

Dental caries is the main cause of irreversible pulpal inflammation. Restorative dentistry plays an important role in active caries control, which itself may cause significant irritation to the dental pulp.[1] Besides, the effects of pulpal insults are considered cumulative. With each succeeding irritation, the pulp has a diminished capacity to remain vital.[1] Apart from the well-known chemical irritants like restorative materials, physical irritations during cavity preparation such as from heat, desiccation, or vibration are reported factors that may adversely affect the dental pulp.[3,17-19] However, most of the studies were conducted on human or experimental animal teeth with normal pulp, which may not reveal the true effects of these procedures on teeth with carious lesions that are already causing inflammation of the pulp. Moreover, reduced PBF and its underlying effect by a local anesthetic with vasoconstrictors were not considered.

Because the pulp tissue cannot be directly inspected, indirect methods like thermal and electric pulp tests were frequently used to evaluate the pulp vitality. The cold test is a more reliable test (sensitivity and specificity) to prove the vitality of the tooth and the success for anesthesia as found by Petersson and others.[20]

It is generally accepted that assessment of the blood supply within the dental pulp (pulp vitality) is the earliest indicator and may be the only available true indicator of the actual state of pulpal health.[21,22] However, thermal and electric pulp tests actually assess the condition of the nerves within the dental pulp rather than the pulp blood supply. Laser Doppler flowmetry (LDF) is reportedly able to assess changes of blood flow within the dental pulp noninvasively.[23] It is considered the most accurate method for diagnosing the state of pulpal health and came closest to serving as a gold standard.[22,24]

To date, no study has assessed the effect of infiltration anesthesia and following cavity cutting on the PBF of carious teeth. The aims of the present study were to (1) evaluate the anesthesia effect at two injection sites for carious maxillary left central incisors using 2% lidocaine with 1:100,000 adrenaline and (2) monitor the dynamic PBF changes induced by the injection and following cavity cutting by LDF. We compared two injection target sites in this study: site X was centered over the root apex of the carious maxillary left central incisors and site Y was over the midpoint of the line connecting the root apexes of both maxillary left central and lateral incisors. The null hypothesis tested was that there are no differences in the efficiency of anesthesia and PBF reduction between maxillary infiltrations at the two injection sites.

## METHODS AND MATERIALS

### Subjects

All patients were informed about the purpose and procedures used in the study, and they gave their written informed consent to the protocol approved by the Sichuan University Committee for Research on Human Subjects (WCHSIRB-D-2015-104R1).

Eighty participants with unrestored carious maxillary left central incisors and 40 participants with healthy maxillary left central incisors were included in this study, for a total of 120 subjects (66 men and 54 women; age, 18 to 39 years). All subjects were randomly selected from the pool of patients referred to the Department of Conservative Dentistry, West China Hospital of Somatology, from November 2015, until the number of subjects achieved the requirement of this study.

Tooth responsiveness to external stimuli was evaluated by conventional electrical and thermal tests. Radiographs were taken to ensure that the pulp chamber was visible and the periapical status of the tooth was normal. The extent of caries was determined by radiographic combined with clinical examination. Criteria for exclusion were as follows: caries lesion offended the cervical area of the tooth crown or caused pulp exposure, gingivitis, or periodontitis; a history of spontaneous pain of teeth; a history of recent trauma or orthodontic treatment; active sites of pathology in the area of injection; allergic to anesthesia; a significant medical history or medication that might influence anesthesia and PBF; a history of smoking or drinking; and being pregnant or breastfeeding.

Participants were divided into three groups according to the degree of caries lesion. The moderate caries group included participants with moderate caries lesions limited to the shallow dentin layer (n=40). The deep caries group was participants with deep caries lesions to the inner dentin layer (n=40). The no caries group was participants with healthy intact maxillary left central incisors (n=40). Each subject was randomly assigned to infiltration at one of the examined sites.

### Laser Doppler Flowmetry

PBF was measured using a LDF (moorVMS-LDF1-HP, Moor, Axminster, England) with light delivered at a wavelength of 785 nm produced by a 2.5-mW laser and a type CP3 probe (Moor) with a cross-sectional diameter of 1.6 mm. Before each data collection session, the LDF was calibrated using a motility standard liquid consisting of a low concentration of polystyrene microspheres in water undergoing thermal motion (Moor Motility Standard, Moor).

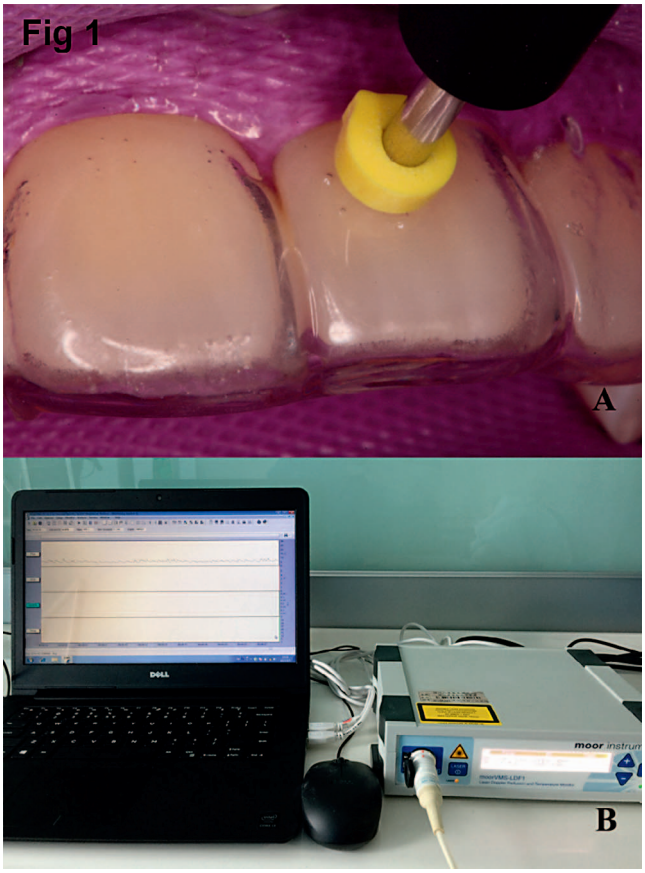



Figure 1. *Photographs showing the recording of laser Doppler. (A): View of the placement of rubber dam, acrylic guard, and application of the LDF tester. (B): Setup for the recording of the laser Doppler.*

Customized acrylic resin splints were used to secure the probe in the appropriate positions. Channels were drilled through the splint to allow for the insertion of the optical probe for pulpal recordings. The probe was placed perpendicular on the labial enamel surface of the tooth crown approximately 3 mm from the gingival margin, over the central long axis of the crown (Fig. 1). An opaque, heavy-gauge rubber dam was placed, and the participant was allowed to rest in a supine position in the dental chair for 15 minutes prior to LDF measurement. Heart rate and blood pressure were taken throughout the measurement sessions. The PBF was expressed in perfusion units (PUs), monitored, and subsequently analyzed using data processing software (Moor VMS-PC) (Figure 1). All measurements were performed by the same operator under standardized environmental conditions at a constant room temperature. The operator only

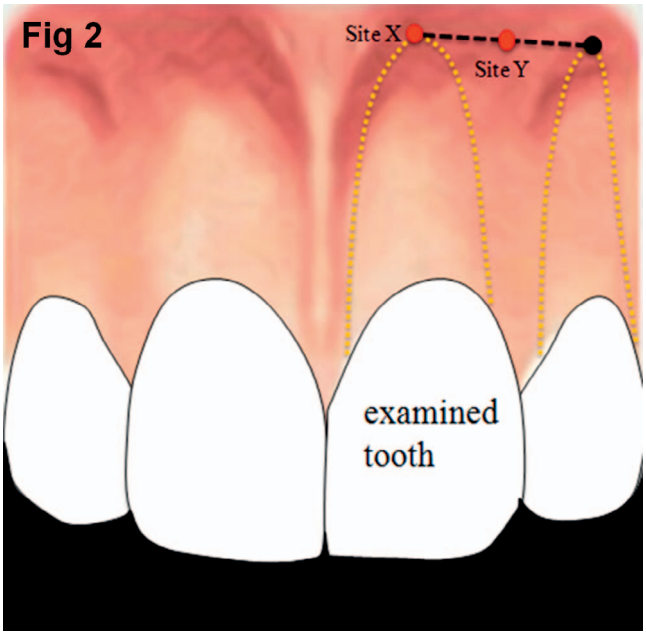



Figure 2. *Schematic diagram of injection sites.*

recorded the PU values and did not know the aim and design of the study.

**Experimental Procedures**

An electric pulp tester (Analytic Technology, Redmond, WA, USA) was used to record pulpal sensitivity of the subjects in each group in machine units before the experiment to determine the baseline response. The contralateral maxillary canine was used as a control to ensure that the pulp tester was operating properly and that the volunteers were responding appropriately during the study. The current rate was set at 25 seconds to increase from no output (0) to the maximum output (80). All the preinjection and postinjection pulp tests were performed by the same trained person who was blinded to the anesthetic target site administered.

After the measurement of the baseline response using the electric pulp tester, the rubber dam and splint were put on, and the LDF measurement was made to obtain the baseline PU value of the subjects. The rubber dam and splint were then removed, and the participants in each group randomly had buccal infiltration at one of the two sites using 0.9 mL 2% lidocaine hydrochloride with 1:100,000 adrenaline. Site X was centered over the root apex of the maxillary left central incisor, the common recommended site (n=20), and site Y was centered over the midpoint of the line connecting the root apexes of both maxillary left central and lateral incisors (n=20) (Figure 2).The randomization process involved selection of one from among 40 sealed envelopes for each group by the operator immediately before the anesthesia, and this revealed to the operator the target sites to inject. The participants did not know the site of injection. All administration was done quantitatively with a 31-gauge needle (CK-JECT, CK Dental Ind Co, Bucheon, Korea) placed in the submucosa over the estimated position by a computer-controlled local anesthetic delivery system at default mode (KM-7500, KMG Co, Busan, Korea). Criterion for pulpal anesthesia was no response from the subject at the maximum output (80 reading) of the pulp tester. The onset of anesthesia was defined as the time from the end of injection to the first of two consecutive 80 readings. The duration of pulpal anesthesia was defined as the time from the onset of pulpal anesthesia to the time recorded before two positive responses to the pulp tester were obtained.[9]

After the administration, PU value was recorded for 12 minutes to evaluate the PBF change during the time from the end of injection to the beginning of cavity cutting. Pulp sensitivity was recorded at five and 10 minutes after injection. If the teeth showed response at the maximum output, one more pulp electrical test was done five minutes later.

Cavity cutting began on the moderate and deep caries groups 12 minutes after injection. Each two minutes, drilling was followed by four minutes recording of the PU value. The drilling session was repeated three times to ensure the complete excavation of carious lesions. All the drillings were done by the same experienced doctor. Teeth in the no caries group remained undrilled. The PBF was then monitored continuously, and pulp sensitivity was recorded at five-minute intervals until the end of the observation time.

Participants were instructed to definitively rate any pain felt during the cavity cutting using the Heft-Parker Visual Analog Scale (VAS)[25] on a 170-mm marked line. The VAS was divided into four categories: no pain (0 mm), mild pain (1-54 mm), moderate pain (55-113 mm), and severe pain (114-170 mm). A designated doctor not involved with the clinical phase of the study collected and analyzed the VAS forms. The success of the maxillary infiltration was defined as the ability to clean the carious lesion of the tooth without pain (VAS score of 0) or with mild pain (VAS score of $\leq$54 mm). After the study ended, the cavities were restored following routine procedures.

Table 1: *Duration of anesthesia and mean pain ratings after cavity cutting in each group (mean±SD)*

| Injection target sites | Participants | | | | | |
|---|---|---|---|---|---|---|
| | Moderate caries group | | Deep caries group | | No caries group | |
| | X | Y | X | Y | X | Y |
| Duration of anesthesia (min) | 53.2±7.4 | 47.9±5.1 | 51.9±6.8 | 46.2±5.7 | 52.6±7.0 | 47.0±5.3 |
| Mean pain ratings | 14.6±6.7 | 15.0±5.9 | 15.1±7.1 | 17.4±7.3 | — | — |

### Statistical Analysis

Assuming a 70% anesthetic success rate and a nondirectional $\alpha$ risk of 0.05, a total sample size of 110 subjects and almost 18 subjects for each of the six subgroups would be required to show a difference in anesthetic success of a 30% decrease with a power of 0.90. The effects of the degree of caries and the injection sites on the results and their interaction were analyzed using analysis of variance for factorial design. Comparisons between and within groups for the duration of anesthesia, baseline PU value, and the percentage of fluctuation amplitude (percentage of fluctuation amplitude of PU value to baseline PU value) during drilling were analyzed using Tukey's test. The data of percentage reduction of the PU value (percentage of maximal reduction of PU value to baseline PU value) within 12 minutes after injection was analyzed using the Games-Howell test. $p<0.05$ was considered significant.

## RESULTS

All the subjects had successful anesthesia. None of the subjects showed response to the electric pulp tester at five minutes after injection. The mean durations of anesthesia are presented in Table 1, and no statistical differences were found between the groups.

The baseline PBF values, the mean percentage reduction of PBF within 12 minutes after injection, and the mean percentage fluctuate amplitude of PBF during cavity cutting in each group are listed in Table 2. The deep caries group has a higher baseline PBF than the other groups ($p<0.05$). The subgroups with injection at site Y showed a lower percentage reduction of PBF than those injected at site X ($p<0.05$). The degree of caries showed no obvious effect on the PBF change and had no interaction with the injection sites ($p>0.05$). None of the subjects returned to baseline PBF by the end of the observation time.

Subjects who had the same treatments showed a similar trend of PBF change within 60 minutes after injection. The decrease in PBF is illustrated in Figure 3. During the first 12 minutes after injection, the reduction of PBF seemed to be more abrupt in the subgroups with injection at site X than at site Y (Figure 4). Obvious fluctuations of PBF were found during cavity cutting in all study groups (Figure 5). The percentage fluctuation amplitude of PBF during the cavity-cutting procedures presented no statistical differences among the groups ($p>0.05$). No obvious fluctuation of PBF was found in the group with healthy maxillary left incisors (no cavity cutting).

## DISCUSSION

We used the 80 reading (maximum output) of the electric pulp test as a criterion for pulpal anesthesia based on the studies of Drevenet and others[26] and Certosimo and Archer.[27] Petersson and others[20] reported that the probability of a sensitive reaction representing a vital pulp was 90% with the cold test using ethyl chloride and 84% with the electrical test. Due to the lack of ethyl chloride in the clinical department during the period of our study, we used the electrical test instead of the cold test.

All the subjects had successful infiltration, regardless of the injection site and degree of caries, which

Table 2: *Baseline PBF, percentage reduction and percentage fluctuation amplitude of PBF in each group (mean±SD)*[a]

| Injection target sites | Participants | | | | | |
|---|---|---|---|---|---|---|
| | Moderate caries group | | Deep caries group | | No caries group | |
| | X | Y | X | Y | X | Y |
| Baseline PBF | 5.79±0.90[b] | 5.75±0.81[b] | 9.91±0.85[a] | 10.09±1.18[a] | 5.98±0.86[b] | 6.02±0.86[b] |
| Maximal reduction of PBF to baseline PBF (%) | 57±6[a] | 39±8[b] | 58±5[a] | 32±10[b] | 57±5[a] | 36±7[b] |
| Fluctuation amplitude of PBF to baseline PBF (%) | 13±3 | 14±2 | 14±1 | 12±2 | — | — |

[a] *In each row, statistical groups are designated by superscript letters (p<0.05).*

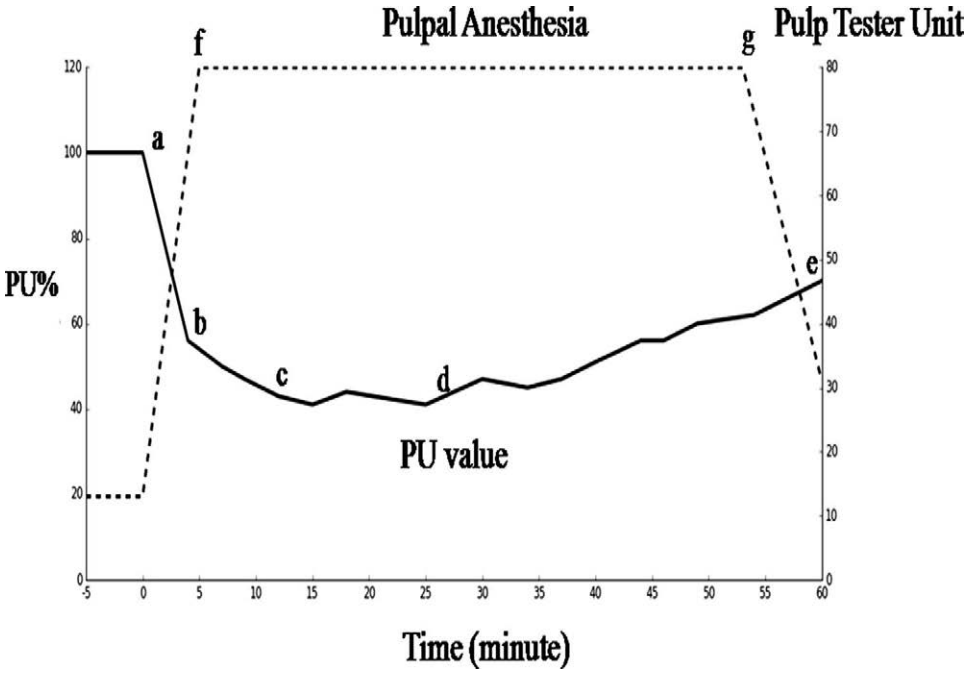



Figure 3. *Dynamic changes of PBF and tooth response to electric pulp tester of one subject's injection at site X in moderate caries group 60 minutes after injection. PU%, the percentage of the real-time PU value to the baseline PU value; a, complement of injection; c, beginning of cavity cutting; ab curve, stage of PBF abruptly reduced after injection; bc curve, phase that PBF slowly declines; d, complement of cavity cutting; e, the percentage PU value at 60 minutes after injection; fg segment, tooth showed no response at 80 reading of the pulp tester.*

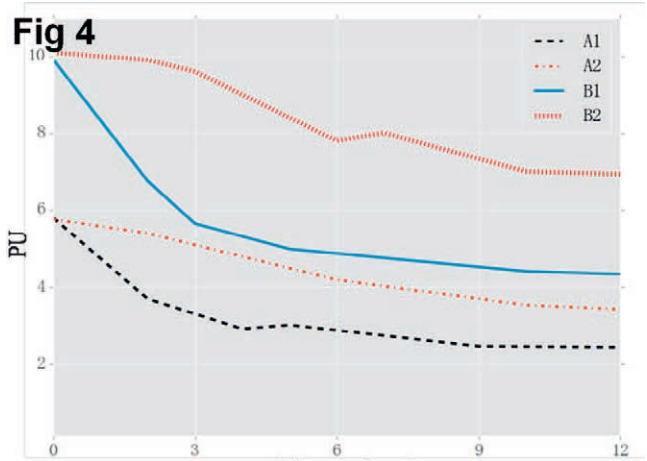
means that no obvious pain was reported during the cavity-cutting procedure. The reported success rate of infiltration anesthesia for healthy maxillary central incisors ranged from 72% to 100%.[12,13,28,29] These variations may be partly due to the different kind and dosage of the anesthetic agents, as well as the population and operator difference.[8]

The information about the onset and duration of infiltration is important for clinic treatment. Kämmerer and others[28] reported that the mean onset of infiltration for maxillary central incisors using 4% articaine with varying dosages of epinephrine was 4.2 to 5.3 minutes. For PBF evaluation, we performed the electric pulp test at five and 10 minutes after the injection instead of evaluating the timely onset of the anesthesia. We found that five minutes was enough for the left maxillary central incisors in all groups to show no response to a reading of 80. It is difficult to compare our study with that of Kämmerer and others[28] because of the different kinds of anesthetics. The present results were close to those found in research on maxillary lateral incisors with a similar study design.[8,9] Whether there are differences in the anesthesia effects between the central and lateral incisors needs further investigation.

The mean duration of anesthesia of each group was greater than 45 minutes but limited to one hour, and no differences were found at the two injection sites. There are controversies regarding the duration of infiltration anesthesia for maxillary central incisors using 2% lidocaine with 1:100,000 epinephrine. Similar to our study, Lawaty and others[29] reported that anesthesia of short duration (less than one hour) occurred in 42% of the subjects. In contrast, Pitt and others[13] reported that the duration of anesthesia exceeded 80 minutes in all of the maxillary central incisors. Adding the dose of anesthetics was considered helpful for prolonging the duration of anesthesia.[7] In our pilot study, anesthesia using 1.8 mL 2% lidocaine with 1:100,000 epinephrine can provide a longer duration of anesthesia. However, considering the possible side effects induced by prolonged vasoconstriction,[2,14] 45 minutes was adequate for short dental operative procedures to minimize injury to the pulp.

The baseline PBF of the subjects in the deep caries group was much higher than the other groups, which indicated that there may be primary pulpal congestion in teeth with deep caries, although these subjects showed normal responses to the thermal and electrical tests. The increase in baseline PBF seemed to have no obvious effect on the success rate, onset, and duration of infiltration for maxillary central incisors in the present study, yet the long-term side effects on these teeth should be monitored.

A significant reduction in PBF occurred after the injection in all subjects, as reported by other studies.[13,14,30,31] Pitt and others[13] reported a prom-



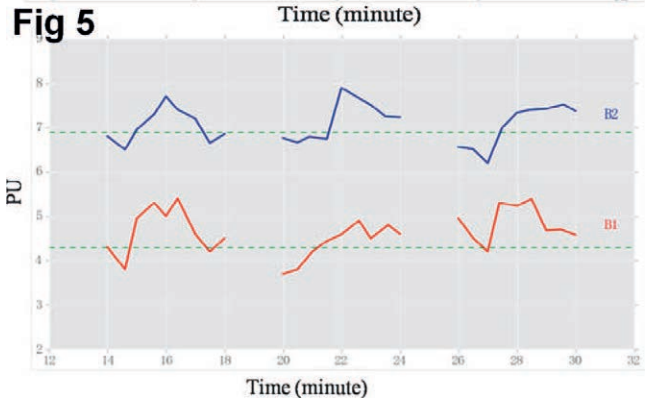




Figure 4. *Curve of the mean PBF change within 12 minutes after injection in four study groups. A1, moderate caries group with injection at site X; A2, moderate caries group with injection at site Y; B1, deep caries group with injection at site X; B2, deep caries group with injection at site Y.*

Figure 5. *An illustration of fluctuation of PU values after each cavity-cutting section of one subject for each subgroup in the deep caries group. The horizontal dotted line represents the mean PU value at the beginning of cavity cutting. B1, deep caries group with injection at site X; B2, deep caries group with injection at site Y.*

inent reduction (31%) of PBF in maxillary central incisors when 1 mL 2% lidocaine with 1:80,000 adrenalin was administered. Ahn and others[14] found a 73% reduction of PBF in the maxillary premolars after infiltration using 2% lidocaine with 1:100,000 epinephrine. The variations in PBF reduction between each study were partly attributed to the tooth position, dosage of vasoconstrictor, and population. Our results showed that reduction of PBF in subgroups injected at site Y was significantly less than those injected at site X. It is difficult to compare this result with other studies because none of the previous studies determined the effect of injection sites. Site X subgroups showed a 3- to 4-minute linear decrease of PBF soon after injection followed by a gradual reduction. Most studies reported that PBF after injection showed a rapid decrease and a gradual return toward the baseline value.[13,31] However, site Y subgroups exhibited a more moderate change of PBF, which was not reported in previous studies. The results mentioned above suggest that site Y may induce less effect on the PBF of the ill tooth, which may benefit pulp health. Despite the reduction speed of PBF, the PBF became comparatively stable at the time the cavity cutting procedure began in this study.

Cavity cutting was considered to have adverse effects on the PBF of the teeth.[32-34] In the present study, the PBF of the subjects experiencing cavity cutting exhibited prominent fluctuations after every two minutes of drilling, whereas the teeth without drilling showed no obvious variation in the amplitude of PBF. No statistically relevant differences were found in the percentage of fluctuation amplitude of PBF to baseline PBF between each study group, which suggests that cavity-cutting procedures may independently affect the PBF. Whether the accumulation effect of the reduction of PBF after anesthesia and the repeated fluctuation in PBF after dentin cutting found in our study has short- or long-term effects on the pulp status of teeth still needs further investigation.

By the end of the observation period in this study, none of the teeth returned to its baseline PBF, which was similar to the results reported by Ahn and others[14] and Pitt and others.[13] Although blood flow measurements were not continued past 60 minutes, a rebound hyperemic effect may be expected.

Assessments of PBF using laser Doppler may be highly susceptible to environmental and technique-related factors, including characteristics of the laser beam, probe position, tooth type, and nonpulpal signals obtained from nearby tissue.[24,35] In this study, the combination of the splint and rubber dam ensures the greatest possible accurate repositioning of the probe and isolation of the periodontal signal. Besides, maxillary central incisors were reported as having no significant interindividual differences on PBF characteristics[36]; the interindividual controls in our study leads to a convincing result.

## CONCLUSION

With the limitations of this present study, we concluded that, independent of cavity depth, carious anterior teeth anesthetized by infiltration further from the apex had significantly less reduction on the pulpal blood flow compared with teeth anesthetized by infiltration at the apex, which may be beneficial for the prognosis of teeth with suspicious pulp congestion.

**Acknowledgments**

This study was supported by the National Natural Science Foundation of China (11402154) and Innovation Team of Sichuan Province (2015TD0011).

**Regulatory Statement**

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Sichuan University Committee for Research on Human Subjects. The approval code for this study is WCHSIRB-D-2015-104R1.

**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 7 March 2017)*

**REFERENCES**

1. Fouad AF LL (2016) Pulpal reaction to caries and dental procedures In: Cohen SBR (ed) *Pathways of The Pulp* **11th edition** Elsevier Inc, St. Louis, Mo, 573-598.
2. Kim S (1985) Regulation of pulpal blood flow *Journal of Dental Research* **64(Special Number 3)** 590-596.
3. Kim S (1990) Neurovascular interactions in the dental pulp in health and inflammation *Journal of Endodontics* **16(2)** 48-53.
4. Pashley DH (1979) The influence of dentin permeability and pulpal blood flow on pulpal solute concentrations *Journal of Endodontics* **5(12)** 355-361.
5. Caviedes-Bucheli J, Munoz HR, Azuero-Holguin MM, & Ulate E (2008) Neuropeptides in dental pulp: The silent protagonists *Journal of Endodontics* **34(7)** 773-788.
6. Gross R, McCartney M, Reader A, & Beck M (2007) A prospective, randomized, double-blind comparison of bupivacaine and lidocaine for maxillary infiltrations *Journal of Endodontics* **33(9)** 1021-1024.
7. Mikesell A, Drum M, Reader A, & Beck M (2008) Anesthetic efficacy of 1.8 mL and 3.6 mL of 2% lidocaine with 1:100,000 epinephrine for maxillary infiltrations *Journal of Endodontics* **34(2)** 121-125.
8. Evans G, Nusstein J, Drum M, Reader A, & Beck M (2008) A prospective, randomized, double-blind comparison of articaine and lidocaine for maxillary infiltrations *Journal of Endodontics* **34(4)** 389-393.
9. Mason R, Drum M, Reader A, Nusstein J, & Beck M (2009) A prospective, randomized, double-blind comparison of 2% lidocaine with 1:100,000 and 1:50,000 epinephrine and 3% mepivacaine for maxillary infiltrations *Journal of Endodontics* **35(9)** 1173-117.
10. Katz S, Drum M, Reader A, Nusstein J, & Beck M (2010) A prospective, randomized, double-blind comparison of 2% lidocaine with 1:100,000 epinephrine, 4% prilocaine with 1:200,000 epinephrine, and 4% prilocaine for maxillary infiltrations *Anesthesia Progress* **57(2)** 45-51.
11. Younkin K, Reader A, Drum M, Nusstein J, & Beck M (2014) Anesthetic efficacy of a combination of 0.5 M mannitol plus 36.8 mg of lidocaine with 18.4 mug epinephrine in maxillary infiltration: a prospective, randomized, single-blind study *Anesthesia Progress* **61(2)** 63-68.
12. Parirokh M, Sadeghi AS, Nakhaee N, Pardakhty A, Abbott PV, & Yosefi MH (2012) Effect of topical anesthesia on pain during infiltration injection and success of anesthesia for maxillary central incisors *Journal of Endodontics* **38(12)** 1553-1556.
13. Pitt Ford TR (1993) Action of adrenaline on the effect of dental local anaesthetic solutions *Endodontics & Dental Traumatology* **9(1)** 31-35.
14. Ahn J, & Pogrel MA (1998) The effects of 2% lidocaine with 1:100,000 epinephrine on pulpal and gingival blood flow *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics* **85(2)** 197-202.
15. Chng HS, Pitt Ford TR, & McDonald F (1996) Effects of prilocaine local anaesthetic solutions on pulpal blood flow in maxillary canines *Endodontics & Dental Traumatology* **12(2)** 89-95.
16. Kim S, Edwall L, Trowbridge H, & Chien S 1984 Effects of local anesthetics on pulpal blood flow in dogs *Journal of Dental Research* **63(5)** 650-652.
17. Cox CF, White KC, Ramus DL, Farmer JB, & Snuggs HM (1992) Reparative dentin: Factors affecting its deposition *Quintessence International* **23(4)** 257-270.
18. Byers MR, Narhi MV, & Mecifi KB (1988) Acute and chronic reactions of dental sensory nerve fibers to cavities and desiccation in rat molars. *Anatomical Record* **221(4)** 872-883.
19. Nyborg H, & Brannstrom M (1968) Pulp reaction to heat. *Journal of Prosthetic Dentistry* **19(6)** 605-612.
20. Petersson K, Söderström C, Kiani-Anaraki M, & Lévy G (1999) Evaluation of the ability of thermal and electrical tests to register pulp vitality *Endodontics & Dental Traumatology* **15(3)** 127-131.
21. Baumgardner KR, Walton RE, Osborne JW, & Born JL (1996) Induced hypoxia in rat pulp and periapex demonstrated by 3H-misonidazole retention *Journal of Dental Research* **75(10)** 1753-1760.
22. Alghaithy RA, & Qualtrough AJ (2017) Pulp sensibility and vitality tests for diagnosing pulpal health in permanent teeth: A critical review *International Endodontic Journal* **50(2)** 135-142.
23. Gazelius B, Olgart L, & Edwall B (1988) Restored vitality in luxated teeth assessed by laser Doppler flowmeter *Endodontics & Dental Traumatology* **4(6)** 265-268.
24. Jafarzadeh H (2009) Laser Doppler flowmetry in endodontics: A review *International Endodontic Journal* **42(6)** 476-490.
25. Heft MW, Parker SR (1984) An experimental basis for revising the graphic rating scale for pain *Pain* **19(2)** 153-161.
26. Dreven LJ, Reader A, Beck M, Meyers WJ, & Weaver J (1987) An evaluation of an electric pulp tester as a

measure of analgesia in human vital teeth. *Journal of Endodontics* **13(5)** 233-238.

27. Certosimo AJ, & Archer RD (1996) A clinical evaluation of the electric pulp tester as an indicator of local anesthesia *Operative Dentistry* **21(1)** 25-30.
28. Kammerer PW, Seeling J, Alshihri A, & Daublander M (2014) Comparative clinical evaluation of different epinephrine concentrations in 4% articaine for dental local infiltration anesthesia *Clinical Oral Investigations* **18(2)** 415-421.
29. Lawaty I, Drum M, Reader A, & Nusstein J (2010) A prospective, randomized, double-blind comparison of 2% mepivacaine with 1:20,000 levonordefrin versus 2% lidocaine with 1:100,000 epinephrine for maxillary infiltrations *Anesthesia Progress* **57(4)** 139-144.
30. Hashimoto S, Yamashiro M, Fujita K, Yasuda A, & Sunada K (2014) Effects of epinephrine on lidocaine pharmacokinetics and blood volume in the dental pulp *Journal of Endodontics* **40(9)** 1370-1374.
31. Yoon MJ, Lee SJ, Kim E, & Park SH (2012) Doppler ultrasound to detect pulpal blood flow changes during local anaesthesia. *International Endodontic Journal* **45(1)** 83-87.
32. Ottl P, & Lauer HC (1998) Temperature response in the pulpal chamber during ultrahigh-speed tooth preparation with diamond burs of different grit *The Journal of Prosthetic Dentistry* **80(1)** 12-19.
33. Ohmoto K, Taira M, Shintani H, & Yamaki M (1994) Studies on dental high-speed cutting with carbide burs used on bovine dentin. *Journal of Prosthetic Dentistry* **71(3)** 319-323.
34. Zach L (1972) Pulp lability and repair: Effect of restorative procedures *Oral Surgery, Oral Medicine, and Oral Pathology* **33(1)** 111-121.
35. Akpinar KE, Er K, Polat S, & Polat NT (2004) Effect of gingiva on laser Doppler pulpal blood flow measurements *Journal of Endodontics* **30(3)** 138-140.
36. Norer B, Kranewitter R, & Emshoff R (1999) Pulpal blood-flow characteristics of maxillary tooth morphotypes as assessed with laser Doppler flowmetry *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontics* **87(1)** 88-92.

©Operative Dentistry, 2018, **43-1,** 31-37

# Effectiveness of LED/Laser Irradiation on In-Office Dental Bleaching after Three Years

RFL Mondelli • FAP Rizzante • ER Rosa • AFS Borges • AY Furuse • JFS Bombonatti

### Clinical Relevance

Use of a hybrid light (laser/LED) for in-office dental bleaching shows the same degree of color change with lower bleaching time and sensitivity compared with conventional in-office bleaching.


## SUMMARY

**The present *in vivo* randomized, triple-blinded, and split-mouth clinical study evaluated the effectiveness of a hybrid light (HL) source on the color change, stability, and tooth sensitivity in patients submitted to different in-office bleaching techniques. Twenty volunteers were divided into two groups and four subgroups. A split-mouth design was conducted to compare two in-office bleaching techniques (with and without light activation): 35% Lase Peroxide Sensy (LPS) + HL: 35% hydrogen peroxide (HP) + HL; 35% LPS: 35% HP; 25% LPS + HL: 25% HP + HL; and 35% Whiteness HP (WHP): 35% HP. For the groups activated with HL, the HP was applied on the enamel surface three consecutive times using a 3 × 2-minute protocol (three HL activations for two minutes each, with a 30-second interval for a total of seven minutes and 30 seconds) for each gel application, totaling 22 minutes and 30 seconds. For the other groups, HP was applied 3 × 15 minutes, totaling 45 minutes. A spectrophotometer was used to measure the color change (ΔE) before the treatment and 24 hours, one week, and one, 12, and 36 months after. A visual analog scale was used to evaluate the tooth sensitivity before the treatment, immediately following treatment, 24 hours, and one week after. Anal-**



Rafael Francisco Lia Mondelli, DDS, MSc, PhD, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Sao Paulo, Brazil

*Fabio Antonio Piola Rizzante, DDS, MSc, PhD candidate, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Sao Paulo, Brazil

Enio Ratton Rosa, DDS, MSc, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Sao Paulo, Brazil

Ana Flavia Sanches Borges, DDS, MSc, PhD, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Sao Paulo, Brazil

Adilson Yoshio Furuse, DDS, MSc, PhD, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Sao Paulo, Brazil

Juliana Fraga Soares Bombonatti, DDS, MSc, PhD, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Sao Paulo, Brazil

*Corresponding author: Alameda Dr. Octávio Pinheiro Brisola, 9-75, CEP 17012-901, Brazil; e-mail: fabioantonio_7@hotmail.com


DOI: 10.2341/16-208-C

**ysis of variance, Tukey's, Kruskall-Wallis, and Wilcoxon tests, all with $\alpha = 0.05$ were performed. Statistical analysis did not reveal any significant differences ($\Delta E$) between the in-office bleaching techniques with or without HL in the periods evaluated; the activation with HL required 50% less time to achieve such results. The groups without HL presented statistical differences for $\Delta E$ when comparing 24 hours with the other follow-up times (inter-group) and an increase in tooth sensitivity in the initial periods. All techniques and bleaching agents were effective on bleaching during a 36-month evaluation of color stability. The groups activated with HL presented lower sensitivity and required a lower activation time.**

## INTRODUCTION

Tooth whitening is one of the most conservative dental treatments that can improve or enhance the smile and has gained popularity in basic oral care. Currently, tooth bleaching has been recognized as an effective and safe method to treat discolored teeth.[1-3]

Although at-home bleaching has increased dramatically in popularity, in-office bleaching products are still in demand for several reasons. Some patients do not adapt well to an at-home protocol due to the treatment time and because of the bleaching tray.[4] Another contra-indication for home bleaching consists of patients presenting sensitivity who need to be closely monitored for extensive tissue recession or deep, unrestored abfraction lesions.[5]

The use of hydrogen peroxide (HP) in high concentrations (35% and 38%) applied by a dental professional allows the patient to obtain visible results even after only one clinical session.[6] Power bleaching reduces the total in-office time by catalyzing the bleaching agent with light sources, such as lasers, light-emitting diodes (LEDs), or plasma arc light. The theoretical benefit lies in the light sources' ability to heat the HP, increasing the rate of decomposition of oxygen to form oxygen-free radicals enhancing the release of pigment-containing compounds.[6-8]

The activation of a bleaching agent by the thermocatalytic technique has been questioned due to its deleterious effects on the tooth structure.[6] Tooth sensitivity is one of the most common side effects of a bleaching treatment and its occurrence is directly dependent on the bleaching agent concentration, the application time, and the thickness of the dentin. Therefore, high-concentration agents used in in-office procedures usually generate discomfort. Tooth sensitivity can persist for up to 10 days after the conclusion of the bleaching treatment.[9-12]

Other effects such as small alterations in the enamel structure after bleaching treatments, such as surface roughness, porosity, microhardness, and ion release have also raised concerns from a few researchers.[13] These alterations can be easily reversed by polishing, remineralization of the enamel by contact with the calcium and phosphate present in the saliva and/or by the application of fluoride.[6,13]

Considering all these factors, the practitioner is faced with many treatment options. Therefore, the objective of the present *in vivo* study was to compare different in-office bleaching techniques using two concentrations of HP (25 and 35%) and the activation or not with a hybrid light (HL) source (LED/laser).

The null hypothesis was that there should not be any differences in relation to the degree of color change, sensitivity and color stability between bleaching protocols.

## METHODS AND MATERIALS

### Study Design and Patient Recruitment

The present interventional, triple-blinded (patient, outcomes measurements operator, and statistician), split-mouth, randomized clinical trial had as study factors the bleaching protocols in four levels (35% HP, used with HL or without HL, 25% HP with HL, and 35% HP, used without HL), assessed through the evaluation of the degree of color change and stability, as well as through postoperative sensitivity by a visual analog scale (VAS). For treatment comparisons, an $\alpha = 0.05$ and test power = 80% were set. Also considering a 30% estimate dropout rate after 36 months, a minimum of 10 patients were needed for each group.

After approval by the local Research Ethics Committee, 20 patients from a total of 38, aged 18-30 years, were selected based on the inclusion and exclusion criteria (Table 1). All patients signed the informed consent after an explanation of the nature and possible risks of their voluntary participation.

The patients were randomly divided into two groups (n=10), and all bleaching gels were used according to the manufacturer's instructions (Table 2). Following a split-mouth design, each group of patients was submitted to two bleaching treatments with a one-week interval: one on the right maxillary

Table 1: *Inclusion and exclusion criteria*

| Inclusion criteria | Exclusion criteria |
|---|---|
| Sign the consent form | Presence of restorations or decay in the anterior teeth |
| Agree to return to scheduled follow-up sessions | Gingivitis or periodontitis |
| Good general health | Tobacco use |
| A3 or darker shade in at least four teeth | Allergic to peroxides |
| Tooth sensitivity lower than 2 on the VAS scale | History of oropharyngeal neoplasms |
| | Use of bleaching agents within one year |
| | Pregnant or lactating woman |
| | Tetracycline stained teeth |

and mandibular arches and the other on the left maxillary and mandibular arches (35% Lase Peroxide Sensy [LPS] + HL, 35% LPS or 25% LPS + HL, and 35% Whiteness HP [WHP]). All the random sequences were generated by an assistant through an Excel worksheet (Microsoft, Redmond, WA, USA), which was written on a paper for each and stored in a sealed envelope until the treatment sessions.

### Bleaching Procedures

All teeth were cleaned with a rubber cup at low speed using fine pumice powder and water. Next, the soft tissue was protected with a gum barrier (Lase Protect, DMC Equipamentos Ltda, São Carlos, SP, Brazil) and light-cured for 30 seconds with a 1200-mW/cm$^2$ LED lighting device (Radii-cal, SDI, Victoria, Bayswater, Australia).

In the 35% LPS group, the patients had their hemi-left or hemi-right upper and lower arches bleached with 35% HP (Lase Peroxide Sensy, DMC Equipamentos Ltda), without HL activation. In the 35% LPS + HL group, the patients had their contralateral upper and lower arches bleached with 35% HP and activated with HL.

For the present study, the HL was composed of six blue LEDs (470 nm and 350 mW/cm$^2$ each) and three infrared therapeutic diode lasers (810 nm and 200 mW/cm$^2$; Whitening Lase II, DMC Equipamentos Ltda).

In the 35% WHP group, the hemi-left or hemi-right upper and lower arches were bleached with 35% HP (Whiteness HP, FGM Produtos Odontológicos, Joinville, SC, Brazil), without HL activation. In the 25% LPS + HL group, the contralateral upper and lower arches were bleached with 25% HP (Lase Peroxide Sensy II, DMC Equipamentos Ltda) and activated with HL. For the groups without HL activation, HP was applied on the enamel surface for 3 × 15 minutes, totaling 45 minutes. For the other groups, HP was applied on the enamel surface three consecutive times with HL activation following a 3 × 2-minute protocol (three activations of HL for two minutes each, with a 30-second interval, for a total of 7 minutes and 30 seconds) for each gel application, totaling 22 minutes and 30 seconds.

Immediately after each bleaching session, all groups were polished with impregnated polishing felt discs (Lase Peroxide, DMC Equipamentos Ltda) to reestablish the enamel smoothness. After polishing, a desensitizing gel (Lase Sensy, DMC Equipamentos Ltda) composed of 2% sodium fluoride and 5% potassium nitrate was applied for four minutes. To follow and standardize the manufacturer's recommendation for avoiding and controlling postoperative sensitivity, the groups activated with HL were laser irradiated (25 J for 30 seconds).

All patients were instructed to avoid any staining substances in the first 48 hours following the treatment, such as coffee, black tea, cola, mustard, ketchup, red wine, soy sauce, chocolate, red lipstick, consumption of tobacco products, as well as food and acidic beverages.

### Instrumental Method for Color Measurement

A contact-type intraoral spectrophotometer (Vita Easyshade, Vita-Zahnfabrik, Bad Säckingen, Germany) was used for the color assessment during the different

Table 2: *Groups and treatment descriptions*

| Groups | Bleaching gels | Commercial brands | Manufacturer | Modality |
|---|---|---|---|---|
| 25% LPS + HL | HP (25%) | Lase Peroxide Sensy II (25% HP) | DMC Equipamentos Ltda., São Carlos, SP, Brazil | In office with chemical and/or physical activation |
| 35% LPS and 35% LPS + HL | HP (35%) | Lase Peroxide Sensy (35% HP) | DMC Equipamentos Ltda., São Carlos, SP, Brazil | In office with chemical and/or physical activation |
| 35% WHP | HP (35%) (35% WHP) | Whiteness HP (35% HP) | FGM Produtos Odontológicos Ltda., Joinville, SC, Brazil | In office with chemical and/or physical activation |

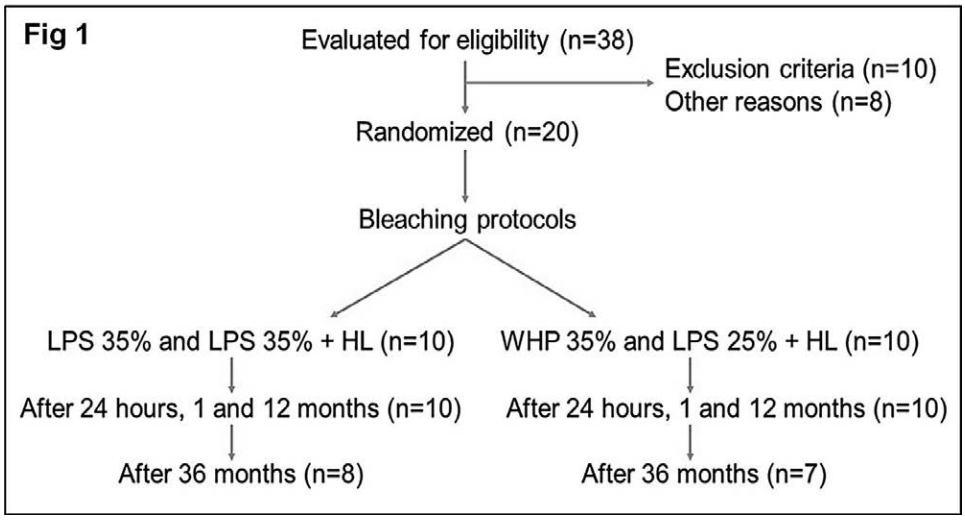



Figure 1. *Study flowchart.*

evaluation times (baseline and after 24 hours, one week, and one, 12, and 36 months). The color assessment was based on the CIELAB system, and color differences were calculated using the following equation: $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.[14] The measurements were performed by a blinded operator. First, the spectrophotometer was calibrated, and after that, the measurements were taken from the median third of each tooth, two times consecutively.[6]

### Tooth Sensitivity Assessment

Each patient was submitted to two different bleaching protocols with a one-week interval. The VAS questionnaire was used to measure the initial tooth sensitivity (baseline), immediately after bleaching, and 24 hours and one week after. The patients had to indicate any tooth or oral sensitivity by marking the level of sensitivity on a horizontal line, which ranged from 0 to 10.

The range of sensitivity scores used were as follows: 0-1, no pain; 2-3, mild pain; 4-6, moderate pain; 7-8, severe pain; 9-10, intolerable pain.

### Statistical Analysis

For the color change (ΔE) analysis, all groups were submitted to two-way analysis of variance (ANOVA) (bleaching agents and HL activation) and the Tukey's test to identify and individually compare the groups. One-way ANOVA was used for intragroup comparison (ΔE over time).

The differences in degree of sensitivity were determined using Kruskal-Wallis and Wilcoxon tests for individual comparisons, both with a 5% significance level.

## RESULTS

In the present study, 20 volunteers were chosen in accordance with the inclusion and exclusion criteria to participate in the clinical research, and a decrease in participation was observed from the initial to the 36-month period (Figure 1).

The intragroup analysis, for ΔE, showed statistically significant differences only for the groups without HL activation ($p \leq 0.05$). The differences were observed between the 24-hour and the other (one-, 12-, and 36-months) evaluations. The comparison between the groups (intergroups) for all periods evaluated did not present any statistically significant difference during the 36 months evaluated (Table 3).

Considering the evaluation of the sensitivity (VAS), a lower degree of sensitivity in the groups in which the HL was used (35% LPS + HL and 25% LPS + HL) in the period immediately after bleaching ($p < 0.034$) was observed. The sensitivity decreased after 24 hours for all groups, without statistical differences between groups after 24 hours and one week (Figure 2).

## DISCUSSION

The purpose of the present *in vivo* study was to compare the effectiveness and stability of in-office bleaching techniques, with or without HL activation, regarding degree of color change (ΔE) up to 36 months and tooth sensitivity up to one week after bleaching.

The null hypothesis was partially verified because the degree of color change in the different assessed times was similar for all groups. Nevertheless, the

Table 3: *Mean, SD, and statistical analysis of ΔE for bleaching groups evaluated during 36 months*[a]

| Groups | 24 Hours (mean ± SD) | One Month (mean ± SD) | 12 Months (mean ± SD) | 36 Months (mean ± SD) |
|---|---|---|---|---|
| 35% LPS (3 × 15 minutes: 45 minutes) | 4.64 ± 1.21 Aa | 6.78 ± 2.11 Ab | 6.61 ± 2.05 Ab | 6.03 ± 1.88 Ab |
| 35% LPS + HL (3 × 7 minutes, 30 seconds: 22 minutes, 30 seconds) | 4.88 ± 1.06 Aa | 5.43 ± 1.09 Aa | 5.45 ± 1.35 Aa | 5.49 ± 1.40 Aa |
| 35% WHP (3 × 15 minutes: 45 minutes) | 4.59 ± 1.04 Aa | 6.51 ± 1.79 Ab | 6.56 ± 1.33 Ab | 6.15 ± 1.97 Ab |
| 25% LPS +HL (3 × 7 minutes, 30 seconds: 22 minutes, 30 seconds) | 4.70 ± 1.38 Aa | 5.10 ± 1.04 Aa | 5.07 ± 1.46 Aa | 5.28 ± 1.06 Aa |

[a] *Lowercase letters show analysis between columns in the same line; capital letters show analysis between lines in the same column.*

groups with HL produced lower sensitivity in the first days compared with the groups without HL.

Two different HP gel concentrations (25% and 35%) were used for the chemical and physical activations. In the groups with physical activation (HL), the bleaching gel time was 22 minutes and 30 seconds, which corresponds to half of the gel application time for groups without light activation (45 minutes). This is the main advantage of using a light source to activate the bleaching gel, allowing lower working time for each bleaching appointment, resulting in lower cost and more convenience for both the patient and professional.[6]

Tooth sensitivity is caused by the passage of HP molecules through the enamel and dentin into the pulp chamber. Therefore, tooth sensitivity may vary with the different factors that affect this passage, such as presence of dental cracks, dentin exposure, and/or pulp chamber dimensions.[5,12] Nevertheless, tooth sensitivity is a temporary side effect that disappears after four days of treatment in most patients, but in some cases can persist for up to 10 days.[6,9-12,15]

The results of sensitivity in the present study indicated that the use of hybrid light activation, allowing a 50% lower bleaching time, promoted lower sensitivity for the patients immediately after the bleaching procedure (Figure 2). The lower gel application time in the groups with HL, associated with the laser therapy during and right after the bleaching procedure, may have contributed to the lower postoperative sensitivity observed. These results are in accordance with Bartolotto and others.[16]

Some studies reported no difference between degree of tooth sensitivity using both LED and HL activation devices, with a greater percentage of patients reporting pain within 12 hours after bleaching.[12,15] The results of the present clinical trial disagree with these results, and this may be due to the higher gel application time in the above related articles (30 minutes per session, about 50% higher than that used in the present study).

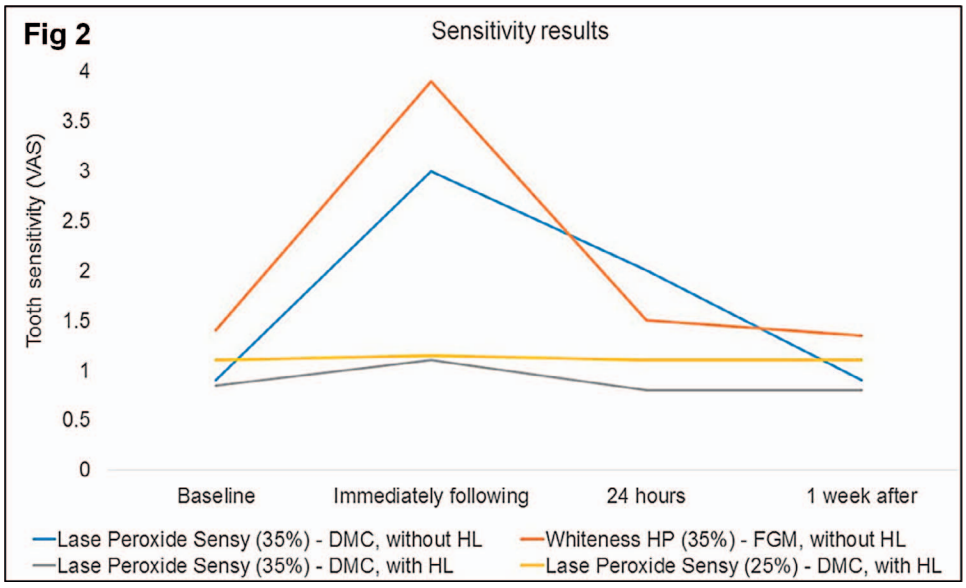



Figure 2. *Mean sensitivity values (VAS) of the groups evaluated for the different periods.*

Nevertheless, the pain was transitory, and the values of color change were similar among all protocols.

The use of CIELAB-based spectrophotometers is the most effective method used for assessment of the color and color changes over time because it is more objective and accurate than using a visual scale and photographs.[6,7,10,11,17]

The results presented in the present three-year study justified the use of HL for similar degrees of color change (ΔE) and stability with in-office bleaching compared with groups without light irradiation, independent of the bleach gel concentration (25% and 35%). Despite this, some researchers present conflicting results regarding the effectiveness of the use of light activation with in-office bleaching.[6,9,18-21] The differences between the light devices, such as the lamp type, wavelength, irradiation, tip design, time used for gel irradiation, and HP gel concentration are some of the reasons for the conflicting results presented in the literature.

All bleaching protocols were similar considering all the evaluation periods. Despite this, the groups with HL produced the same color change results, both immediately after bleaching and over time, but with 50% less bleaching time (22 minutes and 30 seconds versus 45 minutes; Table 3). These results are in agreement with Mondelli and others, who used the same HL device.[6]

The use of HL allows a greater number of gel changes (four to five gel applications) during an appointment and can promote the desired results in a single session.[6] Some clinical studies used the same HL source but left the bleaching gel on the tooth surface for as long as 15 minutes per gel application, increasing the clinical time and tooth sensitivity.[9]

The present study results showed great color change stability with a single bleaching session. The use of the HL should be further explored, using different gels concentrations (such as 10% and 15%) and application times. Such protocols could reduce the risks of tooth sensitivity while promoting the same bleaching effect and could be used in younger patients.

## CONCLUSION

The use of HL produced the same bleaching results as the other protocols without HL, immediately and after 36 months, with 50% gel application time and lower immediate postoperative sensitivity.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Research Ethic Committee of the Bauru School of Dentistry, University of São Paulo. The approval code for this study is 105/2008.

### 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 20 March 2017)*

## REFERENCES

1. Pale M, Mayoral JR, Llopis J, Valles M, Basilio J, & Roig M (2014) Evaluation of the effectiveness of an in-office bleaching system and the effect of potassium nitrate as a desensitizing agent *Odontology* **102(2)** 203-210.
2. Wang Y, Gao J, Jiang T, Liang S, Zhou Y, & Matis BA (2015) Evaluation of the efficacy of potassium nitrate and sodium fluoride as desensitizing agents during tooth bleaching treatment: A systematic review and meta-analysis *Journal of Dentistry* **43(8)** 913-923.
3. Kishi A, Otsuki M, Sadr A, Ikeda M, & Tagami J (2011) Effect of light units on tooth bleaching with visible-light activating titanium dioxide photocatalyst *Dental Materials Journal* **30(5)** 723-729.
4. Machado LS, Anchieta RB, Dos Santos PH, Briso AL, Tovar N, Janal MN, Coelho PG, & Sundfeld RH (2016) Clinical comparison of at-home and in-office dental bleaching procedures: A randomized trial of a split-mouth design *International Journal of Periodontics & Restorative Dentistry* **36(2)** 251-260.
5. Briso AL, Lima AP, Goncalves RS, Gallinari MO, & dos Santos PH (2014) Transenamel and transdentinal penetration of hydrogen peroxide applied to cracked or microabrasioned enamel *Operative Dentistry* **39(2)** 166-173.
6. Mondelli RFL, Azevedo JF, Francisconi AC, Almeida CM, & Ishikiriama SK (2012) Comparative clinical study of the effectiveness of different dental bleaching methods: Two year follow-up *Journal of Applied Oral Science* **20(4)** 435-443.
7. Travassos AC, Rocha Gomes Torres C, Borges AB, & Barcellos DC (2010) In vitro assessment of chemical activation efficiency during in-office dental bleaching *Operative Dentistry* **35(3)** 287-294.
8. Mena-Serrano AP, Parreiras SO, do Nascimento EM, Borges CP, Berger SB, Loguercio AD, & Reis A (2015) Effects of the concentration and composition of in-office bleaching gels on hydrogen peroxide penetration into the pulp chamber *Operative Dentistry* **40(2)** E76-E82.
9. Kossatz S, Dalanhol AP, Cunha T, Loguercio A, & Reis A (2011) Effect of light activation on tooth sensitivity after in-office bleaching *Operative Dentistry* **36(3)** 251-257.
10. Reis A, Tay LY, Herrera DR, Kossatz S, & Loguercio AD (2011) Clinical effects of prolonged application time of an in-office bleaching gel *Operative Dentistry* **36(6)** 590-596.

11. Reis A, Kossatz S, Martins GC, & Loguercio AD (2013) Efficacy of and effect on tooth sensitivity of in-office bleaching gel concentrations: a randomized clinical trial *Operative Dentistry* **38(4)** 386-393.
12. Moncada G, Sepulveda D, Elphick K, Contente M, Estay J, Bahamondes V, Fernandez E, Oliveira OB, & Martin J (2013) Effects of light activation, agent concentration, and tooth thickness on dental sensitivity after bleaching *Operative Dentistry* **38(5)** 467-476.
13. Lia Mondelli RF, Garrido Gabriel TR, Piola Rizzante FA, Magalhaes AC, Soares Bombonatti JF, & Ishikiriama SK (2015) Do different bleaching protocols affect the enamel microhardness? *European Journal of Dentistry* **9(1)** 25-30.
14. Commission Internationale de L'Eclairage (1978) Recommendations on uniform colour spaces, colour-difference equations, and psychometric colour terms *Paris: Bureau Central de la CIE* **CIE #15(Supplement 2)** 9-12.
15. Bernardon JK, Sartori N, Ballarin A, Perdigao J, Lopes GC, & Baratieri LN (2010) Clinical performance of vital bleaching techniques *Operative Dentistry* **35(1)** 3-10.
16. Bortolatto JF, Pretel H, Neto CS, Andrade MF, Moncada G, & Oliveira Junior OB (2013) Effects of LED–laser hybrid light on bleaching effectiveness and tooth sensitivity: A randomized clinical study *Laser Physics Letters* **10(8)**.
17. Ontiveros JC, & Paravina RD (2009) Color change of vital teeth exposed to bleaching performed with and without supplementary light *Journal of Dentistry* **37(11)** 840-847.
18. Goncalves RS, Costa C, Soares D, Santos PD, Cintra L, & Briso A (2016) Effect of different light sources and enamel preconditioning on color change, $H_2O_2$ penetration, and cytotoxicity in bleached teeth *Operative Dentistry* **41(1)** 83-92.
19. Trentino AC, Soares AF, Duarte MA, Ishikiriama SK, & Mondelli RF (2015) Evaluation of pH levels and surface roughness after bleaching and abrasion tests of eight commercial products *Photomedicine and Laser Surgery* **33(7)** 372-377.
20. Mondelli RF, Azevedo JF, Francisconi PA, Ishikiriama SK, & Mondelli J (2009) Wear and surface roughness of bovine enamel submitted to bleaching *European Journal of Esthetic Dentistry* **4(4)** 396-403.
21. Mena-Serrano AP, Garcia E, Luque-Martinez I, Grande R, Loguercio AD, & Reis A (2016) A single-blind randomized trial about the effect of hydrogen peroxide concentration on light-activated bleaching *Operative Dentistry* **41(5)** 455-464.

©Operative Dentistry, 2018, **43-1,** 38-50

## Laboratory Research

# Wear Evaluation of Prosthetic Materials Opposing Themselves

C D'Arcangelo • L Vanini • GD Rondoni • M Vadini • F De Angelis



### Clinical Relevance

Thanks to new composite resins and dental ceramics, an excellent esthetic may be clinically combined with outstanding functional features in terms of their wear behavior, which proved to be very similar to that of the well-known traditional gold alloys.


### SUMMARY

**The purpose of the present *in vitro* study was to compare the two-body wear resistance of a type 3 gold alloy (Aurocast8), two lithium disilicate glass ceramics (IPS e.max CAD and IPS e.max Press), a heat-pressed feldspathic porcelain (Cerabien ZR Press), an yttria-stabilized tetragonal zirconia polycrystal ceramic (Katana Zirconia ML), and three heat-cured composite resins (Ceram.X Universal, Enamel Plus Function, and Enamel Plus HRi) opposing antagonistic cusps made out of the same restorative materials. Ten 6-mm-thick samples and 10 cusp-shaped abraders were manufactured with each test material (n=10) according to standard laboratory procedures. All sample/antagonist pairs made out of the same material were subjected to a two-body wear test in a dual-axis chewing simulator for up to 120,000 loading cycles. The total vertical wear (mm) and the total volumetric loss ($mm^3$) for each sample/antagonist pair were calculated. Data were statistically analyzed using one-way analysis of variance tests. The total vertical wear for the gold alloy was not significantly different compared to Ceram.X Universal, Enamel Plus Function, IPS e.max CAD, and Cerabien ZR Press. Significantly increased wear values were observed for Enamel Plus HRi and IPS e.max Press. The lowest values for total vertical wear and volumetric loss were recorded on the monolithic zirconia.**



*Camillo D'Arcangelo, DDS, Department of Medical, Oral, and Biotechnological Sciences, University of Chieti, Chieti, Italy

Lorenzo Vanini, DDS, private practitioner, Chiasso, Switzerland

Giuseppe Daniele Rondoni, DT, private practitioner, Savona, Italy

Mirco Vadini, PhD, DDS, Department of Medical, Oral, and Biotechnological Sciences, University of Chieti, Chieti, Italy

Francesco De Angelis, PhD, DDS, Department of Medical, Oral, and Biotechnological Sciences, University of Chieti, Chieti, Italy

*Corresponding author: Via dei Vestini, 31, Chieti 66100, Italy; e-mail: cdarcang@unich.it


DOI: 10.2341/16-212-L

### INTRODUCTION

A huge number of dental restorative materials are available today for prosthetic purposes. The ideal restorative should resemble as close as possible the tooth hard tissues that are to be replaced. Among material properties, wear behavior seems of crucial importance, as over time either a reduced wear resistance or an exaggerated abrasiveness may severely jeopardize the esthetic and functional outcome of extensive occlusal rehabilitations, especially when treating patients with parafunctions.

Several studies have analyzed the *in vitro* wear resistance of restorative materials opposing either

human enamel antagonists or dedicated artificial abraders.[1-4] The abrasiveness of gold-based alloys, resin composites, feldspathic porcelains, glass ceramics, and polycrystalline zirconia-based materials toward tooth hard tissues has also been the subject of extensive investigation.[5-9]

In previous research, dental gold-based alloys showed wear characteristics very similar to human enamel.[1,2] Nevertheless, in spite of their excellent marginal accuracy[10,11] and uncontested mechanical/tribological properties,[12] an increasing demand for better esthetics persuaded clinicians to withdraw full-gold restorations in favor of alternative tooth-colored materials.

Dental ceramics exhibit superior optical properties, excellent color stability, and proven biocompatibility.[13-15] Their clinical reliability has also increased[16-20] following the latest advances in adhesive dentistry[21-25] and the recent introduction of strengthened and enhanced ceramic systems.[26] Ceramic materials are wear resistant,[3,4] but they may damage the opposing enamel.[27-30] The general belief that human enamel might be subject to accelerated wear when opposed by traditional porcelain-fused-to-metal crowns[5] was further confirmed *in vivo* in 2011 by Silva and others.[6] Contradictorily, in a similar *in vivo* study by Etman and others,[7] metal-ceramic crowns produced the least tooth wear in comparison to polycrystalline-alumina copings veneered with feldspathic porcelain and to hot-pressed high-leucite glass ceramics. A recent review indicated that some all-ceramic crowns are as wear friendly as metal-ceramic crowns.[8] The author of the same review failed to find a strong association between tooth wear against ceramics and any specific causal agent,[8] including the material hardness or its chemical composition, thus underlying the compelling need for additional studies on this specific research topic. The most recent *in vitro* studies reported for some new all-ceramic systems an abrasiveness very close to that of human enamel[9] as well as a wear resistance similar to that of traditional gold alloys.[2]

In a direct comparison between properties, such as flexural strength, hardness, or optical behavior, ceramic/glass-ceramic materials are generally superior to dental composites.[31] Nevertheless, thanks to continuous innovations in filler composition and particle size/morphology, current micro-/nanohybrid composites definitely show proper esthetic/mechanical features for successful use in all areas of the mouth.[32,33] Additionally, the increasing appeal of composite resins is warranted by their ease of use, the possibility of an easy and invisible intraoral repair of minor defects induced by function, and the opportunity to choose a direct or an indirect approach.[31] Those characteristics are extremely attractive, as minimally invasive solutions today seem to be preferred in every branch of dentistry.[34-36] Composites are traditionally considered more wear friendly than dental ceramics. In general, resin-based materials produce lower enamel antagonist wear than ceramic-based ones, both in the manually polymerized and in the CAD/CAM versions.[37] In a recent *in vitro* study, resin composite antagonists led to the lowest wear on the opposing enamel, being significantly reduced compared to the enamel wear recorded against lithium disilicate glass-ceramic abraders.[38]

Moreover, innovative and enhanced resin composites have been recently introduced, showing promising *in vitro* wear resistance values, statistically similar to those of human enamel and gold-based alloys.[1]

Notwithstanding the great efforts that have been made toward investigating the wear properties of prosthetic materials opposing human enamel or dedicated artificial abraders, little is currently known about the *in vitro* wear behavior of a specific dental restorative material opposing itself.

The likelihood of restorative materials opposing themselves in routine clinical practice appears rather strong. The full-mouth rehabilitation of completely edentulous patients using traditional or implant-supported prostheses likely leads to an artificial occlusion with a prosthetic material on the upper jaw opposing the same prosthetic material on the lower jaw.[39] A similar condition may also occur when partially edentulous patients are treated to replace or restore missing or damaged antagonistic teeth. Today, the esthetic and functional rehabilitation of severely eroded or abraded patients is preferably accomplished with additive ceramic/composite-based restorations, which are almost simultaneously bonded to all teeth, finally leaving each newly restored tooth in functional contact with a similarly restored antagonistic tooth.[40] A full-mouth prosthetic approach has been described to be the best treatment option in the less frequent cases of amelogenesis imperfecta.[41,42] Even when only a single tooth requires a full-coverage restoration, it is not uncommon to ascertain that the antagonist had already been restored by employing the same dental material.

To our knowledge, only one *in vitro* study has investigated the two- and three-body wear between

Table 1: *Summary of the Materials Used in the Experimental Groups. Technical Data Were Provided by the Respective Manufacturers.*

| Material | Lot Number | Shade | Manufacturer | Technical Data |
|---|---|---|---|---|
| Katana Zirconia ML | DNTZC | ML DARK | Kuraray Noritake Dental Inc (Tokyo, Japan) | Multilayered yttria-stabilized tetragonal zirconia polycrystal ceramic |
| Aurocast8 | 15L 02 55 | — | Nobil-Metal S.p.A. (Villafranca d'Asti, Italy) | Type 3 high-gold dental alloy. Composition (W/W): Au = 85.4%, Ag = 9.0%, Cu = 5.0%, Pd < 1.0%, Ir < 1.0% |
| Cerabien ZR Press | BJ5NY | L—A2 | Kuraray Noritake Dental | Heat-pressed feldspathic porcelain. Composition (W/W): $SiO_2$ = 66%, $Al_2O_3$ = 16.5%, $K_2O$ = 10%, $Na_2O$ = 4.5%, CaO = <1%, MgO = <1%, $Li_2O$ = <1%, $B_2O_3$ = <1%, traces of ceramic pigments |
| IPS e.max Press | S42514 | LT—A1 | Ivoclar Vivadent (Schaan, Liechtenstein) | Heat-pressed lithium disilicate ($Li_2Si_2O_5$) glass-ceramic. Composition (W/W): $SiO_2$ = 57%-80%, $Li_2O$ = 11%-19%, $K_2O$ = 0%-13%, $P_2O_5$ = 0%-11%, $ZrO_2$ = 0%-8%, ZnO = 0%-8%, other oxides and ceramic pigments = 0%-10% |
| IPS e.max CAD | V13882 | LT—A1 | Ivoclar Vivadent | Milled lithium disilicate ($Li_2Si_2O_5$) glass-ceramic. Composition (W/W): $SiO_2$ = 57%-80%, $Li_2O$ = 11%-19%, $K_2O$ = 0%-13%, $P_2O_5$ = 0%-11%, $ZrO_2$ = 0%-8%, ZnO = 0%-8%, other oxides and ceramic pigments = 0%-10% |
| Enamel Plus HRi | 2014004972 | UE2 | Micerium S.p.A. (Genova, Italy) | Nanohybrid resin composite. Filler content: 80% W/W (12% zirconium-oxide fillers, 68% innovative proprietary glass-based filler). Mean particle size: 1000 nm |
| Enamel Plus Function | 2014007020 | EF2 | Micerium | Microhybrid resin composite. Filler content: 75% W/W. Mean particle size: 700 nm (including 40 nm fumed silica) |
| Ceram.X Universal | 1504004052 | A2 | Dentsply DeTrey (Konstanz, Germany) | Nanoceramic composite. Filler content: 73% W/W. Particle size: 100 nm-3 μm. Mean particle size: 600 nm |

resin composites used both as samples and as antagonistic abraders.[43] Yet for the reasons outlined above, such information seems particularly important both when planning extensive full-mouth rehabilitations and when selecting the appropriate material to restore one or more teeth that oppose already restored antagonistic teeth.

On these bases, the purpose of the present *in vitro* study was to assess the two-body wear of a type 3 gold alloy, an yttria-stabilized zirconia polycrystalline ceramic, a heat-pressed feldspathic porcelain, a lithium disilicate glass ceramic (milled and heat pressed), and three different heat-cured resin composites opposing standardized antagonistic cusps made out of the same restorative materials. Each sample was subjected to 120,000 mastication simulation cycles. The null hypothesis tested was that no difference could be detected in the wear resistance among the materials under investigation.

## METHODS AND MATERIALS

A complete list of the materials tested in the present study, together with some data about their composition, is given in Table 1.

### Sample Fabrication

Ten IPS e.max Press and 10 Cerabien ZR Press cylindrical specimens were fabricated according to the conventional lost-wax technique by investing and eliminating acrylic resin disks (Plexiglas, Evonik Röhm GmbH, Darmstadt, Germany) 7 mm in diameter and 6 mm thick. The void was filled with the pressable ceramic, following the pressing parameters of each respective manufacturer.

For CAD/CAM materials (IPS e.max CAD and Katana Zirconia ML), ceramic blocks were secured to the arm of a saw (Micromet M, Remet s.a.s., Bologna, Italy) and subjected to consecutive cuts to obtain 6-mm-thick slices. Ten lithium disilicate specimens were produced and subsequently crystallized in a ceramic furnace (Programat EP 5000, Ivoclar Vivadent, Schaan, Liechtenstein) at 840°C to 850°C. Ten zirconia slices were sintered at 1500°C for two hours.

For each of the three resin composites under investigation (Ceram.X Universal, shade A2; Enamel Plus Function, shade EF2; and Enamel Plus HRi, shade UE2), 10 cylinders were manufactured using transparent polyethylene molds measuring 7 mm in diameter and 6 mm in height. The mold was positioned on a glass surface and then filled. The resin composite was applied in three 2-mm-thick layers. Each layer was individually polymerized for 40 seconds (L.E.Demetron I, Sybron/Kerr, Orange, CA, USA) with a 1200-mW/cm$^2$ output. After mold removal, composite cylinders underwent a further heat-curing cycle (Laborlux, Micerium S.p.A., Genova, Italy) at 80°C for 10 minutes.

Gold alloy (Aurocast8) cylindrical specimens (n=10) were made using the traditional lost-wax technique.

### Antagonist Fabrication

An artificial stainless-steel cusp having a slight conical shape and a 2-mm-diameter round tip (Figure 1a,b) was used as a template to produce eight sets of 10 standard antagonists (n=10) employing each one of the eight restorative materials under investigation.

For IPS e.max CAD and Katana Zirconia ML (Figure 1c), the template steel cusp was scanned in order to guide the CAM of the antagonists.

For Ceram.X Universal, Enamel Plus Function (Figure 1g), and Enamel Plus HRi, a polyvinylsiloxane impression of the template steel cusp was taken. The so-achieved cusp-shaped silicon mold was used to produce standard resin-based composite

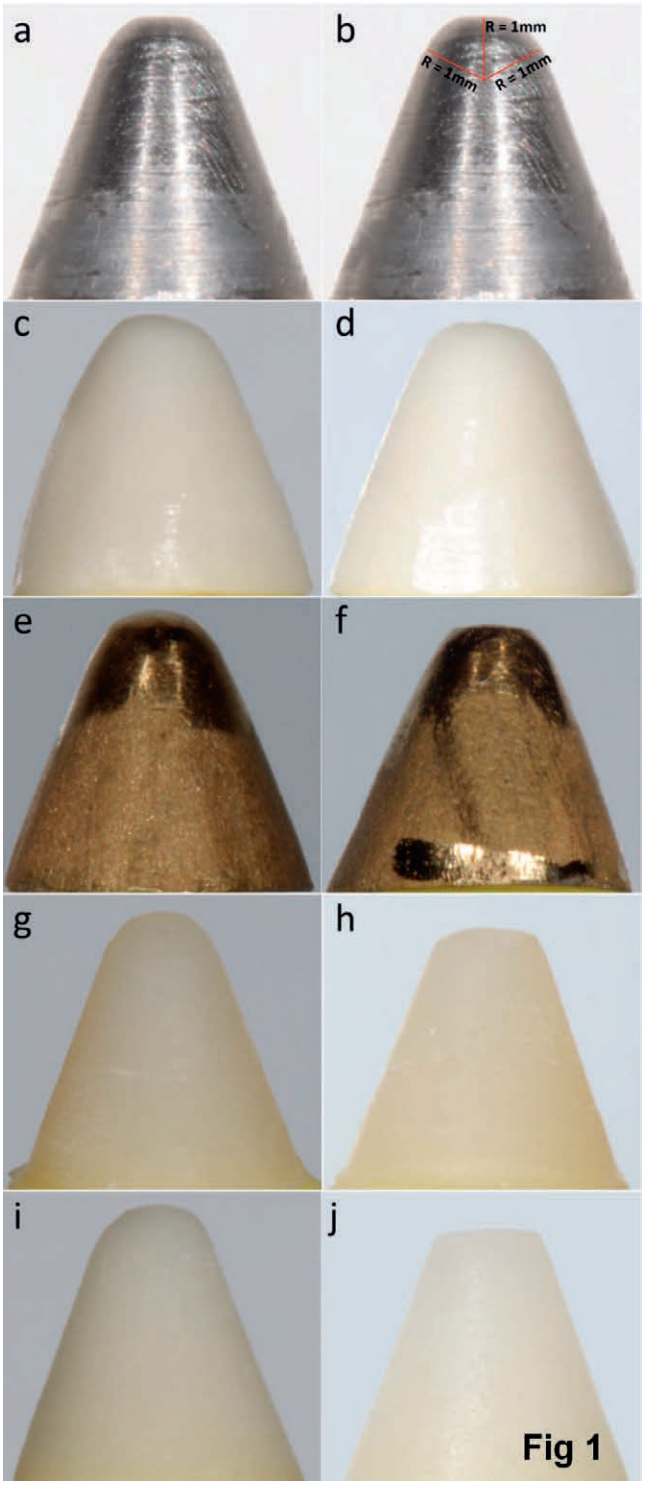



Figure 1. *A stainless-steel cusp (a) having a 1-mm-radius round tip (b) was used as a template to manufacture the standard antagonistic abraders required for chewing simulation testing. The figure shows some representative antagonistic cusps from the following experimental groups: Katana Zirconia ML (before [c] and after [d] testing); Aurocast8 gold alloy (before [e] and after [f] testing); heat-cured Enamel Plus Function (before [g] and after [h] testing); IPS e.max Press lithium disilicate glass-ceramic (before [i] and after [j] testing).*

| Table 2: *Configuration of Parameters Set for Wear Method.* | |
|---|---|
| Number of cycles | 120,000 |
| Force | 49 N |
| Height | 3 mm |
| Lateral movement | −0.7 mm |
| Descendent speed | 60 mm/s |
| Lifting speed | 60 mm/s |
| Feed speed | 40 mm/s |
| Return speed | 40 mm/s |
| Frequency | 1.6 Hz |

antagonists by applying and individually light curing three 2-mm-thick composite layers. After manufacturing, resin composite cusps were heat cured as already described for composite cylindrical specimens.

The same cusp-shaped silicon mold employed for composite antagonists was also used to produce wax replicas of the template steel cusp. The wax replicas were then invested in order to manufacture standard IPS e.max Press (Figure 1i), Cerabien ZR Press, and Aurocast8 (Figure 1e) antagonists following the lost-wax technique and the respective manufacturer instructions.

### Polishing Procedures

Samples and cusps made out of heat-pressed lithium disilicate ceramic, heat-pressed feldspathic porcelain, and milled lithium disilicate ceramic (IPS e.max Press, Cerabien ZR Press, and IPS e.max CAD groups) were hand polished with silicon-carbide silicon polishers (Pink Medium Midgets, RA #15; Dedeco International Inc, Long Eddy, NY, USA) and paper-abrasive cones (L-Red Meister Cones, Kuraray Noritake Dental Inc, Tokyo, Japan), followed by a diamond polishing paste (Signum HP, Heraeus Kulzer GmbH, Hanau, Germany) delivered with a goat hair brush (RA Shiny S, Micerium).

Zirconia samples and cusps (Katana Zirconia ML group) were treated using the same silicon polishers and the same paper-abrasive cones employed on lithium disilicate, but their surface was subsequently polished to a final finish using a zirconia-specific diamond paste (Pearl Surface Z, Kuraray Noritake Dental).

Resin composite samples and cusps were gently finished with a rubber point (Shiny 14, Micerium) and electric hand piece. A polishing procedure was than performed using diamond pastes containing 3-μm (Shiny A, Micerium) and 1-μm (Shiny B, Micerium) diamond particles, delivered with a goat hair brush, followed by an aluminum oxide paste (Shiny C, Micerium), delivered with a felt wheel (Shiny F, Micerium).

Silicone rubber polishers (Blue Fine and Pink Extra-Fine Midgets, HP #15, Dedeco International) and an alloy-specific diamond paste (Dia Past, Nobil-Metal S.p.A., Villafranca d'Asti, Italy), delivered with a felt wheel, were used to polish gold alloy samples and antagonists.

Every step of the above described polishing procedures was performed with an electric hand piece at 15,000 rpm with hand pressure for one minute.

### Wear Testing

After manufacturing, all specimens and cusps were stored for 24 hours at 37°C and then subjected to a two-body wear test in a dual-axis chewing simulator (CS-4.2, SD Mechatronik GmbH, Feldkirchen-Westerham, Germany) according to the methodology described by D'Arcangelo and others.[1] In brief, autopolymerizing acrylic resin was used to secure the cusps on the antagonist holders and to fix each specimen inside the sample chamber.

Each specimen was loaded against a standard cusp made out of the same restorative material at 1.6 Hz for a total of 120,000 chewing cycles. The masticatory cycle in this study consisted of three phases: contact with a vertical force of 5 kg, horizontal sliding of 0.7 mm, and separation of the specimen and its antagonistic cusp. The chewing simulation parameters used are summarized in Table 2.

### Data Analysis

After wear testing, a three-dimensional surface analysis of all specimens was performed with a CAD/CAM contact scanner (Dental Scanner, Renishaw Inc, West Dundee, IL, USA): the sample vertical wear (mm) and its volumetric loss ($mm^3$) were then calculated.[1] Moreover, the difference between the pretest and posttest height of each antagonistic cusp was measured and assumed as the antagonist vertical wear (mm). The vertical wear of each cusp was also used to calculate the volume of the spherical cap corresponding to the antagonist volumetric loss ($mm^3$), according to the following formula:

$$\text{antagonist volumetric loss} = \pi \cdot h^2 \cdot (3R - h)/3,$$

where $h$ is the spherical cap height, corresponding to the antagonist vertical wear (mm), and $R$ is the

Table 3: *Mean values (and standard deviations [SD]) for the sample vertical wear (mm), antagonist vertical wear (mm), and total vertical wear (mm) achieved in the experimental groups (n=10). Total vertical wear mean values were compared using a one-way analysis of variance test. Same letters indicate no statistically significant differences.*

| Material | Sample Vertical Wear (SD) *A* | Antagonist Vertical Wear (SD) *B* | Total Vertical Wear (SD) *A + B* |
|---|---|---|---|
| Katana Zirconia ML | 0.018 (0.011) | 0.092 (0.036) | 0.109 (0.033) C |
| Aurocast8 | 0.073 (0.017) | 0.142 (0.074) | 0.215 (0.085) B |
| Enamel Plus Function (EF2), heat cured | 0.065 (0.033) | 0.207 (0.078) | 0.272 (0.092) B |
| Ceram.X Universal (A2), heat cured | 0.087 (0.018) | 0.204 (0.079) | 0.291 (0.083) B |
| Cerabien ZR Press | 0.104 (0.022) | 0.194 (0.041) | 0.297 (0.061) B |
| IPS e.max CAD | 0.166 (0.029) | 0.147 (0.063) | 0.313 (0.076) B |
| Enamel Plus HRi (UE2), heat cured | 0.234 (0.029) | 0.211 (0.091) | 0.445 (0.087) A |
| IPS e.max Press | 0.181 (0.037) | 0.316 (0.042) | 0.497 (0.059) A |

spherical cap radius, equal to 1 mm for each antagonist because of the standardized manufacturing process.

The total vertical wear (mm) for each sample/antagonist pair was finally calculated as the sum of each sample vertical wear and the corresponding antagonist vertical wear. Similarly, the total volumetric loss ($mm^3$) was calculated as the sum of the sample volumetric loss and the corresponding antagonist volumetric loss.

Means (and standard deviations) for total vertical wear and total volumetric loss were calculated for the eight materials under investigation. Mean values were then compared using one-way analysis of variance (ANOVA) tests and Tukey honestly significant difference tests ($\alpha=0.05$).

### Scanning Electron Microscopy Analysis

After the quantitative wear evaluation, the abraded samples were sputter coated (except the gold alloy samples) and observed under a scanning electron microscope (SEM) (EVO 50 XVP LaB6, Carl Zeiss SMT Ltd, Cambridge, UK) at 50× magnification in order to analyze the wear facets produced throughout the chewing simulation. SEM conditions were set as follows: high vacuum ($2{\cdot}10^{-7}$ Torr), emission current 10 pA, accelerating voltage 10 kV, and working distance around 10 mm.

## RESULTS

Tables 3 and 4 show the total vertical wear and total volumetric loss mean values recorded for each test material after 120,000 mastication simulation cycles against antagonistic cusps made out of the same restorative material. The contribution of mean antagonist wear and mean sample wear to the ultimate calculation of the total wear is also given. Data are presented as box plots in Figures 2 and 3, sorting groups from the least to the greatest wear on the category axis. The one-way ANOVA tests showed that the mean value differences observed for the total vertical wear ($F=26.995$; $p<0.001$) and for the

Table 4: *Mean values (and standard deviations [SD]) for the sample volumetric loss ($mm^3$), antagonist volumetric loss ($mm^3$), and total volumetric loss ($mm^3$) achieved in the experimental groups (n=10). Total volumetric loss mean values were compared using a one-way analysis of variance test. Same letters indicate no statistically significant differences.*

| Material | Sample Volumetric Loss (SD) *C* | Antagonist Volumetric Loss (SD) *D* | Total Volumetric Loss (SD) *C + D* |
|---|---|---|---|
| Katana Zirconia ML | 0.024 (0.019) | 0.029 (0.024) | 0.052 (0.029) E |
| Aurocast8 | 0.146 (0.058) | 0.074 (0.067) | 0.220 (0.119) D |
| Enamel Plus Function (EF2), heat cured | 0.082 (0.059) | 0.139 (0.094) | 0.221 (0.135) D |
| Ceram.X Universal (A2), heat cured | 0.175 (0.044) | 0.137 (0.073) | 0.311 (0.094) CD |
| Cerabien ZR Press | 0.185 (0.066) | 0.114 (0.046) | 0.299 (0.111) CD |
| IPS e.max CAD | 0.346 (0.126) | 0.074 (0.060) | 0.421 (0.155) BC |
| Enamel Plus HRi (UE2), heat cured | 0.362 (0.058) | 0.149 (0.116) | 0.511 (0.100) B |
| IPS e.max Press | 0.512 (0.085) | 0.284 (0.066) | 0.796 (0.127) A |

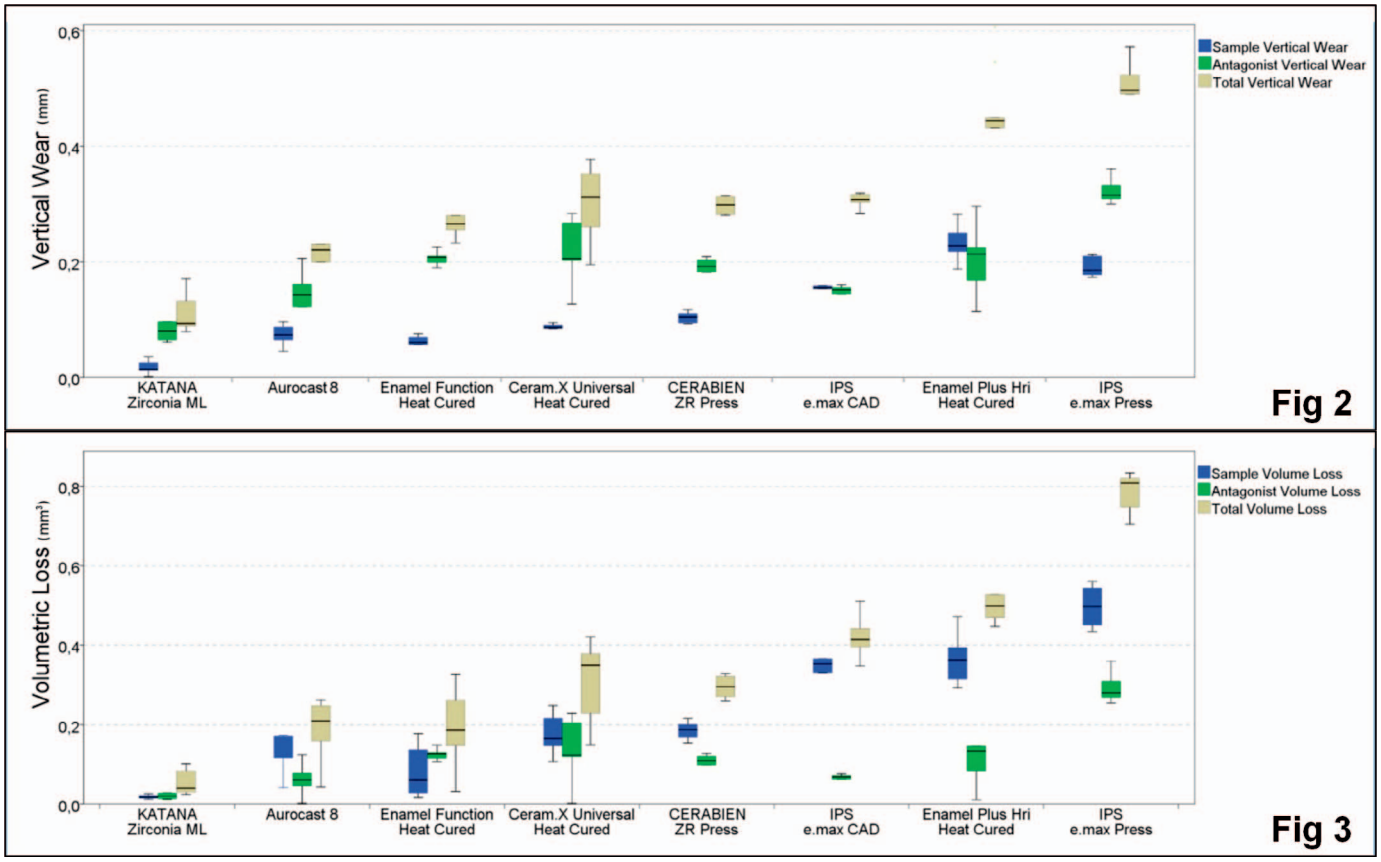



Figure 2. *Data for vertical wear (mm) have been plotted as box plots in order to visualize the distribution of the values observed in the experimental groups. The central line in the box represents the median, and the box represents the middle 50% of values (ie, it ranges from the 25th to the 75th percentile). The whiskers show the extent of the data. Groups on the category axis have been sorted from the least to the greatest observed mean vertical wear values.*

Figure 3. *Data for volumetric loss ($mm^3$) have been plotted as box plots in order to visualize the distribution of the values observed in the experimental groups. The central line in the box represents the median, and the box represents the middle 50% of values (ie, it ranges from the 25th to the 75th percentile). The whiskers show the extent of the data. Groups on the category axis have been sorted from the least to the greatest observed mean volumetric loss values.*

total volumetric loss ($F$=38.957; $p$<0.001) were statistically significant.

The least total vertical wear and total volumetric loss mean values were recorded on zirconia samples opposing zirconia cusps, with a statistically significant difference compared to the total wear of the gold alloy facing gold alloy cusps ($p$=0.044 for vertical wear; $p$=0.033 for volumetric loss). Compared to the gold alloy, slightly increased but not significantly different total mean wear values were registered on heat-cured Enamel Plus Function ($p$=0.671 for vertical wear; $p$=1.000 for volumetric loss), heat-cured Ceram.X ($p$=0.311 for vertical wear; $p$=0.627 for volumetric loss), and Cerabien ZR Press ($p$=0.217 for vertical wear; $p$=0.770 for volumetric loss). The use of e.max CAD led to significantly increased total volumetric loss mean values compared to the gold alloy ($p$=0.005), while no statistically significant differences were recorded between the same two materials in terms of total vertical wear ($p$=0.074). The use of heat-cured Enamel Plus HRi and e.max Press was associated with the highest total vertical wear mean values and significantly increased compared to the vertical wear observed in all the other experimental groups but with no statistically significant difference between one another ($p$=0.775).

Representative SEM images of the wear facets observed on some abraded samples are shown in Figure 4. No wear or shallow depressions were observed on Zirconia (Figure 4a). A slight increase in wear facet dimension was observed on gold alloy (Figure 4b) and on Enamel Plus Function (Figure 4c). The worn surfaces on the heat-cured resin composite appeared smooth, and their borders were clear. Worn ceramic specimens (Figure 4d) showed instead a fairly coarse surface.

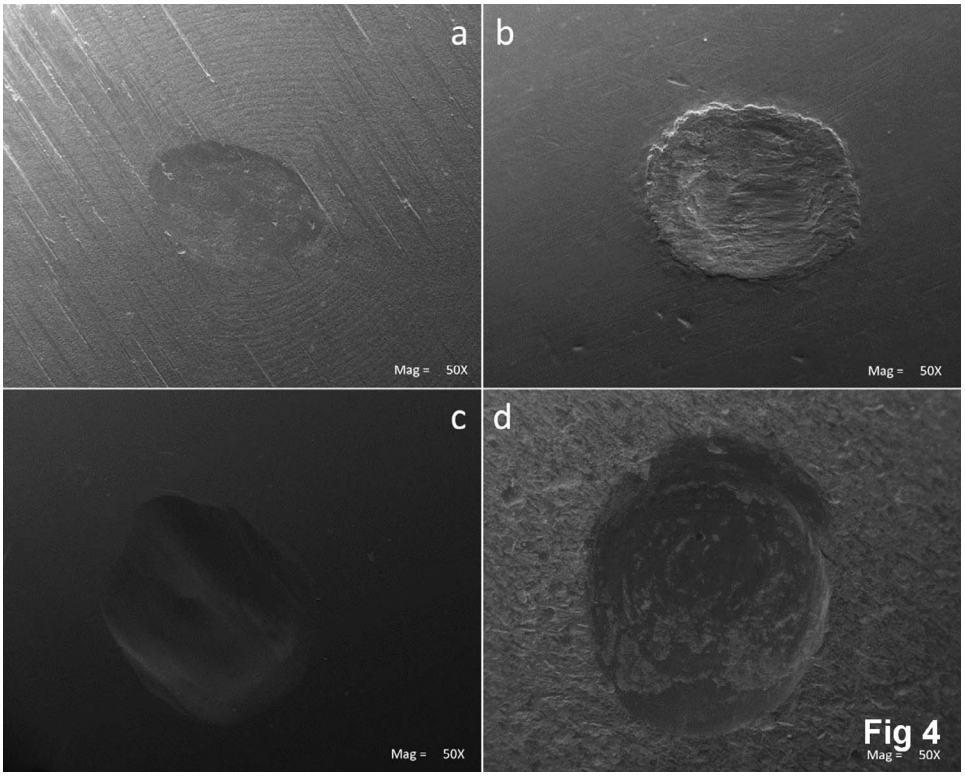



Figure 4. *Scanning electron microphotographs (original magnification 50×) showing the wear facets of some representative samples from the Katana Zirconia ML group (a), Aurocast8 gold alloy group (b), Enamel Plus Function heat-cured group (c), and IPS e.max Press lithium disilicate glass-ceramic group (d).*

## DISCUSSION

The null hypothesis tested in the present study had to be rejected. Significant differences were observed in the wear behavior among the restorative materials under investigation. In an experimental model where every material was tested against an antagonistic cusp made out of the same material, the highest total vertical wear and volumetric loss values were recorded on the heat-pressed lithium disilicate (e.max Press) and on a particular heat-cured nanohybrid composite (Enamel Plus HRi, shade UE2), specifically commercialized by the manufacturer as an esthetic material for anterior restorations.

Sample/antagonist pairs made out of Katana Zirconia ML showed the least total vertical wear and volumetric loss mean values, confirming the high wear resistance exhibited by zirconia-based polycrystalline ceramics in previous investigations.[44]

Two innovative resin-based composites were also tested in the present study. After 120,000 chewing simulation cycles against antagonists made out of the same material, the heat-cured Enamel Plus Function and the heat-cured Ceram.X Universal showed an extremely promising wear behavior, very similar to that of the gold-based alloy, in terms of both vertical wear and volumetric loss. Enamel Plus Function was recently introduced by the manufacturer as a clinical alternative to Enamel Plus HRi for posterior teeth, with the goal of increasing mechanical properties and improving long-term outcomes when used on load-bearing occlusal surfaces.[1] It has been formulated by putting the greatest effort toward optimizing the bond between filler particles and the resin matrix,[1] which might explain the positive wear resistance observed. Ceram.X Universal, on the other hand, is based on a proprietary filler technology called SphereTEC and contains granulated spherical submicron glass fillers. According to the manufacturer, this new filler technology, in combination with an optimized resin matrix, improves the esthetics and polishability and provides high fracture toughness. Even the enhanced wear properties recorded in the present study for this new resin-based composite might be somehow correlated with its unique chemical composition.

The total vertical wear mean values for the milled lithium disilicate ceramic (e.max CAD) and for the heat-pressed feldspathic porcelain (Cerabien ZR

Press) were also not statistically different from the gold alloy. Nevertheless, the wear behavior of ceramics should not be considered similar to that of metal or composite resin. To some extent, metal and composite resins wear through a mechanism involving plastic deformation and adhesion, while ceramics wear through microfractures.[38,45]

The wear properties of the gold alloy are likely due to its inherently increased ductility, provided by metal bonds in its structure, compared to the brittle property of ceramics.[46] The SEM pictures of worn gold alloy samples suggest the occurrence of a plastic deformation over the wear process (Figure 4b). On the other hand, the SEM photographs of worn ceramic specimens (Figure 4d) demonstrated a coarse surface and flaws probably generated by exfoliated hard debris. Even if crack formations of ceramic could not be seen in the SEM observation, fatigue wear might have occurred due to repetitive loading on the brittle ceramic surface. The heat-cured Enamel Plus Function (Figure 4c) displayed a relatively uniform wear surface: the lack of evident irregularities and images of filler dislodgement/protrusion might be correlated to the optimum bond between filler particles and resin matrix.

For decades, the use of metal or gold on the occlusal surfaces has been considered a valid solution in all cases where the prosthetic occlusion was in contact with natural enamel, resin composite, porcelain, or a combination of such materials,[47] causing minimal wear to the antagonist[48] and little interference with the patient occlusal balance.[3] In recent *in vitro* studies, a type 3 gold alloy exhibited the same wear rates of human enamel.[1,2] As a consequence, dental materials that closely resemble the gold alloy in their wear behavior should probably be considered the most physiological substitutes for lost tooth hard tissues.

Excessive wear or exaggerated abrasiveness, on the other hand, should be avoided, as it may lead to unacceptable restoration and/or antagonist damage, with possible alterations of the functional path of masticatory movements. When anterior teeth are involved, both esthetics and anterior guidance function are impaired, finally leading to increased stresses on the masticatory system and possible temporomandibular joint dysfunctions.[49-51]

Many studies have attempted to relate the wear resistance and/or the abrasiveness of dental materials to specific material properties, such as surface topography, fracture toughness, or hardness.[52-54]

According to Fischer and others,[55] for most materials, metal in particular, the wear resistance can indeed be considered directly proportional to the hardness. However, for the abrasion caused by most ceramics, hardness and wear are probably not strictly associated with each other.[56-58] The wear caused by ceramics appears more related to surface roughness and fracture toughness[55,59,60] and should be conveniently considered as a multifactorial condition.[61]

Unlike the case of ceramics, composites produce wear on their antagonist through hard filler protruding from the abraded resin matrix, and the hardness is thought to be a reliable predictor of their abrasiveness.[53,54]

According to the general knowledge about wear between two contacting materials, a softer material is abraded more easily than an opposing harder one.[54] However, in the present study, each tested material was also used to manufacture the respective antagonistic abrader in order to mimic *in vitro* the common clinical situation of two opposing restorations made out of the same dental material. Thus, in this study, every sample was tested against an antagonistic abrader showing exactly the same mechanical properties. Furthermore, the total wear (sample wear plus antagonist wear) was calculated and assumed as the parameter under investigation. In a similar experimental scenario, hardness is maybe less correlated with total wear because, even assuming that a harder material would easily abrade its antagonist, probably it is also less likely to be worn out compared to a softer one and vice versa. Interestingly, even though the manufacturer reports the same Vickers hardness value for both the heat-pressed and the milled versions of lithium disilicate (5800 MPa), in this study a statistically significant difference was detected in the wear properties of e.max Press and e.max CAD. This finding confirmed that the wear behavior of a brittle substrate (like ceramic) is perhaps different from that of a composite, and, consequently, the use of hardness as a wear predictor for all the materials tested did not seem an appropriate solution.

In the present study, both pressed and milled ceramic materials were subjected to a standardized surface polishing procedure before testing. Although in a few studies no statistically significant differences were recorded between the wear properties of polished and glazed lithium disilicate ceramics, a trend toward higher antagonist wear was noted for the glazed ones.[62,63] Moreover, several studies have reported that glazed zirconia may lead to increased

opposing enamel wear compared to polished zirconia.[62,64-71]

As a consequence, assuming that wear-friendly dental materials should always be preferred, it appeared more clinically relevant to deepen the wear properties of a polished ceramic instead of focusing on a glazed one. Furthermore, the wear resistance and the abrasiveness of a glazed ceramic could be influenced by the specific ceramic glaze applied rather than representing an intrinsic property of the material itself.

As a general rule, well-conducted randomized controlled clinical trials should be considered the best method to evaluate the quality of dental materials. However, they are costly, time consuming, and hard to standardize. Therefore, *in vitro* research still remains an indispensable step for initial screening of material properties, and dynamic tests appear to be extremely valuable in predicting the clinical performance of biomaterials subjected to the cyclic solicitations generated by physiological movements.[28,72,73]

## CONCLUSIONS

Within the limitations of an *in vitro* model designed to test the two-body wear resistance of a dental restorative material opposing an antagonist made out of the same material, the following conclusions could be drawn:

1) Among the esthetic and adhesive materials investigated, two heat-cured resin composites (Enamel Plus Function and Ceram.X Universal), a heat-pressed feldspathic porcelain (Cerabien ZR Press), and a milled lithium disilicate glass ceramic (IPS e.max CAD) showed a vertical wear statistically similar to the traditional type 3 gold alloy.
2) Total vertical wear and total volumetric loss observed on the monolithic zirconia (Katana Zirconia ML) were significantly reduced compared to the gold alloy and to all the other tested materials.
3) Total vertical wear and total volumetric loss recorded on the heat-pressed lithium disilicate glass ceramic (IPS e.max Press) were significantly increased compared to what was observed for the milled lithium disilicate glass ceramic (IPS e.max CAD).

### 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 7 March 2017)*

### REFERENCES


1. D'Arcangelo C, Vanini L, Rondoni GD, Pirani M, Vadini M, Gattone M, & De Angelis F (2014) Wear properties of a novel resin composite compared to human enamel and other restorative materials *Operative Dentistry* **39(6)** 612-618.
2. D'Arcangelo C, Vanini L, Rondoni GD, & De Angelis F. Wear properties of dental ceramics and porcelains compared with human enamel *Journal of Prosthetic Dentistry* **115(3)** 350-355, http://dx.doi.org/10.1016/j.prosdent.2015.09.010
3. Elmaria A, Goldstein G, Vijayaraghavan T, Legeros RZ, & Hittelman EL (2006) An evaluation of wear when enamel is opposed by various ceramic materials and gold *Journal of Prosthetic Dentistry* **96(5)** 345-353.
4. Hacker CH, Wagner WC, & Razzoog ME (1996) An in vitro investigation of the wear of enamel on porcelain and gold in saliva *Journal of Prosthetic Dentistry* **75(1)** 14-17.
5. Wiley MG (1989) Effects of porcelain on occluding surfaces of restored teeth *Journal of Prosthetic Dentistry* **61(2)** 133-137.
6. Silva NR, Thompson VP, Valverde GB, Coelho PG, Powers JM, Farah JW, & Esquivel-Upshaw J (2011) Comparative reliability analyses of zirconium oxide and lithium disilicate restorations in vitro and in vivo *Journal of the American Dental Association* **142(Supplement 2)** 4S-9S.
7. Etman MK, Woolford M, & Dunne S (2008) Quantitative measurement of tooth and ceramic wear: In vivo study *International Journal of Prosthodontics* **21(3)** 245-252.
8. Hmaidouch R, & Weigl P (2013) Tooth wear against ceramic crowns in posterior region: A systematic literature review *International Journal of Oral Science* **5(4)** 183-190, http://dx.doi.org/10.1038/ijos.2013.73
9. Zandparsa R, El Huni RM, Hirayama H, & Johnson MI (2016) Effect of different dental ceramic systems on the wear of human enamel: An in vitro study *Journal of Prosthetic Dentistry* **115(2)** 230-237, http://dx.doi.org/10.1016/j.prosdent.2015.09.005
10. Park JM, Hong YS, Park EJ, Heo SJ, & Oh N (2016) Clinical evaluations of cast gold alloy, machinable zirconia, and semiprecious alloy crowns: A multicenter study *Journal of Prosthetic Dentistry* **115(6)** 684-691, http://dx.doi.org/10.1016/j.prosdent.2015.10.018
11. Johnson R, Verrett R, Haney S, Mansueto M, & Challa S (2017) Marginal gap of milled versus cast gold restorations *Journal of Prosthodontics* **26(1)** 56-63, http://dx.doi.org/10.1111/jopr.12432
12. Tucker RV (2008) Why gold castings are excellent restorations *Operative Dentistry* **33(2)** 113-115.
13. Rosenblum MA, & Schulman A (1997) A review of all-ceramic restorations *Journal of the American Dental Association* **128(3)** 297-307.

14. Kelly JR, & Benetti P (2011) Ceramic materials in dentistry: Historical evolution and current practice *Australian Dental Journal* **56(Supplement 1)** 84-96, http://dx.doi.org/10.1111/j.1834-7819.2010.01299.x

15. McLean JW (2001) Evolution of dental ceramics in the twentieth century *Journal of Prosthetic Dentistry* **85(1)** 61-66.

16. Sailer I, Pjetursson BE, Zwahlen M, & Hämmerle CH (2007) A systematic review of the survival and complication rates of all-ceramic and metal-ceramic reconstructions after an observation period of at least 3 years. Part II: Fixed dental prostheses *Clinical Oral Implants Research* **18(Supplement 3)** 86-96.

17. Pjetursson BE, Sailer I, Zwahlen M, & Hämmerle CH (2007) A systematic review of the survival and complication rates of all-ceramic and metal-ceramic reconstructions after an observation period of at least 3 years. Part I: Single crowns *Clinical Oral Implants Research* **18(Supplement 3)** 73-85.

18. Malament KA, & Socransky SS (1999) Survival of Dicor glass-ceramic dental restorations over 14 years: Part I. Survival of Dicor complete coverage restorations and effect of internal surface acid etching, tooth position, gender, and age *Journal of Prosthetic Dentistry* **81(1)** 23-32.

19. Malament KA, Socransky SS, Thompson V, & Rekow D (2003) Survival of glass-ceramic materials and involved clinical risk: Variables affecting long-term survival *Practical Procedures and Aesthetic Dentistry* **2003(Supplement)** 5-11.

20. Malament KA (1988) Considerations in posterior glass-ceramic restorations *International Journal of Periodontics and Restorative Dentistry* **8(4)** 32-49.

21. D'Arcangelo C, Vanini L, Prosperi GD, Di Bussolo G, De Angelis F, D'Amario M, & Caputi S (2009) The influence of adhesive thickness on the microtensile bond strength of three adhesive systems *Journal of Adhesive Dentistry* **11(2)** 109-115.

22. Burke FJ, Fleming GJ, Nathanson D, & Marquis PM (2002) Are adhesive technologies needed to support ceramics? An assessment of the current evidence *Journal of Adhesive Dentistry* **4(1)** 7-22.

23. D'Arcangelo C, De Angelis F, Vadini M, D'Amario M, & Caputi S (2010) Fracture resistance and deflection of pulpless anterior teeth restored with composite or porcelain veneers *Journal of Endodontics* **36(1)** 153-156, http://dx.doi.org/10.1016/j.joen.2009.09.036

24. D'Arcangelo C, D'Amario M, De Angelis F, Zazzeroni S, Vadini M, & Caputi S (2007) Effect of application technique of luting agent on the retention of three types of fiber-reinforced post systems *Journal of Endodontics* **33(11)** 1378-1382.

25. D'Arcangelo C, D'Amario M, Vadini M, Zazzeroni S, De Angelis F, & Caputi S (2008) An evaluation of luting agent application technique effect on fibre post retention *Journal of Dentistry* **36(4)** 235-240, http://dx.doi.org/10.1016/j.jdent.2008.01.004

26. Santos MJ, Costa MD, Rubo JH, Pegoraro LF, & Santos GC Jr (2015) Current all-ceramic systems in dentistry: A review *Compendium of Continuing Education in Dentistry* **36(1)** 31-37.

27. ISO-Standards (2001) *ISO 14569-2 Dental Materials Guidance on Testing of Wear. Part 2: Wear by Two- and/or Three-Body Contact. Technical Specification* International Organization for Standardization, Geneva.

28. Heintze SD (2006) How to qualify and validate wear simulation devices and methods *Dental Materials* **22(8)** 712-734.

29. Heintze SD, Barkmeier WW, Latta MA, & Rousson V (2011) Round robin test: Wear of nine dental restorative materials in six different wear simulators—Supplement to the round robin test of 2005 *Dental Materials* **27(2)** e1-e9, http://dx.doi.org/10.1016/j.dental.2010.09.003

30. Heintze SD, Zellweger G, Grunert I, Muñoz-Viveros CA, & Hagenbuch K (2012) Laboratory methods for evaluating the wear of denture teeth and their correlation with clinical results *Dental Materials* **28(3)** 261-272, http://dx.doi.org/10.1016/j.dental.2011.10.012

31. Ruse ND, & Sadoun MJ (2014) Resin-composite blocks for dental CAD/CAM applications *Journal of Dental Research* **93(12)** 1232-1234, http://dx.doi.org/10.1177/0022034514553976

32. Ferracane JL (2011) Resin composite—State of the art *Dental Materials* **27(1)** 29-38, http://dx.doi.org/10.1016/j.dental.2010.10.020

33. Ilie N, & Hickel R (2009) Investigations on mechanical behaviour of dental composites *Clinical Oral Investigations* **13(4)** 427-438, http://dx.doi.org/10.1007/s00784-009-0258-4

34. Ryder MI, & Armitage GC (2016) Minimally invasive periodontal therapy for general practitioners *Periodontology 2000* **71(1)** 7-9, http://dx.doi.org/10.1111/prd.12132

35. D'Amario M, Baldi M, Petricca R, De Angelis F, El Abed R, & D'Arcangelo C (2013) Evaluation of a new nickel-titanium system to create the glide path in root canal preparation of curved canals *Journal of Endodontics* **39(12)** 1581-1584, http://dx.doi.org/10.1016/j.joen.2013.06.037

36. Opal S, Garg S, Dhindsa A, & Taluja T (2014) Minimally invasive clinical approach in indirect pulp therapy and healing of deep carious lesions *Journal of Clinical Pediatric Dentistry* **38(3)** 185-192.

37. Stawarczyk B, Özcan M, Trottmann A, Schmutz F, Roos M, & Hämmerle C (2013) Two-body wear rate of CAD/CAM resin blocks and their enamel antagonists *Journal of Prosthetic Dentistry* **109(5)** 325-332, http://dx.doi.org/10.1016/S0022-3913(13)60309-1

38. Sripetchdanond J, & Leevailoj C (2014) Wear of human enamel opposing monolithic zirconia, glass ceramic, and composite resin: An in vitro study *Journal of Prosthetic Dentistry* **112(5)** 1141-1150, http://dx.doi.org/10.1016/j.prosdent.2014.05.006

39. Bencharit S, Schardt-Sacco D, Border MB, & Barbaro CP (2010) Full mouth rehabilitation with implant-supported prostheses for severe periodontitis: A case report *Open Dentistry Journal* **13(4)** 165-171, http://dx.doi.org/10.2174/1874210601004010165

40. Grütter L, & Vailati F (2013) Full-mouth adhesive rehabilitation in case of severe dental erosion, a minimally invasive approach following the 3-step technique *European Journal of Esthetic Dentistry* **8(3)** 358-375.

41. Pinky, Naik S, & Nd S (2010) Amelogenesis imperfecta: Full mouth rehabilitation in deciduous dentition *International Journal of Clinical Pediatric Dentistry* **4(2)** 171-175, http://dx.doi.org/10.5005/jp-journals-10005-1105

42. Sabandal MM, & Schäfer E (2016) Amelogenesis imperfecta: Review of diagnostic findings and treatment concepts *Odontology* **104(3)** 245-256, http://dx.doi.org/10.1007/s10266-016-0266-1

43. Osiewicz MA, Werner A, Pytko-Polonczyk J, Roeters FJ, & Kleverlaan CJ (2015) Contact- and contact-free wear between various resin composites *Dental Materials* **31(2)** 134-140, http://dx.doi.org/10.1016/j.dental.2014.11.007

44. Nakashima J, Taira Y, & Sawase T (2016) In vitro wear of four ceramic materials and human enamel on enamel antagonist *European Journal of Oral Sciences* **124(3)** 295-300, http://dx.doi.org/10.1111/eos.12272

45. Oh WS, Delong R, & Anusavice KJ (2002) Factors affecting enamel and ceramic wear: A literature review *Journal of Prosthetic Dentistry* **87(4)** 451-459.

46. Ramp MH, Suzuki S, Cox CF, Lacefield WR, & Koth DL (1997) Evaluation of wear: Enamel opposing three ceramic materials and a gold alloy *Journal of Prosthetic Dentistry* **77(5)** 523-530.

47. Elkins WE (1973) Gold occlusal surfaces and organic occlusion in denture construction *Journal of Prosthetic Dentistry* **30(1)** 94-98.

48. Barco MT Jr, & Synnott SA (1989) Precision metal occlusal surfaces for removable partial dentures *International Journal of Prosthodontics* **2(4)** 365-367.

49. Schuyler CH (1951) Full denture service as influenced by tooth forms and materials *Journal of Prosthetic Dentistry* **1(1-2)** 33-37.

50. Bauer W, van den Hoven F, & Diedrich P (1997) Wear in the upper and lower incisors in relation to incisal and condylar guidance *Journal of Orofacial Orthopedics* **58(6)** 306-319.

51. Wedel A, Borrman H, & Carlsson GE (1998) Tooth wear and temporomandibular joint morphology in a skull material from the 17th century *Swedish Dental Journal* **22(3)** 85-95.

52. Anusavice KJ (2013) *Phillips' Science of Dental Materials* Elsevier, St Louis MO 48-68.

53. Suzuki S, & Leinfelder KF (1993) Wear of enamel cusps opposed by posterior composite resin *Quintessence International* **24(12)** 885-890.

54. Shimane T, Endo K, Zheng JH, Yanagi T, & Ohno H (2010) Wear of opposing teeth by posterior composite resins—Evaluation of newly developed wear test methods *Dental Materials Journal* **29(6)** 713-720.

55. Fischer TE, Anderson MP, & Jahanmir S (1989) Influence of fracture toughness on the wear resistance of yttria-doped zirconium oxide *Journal of the American Ceramic Society* **72(2)** 252-257, http://dx.doi.org/10.1111/j.1151-2916.1989.tb06110.x

56. DeLong R, Douglas WH, Sakaguchi RL, & Pintado MR (1986) The wear of dental porcelain in an artificial mouth *Dental Materials* **2(5)** 214-219.

57. Seghi RR, Rosenstiel SF, & Bauer P (1991) Abrasion of human enamel by different dental ceramics in vitro *Journal of Dental Research* **70(3)** 221-225.

58. Magne P, Oh WS, Pintado MR, & DeLong R (1999) Wear of enamel and veneering ceramics after laboratory and chairside finishing procedures *Journal of Prosthetic Dentistry* **82(6)** 669-679.

59. Metzler KT, Woody RD, Miller AW III, & Miller BH (1999) In vitro investigation of the wear of human enamel by dental porcelain *Journal of Prosthetic Dentistry* **81(3)** 356-364.

60. Fischer TE, Zhu Z, Kim H, & Shin DS (2000) Genesis and role of wear debris in sliding wear of ceramics *Wear* **245(1-2)** 53-60.

61. Faria AC, de Oliveira AA, Alves Gomes E, Silveira Rodrigues RC, & Faria Ribeiro R (2014) Wear resistance of a pressable low-fusing ceramic opposed by dental alloys *Journal of the Mechanical Behavior of Biomedical Materials* **32(April)** 46-51, http://dx.doi.org/10.1016/j.jmbbm.2013.12.018

62. Preis V, Weiser F, Handel G, & Rosentritt M (2013) Wear performance of monolithic dental ceramics with different surface treatments *Quintessence International* **44(5)** 393-405, http://dx.doi.org/10.3290/j.qi.a29151

63. Lawson NC, Janyavula S, Syklawer S, McLaren EA, & Burgess JO (2014) Wear of enamel opposing zirconia and lithium disilicate after adjustment, polishing and glazing *Journal of Dentistry* **42(12)** 1586-1591, http://dx.doi.org/10.1016/j.jdent.2014.09.008

64. Burgess JO, Janyavula S, Lawson NC, Lucas TJ, & Cakir D (2014) Enamel wear opposing polished and aged zirconia *Operative Dentistry* **39(2)** 189-194, http://dx.doi.org/10.2341/12-345-L

65. Janyavula S, Lawson N, Cakir D, Beck P, Ramp LC, & Burgess JO (2013) The wear of polished and glazed zirconia against enamel *Journal of Prosthetic Dentistry* **109(1)** 22-29, http://dx.doi.org/10.1016/S0022-3913(13)60005-0

66. Mitov G, Heintze SD, Walz S, Woll K, Muecklich F, & Pospiech P (2012) Wear behavior of dental Y-TZP ceramic against natural enamel after different finishing procedures *Dental Materials* **28(8)** 909-918, http://dx.doi.org/10.1016/j.dental.2012.04.010

67. Park JH, Park S, Lee K, Yun KD, & Lim HP (2014) Antagonist wear of three CAD/CAM anatomic contour zirconia ceramics *Journal of Prosthetic Dentistry* **111(1)** 20-29, http://dx.doi.org/10.1016/j.prosdent.2013.06.002

68. Preis V, Behr M, Kolbeck C, Hahnel S, Handel G, & Rosentritt M (2011) Wear performance of substructure ceramics and veneering porcelains *Dental Materials* **27(8)** 796-804, http://dx.doi.org/10.1016/j.dental.2011.04.001

69. Stawarczyk B, Özcan M, Schmutz F, Trottmann A, Roos M, & Hämmerle CH (2013) Two-body wear of monolithic, veneered and glazed zirconia and their corresponding enamel antagonists *Acta Odontologica Scandinavica* **71(1)** 102-112, http://dx.doi.org/10.3109/00016357.2011.654248

70. Jung YS, Lee JW, Choi YJ, Ahn JS, Shin SW, & Huh JB (2010) A study on the in-vitro wear of the natural tooth structure by opposing zirconia or dental porcelain *Journal of Advanced Prosthodontics* **2(3)** 111-115, http://dx.doi.org/10.4047/jap.2010.2.3.111

71. Heintze SD, Cavalleri A, Forjanic M, Zellweger G, & Rousson V (2008) Wear of ceramic and antagonist—A systematic evaluation of influencing factors in vitro *Dental Materials* **24(4)** 433-449.

72. Morresi AL, D'Amario M, Capogreco M, Gatto R, Marzo G, D'Arcangelo C, & Monaco A (2014) Thermal cycling for restorative materials: Does a standardized protocol exist in laboratory testing? A literature review *Journal of the Mechanical Behavior of Biomedical Materials* **29(January)** 295-308, http://dx.doi.org/10.1016/j.jmbbm.2013.09.013

73. Mazzitelli C, Monticelli F, Toledano M, Ferrari M, & Osorio R (2012) Effect of thermal cycling on the bond strength of self-adhesive cements to fiber posts *Clinical Oral Investigations* **16(3)** 909-915, http://dx.doi.org/10.1007/s00784-011-0576-1

©Operative Dentistry, 2018, **43-1,** 51-59

# Effect of Vital Tooth Bleaching on Surface Roughness and Streptococcal Biofilm Formation on Direct Tooth-Colored Restorative Materials

I Wongpraparatana • O Matangkasombut • P Thanyasrisung • M Panich

### Clinical Relevance

Vital tooth bleaching with 10% carbamide peroxide or 40% hydrogen peroxide increased both the surface roughness and biofilm formation on resin composite and resin-modified glass ionomer cement restorative materials, suggesting that existing restorations should be polished or replaced after bleaching.




## SUMMARY

**Objective: To compare the effect of simulated bleaching with a 10% carbamide peroxide (CP) or a 40% hydrogen peroxide (HP) system on surface roughness of resin composite and resin-modified glass ionomer cement (RMGI) and streptococcal biofilm formation on these surfaces.**

**Methods and Materials: Specimens of nano-filled resin composite and RMGI (n=108 each) were randomly divided into three groups (n=36 each): no treatment control, 10% CP, and 40% HP. The surface roughness values (Ra) were measured before and after treatments. The specimens in each group were randomly divided into three subgroups (n=12) and incubated with *Streptococcus mutans*, *Streptococcus sanguinis*, and trypticase soy broth control for 24 hours. Biofilm formation was quantified by crystal violet staining, and the structure was visualized by scanning electron microscopy. The differences between the mean changes in Ra between the 10% CP and 40% HP groups of each material were evaluated with an independent *t*-test. The quantity of biofilm forma-**



Issara Wongpraparatana, DDS, Chulalongkorn University, Faculty of Dentistry, Operative Dentistry, Bangkok, Thailand

*Oranart Matangkasombut, DDS, PhD, Chulalongkorn University, Faculty of Dentistry, Microbiology and Research Unit on Oral Microbiology and Immunology, Bangkok, Thailand; Laboratory of Biotechnology, Chulabhorn Research Institute, Bangkok, Thailand

Panida Thanyasrisung, DDS, PhD, Chulalongkorn University, Faculty of Dentistry, Microbiology and Research Unit on Oral Microbiology and Immunology, Bangkok, Thailand

*Muratha Panich, DDS, MSD, Chulalongkorn University, Faculty of Dentistry, Operative Dentistry, Bangkok, Thailand

*Corresponding authors: Henri Dunant Rd., Pathumwan, Bangkok 10330, Thailand; e-mail oranart.m@g.chula.edu, mupanich@yahoo.com


DOI: 10.2341/16-366-L

**tion on each material was analyzed with one-way analysis of variance with the *post hoc* Tukey test ($\alpha$=0.05).**

**Results: Surface roughness significantly increased after bleaching in all groups. There was no significant difference between the 10% CP and 40% HP groups of each material. For *S. mutans* biofilm formation, bleaching with 10% CP and 40% HP increased biofilm on both materials compared to controls. However, *S. sanguinis* biofilm formation was significantly higher on bleached resin composite but not on RMGI specimens.**

**Conclusions: Simulated bleaching with 10% CP or 40% HP increased both surface roughness and biofilm formation on resin composite and RMGI, except for *S. sanguinis* biofilm on RMGI.**

## INTRODUCTION

Tooth discoloration poses a common esthetic problem, and its treatment is in high demand. The most conservative and noninvasive procedure is vital tooth bleaching, which includes home bleaching, in-office bleaching and over-the-counter products.[1] Home bleaching using a low concentration of carbamide peroxide (CP) is the most popular procedure due to its high success rate and few side effects.[2,3] However, if the patients expect immediate results or refuse an at-home tray delivery technique, then in-office bleaching with a high concentration of hydrogen peroxide (HP) is an alternative.

Although vital tooth bleaching is a relatively safe procedure, it can have adverse effects on restorative materials.[4] A concern exists for patients with existing restorations or with carious lesions requiring restorations before bleaching treatment. Chemical softening of restorative materials caused by bleaching agents may affect clinical durability of these materials.[4] Surface texture of the restorations is also important because rough surfaces may facilitate bacterial adhesion and biofilm formation[5-7] and may also increase susceptibility to staining.[8]

Among tooth-colored restorative materials, nanofilled resin composites, other types of resin composite (such as microhybrid or nanohybrid), and resin-modified glass ionomer cement (RMGI) are the most commonly used. Previous studies reported various effects of CP or HP on surface roughness of restorative materials. While some studies showed that the surface roughness of resin composite increased after bleaching,[9,10] other studies showed insignificant changes.[11-16] Likewise, for RMGI, some studies showed that the surface roughness increased,[9,13,16] whereas others showed no effect.[12,14]

An increase in surface roughness tends to promote biofilm formation since it provides more area for bacterial adhesion and also protects bacteria from shear force and saliva flow.[17] The formation of dental biofilm begins with the adhesion of early colonizers, such as *Streptococcus sanguinis*, and is followed by the late colonizers. In an acidic environment, *Streptococcus mutans*, a major cariogenic pathogen, becomes dominant in the biofilm community and promotes the risk of dental caries initiation and progression.[18-20] Restorative materials with rough surfaces may promote biofilm formation and could potentially increase the risk for dental caries development.

The aim of this study was to evaluate the effect of two bleaching systems on the surface roughness of two direct restorative materials and the streptococcal biofilm formation on these surfaces. The direct restorative materials—a nanofilled resin composite and a RMGI—were exposed to an at-home bleaching system with 10% CP or an in-office system with 40% HP. The null hypothesis was that bleaching would not increase surface roughness of tooth-colored restorative materials and consequent biofilm formation and that different bleaching systems would not present different effects on the materials.

## METHODS AND MATERIALS

### Specimen Fabrication and Bleaching Procedures

Two restorative materials were used in this study: a nanofilled resin composite material (Filtek Z350, 3M ESPE, St Paul, MN, USA) and an RMGI (Fuji II LC, GC Corp, Tokyo, Japan). One hundred and eight specimens of each material in shade A2 were fabricated into disks of 5 mm in diameter and 2 mm thick. The materials were inserted into metal molds positioned on a transparent plastic matrix strip and a glass slab. A second transparent plastic matrix strip and glass slab were used to compress the restorative materials. Each specimen was cured with an LED light (Demi, SDS Kerr, Danbury, CT, USA) at 800 mW/cm$^2$ for 40 seconds. All specimens were polished in a stepwise manner with medium, fine, and superfine polishing discs (Sof-Lex, 3M ESPE) on a slow-speed hand piece rotating in one direction and cleaned in distilled water in an ultrasonic cleanser for five minutes. All specimens were stored in artificial saliva at 37°C for 24 hours.

Table 1: *Composition and Application of Materials Used in This Study*

| | Composition | Application |
|---|---|---|
| Nanofilled resin composite (Filtex Z350, 3M ESPE Dental Products, St Paul, MN, USA) | Matrix: Bis-GMA, UDMA, TEGDMA and Bis-EMA | Curing time: 20 s |
| | Filler: combination of aggregated Zr/Si cluster filler (0.6-1.4 μm) and nonaggregated 20-nm Si filler (filler volume: 63.3%) | |
| Resin-modified glass ionomer cement (Fuji II LC, GC Corp, Tokyo, Japan) | Powder: fluoroaminosilicate glass | Mixing time: 10 s |
| | Liquid: polyacrylic acid, tartaric acid, distilled water, camphoquinone, dibutyl hydroxy toluene, and three-resin complex (mainly HEMA) | Working time: 3 min, 15 s |
| | | P/L ratio 0.33 g/0.10 g |
| | | Curing time: 20 s |
| At-home bleaching (Opalescence PF, Ultradent Products, South Jordan, UT, USA) | 10% carbamide peroxide, | 8 h/session, 14 sessions |
| | potassium nitrate, 0.11% fluoride ion, | |
| | carbopol, glycerine, flavoring (pH=6.7) | |
| In-office bleaching (Opalescence Boost, Ultradent Products) | 40% hydrogen peroxide, | Two 20-min applications for a total of 40 min of treatment time |
| | potassium nitrate, 0.11% fluoride ion, carbopol, glycerine, flavoring (pH=7) | |

*Abbreviations: Bis-GMA, bis-phenol A-glycidyl dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-EMA, bis-phenol A-ethoxylated dimethacrylate; Zr, Zirconium; Si, Silicon; HEMA, hydroxyethylmethacrylate; min, minute(s); s, second(s); g, gram(s); P/L ratio, powder to liquid ratio.*

The specimens of each restorative material type were randomly divided into three groups (n=36 each): no treatment control, 10% CP, and 40% HP treatment groups. The control specimens were stored in artificial saliva for 112 hours at 37°C, and the artificial saliva was changed daily. In the 10% CP group, specimens were bleached with 10% CP (Opalescence PF, Ultradent Products Inc, South Jordan, UT, USA) for 14 cycles of eight-hour applications to simulate home bleaching conditions according to manufacturer recommendations. Between each cycle, bleaching agents were rinsed off with distilled water for 20 seconds. In the 40% HP group, specimens were bleached with 40% HP (Opalescence Boost, Ultradent Products) for two cycles of 20-minute applications to simulate in-office bleaching according to manufacturer recommendations. After bleaching, all specimens were rinsed off with distilled water for 20 seconds and air-dried for 30 seconds. Information on material compositions and bleaching material applications is shown in Table 1.

### Surface Roughness Measurement

The surface roughness values (Ra) were measured by a high-resolution three-dimensional optical surface measurement device (InfiniteFocusSL, Alicona Imaging GmbH, Graz, Austria) at 50× magnification in five different areas before and after treatment procedures. Specimens were fixed with a special jig to place them in the same positions for the measurements. The average surface roughness (Ra) was determined and recorded.

### Bacterial Cultures

Bacterial cultures were prepared from frozen stocks of *S. mutans* ATCC25175 and *S. sanguinis* ATCC10556 as described by Ittatirut and others.[21] *S. mutans* and *S. sanguinis* were cultured in trypticase soy agar plates and incubated at 37°C with 5% $CO_2$ for 48 hours. For each experiment, a single colony was inoculated into sterile trypticase soy broth and incubated at 37°C with 5% $CO_2$ for 16 hours. The $OD_{550nm}$ of the cultures was adjusted to 0.1 as measured by a spectrophotometer (Pharmacia LKB Biotechnology Inc, Uppsala, Sweden) and incubated at 37°C for two hours until the $OD_{550nm}$ reached 0.3. The cultures were then centrifuged and resuspended in trypticase soy broth with 4% sucrose for biofilm formation assays.

### Biofilm Formation Assays

The specimens were mounted into 96-well plates and disinfected with ethylene oxide gas. Each group of the specimens was randomly divided into three subgroups (n=12) for biofilm formation assays with *S. mutans*, *S. sanguinis*, and no bacteria control. The assay was done as described by Ittatirut and others[21] with minor modifications. Each well containing a specimen was filled with 100 μL of filter-sterilized artificial saliva and incubated at 37°C for two hours. Then 100 μL of bacterial suspensions with 4% sucrose in trypticase soy broth were dispensed onto each specimen, except for the control group, which received only media without bacteria. All specimens were incubated at 37°C with 5% $CO_2$ for 24 hours.

Table 2: *Surface Roughness Values (nm) Before and After Bleaching*

| Restorative Materials | Bleaching Agents | Before Bleaching | | After Bleaching | | *p*-Value[a] |
|---|---|---|---|---|---|---|
| | | Mean | SD | Mean | SD | |
| Resin composite | 10% CP | 183.65 | 12.48 | 189.21 | 12.35 | <0.001 |
| Resin composite | 40% HP | 179.41 | 15.56 | 185.13 | 15.52 | <0.001 |
| RMGI | 10% CP | 375.75 | 59.67 | 443.98 | 65.54 | <0.001 |
| RMGI | 40% HP | 365.16 | 63.28 | 427.52 | 75.70 | <0.001 |

*Abbreviations: CP, carbamide peroxide; HP, hydrogen peroxide; RMGI, resin-modified glass ionomer cement.*
[a] *Paired* t*-test.*

The total amount of biofilm formation (n=9 for each subgroup) was quantified by the crystal violet assay. The amount of biofilm was measured by the optical density of extracted crystal violet in the destaining solution at 595 nm ($OD_{595nm}$) with a microplate reader (Biochrom Anthos Zenyth 200rt, Biochrom US, Holliston, MA, USA). Each optical density value of the biofilm group was subtracted by the mean optical density of the respective controls without bacteria to remove background value.

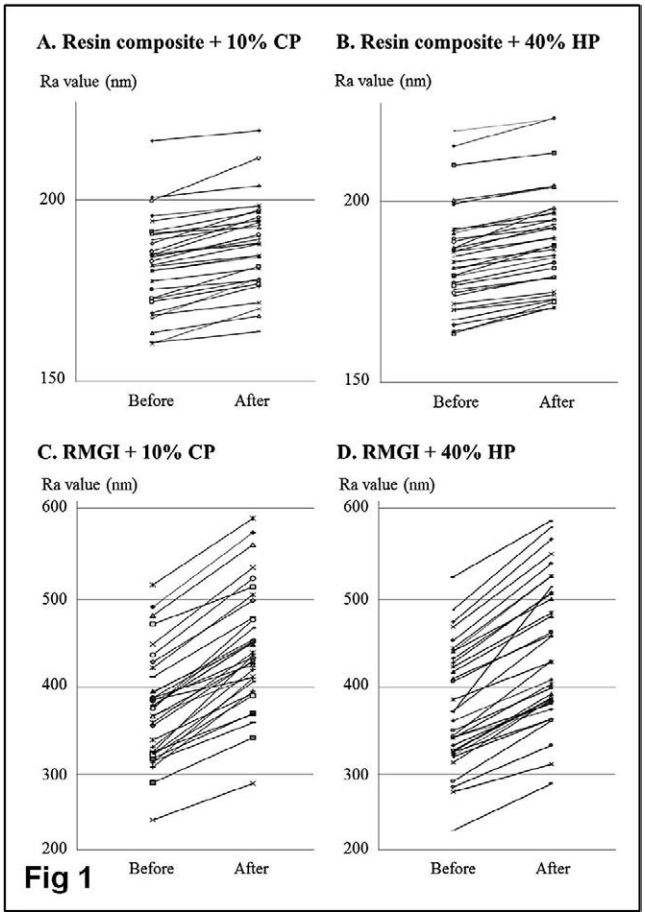



Figure 1. *Bleaching significantly increased surface roughness of both resin composite and resin-modified glass ionomer cement (RMGI). Scatter plots of paired data (before and after bleaching) of surface roughness values are shown. (A): Resin composite before and after bleaching with 10% carbamide peroxide. (B): Resin composite before and after bleaching with 40% hydrogen peroxide. (C): RMGI before and after bleaching with 10% carbamide peroxide. (D): RMGI before and after bleaching with 40% hydrogen peroxide.*

Three specimens of each group were prepared and examined with a scanning electron microscope at 6000× magnification (model JSM-5410LV, JEOL Ltd, Tokyo, Japan).

### Statistical Analysis

Statistical calculations were performed with SPSS version 17.0 software (SPSS Inc, Chicago, IL, USA). The differences in surface roughness values between before and after treatments were evaluated with a paired *t*-test. The differences of mean changes in surface roughness values between the 10% CP and 40% HP groups of each material were evaluated with an independent *t*-test. The amount of biofilm formation among groups of the same material and bacterial species was evaluated with one-way analysis of variance with the *post hoc* Tukey test. The correlation between surface roughness and biofilm formation was analyzed by linear regression. Statistical significance level was set at $\alpha = 0.05$.

## RESULTS

*Surface Roughness*—When comparing the surface roughness values before and after bleaching within each group, we found that surface roughness significantly increased after bleaching treatments for all groups ($p<0.001$) (Table 2; Figure 1). No significant difference was observed when the mean changes in surface roughness were compared between 10% CP and 40% HP for each material (Table 3).

*Biofilm Formation*—On resin composite specimens, bleaching with 10% CP or 40% HP increased both *S. mutans* and *S. sanguinis* biofilm formation when compared to unbleached specimens ($p<0.001$), with no significant difference between the 10% CP and 40% HP groups ($p=0.661$). *S. mutans* biofilm

Table 3: *Mean Changes in Surface Roughness Values (nm) Between 10% Carbamide Peroxide and 40% Hydrogen Peroxide*

| Restorative Materials | 10% Carbamide Peroxide | | 40% Hydrogen Peroxide | | *p*-Value[a] |
|---|---|---|---|---|---|
| | Mean | SD | Mean | SD | |
| Resin composite | 5.56 | 2.75 | 5.72 | 2.11 | 0.931 |
| RMGI | 67.98 | 19.95 | 62.11 | 21.49 | 0.096 |

*Abbreviation: RMGI, resin-modified glass ionomer cement.*
[a] *Independent* t-*test.*

formation on bleached RMGI specimens were also increased ($p<0.001$) with no significant difference between 10% CP and 40% HP ($p=0.487$). However, no significant difference was observed for *S. sanguinis* biofilm formation between bleached and unbleached RMGI specimens ($p=0.063$) (Table 4). When we analyzed the relationship between surface roughness and biofilm formation, regardless of bleaching treatments, simple regression showed a significant correlation only in the RMGI and *S. mutans* group ($p=0.013$, $r=0.473$) (Figure 2).

Representative scanning electron micrographs of biofilm structure are shown in Figure 3 (resin composite) and Figure 4 (RMGI). As expected, the control groups without bacteria did not show any biofilm formation (Figure 3A,B,C and Figure 4A,B,C). For resin composite specimens, *S. mutans and S. sanguinis* biofilm formation was observed in all groups, but the number of cells in unbleached groups appeared lower than the bleached groups (Figure 3D,E,F,G,H,I) For RMGI specimens, all groups showed similar *S. mutans and S. sanguinis* biofilm formation (Figure 4D,E,F,G,H,I).

## DISCUSSION

In this study, we found that surface roughness of both materials significantly increased after bleaching treatment for all groups with no significant difference between 10% CP and 40% HP groups. For *S. mutans* biofilm formation, bleaching with 10% CP and 40% HP increased biofilm on both materials compared to the control group. However, for *S. sanguinis* biofilm, there was significantly higher biofilm formation on bleached resin composite but not on RMGI specimens. Hence, the findings of this study reject the first part of the null hypothesis, showing that the bleaching systems used promoted increased surface roughness and biofilm formation on both materials tested. The second part of the null hypothesis, however, is accepted since no difference in effect between the two bleaching systems used was observed. To our knowledge, this is the first study that evaluates the effect of bleaching on biofilm formation of both *S. mutans* and *S. sanguinis* on tooth-colored restorative materials.

Vital bleaching is a popular treatment option for discolored teeth due to its high success rate, ease of

Table 4: *Streptococcal biofilm formation on restorative materials bleached with 10% carbamide peroxide and with 40% hydrogen peroxide.*

| Restorative materials | Bacteria | Amount of biofilm ($OD_{595\ nm}$) | | | ANOVA | Tukey test | | |
|---|---|---|---|---|---|---|---|---|
| | | | Mean | SD | p-value | pairwise comparison | | p-value |
| Resin composite | *S. mutans* | Unbleached | 0.178 | 0.086 | <0.001 | Unbleached | 10% CP | <0.001 |
| | | 10% CP | 0.442 | 0.134 | | Unbleached | 40% HP | <0.001 |
| | | 40% HP | 0.493 | 0.119 | | 10% CP | 40% HP | 0.661 |
| Resin composite | *S. sanguinis* | Unbleached | 0.173 | 0.063 | <0.001 | Unbleached | 10% CP | <0.001 |
| | | 10% CP | 0.774 | 0.129 | | Unbleached | 40% HP | <0.001 |
| | | 40% HP | 0.340 | 0.123 | | 10% CP | 40% HP | 0.487 |
| RMGI | *S. mutans* | Unbleached | 0.656 | 0.080 | <0.001 | Unbleached | 10% CP | <0.001 |
| | | 10% CP | 0.985 | 0.115 | | Unbleached | 40% HP | <0.001 |
| | | 40% HP | 0.973 | 0.166 | | 10% CP | 40% HP | 0.977 |
| RMGI | *S. sanguinis* | Unbleached | 0.746 | 0.149 | 0.063 | Unbleached | 10% CP | 0.054 |
| | | 10% CP | 0.922 | 0.183 | | Unbleached | 40% HP | 0.273 |
| | | 40% HP | 0.859 | 0.117 | | 10% CP | 40% HP | 0.658 |

*Abbreviations: CP, carbamide peroxide; HP, hydrogen peroxide; RMGI, resin-modified glass ionomer cement.*

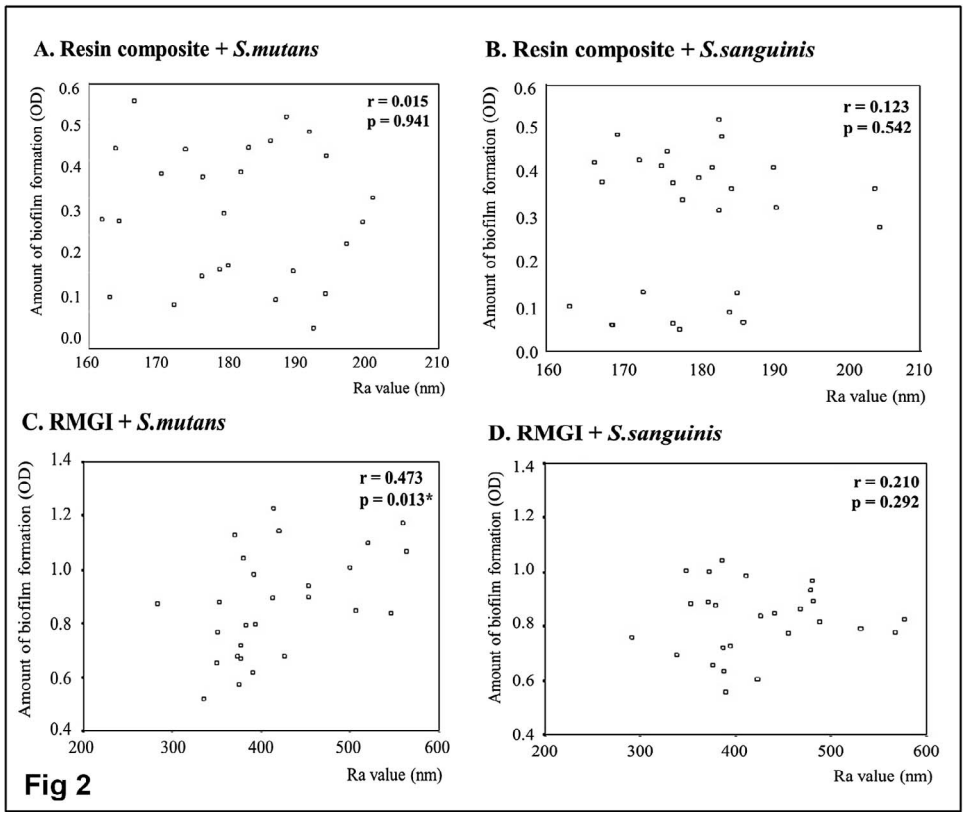



Figure 2. *Relationship between surface roughness values (Ra) and amount of biofilm formation (optical density from crystal violet staining assays) were analyzed with linear regression analysis. (A): Resin composite and* S. mutans *biofilm. (B): Resin composite and* S. sanguinis *biofilm. (C): Resin-modified glass ionomer cement (RMGI) and* S. mutans *biofilm. (D): RMGI and* S. sanguinis *biofilm.* r*, correlation coefficient;* *p $<$ *0.05 is considered significant.*

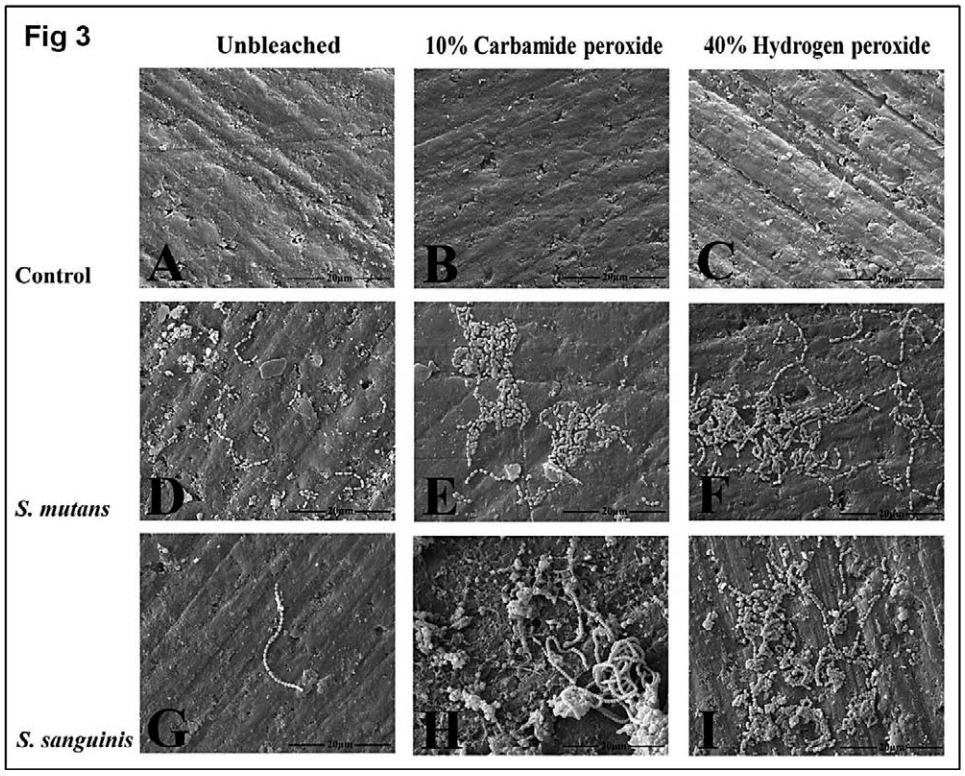



Figure 3. *Scanning electron microscopic images of representative examples of resin composite specimens show biofilm structure on the surfaces. (A): Unbleached without bacteria. (B): Bleached with 10% carbamide peroxide without bacteria. (C): Bleached with 40% hydrogen peroxide without bacteria. (D): Unbleached with* S. mutans*. (E): Bleached with 10% carbamide peroxide with* S. mutans*. (F): Bleached with 40% hydrogen peroxide with* S. mutans*. (G): Unbleached with* S. sanguinis*. (H): Bleached with 10% carbamide peroxide with* S. sanguinis*. (I): Bleached with 40% hydrogen peroxide with* S. sanguinis.

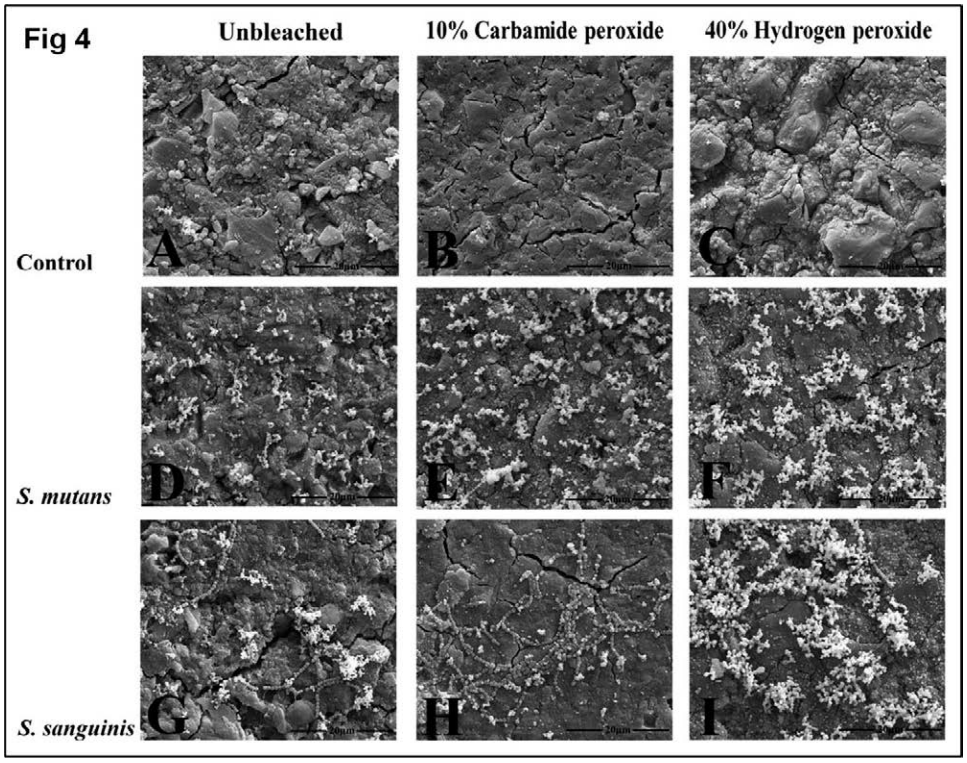



Figure 4. *Scanning electron microscopic images of representative examples of resin-modified glass ionomer cement specimens show biofilm structure on the surfaces. (A): Unbleached without bacteria. (B): Bleached with 10% carbamide peroxide without bacteria. (C): Bleached with 40% hydrogen peroxide without bacteria. (D): Unbleached with* S. mutans. *(E): Bleached with 10% carbamide peroxide with* S. mutans. *(F): Bleached with 40% hydrogen peroxide with* S. mutans. *(G): Unbleached with* S. sanguinis. *(H): Bleached with 10% carbamide peroxide with* S. sanguinis. *(I): Bleached with 40% hydrogen peroxide with* S. sanguinis.

use, and conservativeness. However, bleaching could have adverse effects on existing restorations. Surface roughness is an important property that may affect clinical longevity of restorations, susceptibility to staining, bacterial adhesion, and biofilm formation.[5-7] Previous studies showed an increase in surface roughness of direct tooth-colored restorations after bleaching treatments.[9,10] Moreover, Mor and others[22] showed that bleaching agents may affect adherence of cariogenic microorganisms to the outer surfaces of resin composite restorations.

Our study showed that treatment with 10% CP or 40% HP increased surface roughness of nanofilled resin composite. Compared to previous studies on resin composite, this is in agreement with certain studies[9,10] but differs from others.[11-16] The different results may be due to the use of different types of resin composite, bleaching agents, bleaching systems, and methods of surface roughness measurement. Among the three reports that studied nanofilled resin composite, our results are similar to those using similar measurement methods reported by Markovic and others,[9] who used a three-dimensional optical surface measurement device to examine the effect of 16% CP, 22% CP, and 38% HP, and by Rattacaso and others,[10] who used a contact profilometer to evaluate the effect of 16% CP. In contrast, our results differ from the report of Yu and others,[16] who used scanning electron microscopy to evaluate the effect of 15% CP.

Similarly, we observed that the surface roughness of RMGI also increased after 10% CP or 40% HP application. This result is similar to the findings of Turker and Biskin[13] but is different from those of Wattanapayungkul and Yap.[14] Besides differences in roughness measurement methods among these studies, different bleaching systems differ not only in concentrations of bleaching agents but also in pH and application procedures. It is reasonable to conclude that the effect of bleaching on material surfaces may also be system dependent.

The mechanisms by which bleaching agents could adversely affect resin composite and RMGI are not well understood. Peroxides could induce oxidative cleavage of polymer chains, especially the unreacted double bonds that are the most vulnerable parts of the polymers.[23-25] Durmer and others[26] found that HP reacts with not only the unreacted C-C double bonds but also the C-C single bond in a polymer network in resin composite. Free radicals induced by peroxides may also have an impact on the resin–filler interface and cause a filler–matrix debonding.[23,27,28] Furthermore, water uptake may result in stress corrosion and debonding of fillers.[29]

The effect of bleaching on the surfaces of restorative materials could influence bacterial biofilm

formation. In this study, we used *S. sanguinis and S. mutans* as the representative species of early colonizers and major cariogenic pathogens, respectively.[18-20] On resin composite specimens, bleaching with 10% CP or 40% HP increased both *S. sanguinis* and *S. mutans* biofilm formation compared with unbleached specimens. In contrast, bleaching increased only *S. mutans* biofilm formation on RMGI specimens. Because surface roughness may play an important role in biofilm formation of oral bacteria, we evaluated the relationship between surface roughness and biofilm formation. We found a correlation between *S. mutans* biofilm formation and surface roughness of RMGI. This correlation was similarly observed in an earlier study on enamel surfaces from our group[21] and a study on restorative materials from Filiz and others.[6] Unlike *S. mutans*, we did not observe a correlation between *S. sanguinis* biofilm and surface roughness both in this study and in our previous study on enamel surfaces.[21] This may partly explain why we found an increase in *S. mutans* but not *S. sanguinis* biofilm formation on RMGI with greater surface roughness after bleaching. Moreover, other factors, such as pH and changes in surface chemistry after bleaching, could also influence biofilm formation.[21]

Since cariogenic bacteria are an essential causative factor for the pathogenesis of dental caries, an increase in bacterial adhesion and biofilm formation on restorative materials could potentially increase the risk for secondary caries. Previous studies suggest that reduction of oral mutans streptococcal colonization is associated with lower caries increment[30] and that the use of restorative materials or adhesives with antimicrobial properties may lower the risk of secondary caries.[31,32] However, direct evidence for the association between bacterial adhesion or biofilm formation on restorative materials and secondary caries is still lacking.[33] As secondary caries is a common cause of restoration failures, further investigations into this issue are needed, especially studies in clinical settings.[33,34]

Within the limitation of our *in vitro* study of single-species bacterial biofilm, our results imply that both high and low concentrations of peroxide bleaching agents in in-office and home bleaching systems, respectively, could similarly increase surface roughness and biofilm formation on tooth-colored restorations. Thus, dentists should pay attention to planning appropriate sequences of treatments and should polish existing tooth-colored restorations after bleaching. Nevertheless, additional research on multispecies biofilm and clinical studies is required.

## CONCLUSIONS

Within the limitations of this study, it is possible to conclude that the bleaching systems used, 10% CP or 40% HP, significantly increased both the surface roughness and the streptococcal biofilm formation on resin composite and resin-modified glass ionomer cement.

### Acknowledgements

The authors are grateful for the support of members of the Research Unit on Oral Microbiology and Immunology. This work was supported by the Faculty of Dentistry and the Chulalongkorn University Ratchadaphiseksomphot Endowment Fund (to the Research Unit on Oral Microbiology and Immunology).

### 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 27 March 2017)*

### REFERENCES

1. Joiner A (2006) The bleaching of teeth: A review of the literature *Journal of Dentistry* **34(7)** 412-419.
2. Costa JB, McPharlin R, Paravina RD, & Ferracane JL (2010) Comparison of at-home and in-office tooth whitening using a novel shade guide *Operative Dentistry* **35(4)** 381-388.
3. Auschill TM, Hellwig E, Schmidale S, Sculean A, & Arweiler NB (2005) Efficacy, side-effects and patients' acceptance of different bleaching techniques (OTC, in-office, at-home) *Operative Dentistry* **30(2)** 156-163.
4. Attin T, Hannig C, Wiegand A, & Attin R (2004) Effect of bleaching on restorative materials and restorations—A systematic review *Dental Materials* **20(9)** 852-861.
5. Mei L, Busscher HJ, van der Mei HC, & Ren Y (2011) Influence of surface roughness on streptococcal adhesion forces to composite resins *Dental Materials* **27(8)** 770-778.
6. Aykent F, Yondem I, Ozyesil AG, Gunal SK, Avunduk MC, & Ozkan S (2010) Effect of different finishing techniques for restorative materials on surface roughness and bacterial adhesion *Journal of Prosthestic Dentistry* **103(4)** 221-227.
7. Ikeda M, Matin K, Nikaido T, Foxton RM, & Tagami J (2007) Effect of surface characteristics on adherence of *S. mutans* biofilms to indirect resin composites *Dental Materials Journal* **26(6)** 915-923.
8. Lu H, Roeder LB, Lei L, & Powers JM (2005) Effect of surface roughness on stain resistance of dental resin composites *Journal of Esthetic Dentistry* **17(2)** 102-109.

9. Markovic L, Jordan RA, Glasser M-C, Arnold WH, Nebel J, Tillmann W, Ostermann T, & Zimmer S (2014) Effects of bleaching agents on surface roughness of filling materials *Dental Materials Journal* **33(1)** 59-63.
10. Rattacaso RM, Garcia LFR, Aguilar FG, Consani S, & Pires-de-Souzade FCP (2011) Bleaching agent action on color stability, surface roughness and microhardness of composites submitted to accelerated artificial aging *European Journal of Dentistry* **5(2)** 143-149.
11. Polydorou O, Hellwig E, & Auschill TM (2006) The effect of different bleaching agents on the surface texture of restorative materials *Operative Dentistry* **31(4)** 473-480.
12. Silva MF, Davies RM, Stewart B, DeVizio W, Tonholo J, Júnior JGdS, & Pretty IA (2006) Effect of whitening gels on the surface roughness of restorative materials in situ *Dental Materials* **22(10)** 919-924.
13. Turker SB, & Biskin T (2003) Effect of three bleaching agents on the surface properties of three different esthetic restorative materials *Journal of Prosthestic Dentistry* **89(5)** 466-473.
14. Wattanapayungkul P, & Yap AUJ (2003) Effects of in-office bleaching products on surface finish of tooth-colored restorations *Operative Dentistry* **28(1)** 15-19.
15. Wattanapayungkul P, Yap AUJ, Chooi KW, Lee MFLA, Selamat RS, & Zhou RD (2004) The effect of home bleaching agents on the surface roughness of tooth-colored restoratives with time *Operative Dentistry* **29(4)** 398-403.
16. Yu H, Pan X, Lin Y, Li Q, Hussain M, & Wang Y (2009) Effects of carbamide peroxide on the staining susceptibility of tooth-colored restorative materials *Operative Dentistry* **34(1)** 72-82.
17. Quirynen M, Dierickx K, & van Steenberghe D (2000) Effects of surface roughness and free energy on oral bacterial adhesion In: An YH, Friedman RJ (eds) *Handbook of Bacterial Adhesion: Principles, Methods and Applications* Humana Press, Totowa, NJ 91-102.
18. Hamada S, & Slade H (1980) Biology, immunology, and cariogenicity of *Streptococcus mutans Microbiological Reviews* **44(2)** 331-384.
19. Kolenbrander PE, Andersen RN, Blehert DS, Egland PG, Foster JS, & Palmer RJ (2002) Communication among oral bacteria *Microbiology and Molecular Biology Reviews* **66(3)** 486-505.
20. Loesche W (1986) Role of *Streptococcus mutans* in human dental decay. *Microbiological Reviews* **50(4)** 353-380.
21. Ittatirut S, Matangkasombut O, & Thanyasrisung P (2014) In-office bleaching gel with 35% hydrogen peroxide enhanced biofilm formation of early colonizing streptococci on human enamel *Journal of Dentistry* **42(11)** 1480-1486.
22. Mor C, Steinberg D, Dogan H, & Rottstein I (1998) Bacterial adherence to bleached surfaces of composite resin in vitro *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology and Endodontics* **86(5)** 582-586.
23. Hannig C, Duong S, Becker K, Brunner E, Kahler E, & Attin T (2007) Effect of bleaching on subsurface microhardness of composite and a polyacid modified composite *Dental Materials* **23(2)** 198-203.
24. Wu W, & McKinney JE (1982) Influence of chemicals on wear of dental composites *Journal of Dental Research* **61(10)** 1180-1183.
25. Cullen DR, Nelson JA, & Sandrik JL (1993) Peroxide bleaches: Effect on tensile strength of composite resins *Journal of Prosthestic Dentistry* **69(3)** 247-249.
26. Durner J, Stojanovic M, Urcan E, Spahl W, Haertel U, Hickel R, & Reichl F-X (2011) Effect of hydrogen peroxide on the three-dimensional polymer network in composites *Dental Materials* **27(6)** 573-580.
27. Durner J, Obermaier J, & Ilie N (2014) Investigation of different bleaching conditions on the amount of elutable substances from nano-hybrid composites *Dental Materials* **30(2)** 192-199.
28. De Alexandre RS, Sundfeld RH, Briso ALF, Bedran-Russo AKB, Valentino TA, & Sundefeld MLMM (2006) Effect of 10% carbamide peroxide dental bleaching on microhardness of filled and unfilled sealant materials *Journal of Esthetic and Restorative Dentistry* **18(5)** 273-278.
29. Söderholm KJ, Zigan M, Ragan M, Fischlschweiger W, & Bergman M (1984) Hydrolytic degradation of dental composites *Journal of Dental Research* **63(10)** 1248-1254.
30. Featherstone JDB, White JM, Hoover CI, Rapozo-Hilo M, Weintraub JA, Wilson RS, Zhan L, & Gansky SA (2012) A randomized clinical trial of anticaries therapies targeted according to risk assessment (caries management by risk assessment) *Caries Research* **46(2)** 118-129.
31. Kuper NK, van de Sande FH, Opdam NJM, Bronkhorst EM, de Soet JJ, Cenci MS, & Huysmans MCDJNM (2015) Restoration materials and secondary caries using an in vitro biofilm model *Journal of Dental Research* **94(1)** 62-68.
32. de Moraes MDR, de Melo MAS, Bezerra DdS, Costa LS, Saboía VdPA, & Rodrigues LKA (2016) Clinical study of the caries-preventive effect of resin-modified glass ionomer restorations: Aging versus the influence of fluoride dentifrice *Journal of Investigative and Clinical Dentistry* **7(2)** 180-186.
33. do Amaral GS, Negrini T, Maltz M, & Arthur RA (2016) Restorative materials containing antimicrobial agents: Is there evidence for their antimicrobial and anticaries effects? A systematic review *Australian Dental Journal* **61(1)** 6-15.
34. Nedeljkovic I, Teughels W, De Munck J, Van Meerbeek B, & Van Landuyt KL (2015) Is secondary caries with composites a material-based problem? *Dental Materials* **31(11)** e247-e277.

©Operative Dentistry, 2018, **43-1,** 60-70

# Three-Year Effects of Deproteinization on the *In Vitro* Durability of Resin/Dentin-Eroded Interfaces

FSF Siqueira • AFM Cardenas • GM Gomes • AC Chibinski • OMM Gomes
MC Bandeca • AD Loguercio • JC Gomes



### Clinical Relevance

The use of NaOCl at the interface of resin and eroded dentin may be a viable alternative to minimize the degradation of the etch-and-rinse and self-etch resin–dentin interfaces.


## SUMMARY

**Objective: To evaluate the effect of sodium hypochlorite on the immediate and three-year bonding properties of a resin-eroded dentin interface produced by one of two adhesive strategies.**

**Methods and Materials: Forty-eight molars were randomly assigned to six experimental groups, according to the combination of the adhesive strategy (etch-and-rinse and self-etch) and the dentin surface (control groups without erosion, eroded dentin surface [ED], and eroded dentin surface + NaOCl 5.2% [ED + NaOCl]). After completing restoration, specimens were stored in water (37°C) for 24 hours and then sectioned into resin–dentin beams (0.8 $mm^2$) to be tested under tension (0.5 mm/min) immediately thereafter or after three years of water storage. To assess nanoleakage (NL), specimens were immersed in silver nitrate solution and examined by scanning electron microscopy at both time points. The dentin-etching pattern was examined under a scanning electron microscope. Data were subjected to appropriate statistical analysis ($\alpha$=0.05)**

**Results: In both strategies, a more pronounced and significant reduction of the microtensile**


Fabiana Suelen Figueredo de Siqueira, DDS, MS, PhD student, Department of Restorative Dentistry, School of Dentistry, Ponta Grossa State University, Ponta Grossa, Brazil

Andres Felipe Millan Cardenas, DDS, MS, PhD, professor, Department of Postgraduate Program in Dentistry, CEUMA University, São Luis, Brazil

Giovana Mongruel Gomes, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Ponta Grossa State University, Ponta Grossa, Brazil

Ana Claúdia Rodriguez Chibinski, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Ponta Grossa State University, Ponta Grossa, Brazil

Osnara Maria Mongruel Gomes, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Ponta Grossa State University, Ponta Grossa, Brazil

Matheus Coelho Bandeca, DDS, MS, PhD, professor, Department of Postgraduate Program in Dentistry, CEUMA University, São Luis, Brazil

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

João Carlos Gomes, DDS, MS, PhD, professor, Department of Restorative Dentistry, School of Dentistry, Ponta Grossa State University, Ponta Grossa, Brazil

*Corresponding author: Avenida Carlos Cavalcanti, 4748 Uvaranas, Ponta Grossa, Paraná 84030-900, Brazil; e-mail: aloguercio@hotmail.com

DOI: 10.2341/16-308-L

**bond strength (μTBS) values was observed for the ED groups ($p$=0.0001) after three years. However, in the ED + NaOCl group, μTBS values were maintained after three years of water storage. Furthermore, application of NaOCl to eroded dentin significantly reduced the immediate NL values and also preserved these values after three years of water storage for both adhesive strategies ($p$>0.05). When considering the ED group, a superficial removal of the smear layer and enlarged lumen tubules in comparison to control were present. However, for ED + NaOCl, there was a total removal of the smear layer and significant numbers of collagen fibrils were exposed.**

**Conclusion: The use of NaOCl may maintain the long-term stability of a resin-eroded dentin interface formed by etch-and-rinse and self-etch adhesives.**

## INTRODUCTION

Dental erosion is characterized by the loss of tooth structure due to the action of acids of nonbacterial origin in the short and long term. The prevalence of dental erosion has been increasing due to the increased consumption of acidic drinks, soft drinks, wine, and tea.[1,2] Although erosion wear is typically restricted to the enamel,[3,4] it can result in dentin exposure over time if the etiological factors are not controlled,[5] leading to the necessity for restorative treatment.[6]

An eroded dentin surface has characteristics that differ from those of sound dentin.[7,8] Erosion can remove dentinal plugs and organic intertubular dentin, resulting in increased tubule diameters. In addition, the eroded dentin surfaces have more exposed collagen fibrils and a reduced mineral content in the outermost layer,[7,8] which represent additional challenges for dental adhesion. Furthermore, the superficial layer of exposed collagen is often inadequately infiltrated by resin monomers.[7] When the hybrid layer formed during restoration involves incomplete infiltration by adhesive, the durability of the bonding interface may be jeopardized.[9-11]

Although only a few alternatives for improving the longevity of eroded dentin have been tested,[7,12,13] none of them have evaluated the removal of collagen fibrils by a deproteinization substance.[14-17] Sodium hypochlorite (NaOCl) solution is the substance most commonly evaluated for its nonspecific deproteinizing effect.[18] It is capable of producing chemical alterations that change the structure of demineralized dentin, such as altering the dentin composition to make it more similar to enamel (hydroxyapatite rich).[19] In addition, removal of unsupported collagen could have a beneficial effect on the spread and diffusion of primers and adhesives throughout the dentin[20] and may provide a more permeable substrate.

To date, the long-term stability of resin-eroded dentin interfaces after application of NaOCl has not been examined. The aim of the present study was therefore to compare the dentin microtensile bond strength (μTBS) and the nanoleakage (NL) after application of sodium hypochlorite both in the short term (immediately) and in the long term (after three years) and the dentin-etching pattern. The null hypotheses tested were that pretreatment with NaOCl solution would not influence 1) the immediate and three-year μTBS or 2) the immediate and three-year NL in eroded dentin.

## METHODS AND MATERIALS

### Selection and Preparation of Teeth

Forty-eight extracted, caries-free human molars were used. The ethics committee of the local university approved this research project (protocol no. 667.240). The teeth were disinfected in 0.5% chloramine, stored in distilled water, and used within six months after extraction.

The teeth were sectioned parallel to the occlusal surface using a low-speed diamond saw (Isomet, Buehler, Evanston, IL, USA) under water cooling to expose the mid-coronal dentin. All specimens had a smear layer standardized by polishing the flat dentin surface with 600-grit SiC paper under running water for 60 seconds.

### Experimental Design

The teeth were then randomly assigned to six groups (42 dentin specimens to μTBS and NL and 6 to the dentin-etching pattern) based on the combination of the main variables, that is, the adhesive strategies and the dentin surface. The adhesive strategies used were an etch-and-rinse adhesive (Adper Single Bond 2, 3M ESPE, St Paul, MN, USA) and a self-etch adhesive (Scotchbond Universal Adhesive, 3M ESPE [also known as Single Bond Universal in some countries]) (Table 1). The dentin surfaces tested included a control group without erosion, an eroded dentin surface (ED), and an eroded dentin surface treated with NaOCl 5.2% (ED + NaOCl). In the control group, after preparation, all teeth were ready for restoration. In the other groups, an erosive protocol was performed prior to restoration.

Table 1: *Adhesive System (Batch Number), Composition, and Application Mode*

| Adhesive System/Manufacturer (Batch Number) | Composition | Groups | Application Mode |
|---|---|---|---|
| Adper Single Bond 2 (ER) 3M ESPE, St Paul, MN, USA (395127BR) | Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and poly (itaconic) acids,10% by weight of 5-nm-diameter spherical silica particles | Control | 1. 37% phosphoric acid (Condac, FGM Dental Products, Joinville, SC, Brazil) for 15 s<br>2. Rinsing for 15 s and air-drying for 30 s<br>3. Apply two to three consecutive coats of adhesive for 15 s with gentle agitation<br>4. Gently air-dry for 5 s.<br>5. Light cure for 10 s at 1200 mW/cm$^2$ |
| | | Eroded dentin (ED) | |
| | | Eroded dentin + NaOCl 5.2% (ED + NaOCl) | 1. 37% phosphoric acid (Condac) for 15 s<br>2. Actively apply NaOCl 5.2% for 40 s<br>3. Rinsing for 15 s and air-drying for 30 s<br>4. Apply two to three consecutive coats of adhesive for 15 s with gentle agitation<br>5. Gently air-dry for 5 s<br>6. Light cure for 10 s at 1200 mW/cm$^2$ |
| Scotchbond Universal (SE) 3M ESPE(504834) | MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane | Control | 1. Apply the adhesive with a microbrush and rub it in for 20 s[a]<br>2. Gently air-dry for 5 s<br>3. Light cure for 10 s at 1200 mW/cm$^2$ |
| | | Eroded dentin (ED) | |
| | | Eroded dentin + NaOCl 5.2% (ED + NaOCl) | 1. Actively apply NaOCl for 40 s, washing, and drying<br>2. Apply the adhesive with a microbrush and rub it in for 20 s[a]<br>3. Gently air-dry for 5 s<br>4. Light cure for 10 s at 1200 mW/cm$^2$ |

*Abbreviations: Bis-GMA, bisphenolglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, methacryloyloxydecyldihydrogen phosphate.*
[a] *The materials were applied according to the recommendations of their respective manufacturers.*

## Erosive Protocol

For eight days, teeth were exposed to erosive cycles by immersion in a demineralization solution of citric acid for five minutes, followed by immersion in a remineralization solution for 3.5 hours, repeated six times per day; teeth were then rinsed with demineralized water.[7] The pH of the solutions was monitored periodically with the aid of a pH meter. The compositions of the demineralization and remineralization solutions are listed in Table 2.

## Restorative Procedures

All step-by-step bonding procedures are described in Table 1. Composite resin buildups (Filtek Z350, 3M ESPE) were constructed incrementally (three increments of 1.5 mm each) and each increment light activated for 40 seconds using a light-emitting-diode light-curing unit set at 1200 mW/cm$^2$ (Radii, SDI, Bayswater, Australia). A single operator carried out all bonding procedures. Seven teeth were used for each experimental group.

After storage in distilled water at 37°C for 24 hours, the specimens were sectioned longitudinally in mesial-to-distal and buccal-to-lingual directions across the bonded interface, using a low-speed diamond saw (Isomet) to obtain resin–dentin bonded sticks with a cross-sectional area of approximately 0.8 mm$^2$ as measured with digital calipers (Digimatic Caliper, Mitutoyo, Tokyo, Japan). The number of sticks showing premature failure during specimen preparation was recorded for each tooth. The bonded sticks originating from the same tooth were randomly assigned for immediate testing or

Table 2: *Composition of Demineralization and Remineralization Solution (pH Cycling)*[a]

| Solution (at 37°C) | Composition |
|---|---|
| Demineralization | 0.5% citric acid with pH of 3.5 |
| Remineralization | 0.004 g ascorbic acid; 1.16 g NaCl; 0.34 g $CaCl_2$; 0.32 g $NH_4Cl$; 2.54 g KCl; 0.38 g NaSCN; 0.64 g $KH_2PO_4$; 0,64 g $Na_2HPO_4$ dissolved in 1 l of demineralized water; pH is set to 6.4 with HCl |

[a] *Adapted in accordance with Zimmerli.*

testing after three years of storage in distilled water at 37°C. The distilled water was changed monthly.[21]

### Microtensile Bond Strength

Resin–dentin bonded sticks were attached to a Geraldeli jig[22] with cyanoacrylate adhesive and tested under tension (Kratos Dinamometros, Cotia, Brazil) at 0.5 mm/min until failure. The μTBS values (MPa) were calculated by dividing the load at failure by the cross-sectional bonding area.

The failure mode of the resin–dentin bonded sticks was classified as cohesive (C; failure exclusively within the dentin or the resin composite) or adhesive/mixed (A/M; failure at the resin–dentin interface or failure at the resin–dentin interface with partial cohesive failure of the neighboring substrates). This classification was performed under a stereomicroscope at 100× magnification (SZ40, Olympus, Tokyo, Japan). Specimens with premature failures were not included in the tooth mean for statistical analysis.

### Nanoleakage Evaluation

Three bonded sticks from each tooth that were not used in the microtensile test were evaluated for nanoleakage at each time point. Ammoniacal silver nitrate was prepared according to the protocol previously described by Tay and others.[23] The sticks were placed in ammoniacal silver nitrate solution in the dark 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 to metallic silver grains within spaces along the bonded interface. Specimens were polished with wet 600-, 1000-, 1200-, 1500-, 2000-, and 2500-grit SiC paper and 1- and 0.25-mm diamond paste (Buehler) using a polishing cloth. They were ultrasonically cleaned, air-dried, mounted on stubs, and coated with carbon-gold (MED 010, Balzers Union, Balzers, Liechtenstein). Resin–dentin interfaces were analyzed in a field-emission scanning electron microscope operated in the backscattered mode (LEO 435 VP, LEO Electron Microscopy Ltd, Cambridge, UK).

Three images of each resin–dentin bonded stick were captured.[24] The relative percentage of NL within the adhesive and hybrid layers in each specimen was measured in all images using the UTHSCSA ImageTool 3.0 software (Department of Dental Diagnostic Science, University of Texas Health Science Center, San Antonio, TX, USA) by a blinded researcher. The mean NL of all sticks from the same tooth was averaged for statistical purposes.

### Dentin-Etching Pattern: Occlusal and Lateral Surfaces

For this part of the study, six teeth were used. A flat dentin surface was exposed on each tooth after wet grinding the occlusal enamel with 180-grit SiC paper. The enamel-free, exposed dentin surfaces were further polished with 600-grit silicon-carbide paper for 60 seconds to standardize the smear layer. The crowns of teeth were longitudinally sectioned in a buccal-to-lingual direction with a water-cooled low-speed diamond saw (Isomet). After that, each third was transversely sectioned in a buccal-to-lingual direction to obtain three slices per tooth (n=18 specimens). The unique difference was that for lateral analysis of the dentin-etching pattern, in half the teeth a precut groove was made on the pulpal side to allow segmentation.

Specimens from each tooth were divided according to control group without erosion, ED, and ED + NaOCl. Then the surfaces were rinsed with tap water for 30 seconds and air-dried with an air spray for five seconds, keeping the dentin wet. The specimens were fixed in 2.5% glutaraldehyde in 0.1 M sodium cacodylate buffer at pH 7.4 for 12 hours at 4°C, rinsed with 20 mL (outer diameter) of 0.2 M sodium cacodylate buffer at pH 7.4 for one hour, and dehydrated in ascending grades of ethanol: 25% (20 minutes), 50% (20 minutes), 75% (20 minutes), 95% (30 minutes), and 100% (60 minutes).[25]

The specimens were segmented and sputter coated with gold-palladium in a vacuum evaporator (SCD 050, Balzers). The entire surface was examined under a scanning electron microscope (MIRA3 LM, Tescan Orsay Holding, Warrendale, PA, USA). Three photomicrographs of representative surface

Table 3: *Number of Specimens According to Fracture Pattern Mode for Each Group*[a]

| | Etch-and-Rinse Adhesive | | Self-Etch Adhesive | |
|---|---|---|---|---|
| | Immediate | 3 y | Immediate | 3 y |
| Control | 51/2/0 | 59/0/3 | 62/3/1 | 58/1/3 |
| ED | 50/0/3 | 62/1/6 | 57/0/3 | 61/2/5 |
| ED + NaOCl | 58/2/2 | 59/1/5 | 63/0/1 | 59/1/3 |

[a] *The numbers represent the number of bonded sticks that showed adhesive-mixed/cohesive/premature failures for all experimental conditions.*

areas were taken at 2500× and 20,000× magnification.

### Statistical Analysis

The experimental unit in the current study was the hemitooth since half the sample was tested immediately and the other half after three years. The μTBS (MPa) and NL (%) values of all sticks from the same tooth were averaged for statistical purposes. The μTBS and NL means for every test group were the average of the seven hemiteeth used per group. Data were compared by three-way repeated measures analysis of variance (adhesive strategies vs. surface treatment vs storage time) and the Tukey *post hoc* test for pairwise comparisons ($\alpha$=0.05).

## RESULTS

### Microtensile Bond Strength

The majority of specimens were classified as having had adhesive/mixed failures (93.6%). A low percentage of cohesive failures (1.54%) in the dentin and the resin composite occurred for both adhesives after three years. Although the number of specimens with pretest failures increased after three years, it did not reach statistical significance for any of the tested conditions (Table 3, data not shown).

The cross-product interaction was statistically significant ($p$=0.0001; Table 4). The control group for the etch-and-rinse adhesive showed the highest μTBS values. On the other hand, for self-etch adhesive, higher μTBS values were observed for the eroded dentin after NaOCl application ($p$=0.0001; Table 4).

In the long term, for both adhesives, a more pronounced and significant reduction in the μTBS values were observed in the ED groups ($p$=0.0001; Table 4). However, when NaOCl was applied to the eroded dentin, the μTBS values were maintained after the three-year water storage period for both adhesive strategies tested ($p$>0.05; Table 4).

### Nanoleakage Evaluation

The cross-product interaction was statistically significant ($p$=0.001; Table 5). When immediate NL values were observed, statistically significant differences in NL values were observed for the ED groups in both adhesive approaches as compared to control groups ($p$=0.001; Table 5). The application of NaOCl to the eroded dentin significantly diminished the immediate values of NL ($p$=0.001; Table 5) and also preserved the NL values after three years of water storage ($p$>0.05; Table 5). Application of NaOCl to the eroded dentin showed similar NL values to the control group for both adhesive strategies evaluated ($p$>0.05; Table 5).

Figure 1 shows scanning electron microscopic images representative of NL for all groups after immediate and three years. The amount of NL was lower and practically limited to the hybrid layer in the control group and the ED + NaOCl group. For the ED group, in contrast, the amount of NL was higher, with silver nitrate uptake occurring virtually throughout the entire thickness of the hybrid layer.

### Dentin-Etching Pattern: Occlusal and Lateral Surfaces

Scanning electron microscopic images of the dentin surface of different groups are shown in Figure 2. For the control group (A and B), an irregular smear layer was observed on the dentin surface without any change in the tubular diameter. For the lateral view (C), it was possible to observe the presence of a smear layer in the lumen of the tubule. For the ED group, superficial demineralization of dentin was found with

Table 4: *Mean Values (MPa) and Standard Deviations of Microtensile Bond Strength for Each Experimental Group*[a]

| | Etch-and-Rinse Adhesive | | Self-Etch Adhesive | |
|---|---|---|---|---|
| | Immediate | 3 y | Immediate | 3 y |
| Control | 53.9 ± 3.4 A | 45.7 ± 5.9 B | 32.5 ± 2.0 D | 30.1 ± 5.8 DE |
| ED | 35.0 ± 3.7 C | 26.2 ± 6.1 EF | 34.5 ± 5.4 CD | 21.4 ± 5.4 F |
| ED + NaOCl | 33.4 ± 4.4 CD | 27.3 ± 6.1 DE | 42.9 ± 3.3 B | 40.2 ± 3.1 B |

[a] *Different letters show statistically significant differences (p≤0.05).*

Table 5: *Mean Values (%) and Standard Deviations of Nanoleakage (NL) for Each Experimental Group*[a]

| | Etch-and-Rinse Adhesive | | Self-Etch Adhesive | |
|---|---|---|---|---|
| | Immediate | 3 y | Immediate | 3 y |
| Control | 6.5 ± 2.5 A | 12.3 ± 2.9 AB | 9.3 ± 2.7 AB | 11.3 ± 3.6 AB |
| ED | 21.3 ± 3.3 C | 43.8 ± 4.5 D | 40.9 ± 2.9 D | 46.4 ± 4.0 D |
| ED + NaOCl | 11.9 ± 2.9 AB | 14.2 ± 2.8 B | 10.5 ± 2.5 AB | 12.1 ± 3.0 AB |

[a] *Different letters show statistically significant differences ($p \leq 0.05$).*

absence of dentinal plugs, enlarged tubules, and a fibrous intertubular collagen network (A and B). The lateral view revealed the presence of a higher portion of the collagen fibril layer collapsed, and the border of the demineralized and the mineralized tissue (C). For the ED + NaOCl group, it was evident that the majority of the collagen network covering the intertubular dentin and the peritubular dentin was removed. This was also observed in the lateral view (C). An enlargement in the tubular diameter was also noted when compared to ED group (A and B).

## DISCUSSION

The composition of eroded dentin differs structurally and chemically from that of sound dentin because of the mineral/organic composition.[26,27] Eroded dentin is characterized by dissolution of the mineral component and the presence of a zone comprising a dense fibrous collagen network.[28] The presence of this highly disorganized collagen on the surface hinders adequate infiltration of the adhesive into the underlying dentin, affecting the quality of bonding.[29,30]

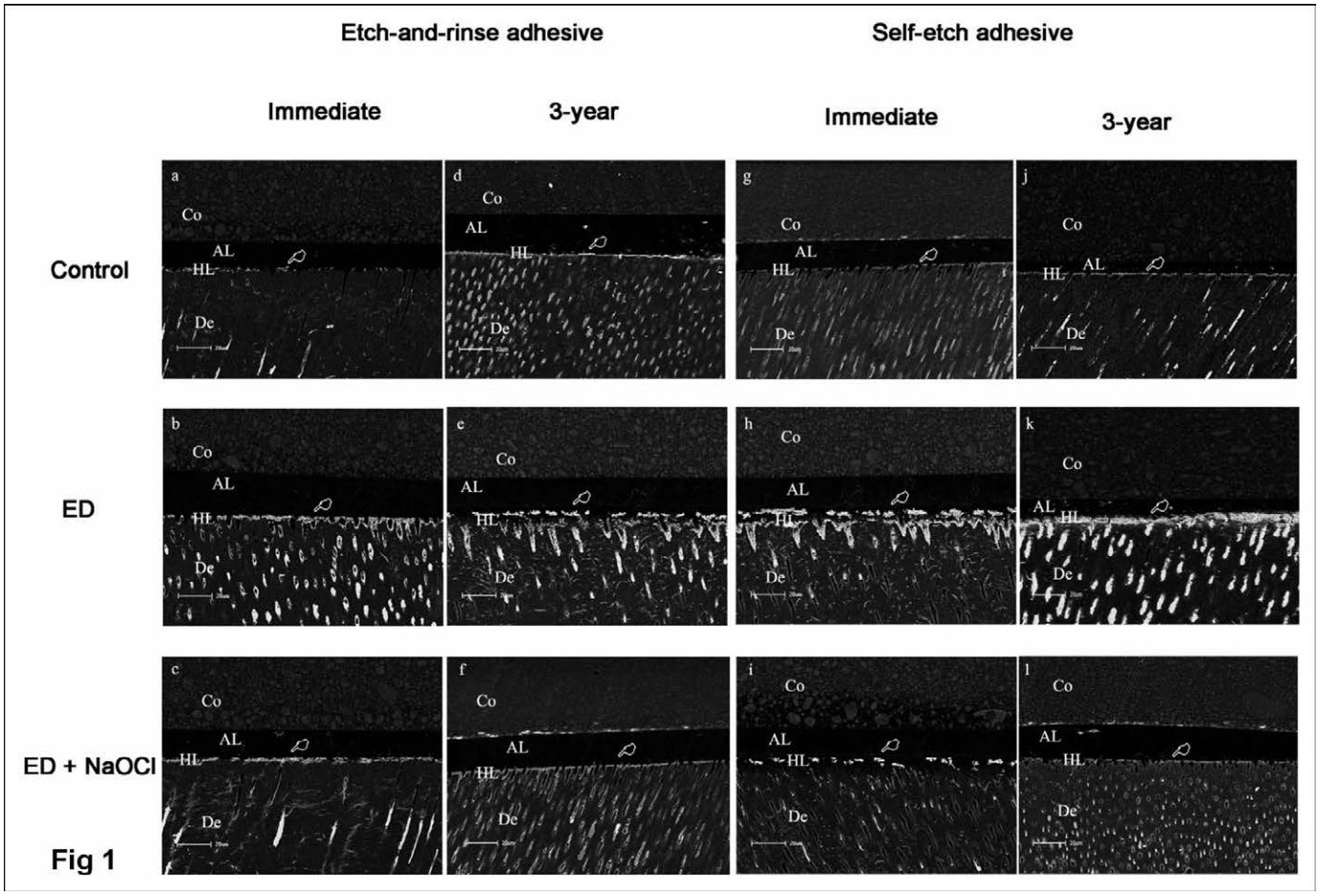



Figure 1. *Backscatter scanning electron microscopic micrographs of the adhesive interface of the experimental groups. Silver nitrate deposit (white hands) in all groups is mainly within the hybrid layer. However, after three years of the water storage, this deposition was more pronounced in ED (e and k) when compared to control (d and j) and ED + NaOCl (f and l). Co, composite resin; AL, adhesive layer; HL, hybrid layer; De, dentin.*

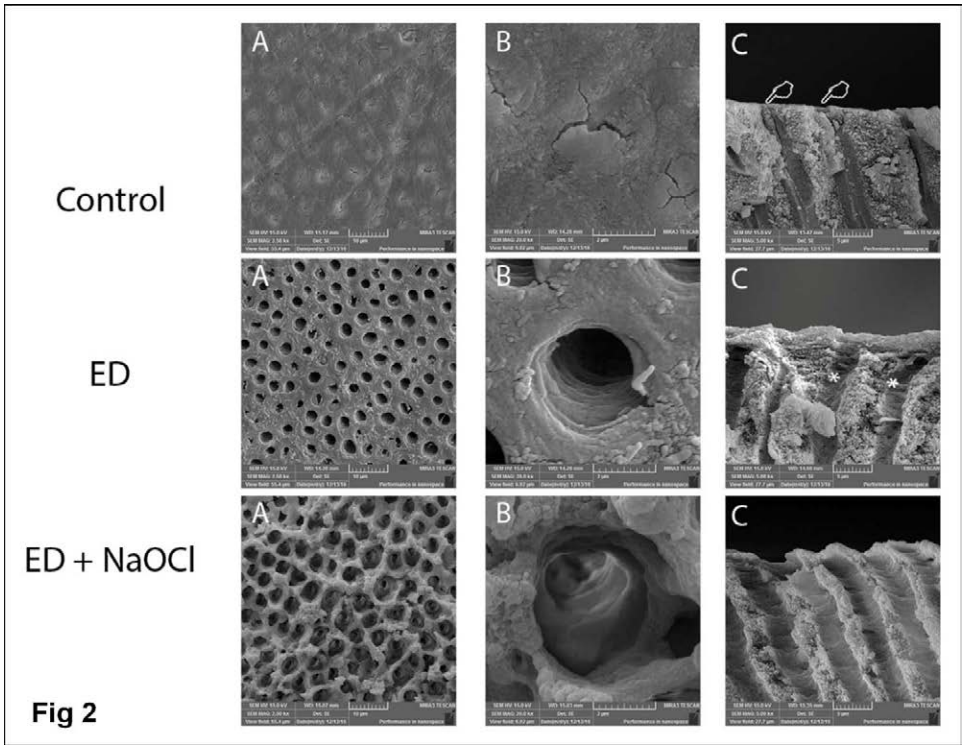



Figure 2. *Scanning electron microscopic images of the dentin surface of the experimental groups. For the control group, a smear layer is observed covering all surfaces (A and B). It was possible to observe the presence of a smear layer in the lumen of the tubule (white arrows in C). For the ED group, decalcified organic matrix was found with enlarged lumen tubules (A and B). The lateral view revealed the presence of a higher portion of the collagen fibril layer collapsed and the border of the demineralized and the mineralized tissue (asterisk in C). For the ED + NaOCl group, the removal of the majority of collagen network covering the intertubular dentin and the peritubular dentin occurred, this was also observed in the lateral view (A-C).*

In the present study, the appropriate hybridization of resin with the deeply eroded dentin surface, in the absence of NaOCl treatment, appeared difficult regardless of the adhesive strategy used, as confirmed by the significantly reduced μTBS values and increased NL after three years of water storage. These findings are in accordance with those of Zimmerli and others.[7] In their study, after one year of water storage, μTBS values were significantly reduced for an eroded surface.[7] This can most likely be attributed to the collapse of the demineralized collagen fibrils and to the higher water content, which prevented the adhesive from infiltrating fully as well as polymerizing properly. Such inefficient hybridization enhanced NL and consequently accelerated bond degradation.[7,9,10]

It is known that dentin erosion can anticipate or potentiate proteolytic activity in dentin, similar to the process of carious lesion progression.[31-34] Metalloproteinases represent a family of zinc- and calcium-dependent endopeptidases present in the dentin and saliva that are capable of degrading extracellular matrix components, including collagen, in their native and denatured forms.[35] These enzymes are exposed and activated when dentin is solubilized.[35] This may also have contributed to the decrease in μTBS values after three years of water storage in the eroded dentin groups. Thus, the poor infiltration of demineralized collagen and the increased activity of the metalloproteinases in eroded dentin may have contributed to increased NL both immediately and after three years of storage.

On the other hand, when use of NaOCl was incorporated into the bonding protocol, the immediate and three-year performance of resin-eroded dentin interfaces was maintained or improved, leading to rejection of both null hypotheses. Sodium hypochlorite is a nonspecific proteolytic agent that effectively promotes dissolution of collagen and proteoglycans, thus facilitating the intertubular and intratubular infiltration of resin, which minimizes the degradation of the denuded collagen matrix.[18,36-38] For this reason, NaOCl is usually indicated for use with an etch-and-rinse adhesive, particularly after applying the etch acid.[18,36-38] The use of NaOCl also significantly alters the mineral content of dentin, leaving a smear layer of mineralized tissue, which increases the Ca/P ratio on the dentin surface,[39,40] and yielding a dentin surface with characteristics similar to those of enamel.

However, it is noteworthy that the bonding efficacy to eroded dentin did not depend on the adhesive strategies applied when NaOCl was used in the bonding protocol. The etch-and- rinse adhesive maintained effective bonding at the resin-eroded dentin interface after three years of water aging. Similarly, when the self-etch adhesive was used,

although the performance was slightly better than that of the etch-and-rinse adhesive, the bond strength was also maintained after three years of water storage if NaOCl solution was first applied to the eroded dentin.

It has been reported that when NaOCl solution was applied to a smear layer–covered dentin, the mineral ratio increased and the smear layer became thinner due to dissolution of its collagen portion.[41] This NaOCl-treated smear layer with fewer organic components may improve the bonding performance of adhesive systems.[42-44]

However, it is worth mentioning that a significant reduction of immediate μTBS for the etch-and-rinse adhesive was observed when compared to the control group. The etch-and-rinse adhesive requires an acid pre-etching with phosphoric acid to demineralize the 6- to 9-μm surface of the intertubular dentin and create porosities within the underlying collagen fibrillary matrix.[45-47] It is known that NaOCl is able to remove the majority of collagen fibrils[18,36,37] and transform a demineralized substrate to a more mineralized and porous substrate. Thus, we hypothesized that the lower amount of intertubular dentin created by the acid etching following the NaOCl pretreatment promoted the lower μTBS. Furthermore, the excessive etching associated with the erosion, before the acid etching and NaOCl pretreatment, caused deeper demineralization of the intertubular and peritubular dentin, which challenged complete infiltration by the etch-and-rinse adhesive.[48-50]

On the other hand, the NaOCl pretreatment is capable of partially removing the organic components and thinning the smear layer, as observed in the scanning electron microscopic images.[41,51] This might enhance the bonding performance of the self-etch adhesive system.[44,52] A thinner smear layer with a reduced organic component might promote the infiltration self-etch adhesives into the smear layer and the underlying dentin, leading to higher bond strengths.[43,44]

In addition, the compositional difference between adhesives may play an important role in adhesive performance. The self-etching adhesive used in the present study contained a 10-methacryloyloxydecyl dihydrogen phosphate monomer capable of chemically interacting with hydroxyapatite by forming a stable nanolayered adhesive interface.[53-55] Furthermore, it contains a polyalkenoic acid copolymer in its formulation. Although these molecules may compete by binding to the calcium in hydroxyapatite,[54] they are usually associated with improved adhesive performance,[56,57] which may explain the good performance of this adhesive in the present study, both immediately and after three years of storage, in eroded dentin to which NaOCl was applied.

The slight increase in NL after three years of water storage in the control group and eroded dentin after application of NaOCl can be attributed to polymer degradation. Over time, water sorption and swelling of polymers facilitate leaching of hydrophilic monomers within the adhesive blends,[58,59] thus contributing to an increase in the number of spaces within the polymer network[24,58] and exposing additional collagen fibrils to activated MMPs.

Although the results observed in the present study are promising, when associating ethylenediaminetetraacidic acid and NaOCl or other chemical agents, promising results are usually shown in terms of bond strength to dentin.[60,61] However, Dikmen and others[60] and Chauhan and others[61] evaluated different chemical agents in noneroded dentin. Future studies are needed to compare the association of different chemical agents with NaOCl in the adhesive performance of eroded dentin.

Even considering the limitations of the *in vitro* methodology, which only partially simulated intraoral conditions, the long-term benefits of deproteinization presented herein are encouraging and can justify this as an additional step in clinical practice. However, the benefits of NaOCl may need verification by longitudinal clinical trials.

## CONCLUSION

The use of NaOCl at the interface of resin and eroded dentin minimized the degradation of the etch-and-rinse and self-etch resin–dentin interfaces after three years of water storage.

#### Acknowledgement

This study was partially supported by the Conselho Nacional de Desenvolvimento Científico e Tecnológico(CNPq) under grants 304104/2013-9 and 305588/2014-1.

#### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the State University of Ponta Grossa. The approval code for this study is 667.240/2102.

#### 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 23 January 2017)*

## REFERENCES


1. Jaeggi T, & Lussi A (2014) Prevalence, incidence and distribution of erosion *Monographs in Oral Science* **25** 55-73, DOI: 10.1159/000360973.
2. Schlueter N, & Tveit AB (2014) Prevalence of erosive tooth wear in risk groups *Monographs in Oral Science* **25** 74-98, DOI: 10.1159/000359938.
3. Murakami C, Oliveira LB, Sheiham A, Nahas Pires Correa MS, Haddad AE, & Bonecker M (2011) Risk indicators for erosive tooth wear in Brazilian preschool children *Caries Research* **45(2)** 121-129, DOI: 10.1159/000324807.
4. Dugmore CR, & Rock WP (2004) The prevalence of tooth erosion in 12-year-old children *British Dental Journal* **196(5)** 279-282; discussion 273, DOI: 10.1038/sj.bdj.4811040.
5. Jaeggi T, Gruninger A, & Lussi A (2006) Restorative therapy of erosion *Monographs in Oral Science* **20** 200-214, DOI: 10.1159/000093364.
6. Wang X, & Lussi A (2010) Assessment and management of dental erosion *Dental Clinics of North America* **54(3)** 565-578, DOI: 10.1016/j.cden.2010.03.003.
7. Zimmerli B, De Munck J, Lussi A, Lambrechts P, & Van Meerbeek B (2012) Long-term bonding to eroded dentin requires superficial bur preparation *Clinical Oral Investigations* **16(5)** 1451-1461, DOI: 10.1007/s00784-011-0650-8.
8. Prati C, Montebugnoli L, Suppa P, Valdre G, & Mongiorgi R (2003) Permeability and morphology of dentin after erosion induced by acidic drinks *Journal of Periodontology* **74(4)** 428-436, DOI: 10.1902/jop.2003.74.4.428.
9. Hashimoto M, Ohno H, Sano H, Kaga M, & Oguchi H (2003) Degradation patterns of different adhesives and bonding procedures *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **66(1)** 324-330, DOI: 10.1002/jbm.b.10010.
10. Sano H, Ciucchi B, Horner JA, Matthews WG, & Pashley DH (1995) Nanoleakage: Leakage within the hybrid layer. *Operative Dentistry* **20(1)** 18-25.
11. Vaidyanathan TK, & Vaidyanathan J (2009) Recent advances in the theory and mechanism of adhesive resin bonding to dentin: A critical review *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **88(2)** 558-578, DOI: 10.1002/jbm.b.31253.
12. Francisconi-dos-Rios LF, Casas-Apayco LC, Calabria MP, Francisconi PA, Borges AF, & Wang L (2015) Role of chlorhexidine in bond strength to artificially eroded dentin over time *Journal of Adhesive Dentistry* **17(2)** 133-139, DOI: 10.3290/j.jad.a34059.
13. Ramos TM, Ramos-Oliveira TM, de Freitas PM, Azambuja N Jr, Esteves-Oliveira M, Gutknecht N, & de Paula Eduardo C (2015) Effects of Er:YAG and Er,Cr:YSGG laser irradiation on the adhesion to eroded dentin *Lasers in Medical Science* **30(1)** 17-26, DOI: 10.1007/s10103-013-1321-6.
14. Pimenta LA, Amaral CM, Bedran de Castro AK, & Ritter AV (2004) Stability of dentin bond strengths using different bonding techniques after 12 months: Total-etch, deproteinization and self-etching *Operative Dentistry* **29(5)** 592-598.
15. Toledano M, Perdigao J, Osorio R, & Osorio E (2000) Effect of dentin deproteinization on microleakage of class V composite restorations *Operative Dentistry* **25(6)** 497-504.
16. Perdigao J, Lopes M, Geraldeli S, Lopes GC, & Garcia-Godoy F (2000) Effect of a sodium hypochlorite gel on dentin bonding *Dental Materials* **16(5)** 311-323.
17. Barbosa de Souza F, Silva CH, Guenka Palma Dibb R, Sincler Delfino C, & Carneiro de Souza Beatrice L (2005) Bonding performance of different adhesive systems to deproteinized dentin: Microtensile bond strength and scanning electron microscopy *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **75(1)** 158-167, DOI: 10.1002/jbm.b.30280.
18. Marshall GW, Yücel N, Balooch M, Kinney JH, Habelitz S, & Marshall SJ (2001) Sodium hypochlorite alterations of dentin and dentin collagen *Surface Science* **491(3)** 444-445.
19. Tanaka J, & Nakai H (1993) Application of root canal cleaning agents having dissolving abilities of collagen to the surface treatment for enhanced bonding of resin to dentin *Dental Materials Journal* **12(2)** 196-208.
20. Prati C, Chersoni S, & Pashley DH (1999) Effect of removal of surface collagen fibrils on resin-dentin bonding *Dental Materials* **15(5)** 323-331.
21. Skovron L, Kogeo D, Gordillo LA, Meier MM, Gomes OM, Reis A, & Loguercio AD (2010) Effects of immersion time and frequency of water exchange on durability of etch-and-rinse adhesive *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **95(2)** 339-346, DOI: 10.1002/jbm.b.31718.
22. 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.
23. 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.
24. 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, DOI: 10.1016/j.dental.2006.05.005.
25. Kenshima S, Francci C, Reis A, Loguercio AD, & Filho LE (2006) Conditioning effect on dentin, resin tags and hybrid layer of different acidity self-etch adhesives applied to thick and thin smear layer *Journal of Dentistry* **34(10)** 775-783, DOI: 10.1016/j.jdent.2006.03.001.
26. Lussi A, Schlueter N, Rakhmatullina E, & Ganss C (2011) Dental erosion—An overview with emphasis on chemical and histopathological aspects *Caries Reserach* **45(Supplement 1)** 2-12, DOI: 10.1159/000325915.
27. Zavgorodniy AV, Rohanizadeh R, & Swain MV (2008) Ultrastructure of dentine carious lesions *Archives of Oral Biology* **53(2)** 124-132, DOI: 10.1016/j.archoralbio.2007.08.007.

28. Breschi L, Gobbi P, Mazzotti G, Falconi M, Ellis TH, & Stangel I (2002) High resolution SEM evaluation of dentin etched with maleic and citric acid *Dental Materials* **18(1)** 26-35.

29. Pashley DH, Ciucchi B, Sano H, & Horner JA (1993) Permeability of dentin to adhesive agents *Quintessence International* **24(9)** 618-631.

30. Wang Y, & Spencer P (2002) Analysis of acid-treated dentin smear debris and smear layers using confocal Raman microspectroscopy *Journal of Biomedical Materials Research* **60(2)** 300-308.

31. Hebling J, Pashley DH, Tjaderhane L, & Tay FR (2005) Chlorhexidine arrests subclinical degradation of dentin hybrid layers in vivo *Journal of Dental Research* **84(8)** 741-746.

32. Tjaderhane L, Buzalaf MA, Carrilho M, & Chaussain C (2015) Matrix metalloproteinases and other matrix proteinases in relation to cariology: The era of "dentin degradomics" *Caries Research* **49(3)** 193-208, DOI: 10.1159/000363582.

33. Nascimento FD, Minciotti CL, Geraldeli S, Carrilho MR, Pashley DH, Tay FR, Nader HB, Salo T, Tjaderhane L, & Tersariol IL (2011) Cysteine cathepsins in human carious dentin *Journal of Dental Research* **90(4)** 506-511, DOI: 10.1177/0022034510391906.

34. Komori PC, Pashley DH, Tjaderhane L, Breschi L, Mazzoni A, de Goes MF, Wang L, & Carrilho MR (2009) Effect of 2% chlorhexidine digluconate on the bond strength to normal versus caries-affected dentin *Operative Dentistry* **34(2)** 157-165, DOI: 10.2341/08-55.

35. Carrilho MR, Tay FR, Donnelly AM, Agee KA, Tjaderhane L, Mazzoni A, Breschi L, Foulger S, & Pashley DH (2009) Host-derived loss of dentin matrix stiffness associated with solubilization of collagen *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **90(1)** 373-380, DOI: 10.1002/jbm.b.31295.

36. Toledano M, Osorio R, Perdigao J, Rosales JI, Thompson JY, & Cabrerizo-Vilchez MA (1999) Effect of acid etching and collagen removal on dentin wettability and roughness *Journal of Biomedical Materials Research* **47(2)** 198-203.

37. de Castro AK, Hara AT, & Pimenta LA (2000) Influence of collagen removal on shear bond strength of one-bottle adhesive systems in dentin *Journal of Adhesive Dentistry* **2(4)** 271-277.

38. Inai N, Kanemura N, Tagami J, Watanabe LG, Marshall SJ, & Marshall GW (1998) Adhesion between collagen depleted dentin and dentin adhesives *American Journal of Dentistry* **11(3)** 123-127.

39. Baumgartner JC, & Mader CL (1987) A scanning electron microscopic evaluation of four root canal irrigation regimens *Journal of Endodontics* **13(4)** 147-157.

40. Baumgartner JC, & Cuenin PR (1992) Efficacy of several concentrations of sodium hypochlorite for root canal irrigation *Journal of Endodontics* **18(12)** 605-612, DOI: 10.1016/S0099-2399(06)81331-2.

41. Mountouris G, Silikas N, & Eliades G (2004) Effect of sodium hypochlorite treatment on the molecular composition and morphology of human coronal dentin *Journal of Adhesive Dentistry* **6(3)** 175-182.

42. Montagner AF, Skupien JA, Borges MF, Krejci I, Bortolotto T, & Susin AH (2015) Effect of sodium hypochlorite as dentinal pretreatment on bonding strength of adhesive systems *Indian Journal of Dental Research* **26(4)** 416-420, DOI: 10.4103/0970-9290.167633.

43. Fawzy AS, Amer MA, & El-Askary FS (2008) Sodium hypochlorite as dentin pretreatment for etch-and-rinse single-bottle and two-step self-etching adhesives: Atomic force microscope and tensile bond strength evaluation *Journal of Adhesive Dentistry* **10(2)** 135-144.

44. Taniguchi G, Nakajima M, Hosaka K, Iwamoto N, Ikeda M, Foxton RM, & Tagami J (2009) Improving the effect of NaOCl pretreatment on bonding to caries-affected dentin using self-etch adhesives *Journal of Dentistry* **37(10)** 769-775, DOI: 10.1016/j.jdent.2009.06.005.

45. Pashley DH, Tay FR, Breschi L, Tjaderhane L, Carvalho RM, Carrilho M, & Tezvergil-Mutluay A (2011) State of the art etch-and-rinse adhesives *Dental Materials* **27(1)** 1-16, DOI: 10.1016/j.dental.2010.10.016.

46. Yousry MM (2012) Effect of re-etching oxalate-occluded dentin and enamel on bonding effectiveness of etch-and-rinse adhesives *Journal of Adhesive Dentistry* **14(1)** 31-38, DOI: 10.3290/j.jad.a22744.

47. Kanca J III (1992) Resin bonding to wet substrate. 1. Bonding to dentin *Quintessence International* **23(1)** 39-41.

48. Perdigao J (2001) The effect of etching time on dentin demineralization *Quintessence International* **32(1)** 19-26.

49. Hashimoto M, Ohno H, Endo K, Kaga M, Sano H, & Oguchi H (2000) The effect of hybrid layer thickness on bond strength: Demineralized dentin zone of the hybrid layer *Dental Materials* **16(6)** 406-411.

50. 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.

51. Montes MA, de Goes MF, & Sinhoreti MA (2005) The in vitro morphological effects of some current pre-treatments on dentin surface: A SEM evaluation *Operative Dentistry* **30(2)** 201-212.

52. Hiraishi N, Kitasako Y, Nikaido T, Nomura S, Burrow MF, & Tagami J (2003) Effect of artificial saliva contamination on pH value change and dentin bond strength *Dental Materials* **19(5)** 429-434.

53. 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, DOI: 10.1016/j.actbio.2010.03.024.

54. 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, DOI: 10.1177/0022034512437375.

55. 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.

56. Perdigao J, Sezinando A, & Monteiro PC (2013) Effect of substrate age and adhesive composition on dentin bonding *Operative Dentistry* **38(3)** 267-274, DOI: 10.2341/12-307-L.
57. 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, DOI: 10.1016/j.dental.2010.10.023.
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, DOI: 10.1016/j.dental.2007.02.009.
59. 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, DOI: 10.1177/0022034510391799.
60. Dikmen B, Gurbuz O, Ozsoy A, Eren MM, Cilingir A, & Yucel T (2015) Effect of different antioxidants on the microtensile bond strength of an adhesive system to sodium hypochlorite-treated dentin *Journal of Adhesive Dentistry* **17(6)** 499-504, DOI: 10.3290/j.jad.a35257.
61. Chauhan K, Basavanna RS, & Shivanna V (2015) Effect of bromelain enzyme for dentin deproteinization on bond strength of adhesive system *Journal of Conservative Dentistry* **18(5)** 360-363, DOI: 10.4103/0972-0707.164029.

©Operative Dentistry, 2018, **43-1,** 71-80

# Effect of Light Activation of Pulp-Capping Materials and Resin Composite on Dentin Deformation and the Pulp Temperature Change

CJ Soares • MS Ferreira • AA Bicalho • M de Paula Rodrigues • SSL Braga • A Versluis



### Clinical Relevance

Light curing of pulp-capping materials caused deformation of pulpal dentin and increased pulpal temperature by light curing and exotherm effects. The use of Vitrebond or Ultra-Blend Plus in deep cavities resulted in better performance through a combination of high degree of cure, lower temperature, and dentin strain.


## SUMMARY

**Objectives: To analyze the effect of pulp-capping materials and resin composite light activation on strain and temperature development in the pulp and on the interfacial integrity at the pulpal floor/pulp-capping materials in large molar class II cavities.**

**Methods: Forty extracted molars received large mesio-occlusal-distal (MOD) cavity bur preparation with 1.0 mm of dentin remaining at the pulp floor. Four pulp-capping materials (self-etching adhesive system, Clearfil SE Bond [CLE], Kuraray), two light-curing calcium hydroxide cements (BioCal [BIO], Biodinâmica, and Ultra-Blend Plus [ULT], Ultradent), and a resin-modified glass ionomer cement– (Vitrebond [VIT], 3M ESPE) were applied on the pulpal floor. The cavities were incrementally restored with resin composite (Filtek Z350 XT, 3M ESPE). Thermocouple (n=10) and strain gauge (n=10) were placed inside the pulp chamber in contact with the top of the pulpal floor to detect temperature changes and dentin strain during light curing of the pulp-capping materials and during resin composite restoration. Exotherm was calculated by subtracting postcure from polymerization temperature (n=10). Interface integrity at the pulpal**



*Carlos José Soares, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil

Mariana Santos Ferreira, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil

Aline Aredes Bicalho, Health Technical School, Federal University of Uberlândia, Minas Gerais, Brazil

Monise de Paula Rodrigues, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil.

Stella Sueli Lourenço Braga, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Minas Gerais, Brazil

Antheunis Versluis, Bioscience Research, University of Tennessee Health Science Center, Memphis, TN, USA

*Corresponding author: Av. Pará, no. 1720, CEP 38400-902, Bloco 4LA, Uberlândia, Minas Gerais, Brazil; e-mail: carlosjsoares@umuarama.ufu.br


DOI: 10.2341/16-325-L

**floor was investigated using micro-CT (Sky-Scan 1272, Bruker). The degree of cure of capping materials was calculated using the Fourier transform infrared and attenuated total reflectance cell. Data were analyzed using one-way analysis of variance followed by the Tukey test ($\alpha$=0.05).**

**Results: Pulpal dentin strains (μs) during light curing of CLE were higher than for other pulp-capping materials ($p<0.001$). During resin composite light activation, the pulpal dentin strain increased for ULT, VIT, and CLE and decreased for BIO. The pulpal dentin strain was significantly higher during pulp-capping light activation. The temperature inside the pulp chamber increased approximately 3.5°C after light curing the pulp-capping materials and approximately 2.1°C after final restoration. Pulp-capping material type had no influence temperature increase. The micro-CT showed perfect interfacial integrity after restoration for CLE and ULT; however, gaps were found between BIO and pulpal floor in all specimens. BIO had a significantly lower degree of conversion than ULT, VIT, and CLE.**

**Conclusions: Light curing of pulp-capping materials caused deformation of pulpal dentin and increased pulpal temperature in large MOD cavities. Shrinkage of the resin composite restoration caused debonding of BIO from the pulpal floor.**

## INTRODUCTION

Advanced caries in posterior teeth frequently results in deep cavities that require protection of the dentin–pulp complex tissues. The capping procedure consists of application of a capping material in order to maintain pulp vitality, stimulate pulp and dentin repair, and minimize postoperative sensitivity.[1] Many current pulp-capping materials require light activation, which may result in thermal and shrinkage stress.[1] Subsequent resin composite light-curing activation will cause further thermal and shrinkage effects that may induce enamel microcracks and postoperative sensitivity due to dentin deformation.[2,3]

For deep cavities, the use of pulp capping is a common protocol to prevent postoperative sensitivity.[4] The pulp is a highly vascularized tissue that responds to thermomechanical stimuli.[5] To avoid tissue damage, pulpal temperature and deformation should be limited.[6] The pulp/dentin interface should therefore protect against thermal and electrical effects as well as maintain structural integrity during curing irradiation of restorative materials.[7,8] Various light-curing materials have been used for dentin capping, such as resin-modified glass ionomer cements, self-etching adhesive systems, and calcium hydroxide cement.[9-11]

Few studies have reported the effects of temperature rise and dentin deformation within the pulp chamber due to the curing reaction of the capping material. Therefore, the aim of this study was to evaluate the effect of the light-curing process of capping materials and resin composite on the dentin deformation and the pulp temperature change in deep class II mesio-occlusal-distal (MOD) restorations. The null hypothesis was that the capping material and resin composite filling technique had no effect on the dentin deformation and temperature change in the pulp chamber.

## METHODS AND MATERIALS

### Tooth Selection and Cavity Preparation

Eighty extracted intact caries-free human molars were used in this study (Ethics Committee in Human Research approval no. 06257012.1.0000.5152). The teeth were selected to have an intercuspal width within a maximum deviation of 10% from the determined mean.[12] The intercuspal width varied between 4.8 and 5.9 mm. The roots were sectioned 5.0 mm below the cementum–enamel junction using a precision saw (Isomet 1000, Buehler, Lake Bluff, IL, USA). The pulp chamber was manually accessed with a spherical diamond bur (#1016 HL, KG Sorensen, Barueri, SP, Brazil) in a high-speed hand piece with copious air–water spray, preventing damage to the pulp dentin above the pulp chamber (Figure 1). To simulate bone support, the teeth were embedded in a polystyrene resin (Cristal, Piracicaba, SP, Brazil) up to 2 mm below the cementum–enamel junction. The teeth were cleaned using a rubber cup and fine pumice water slurry. Class II MOD cavities with four-fifths of the intercuspal width and 4-mm depth were prepared using a cylindrical-with-round-end diamond bur (#3099, KG Sorensen) with copious air–water spray using a cavity preparation machine (Figure 1).[13] This machine consisted of a high-speed hand piece (Extra Torque 605 C, Kavo do Brasil, Joinville, SC, Brazil) coupled to a mobile base. The mobile base moves vertically and horizontally with three precision micrometric heads (152-389, Mitutoyo Sul Americana Ltda, Suzano, SP, Brazil), attaining a 2-μm level of accuracy (Figure 1). The 1.0-mm-thick remaining pulp dentin was verified using an X-ray image (Kodak Dental Systems, Carestream Health, Rochester, NY, USA).

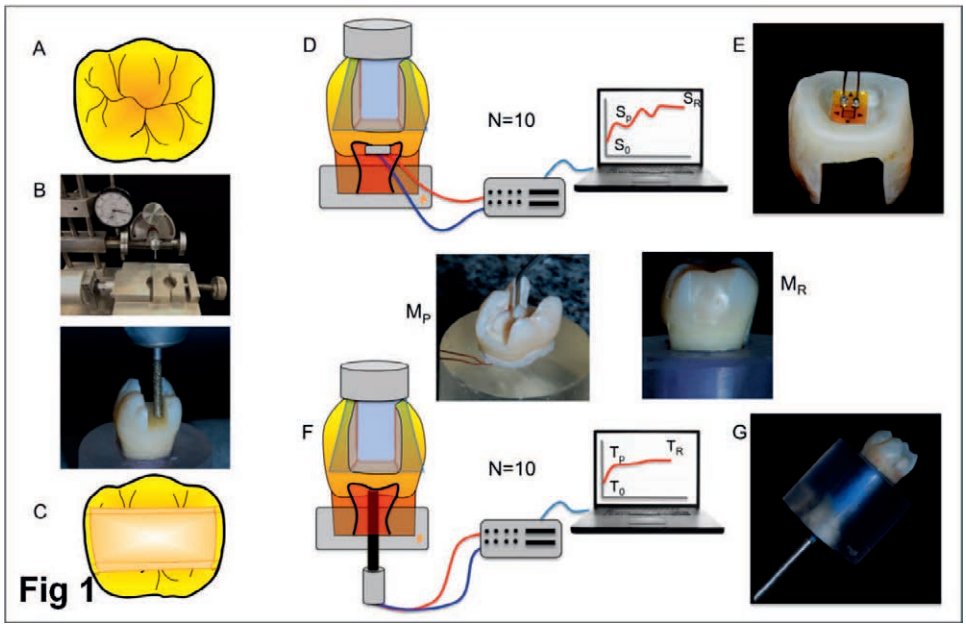



Figure 1. *Schematic diagram of study design. (A): Intact molar. (B): Cavity preparation machine. (C): Mesio-occlusal-distal deep cavity preparation. (D): Measurement of deformation of pulp floor dentin at $M_P$ (pulp capping) and $M_R$ (final restoration). (E): Strain gauge bonded to pulp inner dentin. (F): Measurement of pulpal dentin temperature at $M_P$ (pulp capping) and $M_R$ (final restoration). (G): Thermocouple inserted into the pulp chamber contacting the pulp inner dentin.*

### Temperature Rise Measurement

Forty of the prepared teeth were used for temperature rise measurement (n=10/group). A J-type thermocouple (5TC-TT-J-40-36, Alutal Siebeck Sensors, São Paulo, SP, Brazil) was inserted into the pulp chamber through a root perforation, maintaining contact with the pulp–dentin floor above the pulp chamber (Figure 1). The thermocouple was made of iron and constantan alloys (Cu 55%+Ni 45%) represented by two terminals: a positive pole with a white end and a negative pole with a red end. Externally, the thermocouple was coated with black rubber and an active circular tip (1.0-mm diameter). The J-type thermocouple could capture temperature variations ranging from −20°C to +800°C with a ±0.2°C accuracy. The thermocouple was connected to a data acquisition device (ADS2000IP, Lynx Technology, São Paulo, SP, Brazil). The data were transferred to a computer, using acquisition signal transformation by data analysis software (AqDados 7.02 and AqAnalisys, Lynx). The temperature value was recorded at 0.25-second intervals to detect temperature changes during the light irradiation of the pulp capping of four materials: self-etching adhesive system (Clearfil SE Bond [CLE], Kuraray, Osaka, Japan), light-curing calcium hydroxide cement (BioCal [BIO], Biodinâmica, Ibiporã, PR, Brazil), light-curing calcium hydroxide cement (Ultra-Blend Plus [ULT], Ultradent, South Jordan, UT, USA), and resin-modified glass ionomer cement (Vitrebond [VIT], 3M ESPE, St Paul, MN, USA). The capping material information is shown in Table 1. For the CLE group, the primer was applied followed by gentle air spray, then the adhesive was applied and irradiated for 20 seconds using a QTH light-curing unit with 550-mW/cm$^2$ output (Demetron 501, Kerr, Orange, CA, USA). For ULT, BIO, and VIT groups, the capping materials were manipulated and inserted in a 1.0-mm-thick layer and irradiated using the QTH light-curing unit. The MOD cavity was filled with a restorative resin composite (Filtek Z350 XT, 3M ESPE; see Table 1) using eight increments, each light cured for 20 seconds using the QTH light from the occlusal direction with the tip placed as close as possible to the cavity.

### Exotherm of Pulp-Capping Material

The exotherm temperatures of the light-cured capping materials were determined in a separate experiment, where the materials were placed directly on top of a custom thermocouple tip (n=10). To standardize the amount and contain the capping material, transparent adhesive tape (Durex, 3M ESPE) was placed around the 3.0-mm circular custom-made brass tip of a J-type thermocouple (5TC-TT-J-40-36, Alutal Siebeck Sensors), creating a 1-mm-tall "container" (Figure 2).[14] The capping material was inserted into the container, and the light-curing tip was positioned 1.0 mm above it. The capping material was irradiated for 20 seconds using the QTH light-curing unit. Temperature changes were recorded during the curing run and during a second 20-second irradiation run with the cured material. Exotherm temperature ($T_r$) was deter-

Table 1: *Restorative Materials Used in the Study*

| Material Type | Commercial Name | Manufacturer | Composition |
|---|---|---|---|
| Resin-modified glass ionomer | Vitrebond | 3M ESPE (St Paul, MN, USA) | Powder: fluoroaluminosilicate glass powder with $SiO_2$, $AlF_3$, ZnO, SrO, cryolite, $NH_4F$, MgO, and $P_2O_5$; liquid: modified polyacrylic acid with pendant methacrylate groups, HEMA, water, and photoinitiator |
| Light-curing calcium hydroxide cement | Ultra-Blend Plus | Ultradent Products, Inc (South Jordan, UT, USA) | Urethane dimethacrylate (58%), calcium hydroxide (10%) |
| Light-curing calcium hydroxide cement | BioCal | Biodinâmica (Ibiporã, PR, Brazil) | Calcium (3.53%), ethylene urethane dimethacrylate, inorganic fillers, barium sulfate, photoactivator, titanium dioxide, and iron oxide |
| Self-etching adhesive system | Clearfil SE Bond | Kuraray Medical, Inc (Okayama, Japan) | Primer: MDP, HEMA, hydrophilic dimethacrylate,N,N-diethanol-p-toluidine, water; bonding: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-camphorquinone,N,N-diethanol-p-toluidine, and silanated silicate |
| Nanofilled resin composite | Filtek Z350XT | 3M ESPE | Bis-GMA, Bis-EMA, UDMA, TEGDMA, silica and zirconia nanofillers, and agglomerated zirconia-silica nanoclusters |

*Abbreviations: Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate; Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; HEMA, 2-hydroxyethylmethacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.*

mined by subtracting the temperature rise of the second run ($T_{p1}$, which represented the thermal change due to irradiation only) from the first ($T_{La}$, which contained both irradiation and exotherm contributions): $T_r = T_{La} - T_{p1}$ (Figure 2).

## Pulpal Dentin Deformation

The other 40 prepared teeth were used for the determination of pulp–dentin deformation (n=10 per group). Pulp deformation was measured with strain gauges (PA-06-038AA-120LEN, Excel Sensores, Embú, SP, Brazil), which had an internal electrical resistance of 120 Ω, a gauge factor of 2.07, and a grid size of 1 $mm^2$. The strain gauge was attached to the pulpal dentin above the pulp chamber (Figure 1). For the strain gauge attachment, the teeth were sectioned 1 mm below the cemento–enamel junction using a precision saw (Isomet 1000, Buehler). The dentin located above the pulp chamber was etched with 37% phosphoric acid for 15 seconds, rinsed with an air–water spray, and air-dried. The strain gauge was fixed using cyanoacrylate adhesive (Super Bonder, Loctite, Gulph Mills, PA, USA) (Figure 1). The strain gauges were placed consistently in the buccal-lingual direction to standardize the strain measurement. In addition, a second strain gauge was fixed to another intact tooth to compensate for dimensional deviations due to temperature effects. The strain gauges were connected to a data acquisition device (ADS2000IP, Lynx). Strain values were recorded at 0.25-second intervals during the light irradiation of the pulp-capping materials and the restorative procedure.

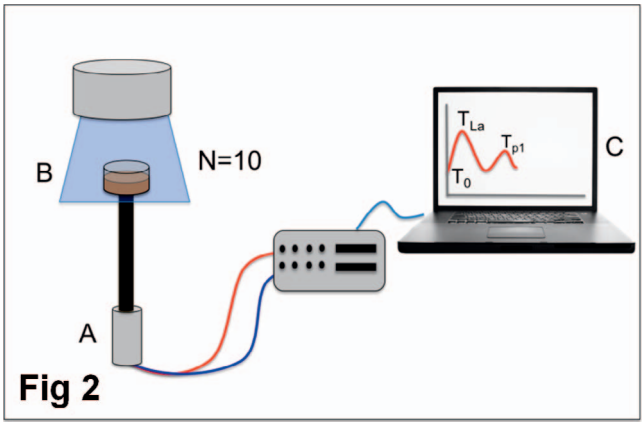



Figure 2. *Schematic diagram of test design to measure temperature increase and exotherm of capping materials. (A): Thermocouple. (B): Light-curing source positioned 1.0 mm from the capping material. (C): Temperature recorded at $T_0$ (temperature before the light activation). $T_{La}$, maximum temperature reached during the light activation of capping material; $T_{p1}$, temperature measured at two minutes after 20 seconds of light activation.*

## Pulp-Capping Material Interface Integrity

To verify the integrity between capping material and the pulp–dentin floor, micro-CT analyses were performed after the final restoration (SkyScan 1272, Bruker microCT, Kontich, Belgium). The crowns were mounted on a custom attachment and scanned in the micro-CT scanner. The X-ray scanner

contains an X-ray microsource, an X-ray detector (chamber), an object handle for positioning and rotating the sample during the tomographic acquisition, and associated electronics to power the X-ray source and the chamber. The scanner operated at 100 kV and 111 mA (0.11-mm Cu filter). The resolution used was 1224/820 pixels (15 μm). The scanning was performed by 180° rotation around the vertical axis with a rotation step of 0.5, average image acquisition 3, position height of 30 mm inside the apparatus, and 35 minutes of scanning time. Images of each specimen were reconstructed (NRecon version 1.6.10.1, Bruker microCT), providing axial cross sections of their inner structure, and the same gray ranking for all samples was selected with the standard minimum of 0 and a maximum of 0.19635. No image correction was performed. The reconstructed samples were saved in bitmap format. Then, using DataViewer software (Bruker microCT), all samples were visualized to check acquisition quality. CTAn version 1.13 software (Bruker microCT) was used for the three-dimensional (volume, surface area, and structure model index) evaluation. CTVol version 2.0 software (Bruker microCT) was used for visualization and qualitative evaluation of the specimens.

### Degree of Conversion of Pulp-Capping Material

The degree of conversion (DC) of capping materials (n=10) was assessed using Fourier transform infrared (FTIR) spectroscopy (Vertex 70, Bruker Optik GmbH, Ettlingen, Germany) with attenuated total reflectance crystal sampling, mid-infrared, and deuterated triglycine sulfate detector elements (Bruker Optik). Capping material in a similar amount as used in the prepared tooth cavities was inserted on the crystal sensor of the FTIRs. The spectra were obtained between aromatic C=C bond stretching vibrations (1608 $cm^{-1}$) and aliphatic C=C bond stretching vibrations (1638 $cm^{-1}$), with 4-$cm^{-1}$ resolution and 32 scans coaddition. All analyses were performed under controlled temperature (25±1°C) and humidity (60%±5%) conditions. DC was calculated from the equivalent aliphatic (1638 $cm^{-1}$) aromatic (1608 $cm^{-1}$) molar ratios of cured (C) and uncured (U) CLE and BIO capping materials. For the VIT, the reference peak was the ester bonds (1712 $cm^{-1}$), and for the Ultra-Blend, the reference peak was the carbonyl (1720 $cm^{-1}$), due to the absence of the aromatic carbon bond. The formula to calculate the DC was $DC(\%) = (1 - C/U) \times 100$.

Table 2: *Means (Standard Deviation) of Maximum Temperature Change (°C) at Dentin Floor of the Pulp Chamber During Pulp Capping and Resin Composite Restoration Polymerization (n=10)*[a]

| Materials | Pulp/Dentin Capping | Cumulative at Final Restoration |
|---|---|---|
| BioCal/Z350XT (BIO) | 4.0 (0.4) A | 6.7 (0.8) A |
| Ultra-Blend Plus/Z350XT (ULT) | 3.9 (0.4) A | 6.6 (0.3) A |
| Vitrebond/Z350XT (VIT) | 3.8 (0.4) A | 6.2 (0.6) A |
| Clearfil SE Bond/Z350XT (CLE) | 6.4 (0.8) B | 8.8 (0.6) B |

[a] *Different letters in columns indicate significant difference among capping materials for each moment of measurement (p<0.05).*

### Statistical Analyses

The data of pulpal temperature rise, pulp dentin strain, exotherm temperature, and DC of capping materials were analyzed for normal distribution and homoscedasticity using the Shapiro–Wilk test and the Levene test, respectively. One-way analysis of variance (ANOVA), followed by the Tukey test, was used for comparing capping materials. All tests were performed at a significance level of $\alpha = 0.05$, and all analyses were performed using the Sigma Plot version 13.1 statistical package (Systat Software Inc, San Jose, CA, USA). The gap formation was analyzed descriptively.

## RESULTS

### Temperature Rise Measurement

The means and standard deviations of the maximum temperature change at the dentin floor for all groups are presented in Table 2. A significant effect was observed for interaction between capping material and temperature increase during the restoration ($p<0.001$). During the light activation of the capping material and the final restoration, the light curing of the CLE resulted in a higher temperature change than other materials ($p=0.021$). No difference was observed among BIO, ULT, and VIT ($p=0.712$). The temperature change was significantly higher during the light activation of pulp capping than during the incremental filling. The means and standard deviations of the temperature change variation measured during the resin composite filling only (calculated as final temperature change minus temperature change at pulp capping) for all groups are presented in Figure 3. No significant difference was observed in temperature change after light activation during the incremental filling procedure among the capping material groups ($p=0.321$).

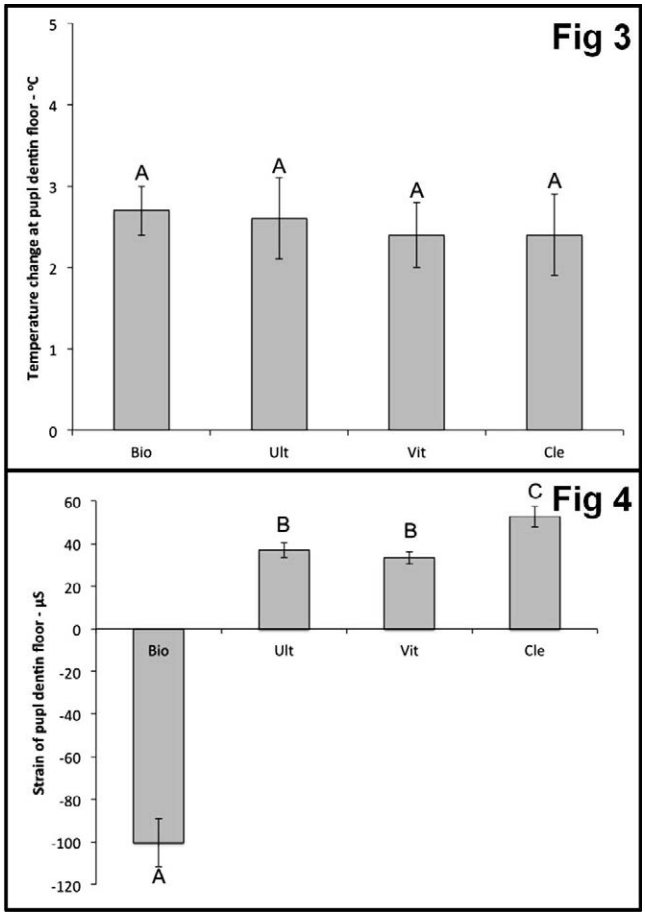



Figure 3. *Temperature changes at pulp dentin floor (°C) measured during resin composite placement (final temperature change – pulp capping temperature change). Different uppercase letters indicate significant difference among capping materials for each moment of measurement (*p*<0.05).*
Figure 4. *Strain values at pulp dentin floor (μs) measured during resin composite placement (total strain – pulp-capping strain values). Different uppercase letters indicate significant difference among capping materials (*p*<0.05).*

## Pulp–Dentin Deformation

The means and standard deviations of the maximum strain at the pulp–dentin floor for all groups are presented in Table 3. A significant effect was observed for interaction between capping material and moment of the restoration ($p<0.001$). During the light activation of the capping material, CLE resulted in higher strains at the pulp–dentin floor than the other materials ($p=0.010$). No difference was observed among BIO, ULT, and VIT ($p=0.124$). After the incremental filling, BIO had the lowest and CLE the highest strains at the pulp–dentin floor of all groups ($p<0.001$). No difference was found between ULT and VIT ($p=0.802$). The means and standard deviations of the strain in dentin due to the restoration filling only (final strain value minus strain values measured during pulp capping) for all groups are presented in Figure 4. BIO had the lowest and CLE the highest strain of all groups ($p<0.001$). No difference was found between ULT and VIT ($p=0.707$).

Table 3: *Means (Standard Deviation) of Maximum Strain (μs) in Dentin Floor of the Pulp Chamber During Pulp Capping and Resin Composite Restoration Polymerization (n=10)*[a]

| Materials | Pulp/Dentin Capping | Cumulative at Final Restoration |
|---|---|---|
| BioCal/Z350XT (BIO) | 183.9 (44.3) A | 83.5 (28.3) A |
| Ultra-Blend Plus/Z350XT (ULT) | 179.9 (55.4) A | 216.9 (45.7) B |
| Vitrebond/Z350XT (VIT) | 176.4 (56.3) A | 210.3 (42.1) B |
| Clearfil SE Bond/Z350XT (CLE) | 215.4 (60.6) B | 268.1 (46.6) C |

[a] *Different letters in columns indicate significant difference among capping materials for each moment of measurement (*p*<0.05).*

## Exotherm of Pulp-Capping Material

Temperatures increased during light activation. The means and standard deviations of the maximum peak temperature are shown in Table 4. One-way ANOVA showed significant differences among the temperature increases ($p<0.001$) and among the exotherm values ($p<0.001$). The CLE had significantly higher temperature increases than all other capping materials. No difference was found between ULT, BIO, and VIT regardless of the testing moment.

## Pulp-Capping Material Interface Integrity

The micro-CT images showed gaps between restoration and the pulp–dentin floor caused by detachment of the BIO (Figure 5A). Perfect integrity between the restoration and the pulp–dentin floor was observed for all other groups (Figure 5B,C,D).

Table 4: *Means (Standard Deviation) of Maximum Temperature Increases (°C) and Exotherm (Mean ± Standard Deviation) for Capping Materials (n=10)*[a]

| Capping Material | Temperature Increases (°C) | Exotherm (°C) |
|---|---|---|
| Clearfil SE Bond (CLE) | 29.0 (2.4) A | 16.8 (1.8) A |
| Ultra-Blend Plus (ULT) | 14.6 (1.2) B | 8.1 (1.8) B |
| Vitrebond (VIT) | 17.5 (3.3) B | 8.9 (3.6) B |
| BioCal (BIO) | 18.2 (2.0) B | 9.8 (2.4) B |

[a] *Different letters in columns indicate significant difference among capping materials for each moment of measurement (*p*<0.05).*

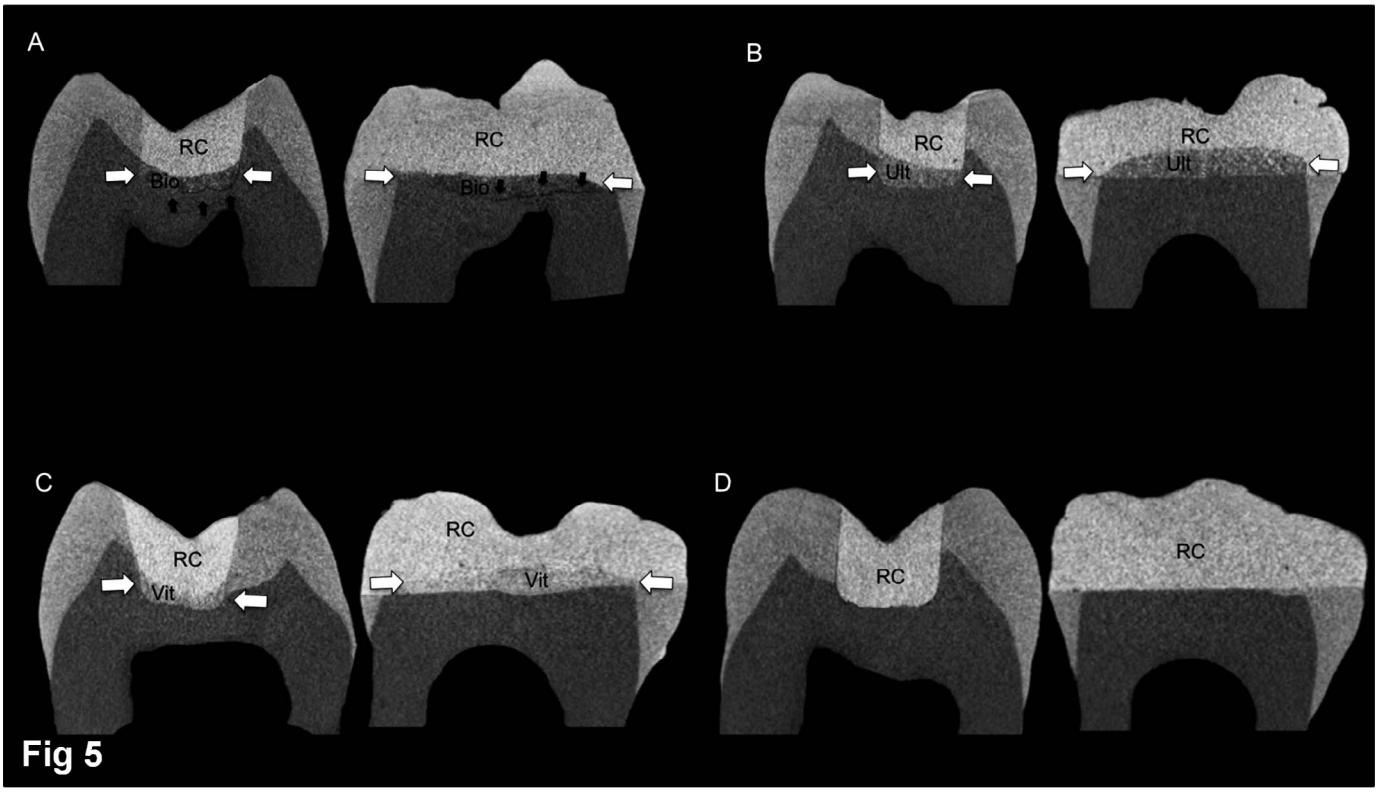



Figure 5. *Micro-CT images of the molar restorations. (A): BIO/Z350XT group (black arrow indicates gap between BioCal and pulp floor dentin). (B): ULT/Z350XT group. (C): VIT/Z350XT group and CLE/Z350XT group (white arrows on A, B, and C indicate pulp protection material layers).*

### Degree of Conversion of Pulp-Capping Material

The means and standard deviations of the degree of cure for all capping materials are shown in Figure 6. One-way ANOVA showed significant difference among the degree of cure ($p$=0.012). The BIO had a significantly lower degree of cure than all other capping materials. No difference was found among ULT, VIT, and CLE.

## DISCUSSION

The null hypothesis was rejected; the light activation of capping material and the resin composite incremental filling technique caused dentin deformation and temperature changes in the pulp chamber. The biomechanical behavior of restorative materials is important in understanding restorative procedures.[15] Light-curing materials produce temperature rises that are influenced by factors such as intensity of light, composition and transmission properties of resin composites, exotherm reaction, depth of the cavity and restoration, duration of light exposure, and type of light source.[16] Thermal stimuli applied externally have been shown to result in rapid development of strains before a change in temperature is detected at the pulp surface.[6]

Particularly in deep cavities, the amount of remaining dentin thickness, the type of pulp-capping materials, and their degree of cure can affect heat generation during restorative procedures, which could lead to irreversible pulpal damage.[17] Temperature increases originate from the exothermic reaction during the curing process of the material and the energy absorbed from the light-curing unit.[18] In this study, we measured the exotherm and the temperature increases during the application of the capping materials. A limitation of this study may be the absence of the pulpal fluid and pulpal pressure, which can reduce the effect of temperature rise inside the pulp chamber. Presence of pulpal fluid may also exacerbate the dentin strain because the humidity makes dentin more prone to deformation.[19]

In this study, we tested a resin-modified glass ionomer cement, a self-etching adhesive system, and two light-curing calcium hydroxide cements. The self-etching adhesive system proved to be the material that blocked the least amount of heat coming from the light used for the activation of the pulp-capping material, maybe because it was the thinnest layer.

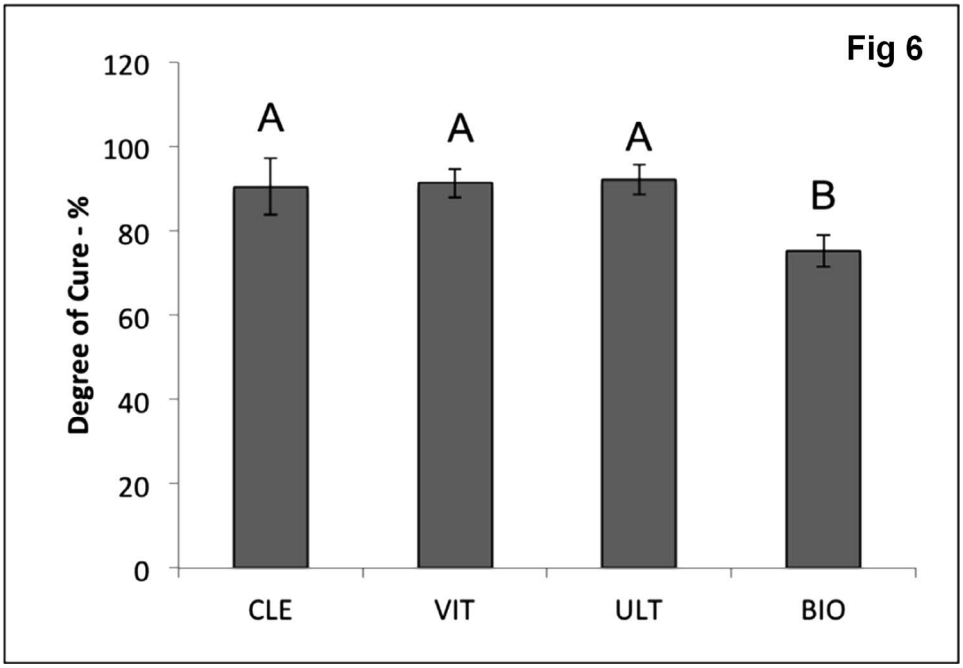



Figure 6. *Means and standard deviations for degree of cure (n=5) for capping materials. BIO, BioCal; ULT, Ultra-Blend Plus; VIT, Vitrebond; CLE, Clearfil SE Bond. Different uppercase letters indicate significant difference among capping materials (p<0.05).*

Additionally, the higher exotherm of the adhesive system may contribute to the higher temperature values at the pulp surface. Despite its popularity, the physical properties of conventional calcium hydroxide cement, such as water solubility, the absence of bond strength to dental hard tissues, and low compressive strength, have limited its use.[20] Light-curing pulp-capping materials have been developed to avoid such disadvantages and to extend the working time. The light-curing calcium hydroxide cements (BIO and ULT) and the resin-modified glass ionomer cement (VIT) had similar temperature increases when they were light activated and similar exotherms that were lower than the adhesive system. The thickness of these materials and their opacity may also help reduce the heat transfer during the following restoration procedure with the resin composite.

The cumulative temperature rises at the pulp–dentin floor during the incremental filling procedure showed that the final restoration temperature rise was similar for all the materials. This result indicates that the resin-modified glass ionomer cement or light-curing calcium hydroxide cement were not more effective in preventing pulp temperature increases than the adhesive system and that the capping material could not prevent the temperature rise in the pulp during polymerization of the resin composite. The use of the capping materials should thus be determined by the necessity to prevent biologic damage of pulp tissues and stimulate dentin and pulp repair.[21-23] Capping materials require a high degree of cure to prevent complications in pulp response.[24,25] Especially in deep cavities, the proximity of capping material to pulp tissue and the higher permeability of the deep dentin may result in free nonconverted monomers ending up in pulp tissues.[26] The BioCal, which is expected to be a "stimulating capping material" resulting in dentin neo-formation by calcium hydroxide composition, had a lower DC with 25% of the uncured monomers. This lower DC warrants further study for the use of this material in deep cavities due to a potential toxicity effect from uncured monomers.

Strain gauges were used for measuring the deformation inside the pulp. These deformations are caused by thermal and volumetric changes that take place in the capping materials and resin composite during polymerization. The higher deformation at the pulpal dentin wall that was found when the CLE was light activated can probably be attributed to the higher heating and thus higher thermal expansion. When we separately evaluated the variation of the strain values at the pulp–dentin floor during the resin composite placement, the BIO showed negative deformation values. These indicate compression force. Apparently, the material deformation was not transferred to the remaining dentin walls. The micro-CT images showed gaps between the restoration and the pulp–dentin floor caused by detachment of the BioCal at the final stage of the restoration. This may be explained by the high polymerization shrinkage of BIO that could have

caused detachment of the pulpal wall and the absence of adequate bonding interaction between BIO and dentin. Perfect integrity between the restoration and the pulp–dentin floor was observed for all other groups.

In this study, a QTH light was used for light activation of the materials. For the incremental filling in eight increments, each light cured for 20 seconds, the light curing resulted in 88 J/cm$^2$ of total energy delivered ($8\times20\times550\ 10^{-3}$). Irradiance and temperature values at the light guide tip vary per light-curing unit, and therefore temperatures at the pulpal floor are also likely to vary.[27,28] The choice of restorative resin composite (resin composite type and filling technique) is another variable that can affect the temperatures.[29] Since temperature rise and dissipation are affected by various interacting properties, further study is needed to determine the thermal response to other types of light-curing units and for restorative resin composites with different optical properties or filling techniques (eg, bulk-fill resin composites).

This study did not find significant differences in pulp–dentin deformation among all BIO, VIT, and ULT groups. No power analysis was performed to determine sample size, which was a limitation of this study. An increased sample size may be needed to detect potential differences among these groups. However, it should be noted that not all statistically significant differences are clinically relevant. From a clinical perspective, the present study demonstrated that capping material can reduce initial heating and strain on the pulpal dentin floor, thought to be the main cause of pulpal sensitivity. However, pulp-capping materials had no effect on temperature rise and deformation at the pulpal roof caused by the resin composite restoration. In general, the use of capping material is recommended only for deep cavities where the dentin remaining at the pulpal floor is not sufficient to prevent temperature rise and dentin strain.[23,30] The tested materials should be adequately polymerized to prevent future damage of pulp tissue by unreacted monomers.[20,31] The performance of BIO demonstrated that this material cannot be used in deep cavities. Therefore, when a capping material is necessary for deep cavities, a resin-modified glass ionomer may be the best option that combines good biocompatibility[9] and biomechanical performance.

## CONCLUSIONS

Within the limitations of this *in vitro* study, we can conclude that the light curing of pulp-capping materials caused pulp–dentin deformation and increased pulpal temperature. BioCal demonstrated a lower degree of cure and a lower capacity to maintain contact with the pulpal floor after shrinkage of the resin composite restoration. Vitrebond and Ultra-Blend Plus demonstrated a better combination of strains and temperature changes at the pulpal surface.

### Acknowledgements

This project was funded by grants from the CNPq—National Council for Scientific and Technological Development from CAPES and FAPEMIG. This research was performed at CPbio-Biomechanics, Biomaterials, and Cell Biology Research Center, Federal University of Uberlândia.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethics Committee in Human Research approval, Federal University of Uberlandia. The approval code for this study is 06257012.1.0000.5152.

### 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 7 March 2017)*

### REFERENCES

1. Schwendicke F, Brouwer F, Schwendicke A, & Paris S (2016) Different materials for direct pulp capping: Systematic review and meta-analysis and trial sequential analysis *Clinical Oral Investigations* **20(6)** 1121-1132.
2. Batalha-Silva S, de Andrada MA, Maia HP, & Magne P (2013) Fatigue resistance and crack propensity of large MOD resin composite restorations: Direct versus CAD/CAM inlays *Dental Materials* **29(3)** 324-331.
3. Burgess J, & Cakir D (2010) Comparative properties of low-shrinkage resin composites *Compendium of Continuing Education in Dentistry* **31(2)** 10-15.
4. Swift EJ Jr (2004) Causes, prevention, and treatment of dentin hypersensitivity *Compendium of Continuing Education in Dentistry* **25(2)** 95-106, 109; quiz 110.
5. Na S, Zhang H, Huang F, Wang W, Ding Y, Li D, & Jin Y (2016) Regeneration of dental pulp/dentine complex with a three-dimensional and scaffold-free stem-cell sheet-derived pellet *Jounal of Tissue Engineering and Regenerative Medicine* **10(3)** 261-270.
6. Linsuwanont P, Versluis A, Palamara JE, & Messer HH (2008) Thermal stimulation causes tooth deformation: A possible alternative to the hydrodynamic theory? *Archives of Oral Biology* **53(3)** 261-272.
7. Masutani S, Setcos JC, Schnell RJ, & Phillips RW (1988) Temperature rise during polymerization of visible light-activated composite resins *Dental Materials* **4(4)** 174-178.

8. Aggarwal V, Singla M, & Miglani S (2011) Effect of thermal and mechanical loading on marginal adaptation and microtensile bond strength of a self-etching adhesive with caries-affected dentin *Journal of Conservative Dentistry* **14(1)** 52-56.

9. Kotsanos N, & Arizos S (2011) Evaluation of a resin modified glass ionomer serving both as indirect pulp therapy and as restorative material for primary molars *European Archives of Paediatric Dentistry* **12(3)** 170-175.

10. Nowicka A, Buczkowska-Radlińska J, Lipski M, Lichota D, Woźniak K, Góra M, Kaczmarek W, & Sikorska-Bochińska J (2009) Response of dental pulp to self-etching adhesive systems used in indirect capping: A literature review *Annales Academiae Medicae Stetinensis* **55(1)** 79-83.

11. Lin M, Xu F, Lu TJ, & Bai BF (2010) A review of heat transfer in human tooth-experimental characterization and mathematical modeling *Dental Materials* **26(6)** 501-513.

12. Bicalho AA, Pereira RD, Zanatta RF, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2014) Incremental filling technique and composite material—Part I: Cuspal deformation, bond strength, and physical properties *Operative Dentistry* **39(2)** 71-82.

13. Soares CJ, Fonseca RB, Gomide HA, & Correr-Sobrinho L (2008) Cavity preparation machine for the standardization of in vitro preparations *Brazilian Oral Research* **22(3)** 281-287.

14. Armellin E, Bovesecchi G, Coppa P, Pasquantonio G, & Cerroni L (2016) LED curing lights and temperature changes in different tooth sites *Biomedical Research International*, DOI: 10.1155/2016/1894672

15. Rosatto CM, Bicalho AA, Veríssimo C, Bragança GF, Rodrigues MP, Tantbirojn D, Versluis A, & Soares CJ (2015) Mechanical properties, shrinkage stress, cuspal strain and fracture resistance of molars restored with bulk-fill composites and incremental filling technique *Journal of Dentistry* **43(12)** 1519-1528.

16. Guiraldo RDS, Consani S, Mastrofrancisco S, Consani RL, Sinhoreti MA, & Correr-Sobrinho L (2008) Influence of light curing unit and ceramic thickness on temperature rise during resin cement photo-activation *Bulletin of Tokyo Dental College* **49(4)** 173-178.

17. Wataha JC, Lockwood PE, Lewis JB, Rueggeberg FA, & Messer RL (2004) Biological effects of blue light from dental curing units *Dental Materials* **20(2)** 150-157.

18. Francisconi LF, de Freitas AP, Scaffa PM, Mondelli RF, & Francisconi PA (2009) Water sorption and solubility of different calcium hydroxide cements *Journal of Applied Oral Science* **17(5)** 427-431.

19. Bicalho AA, de Souza SJ, de Rosatto CM, Tantbirojn D, Versluis A, & Soares CJ (2015) Effect of temperature and humidity on post-gel shrinkage, cusp deformation, bond strength and shrinkage stress—Construction of a chamber to simulate the oral environment *Dental Materials* **31(12)** 1523-1532.

20. Savas S, Botsali MS, Kucukyilmaz E, & Sari T (2014) Evaluation of temperature changes in the pulp chamber during polymerization of light-cured pulp-capping materials by using a VALO LED light curing unit at different curing distances *Dental Materials Journal* **33(6)** 764-769.

21. Murray PE, Smith AJ, Garcia-Godoy F, & Lumley PJ (2008) Comparison of operative procedure variables on pulpal viability in an ex vivo model *International Endodontic Journal* **41(5)** 389-400.

22. Hirschman WR, Wheater MA, Bringas JS, & Hoen MM (2012) Cytotoxicity comparison of three current direct pulp-capping agents with a new bioceramic root repair putty *Journal of Endodontics* **38(3)** 385-388.

23. Costa CA, Ribeiro AP, Giro EM, Randall RC, & Hebling J (2011) Pulp response after application of two resin modified glass ionomer cements (RMGICs) in deep cavities of prepared human teeth *Dental Materials* **27(7)** e158-e170.

24. Caughman WF, Caughman GB, Shiflet RA, Rueggeberg F, & Schuster GS (1991) Correlation of cytotoxicity, filler loading and curing time of dental composites *Biomaterials* **12(8)** 737-740.

25. Salehi S, Gwinner F, Mitchell JC, Pfeifer C, & Ferracane JL (2015) Cytotoxicity of resin composites containing bioactive glass fillers *Dental Materials* **31(2)** 195-203.

26. Thonemann B, Schmalz G, Hiller KA, & Schweikl H (2002) Responses of L929 mouse fibroblasts, primary and immortalized bovine dental papilla-derived cell lines to dental resin components *Dental Materials Journal* **18(4)** 318-323.

27. Gomes M, DeVito-Moraes A, Francci C, Moraes R, Pereira T, Froes-Salgado N, Yamazaki L, Silva L, & Zezell D Temperature increase at the light guide tip of 15 contemporary LED units and thermal variation at the pulpal floor of cavities: An infrared thermographic analysis *Operative Dentistry* **38(3)** 324-333.

28. Mouhat M, Mercer J, Stangvaltaite L, & Örtengren U (2016) Light-curing units used in dentistry: Factors associated with heat development—Potential risk for patients *Clinical Oral Investigation* Oct 1. Epub ahead of print.

29. Andreatta LM, Furuse AY, Prakki A, Bombonatti JF, & Mondelli RF (2016) Pulp chamber heating: An in vitro study evaluating different light sources and resin composite layers *Brazilian Dental Journal* **27(6)** 675-680.

30. Hilton TJ (2009) Keys to clinical success with pulp capping: A review of the literature *Operative Dentistry* **34(5)** 615-625.

31. Chen L, & Suh BI (2017) Cytotoxicity and biocompatibility of resin-free and resin-modified direct pulp capping materials: A state-of-the-art review *Dental Materials Journal* **36(1)** 1-7.

©Operative Dentistry, 2018, **43-1,** 81-89

# The Effects of Cavity Preparation and Composite Resin on Bond Strength and Stress Distribution Using the Microtensile Bond Test

SSL Braga • LRS Oliveira • RB Rodrigues • AA Bicalho • VR Novais
S Armstrong • CJ Soares



### Clinical Relevance

The biomechanical performance of composite resins is strongly influenced by the type of cavity preparation used. Using flat surface preparation to test different composite types may underestimate the effect of C-factor, stress, and shrinkage of composite resins.

## SUMMARY

**Objectives: To evaluate the effect of flowable bulk-fill or conventional composite resin on bond strength and stress distribution in flat or mesio-occlusal-distal (MOD) cavity preparations using the microtensile bond strength (μTBS) test.**

**Methods: Forty human molars were divided into two groups and received either standardized MOD or flat cavity preparations. Restorations were made using the conventional composite resin Z350 (Filtek Z350XT, 3M-ESPE, St Paul, MN, USA) or flowable bulk-fill (FBF) composite resin (Filtek Bulk Fill Flowable, 3M-ESPE). Postgel shrinkage was measured using the strain gauge technique (n=10). The Z350 buildup was made in two increments of 2.0 mm, and the FBF was made in a single increment of 4.0 mm. Six**

Stella Sueli Lourenço Braga, DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlandia, Minas Gerais, Brazil

Laís Rani Sales Oliveira Schliebe, DDS, MSc, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlandia, Minas Gerais, Brazil

Renata Borges Rodrigues, DDS, MS, PhD student, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlandia, Minas Gerais, Brazil

Aline Arêdes Bicalho, DDS, MS, PhD, professor, Health Technical School, Federal University of Uberlandia, Minas Gerais, Brazil

Veridiana Resende Novais, DDS, MS, PhD, professor, Department of Operative Dentistry and Dental Materials, School of Dentistry, University of Uberaba, Minas Gerais, Brazil

Steve Armstrong, DDS, PhD, professor and chair, Department of Operative Dentistry, College of Dentistry, University of Iowa, Iowa City, IA, USA

*Carlos José Soares, DDS, MS, PhD, professor and chair, Department of Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlandia, Minas Gerais, Brazil

*Corresponding author: Avenida Pará, 1720, Bloco 4L, Anexo A, Sala 42, Campos Umuarama, Uberlândia–Minas Gerais, Brazil CEP 38400-902; e-mail: carlosjsoares@ufu.br

DOI: 10.2341/16-338-L

**rectangular sticks were obtained for each tooth, and each section was used for μTBS testing at 1.0 mm/min. Polymerization shrinkage was modeled using postgel shrinkage data. The μTBS data were analyzed statistically using a two-way analysis of variance (ANOVA), and the postgel shrinkage data were analyzed using a one-way ANOVA with Tukey post hoc test. The failure modes were analyzed using a chi-square test ($\alpha$=0.05).**

**Results: Our results show that both the type of cavity preparation and the composite resin used affect the bond strength and stress distribution. The Z350 composite resin had a higher postgel shrinkage than the FBF composite resin. The μTBS of the MOD preparation was influenced by the type of composite resin used. Irrespective of composite resin, flat cavity preparations resulted in higher μTBS than MOD preparations ($p<0.001$). Specifically, in flat-prepared cavities, FBF composite resin had a similar μTBS relative to Z350 composite resin. However, in MOD-prepared cavities, those with FBF composite resin had higher μTBS values than those with Z350 composite resin. Adhesive failure was prevalent for all tested groups. The MOD preparation resulted in higher shrinkage stress than the flat preparation, irrespective of composite resin. For MOD-prepared cavities, FBF composite resin resulted in lower stress than Z350 composite resin. However, no differences were found for flat-prepared cavities.**

**Conclusions: FBF composite resin had lower shrinkage stress than Z350 conventional composite resin. The μTBS of the MOD preparation was influenced by the composite resin type. Flat cavity preparations had no influence on stress and μTBS. However, for MOD preparation, composite resin with higher shrinkage stress resulted in lower μTBS values.**

## INTRODUCTION

Research in the field of adhesive dentistry strives for improvements in composite restorations to increase restoration longevity in tooth-preserving operative procedures. The use of a variety of composite resins with different mechanical properties makes it difficult to analyze the stress distribution at the interface between the tooth and the restoration.[1] Several variables affect the mechanical behavior of the restorations to be studied; therefore, a systematic understanding of the distribution of stress patterns involved in adhesion failure is important for correct interpretation of results. Laboratory bond testing of adhesive restorations using the microtensile bond strength (μTBS) test is the most common method to obtain information about the adhesion between restorative material and tooth structure.[2] The μTBS test is considered to be reliable because of its versatility and reliability *in vitro*.[3] However, this test method is time-consuming and technically demanding, requiring great care during specimen preparation and handling.[3]

During the preparation of samples for μTBS, polymerization of the composite resin produces shrinkage stress. This is influenced by restorative technique, resin elastic modulus, polymerization rate, and configuration of the cavity or "C-factor," which is defined as the ratio between bonded and unbonded composite resin surface area.[4-9] Shrinkage stresses from composite resin cause structural deformation and interfacial integrity failures, decreasing the bond strength between composite resin and tooth.[10] To reduce shrinkage stress, the use of an incremental technique is recommended, which promotes a smaller ratio of bonded to unbonded areas in each composite resin layer, achieving a lower C-factor during polymerization of each layer.[11]

The filling technique and type of composite resin can have a great impact on the adhesion of composite resin with the restoration, in particular in cavities with a high C-factor.[7] Laboratory testing for μTBS in constrained class II cavities are clinically relevant. In high C-factor cavities, the stress relief due to flow is severely limited, and the polymerization shrinkage stress might exceed the bond strength.[9]

New composite resins have been developed to minimize the deleterious clinical effects of polymerization shrinkage.[7,12,13] The use of bulk-fill resin allows a single increment of 4 mm. This thicker increment is possible due to modifications that increase the translucency of the bulk-fill composites, enabling greater light transmission.[14] In addition, formulation of these materials allows modulation of the polymerization reaction by the use of particular stress-relief monomers and more reactive photoinitiators.[12] These features decrease the undesirable effects of polymerization shrinkage.[7,13] To test different adhesive systems on the effects of polymerization shrinkage stress and adhesion, the μTBS test is the most commonly used method for researchers and manufacturers; however, the results of these tests are often difficult to interpret as limited attention has been given to the type of the composite resin used during this testing.

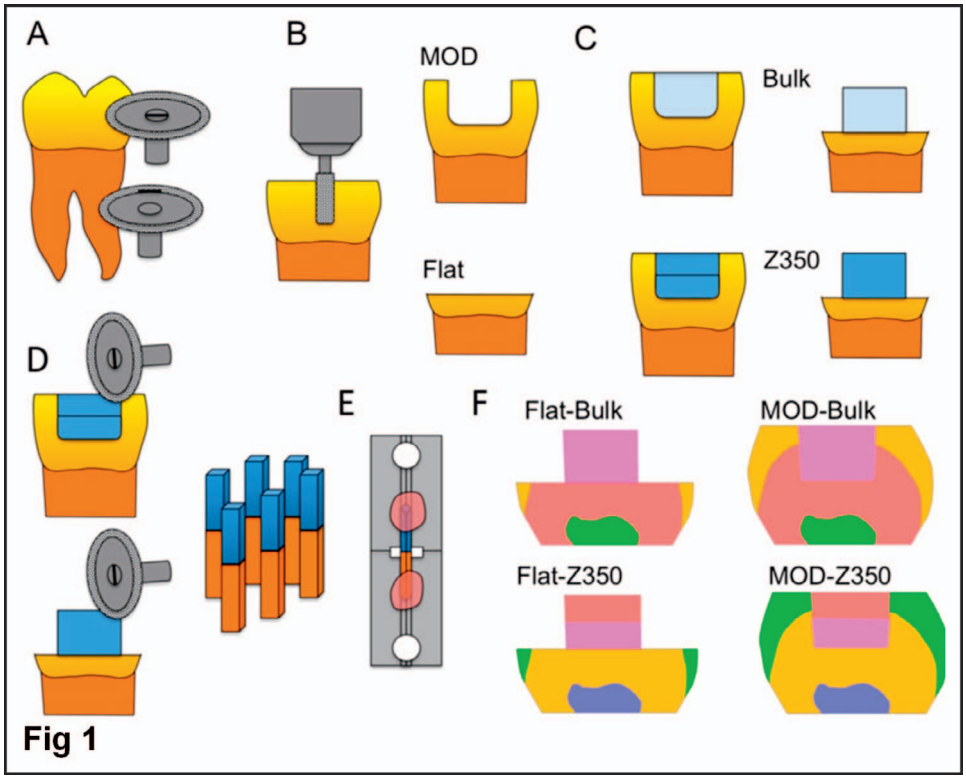



Figure 1. *Stepwise schematic of experimental design. (A): Sectioning of teeth at root and crown. (B): Teeth preparation: MOD, mesio-occlusal-distal preparation; flat, flat surface preparation. (C): Restorations were restored with each composite resin type: bulk, Filtek bulk-fill composite resin (bulk) or incremental filling using Filtek Z350XT (Z350; n=10). (D): Six sticks of restored teeth were obtained for each tooth. (E): Each section was subjected to the μTBS test using Geraldeli's device. (F): Preprocessing of the finite element method.*

The aim of this study was to evaluate the bond strength and stress distribution in flat or mesio-occlusal-distal (MOD) cavity preparations using the same adhesive strategy with flowable bulk-fill or conventional composite resins, as determined using the μTBS test. Therefore, we tested the null hypothesis that the type of cavity preparation or composite resin does not influence bond strength or stress distribution as determined using the μTBS test.

## METHODS AND MATERIALS

Forty extracted, intact human third molars that were caries free were used with approval from the University Ethics Committee in Human Research (No. 1.451.872). Teeth were embedded in a polystyrene resin (Cristal, Piracicaba, SP, Brazil) up to 2.0 mm below the cervical line to simulate alveolar bone and to ease manipulation.[15] The teeth were cleaned using a rubber cup and fine pumice water slurry and randomly distributed into four groups of 10 teeth. Two cavity preparations were defined: the flat preparation was generated by midcoronal cutting using a precision saw machine (Isomet 1000, Buehler, Lake Bluff, IL) resulting in a flat surface; the MOD preparation had an intercuspal width of 4.5 mm and was prepared with a diamond bur (No. 3099, KG Sorensen, Barueri, SP, Brazil) in a high-speed hand piece with copious air-water spray using a cavity preparation machine.[16] The depth of the cavities was the same for both types of preparation (4 mm in depth; Figure 1). A silicone mold was used for composite resin placement on flat cavity preparations. For MOD cavities, the prepared tooth was inserted in a metallic device that simulated proximal teeth, and a unimatrix with an elastic ring (TDV, Pomerode, SC, Brazil) was used in the proximal area. The self-etching adhesive system (ClearFil SE Bond, Kuraray, Japan) was used according to the manufacturer's instructions for all groups. Each type of restoration was made using two different composite resins: Filtek Z350XT (Z350; 3M-ESPE, St Paul, MN, USA) and Filtek bulk-fill flowable (FBF; 3M-ESPE; Table 1). The Z350 composite resin was placed in two horizontal increments for both types of cavity preparation, whereas the FBF composite resin was placed in a single increment. Each increment was light cured for 20 seconds using a multipeak light source with 1200 mW/cm$^2$ exitant irradiance (Bluephase G2, Ivoclar Vivadent, Amherst, NY, USA) from the occlusal direction closest to the cavity or to the composite resin increment.

### μTBS Test

The specimens were sectioned buccolingually into slabs of 1-mm thickness under water cooling using a

Table 1: *Composite Resin Composition*

| Material | Filtek Bulk Fill | Filtek Z350XT |
|---|---|---|
| Code | FBF | Z350 |
| Shade | A2 | A2 |
| Composite resin type | Bulk-fill flowable | Nanofilled composite resin |
| Increment size and light activation time | 4.0 mm/40 s | 2.0 mm/20 s |
| Organic matrix | UDMA, BISGMA, EBPADMA, Procrylat resin | Bis-GMA, Bis-EMA, UDMA, TEGDMA |
| Filler | Silane-treated ceramic and $YbF_3$ | Silica and zirconia nanofillers, agglomerated zirconia-silica nanoclusters |
| Filler % wt/vol | 64/42.5 | 82/60 |
| Manufacturer | 3M-ESPE (St Paul, MN, USA) | 3M-ESPE (St Paul, MN, USA) |

Abbreviations: Bis-GMA, bisphenol-A glycidyl methacrylate; Bis-EMA, ethoxylated bisphenol-A dimethacrylate; UDMA, urethane dimethacrylate; EBPADMA, ethoxylated bisphenol A dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

low-speed diamond saw (Isomet, Buehler). Each slab was then sectioned mesiodistally to produce 1.0 mm × 1.0 mm cross-section sticks (six sticks per tooth). The ends of the specimens were fixed to a Geraldeli's device (Odeme, Joinvile, SC, Brazil) using cyanoacrylate glue (Super Bonder Flex Gel, Henkel Loctite Adesivos Ltda, Itapevi, SP, Brazil) to cover all gripping surfaces of each specimen.[17,18] Each specimen was then subjected to a tensile load at a crosshead speed of 1 mm/min using a microtensile machine (Microtensile ODEME, Luzerna, SC, Brazil). The cross-sectional area of each stick was measured to the nearest 0.01 mm using a digital caliper (Mitutoyo CD15, Mitutoyo Co, Kawasaki, Japan), and the μTBS in MPa was calculated by dividing the fracture load by the surface area. Each tooth was treated as a statistical unit by averaging the μTBS of all six samples from one tooth.

Following the μTBS test, specimens were examined with a stereomicroscope (Mitutoyo, Tokyo, Japan) at 40× magnification. The fractured surfaces were classified as adhesive failure (I), cohesive failure in composite resin (II), cohesive failure in dentin (III), or mixed failure (IV).

## Finite Element Analysis

Residual stress in the tooth was calculated using a digitized buccolingual cross section with similar dimensions and conditions as those used for the μTBS test. The digitized buccolingual cross section of an intact molar with similar dimensions and conditions as those for the μTBS test was used as a reference for construction of the models. Coordinates were obtained using ImageJ software (public domain, Java-based image processing and analysis software developed at the National Institutes of Health, Bethesda, MD, USA).[19] Only the cervical portion of the root was simulated since the rest of the root did not affect the coronal stress distribution.[20] A simplified boundary condition was assumed at the cut plane of the root (fixed zero displacements in both horizontal and vertical directions).

The elastic modulus of enamel was 84 GPa and Poisson's ratio 0.30; the dentin elastic modulus was 18 GPa and the Poisson's ratio 0.23.[21] The elastic modulus of the restorative materials was 14.4 GPa for Z350 and 10.1 GPa for FBF.[13] The Poisson's ratio was chosen to be the same for all composite resins at 0.24.[20] The finite element analysis was performed using MSC.Mentat (preprocessor and postprocessor) and MSC.Marc (solver) software (MSC Software Corporation, Santa Ana, CA, USA). The total number of finite element analyses models was four for the different restorative materials and cavity type. A plane strain condition was assumed for the tooth cross sections. Polymerization shrinkage was simulated by thermal analogy. Temperature was reduced by 1°C, while the linear shrinkage value (postgel shrinkage) was entered as the coefficient of linear thermal expansion.

Modified von Mises equivalent stress was used to express the stress conditions using compressive-tensile strength ratios of 37.3 and 3.0 for the enamel and dentin,[21] respectively, and 6.25 for composite resin.[13] Stress values were recorded at the integration points of each element and node along material interfaces at either aspect (tooth and restoration). The stress values at the interface between composite resin and dentin at two depths in the two-dimensional model correlated with the elastic modulus and postgel shrinkage values at the same depths of the laboratory test restorations. The mean values of the top 5% of stresses were determined for the dentin/composite resin–isolated nodes at the interface and correlated with μTBS values.

Table 2: *Summary of μTBS Values in Flat- or MOD-Prepared Samples Using Filtek Z350XT or Filtek Bulk Fill Composite Resin*[a]

| Group | Conventional Composite Resin (Filtek Z350XT) | | Bulk Fill Composite Resin (Filtek Bulk Fill) | |
|---|---|---|---|---|
| Preparation | Mean (SD) | 95% Confidence Interval | Mean (SD) | 95% Confidence Interval |
| Flat preparation | 45.8 ± 9.0$^{Aa}$ | (39.4-52.2) | 49.2 ± 7.3$^{Aa}$ | (44.0-54.4) |
| MOD preparation | 31.6 ± 7.8$^{Bb}$ | (26.0-37.2) | 38.5 ± 10.8$^{Ba}$ | (30.8-46.2) |

[a] *The mean, standard deviation, and 95% confidence interval of MPa values are provided for each type of preparation and composite resin that was used. n=10 teeth per group. Two-way ANOVA was used to analyze differences between groups. Superscript uppercase letters indicate a significant difference between cavity preparations, and superscript lowercase letters indicate a significant difference between composite resins (p<0.05).*

### Statistical Analysis

The μTBS test data were assessed for normal distribution (Shapiro-Wilk) and equality of variances (Levene test), followed by parametric statistical tests. Two-way analysis of variance (ANOVA) was performed with the study factors represented by the composite resin (Z350 and FBF), cavity preparation (flat and MOD), and their interactions. Multiple comparisons were made using the Tukey post hoc test. The data of failure mode were subjected to the chi-square test. All tests employed a 0.05 level of statistical significance, and all statistical analyses were carried out with Sigma Plot version 13.1 (Systat Software Inc, San Jose, CA, USA).

## RESULTS

We used the μTBS test to assess the effects of cavity preparation and composite resin on the bond strength and stress distribution. Two-way ANOVA of μTBS values revealed a statistically significant difference between the composite resins ($p<0.001$), the cavity preparation ($p=0.001$), and the interaction between composite resins and cavity preparations ($p=0.004$; Table 2). Irrespective of composite resin type, flat preparations had significantly higher μTBS values than MOD preparations. For MOD-prepared samples, FBF composite resin had significantly higher μTBS values than Z350. However, flat-prepared samples showed no difference in μTBS values between FBF or Z350 composite resins.

We also measured the effects of cavity preparation and type of composite resin on stresses in the restoration. Preparations using the Z350 composite resin resulted in higher stress on composite resin buildup, irrespective of the type of cavity preparation (Figure 2). Flat-prepared cavities resulted in lower stresses at the dentin/composite resin interface relative to MOD-prepared cavities (Figure 3A). Within flat cavity preparations, each of the composite resins showed similar stresses at the dentin/composite resin interface. However, in MOD-prepared cavities, the Z350 composite resin resulted in higher stresses at the dentin/composite resin interface compared with MOD-prepared samples with FBF composite resin (Figure 3B). All groups showed similar failure patterns based on fracture mode distributions as represented in Figure 4. Adhesive failures were prevalent for all groups. Therefore, the cavity preparation and type of composite resin used affect the stress at the interface between dentin and composite resin.

## DISCUSSION

The results of our study demonstrate that the type of cavity preparation and type of composite resin affect μTBS and stress distribution. Further, our results also show that each preparation can be influenced by the type of composite resin used. Thus, we reject our null hypothesis that the type of cavity preparation or composite resin does not influence bond strength or stress distribution. Clinically, it is common to find structural loss in posterior teeth, resulting in the formation of large cavities. However, most studies use flat surfaces for testing bond strength, which does not resemble this clinical situation in the oral cavity.[22,23] Therefore, the present study was undertaken to obtain a more clinically relevant idea of the effects of cavity preparation and different composite resins used on (restoration) bond strength.

Restoration bond strength is affected by several factors, with composite resin polymerization shrinkage stress considered to play an important role.[8,24,25] A useful strategy to quantify the stress located at the bonded interface is finite element analysis. Sufficient polymerization that results in adequate mechanical properties associated with favorable bonding is mandatory for the best clinical performance of the composite resin restoration. Previous studies of large MOD restorations have shown that the type of composite resin and filling technique may influence the bond strength value of large MOD restorations.[15] Polymerization stress can be affected by bond strength and elastic modulus values. Bond strength values are indirectly related to polymerization shrinkage stresses,[26,27] while elastic modulus values of the composite resin are directly related to

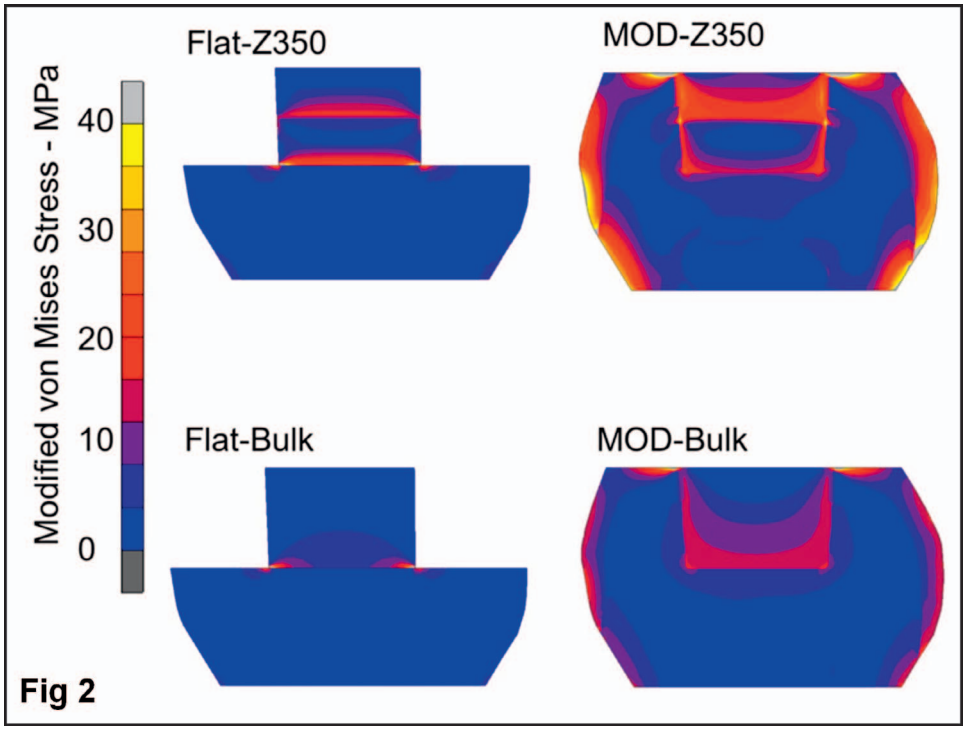



Figure 2. *Stress distributions of the two composite resins, each as calculated by finite element analysis (modified von Mises equivalent stresses; MPa).*

higher stresses in the remaining tooth structure and at the tooth/restoration interface. Materials with high elastic modulus deform less when they are stressed and produce more rigid restorations. This increases the effect of polymerization shrinkage, resulting in residual shrinkage stresses.[28] Thus, when polymerization contraction is restricted by bonding to the cavity walls, a composite resin with a high elastic modulus will result in higher shrinkage stress.[29] FBF composite resin has a lower filler content, which results in a lower elastic modulus and lower postgel shrinkage compared with Z350 composite resin.[13] In this study, Z350 showed higher stress, irrespective of cavity preparation, because of its higher elastic modulus and higher postgel shrinkage. Further, flat cavity preparations resulted in lower stresses at the dentin/composite resin interface than MOD cavity preparations. The free constrain of the composite resin on flat surface releases the shrinkage stress that compromises bond

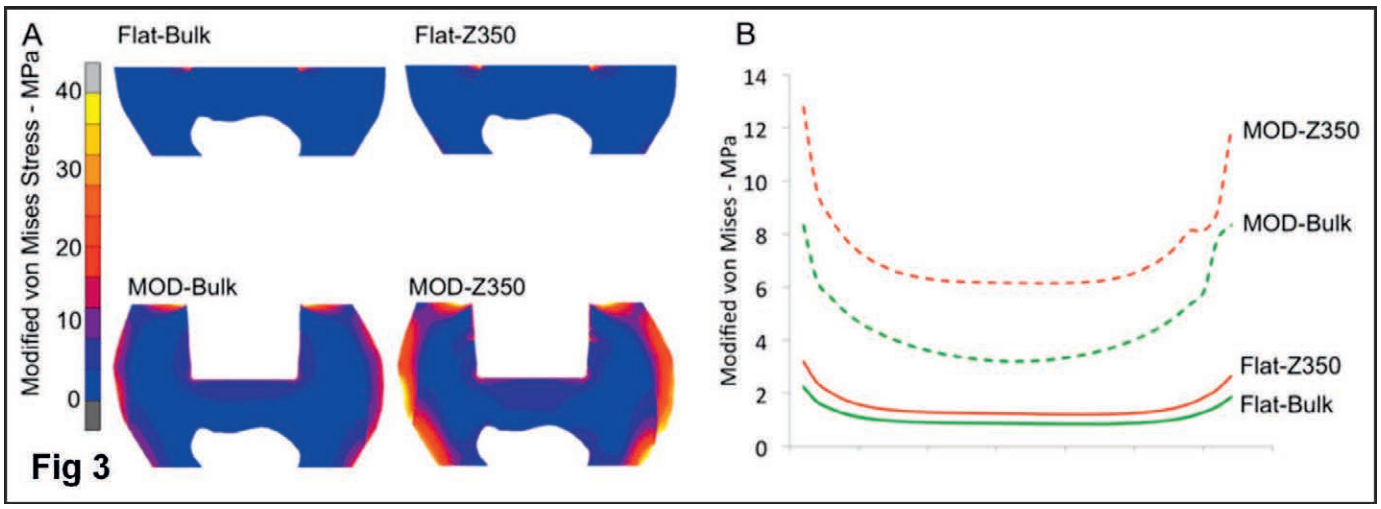



Figure 3. *The Z350 composite resin results in a higher stress distribution irrespective of the type of preparation used. (A): Stress distributions in flat- or MOD-prepared cavities using FBF or Z350 composite resin. (B): Stress along at the dentin/composite resin interface for each group in A calculated by finite element analysis (modified von Mises equivalent stresses; MPa).*

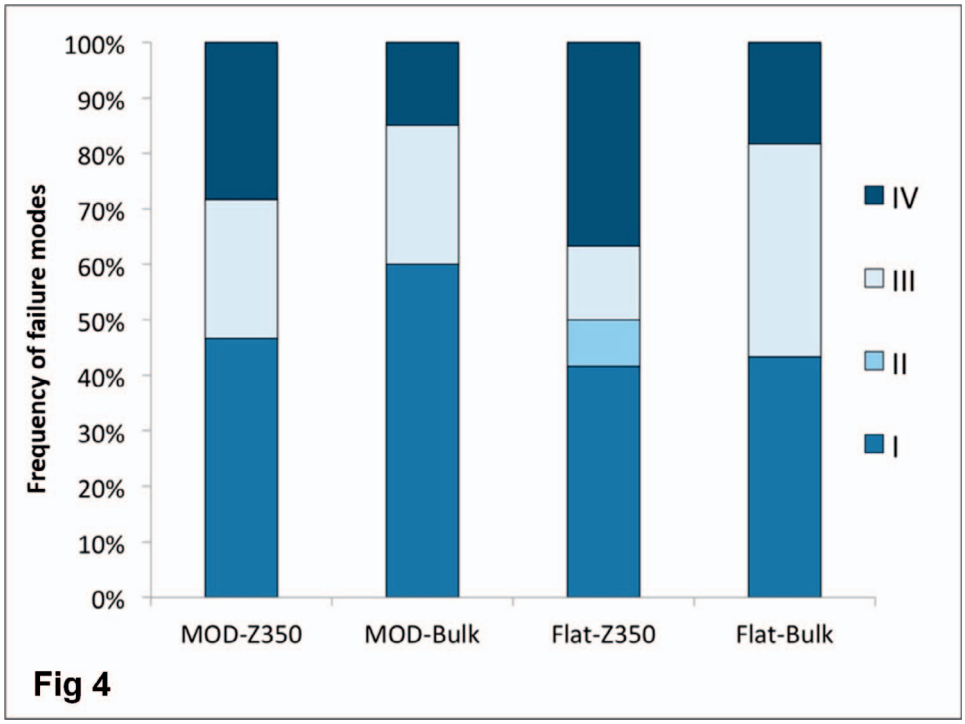



Figure 4. *Frequency of failure mode for flat- or MOD-prepared cavities using FBF (bulk) or Z350 composite resin. Fractured surfaces were classified as follows: I, adhesive failure; II, mixed failure; III, cohesive failure in dentin; IV, cohesive failure in composite resin.*

integrity. Similar to previous reports, our study has confirmed that shrinkage stress is influenced by the elastic modulus of resin, polymerization rates, restorative techniques, and cavity configuration.[23] The horizontal incremental filling used in the MOD cavities joining buccal and lingual walls increased the shrinkage stresses.[30] This stress is initially transferred to the interface, resulting in decreasing μTBS.

Bond strength values tend to decrease directly with the number of walls in the cavity.[6,31] In this study, the bond strength values for flat-prepared cavities were higher than the MOD-prepared cavities, regardless of the type of material that was used. In flat preparations, surrounding walls are absent, contributing to a lower C-factor[11]; thus, composite resin increments deform without restriction of the proximal walls, reducing the residual shrinkage stress, which may be why bond strength values were higher for cavities that underwent the flat preparation in our study. The expected magnitude of stress might be estimated through the ratio of the bonded to the unbonded areas, also known as the configuration factor.[32] The higher the C-factor, the higher the stress level generated; this aspect was observed in the MOD cavity tested in the present study. On the contrary, a higher ratio of unbonded to bonded walls, represented by the flat cavity tested in this study, would be responsible for lower values of stress as shrinkage would freely occur at the unbonded surface areas. In addition, without proximal walls, the increments may receive light energy more effectively, since insufficient curing is associated with lower bond strength and mechanical properties.[33]

In MOD-prepared cavities, FBF composite resin had higher bond strength values than Z350 composite resin. FBF has lower postgel shrinkage and lower elastic modulus. Consequently, shrinkage stress was lower in cavities restored with FBF composite. This is a principal factor to explain the better performance of FBF in MOD cavities. In addition, FBF has better adaptation to the pulp floor of the cavity, generating a more effective union of the Z350 conventional composite resin.[6] The FBF composite resin has a lower filler content and higher translucency,[13] allowing higher light transmission within the material and better photoactivation characteristics than Z350.[6]

The μTBS values were dependent on both the cavity preparation and composite resin type in this study. Therefore, our study highlights the importance of determining how materials were tested when comparing the μTBS results using different adhesive systems within and between laboratories. In addition, the findings of this study demonstrate that in constrained cavities, the use of an FBF composite resin may be a good strategy to produce better interface integrity.

## CONCLUSION

Within the limitations of this study design, we conclude that composite resins and cavity preparations, and the interaction between composite resins and cavity preparations, influence the stress distribution at the restoration/tooth interface and, consequently, the measured bond strength. Flat preparations resulted in lower shrinkage stress and significantly higher μTBS values relative to MOD preparations. FBF composite resin had significantly higher μTBS values than Z350 composite resin when tested using MOD preparation. MOD preparation Z350 resulted in higher stresses at the dentin/composite resin interface. The use of MOD preparations for the treatment of cavities will better reproduce clinical conditions when testing for bond strength using the μTBS test.

### Acknowledgements

This project was funded by grants from the CNPq—National Council for Scientific and Technological Development, from CAPES and FAPEMIG. This research was performed at CPbio-Biomechanics, Biomaterials and Cell Biology Research Center, UFU.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University Ethics Committee in Human Research. The approval code for this study is 1.451.872.

### 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 presented in this article.

*(Accepted 7 January 2017)*

### References

1. Poitevin A, De Munck J, Van Landuyt K, Coutinho E, Peumans M, Lambrechts P, & Van Meerbeek B (2008) Critical analysis of the influence of different parameters on the microtensile bond strength of adhesives to dentin *Journal of Adhesive Dentistry* **10(1)** 7-16.
2. Betamar N, Cardew G, & Van Noort R (2007) Influence of specimen designs on the microtensile bond strength to dentin *Journal of Adhesive Dentistry* **9(2)** 159-168.
3. Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA, & Tay F (1999) The microtensile bond test: a review *Journal of Adhesive Dentistry* **1(4)** 299-309.
4. Kumagai RY, Zeidan LC, Rodrigues JA, Reis AF, & Roulet JF (2015) Bond strength of a flowable bulk-fill resin composite in class II MOD cavities *Journal of Adhesive Dentistry* **17(5)** 427-432.
5. Labella R, Lambrechts P, Van Meerbeek B, & Vanherle G (1999) Polymerization shrinkage and elasticity of flowable composites and filled adhesives *Dental Materials* **15(2)** 128-137.
6. Van Ende A, De Munck J, Van Landuyt K, & Van Meerbeek B (2016) Effect of bulk-filling on the bonding efficacy in occlusal class I cavities *Journal of Adhesive Dentistry* **18(2)** 119-124.
7. Van Ende A, De Munck J, Van Landuyt KL, Poitevin A, Peumans M, & Van Meerbeek B (2013) Bulk-filling of high C-factor posterior cavities: effect on adhesion to cavity-bottom dentin *Dental Materials* **29(3)** 269-277.
8. Van Ende A, Mine A, De Munck J, Poitevin A, & Van Meerbeek B (2012) Bonding of low-shrinking composites in high C-factor cavities *Journal of Dentistry* **40(4)** 295-303.
9. Yoshikawa T, Sano H, Burrow MF, Tagami J, & Pashley DH (1999) Effects of dentin depth and cavity configuration on bond strength *Journal of Dental Research* **78(4)** 898-905.
10. Versluis A, Douglas WH, Cross M, & Sakaguchi RL (1996) Does an incremental filling technique reduce polymerization shrinkage stresses? *Journal of Dental Research* **75(3)** 871-878.
11. 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.
12. Fronza BM, Rueggeberg FA, Braga RR, Mogilevych B, Soares LE, Martin AA, Ambrosano G, & Giannini M (2015) Monomer conversion, microhardness, internal marginal adaptation, and shrinkage stress of bulk-fill resin composites *Dental Materials* **31(12)** 1542-1551.
13. Rosatto CM, Bicalho AA, Verissimo C, Braganca GF, Rodrigues MP, Tantbirojn D, Versluis A, & Soares CJ (2015) Mechanical properties, shrinkage stress, cuspal strain and fracture resistance of molars restored with bulk-fill composites and incremental filling technique *Journal of Dentistry* **43(12)** 1519-1528.
14. Bucuta S, & Ilie N (2014) Light transmittance and micromechanical properties of bulk fill vs. conventional resin based composites *Clinical Oral Investigations* **18(8)** 1991-2000.
15. Bicalho AA, Pereira RD, Zanatta RF, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2014) Incremental filling technique and composite material—part I: cuspal deformation, bond strength, and physical properties *Operative Dentistry* **39(2)** E71-E82.
16. Soares CJ, Fonseca RB, Gomide HA, & Correr-Sobrinho L (2008) Cavity preparation machine for the standardization of *in vitro* preparations *Brazilian Oral Research* **22(3)** 281-287.
17. Raposo LH, Armstrong SR, Maia RR, Qian F, Geraldeli S, & Soares CJ (2012) Effect of specimen gripping device, geometry and fixation method on microtensile bond strength, failure mode and stress distribution: laboratory and finite element analyses *Dental Materials* **28(5)** e50-e62.
18. Soares CJ, Soares PV, Santos-Filho PC, & Armstrong SR (2008) Microtensile specimen attachment and shape—finite element analysis *Journal of Dental Research* **87(1)** 89-93.
19. Schneider CA, Rasband WS, & Eliceiri KW (2012) NIH Image to ImageJ: 25 years of image analysis *Nature Methods* **9(7)** 671-675.

20. Versluis A, & Versluis-Tantbirojn D (2011) Filling cavities or restoring teeth? *Journal of the Tennessee Dental Association* **91(2)** 36-42.
21. Watts DC, el Mowafy OM, & Grant AA (1987) Temperature-dependence of compressive properties of human dentin *Journal of Dental Research* **66(1)** 29-32.
22. Bravis T, Pilecki P, Wilson RF, Fenlon M, Watson TF, & Foxton RM (2012) Effect of loading on the microtensile bond strength and microleakage of a self-etching and etch-and-rinse adhesive in direct class II MOD composite restorations *in vitro Dental Materials Journal* **31(6)** 924-932.
23. He Z, Shimada Y, & Tagami J (2007) The effects of cavity size and incremental technique on micro-tensile bond strength of resin composite in class I cavities *Dental Materials* **23(5)** 533-538.
24. 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.
25. El-Sahn NA, El-Kassas DW, El-Damanhoury HM, Fahmy OM, Gomaa H, & Platt JA (2011) Effect of C-factor on microtensile bond strengths of low-shrinkage composites *Operative Dentistry* **36(3)** 281-292, doi: 10.2341/10-105-L.
26. Boaro LC, Goncalves F, & Braga RR (2010) Influence of the bonding substrate in dental composite polymerization stress testing *Acta Biomaterialia* **6(2)** 547-551.
27. Isaac SZ, Bergamin AC, Turssi CP, Amaral FL, Basting RT, & Franca FM (2013) Evaluation of bond strength of silorane and methacrylate based restorative systems to dentin using different cavity models *Journal of Applied Oral Science* **21(5)** 452-459.
28. Versluis A, Tantbirojn D, Pintado MR, DeLong R, & Douglas WH (2004) Residual shrinkage stress distributions in molars after composite restoration *Dental Materials* **20(6)** 554-564.
29. Bicalho AA, Valdivia AD, Barreto BC, Tantbirojn D, Versluis A, & Soares CJ (2014) Incremental filling technique and composite material—part II: shrinkage and shrinkage stresses *Operative Dentistry* **39(2)** E83-E92.
30. Soares CJ, Bicalho AA, Tantbirojn D, & Versluis A (2013) Polymerization shrinkage stresses in a premolar restored with different composite resins and different incremental techniques *Journal of Adhesive Dentistry* **15(4)** 341-350.
31. Bakhsh TA, Sadr A, Shimada Y, Mandurah MM, Hariri I, Alsayed EZ, Tagami J, & Sumi Y (2013) Concurrent evaluation of composite internal adaptation and bond strength in a class-I cavity *Journal of Dentistry* **41(1)** 60-70.
32. Feilzer AJ, De Gee AJ, & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.
33. Price RB, Doyle G, & Murphy D (2000) Effects of composite thickness on the shear bond strength to dentin *Journal Canadian Dental Association* **66(1)** 35-39.

©Operative Dentistry, 2018, **43-1,** 90-100

# Effect of Finishing and Polishing on Roughness and Gloss of Lithium Disilicate and Lithium Silicate Zirconia Reinforced Glass Ceramic for CAD/CAM Systems

A Vichi • R Fabian Fonzar • C Goracci • M Carrabba • M Ferrari

### Clinical Relevance

Silica-based glass ceramics restorations achieve clinically acceptable, enamel-like roughness and gloss by either glazing or manual finishing and polishing. The latter can be considered an adequate procedure, comparable to furnace-based restorations, and this is noteworthy for chairside monolithic restorations.


## SUMMARY

**Objective: To assess the efficacy of dedicated finishing/polishing systems on roughness and gloss of VITA Suprinity and IPS e.max CAD.**

**Method: A total of 24 blocks of Suprinity and 24 of e.max were cut into a wedge shape using an InLab MC-XL milling unit. After crystallization, the 24 Suprinity wedges were divided into four subgroups: group A.1: Suprinity Polishing Set Clinical used for 30 seconds and group A.2: for 60 seconds; group A.3: VITA Akzent Plus Paste; and group A.4: spray. The 24 e.max wedges (group B) were divided into four subgroups according to the finishing procedure: group B.1: Optrafine Ceramic Polishing System for 30 seconds and group B.2: for 60 seconds; group B.3: IPS e.max CAD Crystall/Glaze paste; and group B.4: spray. After finishing/polishing, gloss was assessed with a glossmeter and roughness evaluated with a profilometer. Results were analyzed by applying a two-way analysis of variance for gloss and another for roughness ($\alpha$=0.05). One specimen per each subgroup was observed with a scanning electron microscope.**



*Alessandro Vichi, DDS, PhD, Department of Medical Biotechnologies, University of Siena, Siena, Italy. Faculty of Medical Sciences, LUdeS HEI Foundation, Lugano, Switzerland – La Valletta, Malta

Riccardo Fabian Fonzar, DDS, PhD, Department of Medical Biotechnologies, University of Siena, Siena, Italy

Cecilia Goracci, DDS, PhD, Department of Medical Biotechnologies, University of Siena, Siena, Italy

Michele Carrabba, DDS, PhD, Department of Medical Biotechnologies, University of Siena, Siena, Italy

Marco Ferrari, MD, DMD, PhD, Department of Medical Biotechnologies, University of Siena, Siena, Italy and Department of Restorative Dentistry, University of Leeds, UK

*Policlinico Le Scotte, viale Bracci Siena, 53100, Italy; email: alessandrovichi1@gmail.com


DOI: 10.2341/16-381-L

**Results: For roughness, materials and surface were significant factors ($p<0.001$). Suprinity exhibited significantly lower roughness than e.max. Also the Material-Surface Treatment interaction was statistically significant ($p=0.026$). For gloss, both material and surface treatment were significant factors ($p<0.001$). VITA Suprinity showed significantly higher gloss than e.max. Also the Material-Surface Treatment interaction was statistically significant ($p<0.001$).**

**Conclusions: Manual finishing/polishing for 60 seconds and glazing paste are the most effective procedures in lowering the roughness of CAD/CAM silica-based glass ceramics. Manual finishing/polishing for 60 seconds allows milled silica-based glass ceramics to yield a higher gloss. VITA Suprinity displayed higher polishability than IPS e.max CAD.**

## INTRODUCTION

Computer-aided design and computer-aided manufacturing (CAD/CAM) technology represents an important part of contemporary prosthetic dentistry.[1] CAD/CAM or "digital" dentistry developed following two main streams. The digital procedure can in fact be carried out by the technicians in their laboratories, with a workflow that can somehow resemble the traditional one, or, alternatively, it can be performed entirely in dental offices. In the so-called chairside procedure, a single-unit restoration can be fabricated in the dental office and delivered in a one-session appointment of reasonable time. Since its launch in 1985, the CEREC system, the first and currently leading system for chairside restorations, has developed hardware, software, and material options.[2,3] Among the several materials available for milling,[4-6] lithium silica-based glass ceramics are relevant for esthetic, physical, and mechanical properties. Despite the different chemistry, both IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein) and VITA Suprinity (VITA Zahnfabrik, Bad Sackingen, Germany) are particularly indicated for monolithic restorations.[7,8] Once milled, the restorations are coarse in texture,[1,9,10] so polishing and finishing are mandatory before delivery.[11] These procedures render the surfaces smoother[1] and more lustrous[12] as well as improve the restoration biocompatibility,[13-15] minimizing the incidence of biological complications such as plaque retention and antagonist-tooth wearing. In addition, well-finished surfaces lead to less technical and esthetic problems because the material becomes tougher,[16,17] glossy,[18] and stable in translucency[19] and color.[20]

Glass ceramics can be finished by handpiece burs, with or without glossy paste, or by furnace-based glazing materials. Because manual polishing and glazing affect differently the surface smoothness and aspect of dental ceramics,[1,11,18,21,22] it appeared of interest to evaluate whether roughness and gloss vary according to the finishing procedures.

To assess *in vitro* the effect of the dedicated manual and furnace-based finishing systems on the surface properties of VITA Suprinity and IPS e.max CAD, a study with a twofold objective was conducted. The first objective was to verify whether differences exist between the two materials in the ability to decrease roughness and increase gloss. The tested null hypothesis was that VITA Suprinity and IPS e.max CAD achieve the same roughness and gloss after finishing with their respective dedicated systems. The second objective was to assess whether for each of the two materials, the manual and the furnace-based recommended systems perform similarly. The tested null hypothesis was that no statistically significant difference in roughness and gloss exist among the different proprietary finishing and polishing systems tested on VITA Suprinity and IPS e.max CAD.

## METHODS AND MATERIALS

### Specimen Preparation

Blocks of zirconia-reinforced lithium silicate (VITA Suprinity, HT A3, VITA Zahnfabrik) and lithium disilicate (IPS e.max CAD, HT A3, Ivoclar Vivadent AG) for the CEREC CAD/CAM system (Sirona Dental, Bernsheim, Germany) were selected for this study. Twenty-four blocks were used for each of the two tested materials.

A model for a 30° wedge-shaped specimen was designed with CEREC InLab software v3.88 (Sirona Dental; Figure 1). Specimens were milled in an InLab MC-XL milling machine (Sirona Dental). In order to standardize the milling procedure, the diamond burs of the milling unit (Step Bur 12S and Cylinder Pointed Bur 12S, both Sirona Dental) were replaced before starting the milling procedure and then every 10 milling cycles. Each milled wedge was finally separated from the block base by means of a low-speed, water-cooled diamond disc.

Final crystallization was performed following manufacturer's instructions in their respective recommended furnaces: VITA Vacumat 6000 (VITA

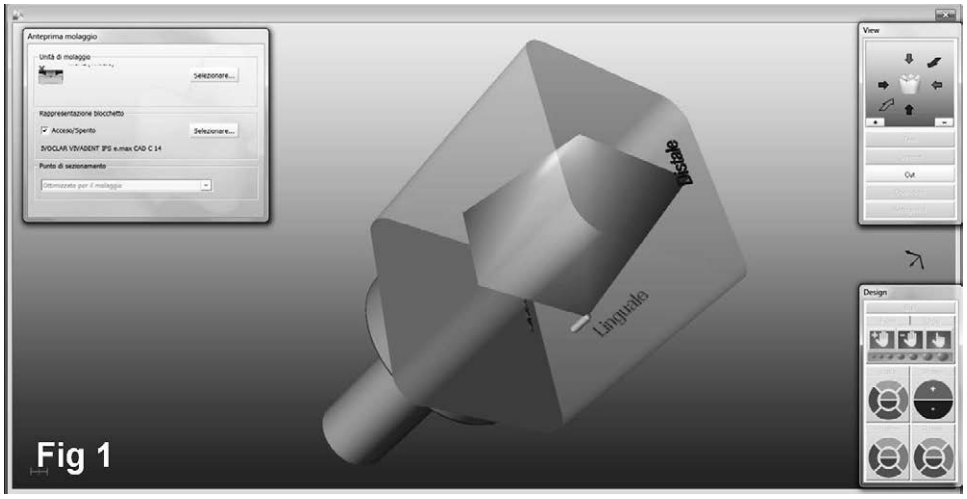



Figure 1. *Software image of the 30° wedges milled from VITA Suprinity and IPS e.max CAD blocks.*

Zahnfabrik) for VITA Suprinity and EP 600 Combi (Ivoclar Vivadent) for IPS e.max CAD.

After crystallization, the 24 VITA Suprinity wedges (group A) were randomly divided into four subgroups according to the finishing procedure (Table 1): group A.1 = VITA Suprinity Polishing Set Clinical used for 30 seconds (VITA Zahnfabrik); group A.2 = VITA Suprinity Polishing Set Clinical used for 60 seconds; group A.3 = VITA Akzent Plus Paste (VITA Zahnfabrik); group A.4 = VITA Akzent Plus Spray (VITA Zahnfabrik). The 24 e.max CAD wedges (group B) were also randomly divided into four subgroups according to the finishing procedure: group B.1 = Optrafine Ceramic Polishing System (Ivoclar Vivadent) used for 30 seconds; group B.2 =

Table 1: *Tested Groups, Materials, and Treatments*

| Groups | Blocks | Finishing and Polishing Systems | Treatments and Abbreviations | |
|---|---|---|---|---|
| A1 | VITA Suprinity (ZLS) | SUPRINITY Polishing Set Clinical | Manual finishing and polishing with contra-angle handpiece First tip: 30 seconds, 10,000 rpm; second tip: 30 seconds, 6,000 rpm. | 30MFP |
| B1 | IPS e.max CAD (LD) | Optrafine Ceramic Polishing System | Manual finishing and polishing with contra-angle handpiece First tip: 30 seconds, 10,000 rpm; second tip: 30 seconds, 6,000 rpm. | |
| A2 | VITA Suprinity (ZLS) | SUPRINITY Polishing Set Clinical | Manual finishing and polishing with contra-angle handpiece First tip: 60 seconds, 10,000 rpm; second tip: 60 seconds, 6,000 rpm. | 60MFP |
| B2 | IPS e.max CAD (LD) | Optrafine Ceramic Polishing System | Manual finishing and polishing with contra-angle handpiece First tip: 60 seconds, 10,000 rpm; second tip: 60 seconds, 6,000 rpm. | |
| A3 | VITA Suprinity (ZLS) | VITA Akzent Plus Paste | Laboratory finishing Smoothly applied on the surface with a brush and fired | GP |
| B3 | IPS e.max CAD (LD) | IPS e.max CAD Crystall/Glaze paste | Laboratory finishing Smoothly applied on the surface with a brush and fired | |
| A4 | VITA Suprinity (ZLS) | VITA Akzent Plus Spray | Laboratory finishing Smoothly sprayed on the surface and fired | GS |
| B4 | IPS e.max CAD (LD) | IPS e.max CAD Crystall/Glaze spray | Laboratory finishing Smoothly sprayed on the surface and fired | |

*Abbreviations: GP, glazing paste; GS, glazing spray; MFP, manual finishing and polishing; rpm, revolutions per minute.*

Optrafine Ceramic Polishing System used for 60 seconds; group B.3 = IPS e.max CAD Crystall/Glaze paste (Ivoclar Vivadent); group B.4 = IPS e.max CAD Crystall/Glaze spray (Ivoclar Vivadent). For each group, five wedges were used for roughness and gloss measurements. Given that both sides of each wedge received the surface treatment, a total of 10 surfaces per group were assessed (n=10). One extra specimen per subgroup was prepared for scanning electron microscopy (SEM) observations.

For the manual finishing procedure (subgroups A.1, A.2, B.1, B.2), rubber cups were used and replaced every two specimens. Finishing was carried out following the manufacturers' instructions with a contra-angle handpiece (Kavo INTRAmatic 20CN, Kavo, Biberach, Germany) under water-cooling. All the manual finishing and polishing procedures were performed by the same operator. The operator was calibrated using a precision scale before and during the procedure, considering a 40*g* force as a reference for light pressure. The calibration was repeated for every subgroup (10 specimens).[23]

For the furnace-based finishing procedure of subgroup A.3, the glazing material was applied and then fired in a VITA Vacumat 6000 furnace (VITA Zahnfabrik, Bad Sackingen, Germany) with the following firing cycle: pre-dry at 400°C for 6 minutes, heat 80°C/minute until 800°C followed by opening after 1 minute of holding time with no vacuum.

For subgroup A.4, the glazing material was applied and then fired in a VITA Vacumat 6000 furnace with the following firing cycle: pre-dry at 400°C for 4 minutes, heat 80°C/minute until 800°C followed by opening after 1 minute of holding time with no vacuum.

For subgroups B.3 and B.4, after the application of the glazing material (paste and spray, respectively), the following firing cycle was performed with EP 600 Combi (Ivoclar, Schaan, Liechtenstein): pre-dry at 403°C for 6 minutes, heat 90°/minute until 820°C, heat 30°C/minute until 840°C, followed by opening after 3 minutes of holding time; vacuum on at 550°C and vacuum off at 820°C.

### Roughness and Gloss Measurement

Before testing, specimens were ultrasonically cleaned in a 95% alcohol solution for three minutes. A profilometer (Mitutoyo SJ-201P, Mitutoyo Corp., Kanagawa, Japan) set with a cutoff value of 0.8 mm, a stylus speed of 0.5 mm/s, and a tracking length of 5.0 mm was used[24] to assess the surface roughness (Ra). The setup was standardized by means of a custom mold for both instrument and specimen. Mean Ra (μm) was recorded.

A glossmeter (Novo-Curve, Rhopoint Instruments Ltd, Bexhill-on-Sea, UK) with a 60° angle was used for the gloss evaluation. ISO 2813 specifications for ceramic materials were followed[25] and gloss units (GU) were recorded. To avoid any ambient light and to control the specimen position during measuring, a custom-made opaque silicone mold was used.

In order to test the formulated null hypothesis, the ability of the material to be finished (group A; group B), and the efficacy of the recommended finishing systems (subgroups A.1-A.4; subgroups B.1-B.4) were compared for roughness and gloss, respectively, using a two-way analysis of variance (ANOVA), having verified that the groups' data distribution was normal ($p$=0.077; Kolmogorov-Smirnov test) and variances were homogeneous ($p$=0.545; Levene test). In all the analyses, the level of significance was set at $\alpha = 0.05$ and PASW Statistic 18.0 software (SPSS, Chicago, IL, USA) was used.

### SEM Evaluation

Specimen preparation for SEM observations involved ultrasonic cleaning in a 95% alcohol solution for three minutes and air drying with an oil-free air spray. Specimens were then secured onto SEM (JSM-6060LV, JEOL, Tokyo, Japan) slabs with gold-conducting tape, and were gold coated in a vacuum sputter coater (SC7620 Sputter Coater, Polaron Range, Quorum Technologies, Newhaven, UK). The treated surfaces were then observed at 500× magnification (Figure 2).

## RESULTS

### Roughness (Ra)

The results of superficial roughness measurements are reported in Table 2. The two-way ANOVA demonstrated that material and surface treatment were significant factors for roughness ($p<0.001$). In particular, regardless of the surface treatment, VITA Suprinity exhibited significantly lower roughness than IPS e.max CAD. When assessing the influence of the polishing system, the two-way ANOVA disclosed that 60 seconds of manual finishing and polishing and glazing paste resulted in significantly lower roughness than glazing spray. Also 60 seconds of manual finishing and polishing yielded significantly lower roughness than 30 seconds of manual finishing and polishing. Also

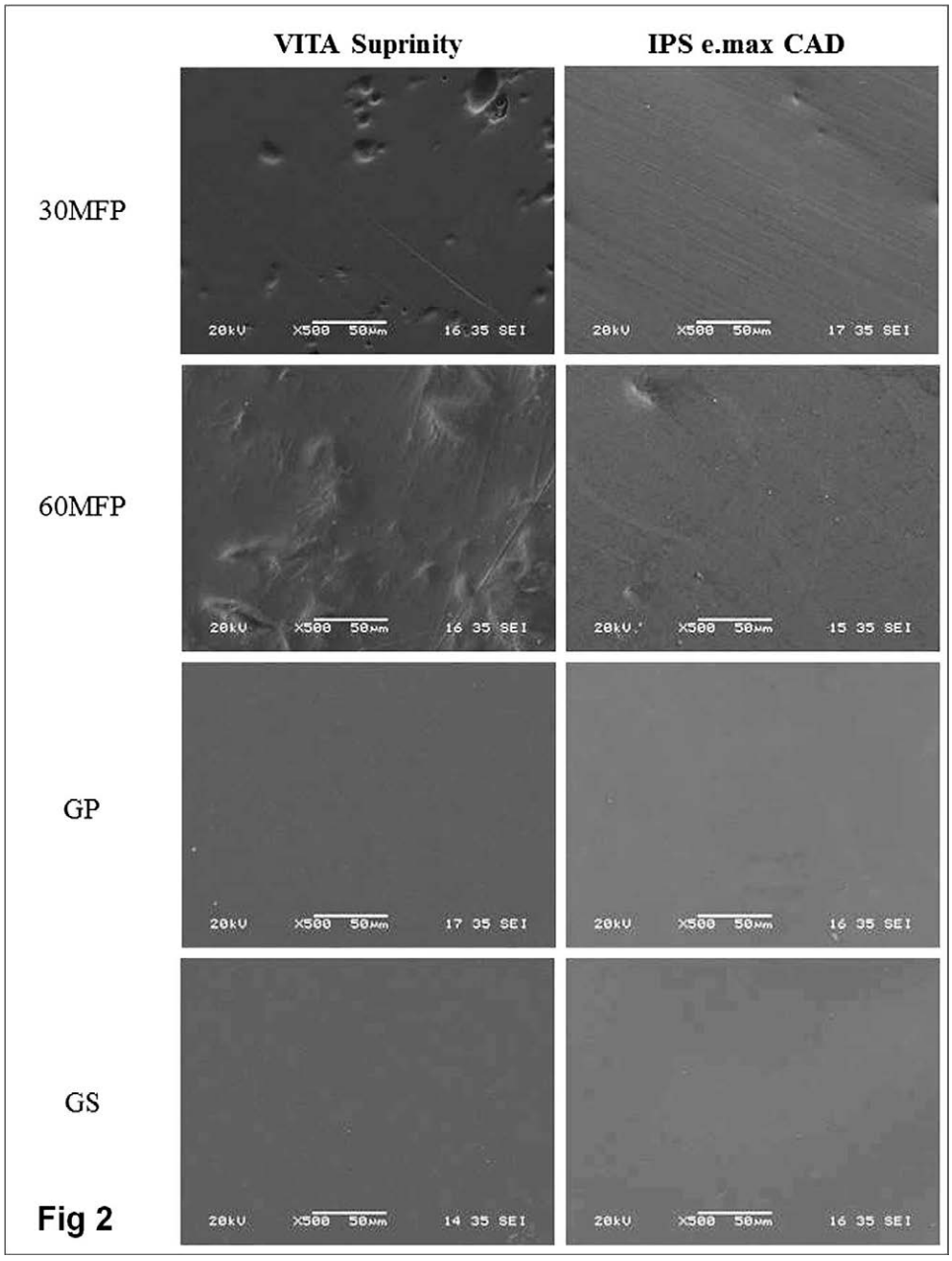



Figure 2. *SEM analysis of VITA Suprinity and IPS e.max CAD at 500 × after 30 and 60 seconds of manual finishing and polishing (30MFP and 60MFP, respectively), glazing paste (GP), and glazing spray (GS).*

the Material × Surface Treatment interaction was statistically significant ($p=0.026$). Specifically, with Suprinity 30 seconds of manual finishing and polishing and glazing spray resulted in significantly lower roughness than 60 seconds of manual finishing and polishing and glazing paste, whereas with IPS e.max CAD, glazing spray produced significantly higher roughness than the other treatments ($p<0.05$). In addition, IPS e.max CAD was significantly rougher than Suprinity following all the treatments except 30 seconds of manual finishing and polishing ($p<0.05$).

**Gloss (GU)**

The results of the gloss measurements are presented in Table 3. The outcome of the two-way ANOVA pointed out that both material and surface treatment were significant factors for gloss ($p<0.001$). In particular, regardless of the surface treatment, VITA Suprinity exhibited significantly higher gloss than IPS e.max CAD. Considering the effect of the polishing system, it emerged that 60 seconds of manual finishing and polishing produced significantly higher gloss than the other investigated treatments. Also the Material × Surface Treatment interaction

Table 2: *Mean (SD) for Surface Roughness of VITA Suprinity and IPS e.max CAD After 30 and 60 Seconds of Manual Finishing and Polishing (30MFP and 60MFP, Respectively), Glazing Paste (GP), and Glazing Spray (GS) and Statistical Significance (Sign.)**

| Treatment | Roughness (μm) | | | | | | Sign. |
|---|---|---|---|---|---|---|---|
| | VITA Suprinity | | | IPS e.max CAD | | | |
| | Mean | SD | Sign. | Mean | SD | Sign. | |
| 30MFP | $0.69^{\alpha}$ | 0.15 | *b* | $0.62^{\alpha}$ | 0.21 | a | BC |
| 60MFP | $0.37^{\alpha}$ | 0.08 | *a* | $0.53^{\beta}$ | 0.13 | a | A |
| GP | $0.42^{\alpha}$ | 0.12 | *a* | $0.66^{\beta}$ | 0.15 | a | AB |
| GS | $0.64^{\alpha}$ | 0.31 | *b* | $0.91^{\beta}$ | 0.21 | b | C |
| Sign. | *A* | | | *B* | | | |

** Italic upper case letters indicate substrate statistical groups. Upper case letters indicate treatment statistical groups. Italic lower case letters indicate statistical groups among treatments within VITA Suprinity. Lower case letters indicate statistical groups among treatments within IPS e.max CAD. Superscript Greek letters indicate statistical groups among the two materials within each treatment.*

Table 3: *Mean (SD) for Surface Gloss of VITA Suprinity and IPS e.max CAD After 30 and 60 Seconds of Manual Finishing and Polishing (30MFP and 60MFP, Respectively), Glazing Paste (GP) and Glazing Spray (GS) and Statistical Significance (Sign.)**

| Treatment | Gloss (GU) | | | | | | Sign. |
|---|---|---|---|---|---|---|---|
| | VITA Suprinity | | | IPS e.max CAD | | | |
| | Mean | SD | Sign. | Mean | SD | Sign. | |
| 30MFP | $49.05^{\beta}$ | 6.17 | *c* | $63.14^{\alpha}$ | 12.13 | a | B |
| 60MFP | $85.02^{\alpha}$ | 12.94 | *a* | $65.77^{\beta}$ | 12.36 | a | A |
| GP | $72.24^{\alpha}$ | 10.60 | *a* | $48.28^{\beta}$ | 9.53 | b | B |
| GS | $69.86^{\alpha}$ | 9.0 | *b* | $54.89^{\beta}$ | 13.91 | ab | B |
| Sign. | *A* | | | *B* | | | |

** Italic upper case letters indicate substrate statistical groups. Upper case letters indicate treatment statistical groups. Italic lower case letters indicate statistical groups among treatments within VITA Suprinity. Lower case letters indicate statistical groups among treatments within IPS e.max CAD. Superscript Greek letters indicate statistical groups among the two materials within each treatment.*

was statistically significant ($p<0.001$). Specifically, with Suprinity, 30 seconds of manual finishing and polishing produced the lowest gloss. However, by increasing the polishing time to 60 seconds, a gloss significantly higher than with either glazing procedure was obtained ($p<0.05$). On IPS e.max CAD using gloss paste, a gloss significantly lower than with either manual polishing procedure was achieved ($p<0.005$). In addition, following 30 seconds of manual finishing and polishing, IPS e.max exhibited significantly higher gloss than Suprinity ($p<0.005$). Conversely, with all the other surface treatments it was Suprinity that achieved superior gloss ($p<0.05$).

### SEM Evaluation

Different surface topographies were observed for the manual and heat-mediated polishing systems (Figure 1). For VITA Suprinity, some irregularities were still present after the 30 seconds of manual polishing. The 60-second polishing group resulted in a more homogeneous surface with few isolated minor defects and scratches. For IPS e.max CAD, scratches resulting from the abrasive action of the polishing rubber cups were particularly evident after 30 seconds of manual polishing. However, no substantial superficial defects were visible. Conversely, a more uniform surface was found for the 60-seconds manual polishing group. For both materials, more extensive defect-free surfaces were observed after paste and spray glazing.

## DISCUSSION

Given that statistically significant differences were detected between IPS e.max CAD and VITA Suprinity in roughness and gloss, the first null hypothesis was rejected. Statistically significant differences were also found in roughness and gloss among the different subgroups, both for IPS e.max CAD and VITA Suprinity. Thus, the second null hypothesis was rejected as well.

Roughness and gloss evaluation allows glass ceramics to be superficially analyzed and screened with regard to their surface characteristic after finishing.[18] Roughness can be described by several linear (Ra, Rq, Rz) or three-dimensional (Sa, Sq, Sz) parameters.[1,22,26] For the present investigation, Ra, which is defined as the mean arithmetical value of all the absolute distances of the profile inside of the measuring length,[11] was assessed because it is the most commonly used parameter for evaluating the effect of finishing protocols on dental ceramics.[27-31]

Gloss (GU) represents the amount of specular reflection from a surface,[12,32] and it is calculated by comparing the magnitude of incident light traveling toward a surface at a 60° angle to the magnitude traveling away from the surface at an equal and opposite angle. Optical properties (refraction index) and surface topography[12] characterize the gloss to the extent that the coarser the texture, the lower the reflectivity.[18,33-36]

The first aim of the present study was to compare finishing systems that are marketed for the proprietary silica-based glass ceramics. The variability observed in roughness and gloss for VITA Suprinity and IPS e.max CAD may be ascribed to the differences in ceramic microstructure as well as to the peculiar properties of the polishing and glazing

Table 4: *Polishing Systems Specifications as Declared by the Manufacturer*

| Instrument | Grit | Contents | Manufacturer |
|---|---|---|---|
| VITA Suprinity Polishing Set clinical (pink) | Diamond powder 500/600 | Polyurethane-rubber/caoutchouc<br>Diamond grains<br>Pigments | VITA Zahnfabrik<br>Bad Sackingen<br>Germany |
| OptraFine F (coarse) | NR | Synthetic rubber<br>Diamond granulate<br>Titanium dioxide | Ivoclar Vivadent AG<br>Schaan<br>Liechtenstein |
| VITA Suprinity Polishing Set Clinical (gray) | Diamond powder 3000 | Polyurethane-rubber/caoutchouc<br>Diamond grains<br>Pigments | VITA Zahnfabrik<br>Bad Sackingen<br>Germany |
| OptraFine P (fine) | NR | Synthetic rubber<br>Diamond granulate<br>Titanium dioxide | Ivoclar Vivadent AG<br>Schaan<br>Liechtenstein |

*Abbreviation: NR, not reported by the manufacturer.*

systems. At present no data are available in the literature concerning VITA Suprinity finishing and polishing. The information available for IPS e.max CAD indicates similar results of the various manual finishing and polishing systems, suggesting that the ability to obtain smooth surfaces mainly depends on the material, rather than on the finishing/polishing system used.[37,38]

Polishing and glazing yielded similar results with regard to roughness. IPS e.max CAD polished with OptraFine for 30 or 60 seconds and finished using glazing paste showed, in fact, comparable roughness. Although Lawson and others[38] reported less efficacy of the glazing paste when compared with 60-second manual polishing, most of the studies agree on the comparability of the two procedures,[39-41] even if different final Ra values are reported. These differences in final Ra might be explained by the baseline roughness of the specimens. Because roughness is conventionally measured with a profilometer on flat surfaces, the after-milling roughness is usually replicated by grinding and polishing the specimens with silicon carbide papers of increasing grit.[42,43] Given that the combination of carbide papers differs among the studies, baseline surfaces might vary and mismatch the effective after-milling roughness. For the present study, the flat specimens were directly milled with the CEREC MC-XL milling unit, with the twofold objective of high repeatability of the specimens and test of the real after-milling surface.

Polishing time played an important role in the final smoothness and luster, as previously reported.[18] Despite the minor differences in composition between the VITA Suprinity Polishing set and Optrafine manual sets (Table 4), polishing time affected roughness and gloss of VITA Suprinity differently than it did IPS e.max CAD. Whereas for VITA Suprinity the obtained values indicated a smoother and glossier surface after 60 seconds of polishing, for IPS e.max CAD, the values did not change. This might be explained by the differences in the microstructure of the two materials.[28,44,45] VITA Suprinity is a zirconia-reinforced, silica-based glass ceramic with a mean crystal size of approximately 0.5 μm, whereas IPS e.max CAD is a lithium disilicate–based glass ceramic with a mean crystal size of 1.5 μm. Because ceramic crystals removed from the surface during polishing might become part of the abrasive system and contribute to characteristics of the surface topography,[46] the finer microstructure of VITA Suprinity might account for its smoother surface compared to IPS e.max CAD after 60 seconds of polishing.[29] In addition, silicon dioxide concentration varies between IPS e.max CAD (57.0-80.0) and VITA Suprinity (56.0-64.0). Because the higher the concentration of silicon dioxide, the greater the crystalline phase,[47] the superior crystalline content of IPS e.max CAD might explain its lower capability to be smoothed after 60 seconds of polishing. Furthermore, the higher content of zirconium dioxide in VITA Suprinity might contribute to its lower superficial roughness after 60 seconds of polishing, because zirconia allows the material to be more efficaciously polished.[48]

The times tested in the present study refer to manufacturers' instructions and are in the range of common clinical use. It could be supposed that either material would display higher superficial smoothness and gloss after longer polishing times.[18] However, longer polishing times might also cause greater substance loss, given that polishing is a subtractive procedure. This occurrence should be given due consideration clinically.

Concerning the efficacy of the furnace-based glazing systems, VITA Suprinity showed lower roughness and higher gloss after paste glazing than spray glazing. Different trends were observed for IPS e.max CAD, given that roughness and gloss were lower for paste than for spray glazing. The differences in composition and characteristic density between IPS e.max CAD Crystall/Glaze Paste and VITA AKZENT Plus Paste resulted in a different glaze spread ability on the ceramic surface. This occurrence might explain the lower gloss of IPS e.max CAD compared with VITA Suprinity, because the less smooth glaze coat might have caused a variation in the superficial refraction index and, therefore, in the gloss.

As reported by Vo and others,[49] IPS e.max CAD treated with glazing spray obtained the highest superficial roughness among the tested finishing systems. Owing to the rougher baseline surfaces of the milled wedges, glazing spray was not able to uniformly coat all the irregularities, thus demonstrating less efficacy at smoothing.[50-51] Monaco and others[52] found Ra values (0.58±0.5 μm) for this procedure lower than the values reported in the present study. A possible explanation might again be found in the baseline roughness of the material. This assumption is supported by other studies indicating that a lower surface roughness can be obtained when the specimens are polished prior to glazing.[1,53]

When assessing the efficacy of the furnace-based systems on the two tested materials, it was observed that glazing paste and spray were more effective on VITA Suprinity than on IPS e.max CAD in terms of both roughness and gloss. As previously reported, a possible reason for this finding may be the differences in microstructure of these glass ceramics. Due to the lower crystalline volume and the smaller crystal size, VITA Suprinity might have exhibited a lower baseline roughness that would have led to lower roughness after glazing.[1,28,53,54] As roughness and gloss have an inverse proportional trend,[12] the lower roughness of VITA Suprinity surfaces may account for their higher gloss.

To better understand the outcome of the present study, the collected data have to be related to clinical requirements. Some *in vivo* studies[55] have suggested an ideal threshold surface roughness of 0.2 μm, above which bacterial retention is facilitated and the incidence of biological complications increased. In addition, superficial roughness greater than 0.5 μm can be detected by the sensorial fibers of the tongue, resulting in discomfort for the patient.[56] Nevertheless, natural enamel roughness is reported to range between 0.64 and 0.90 μm with regard to the tooth type, location, and patient age.[41,57] When referring to the clinical acceptability of the finished surfaces, all the Ra values measured in the present study were far below the abrasive wearing threshold (1.5 μm).[38] Furthermore, 60 seconds of polishing (0.37±0.08 μm) and glazing paste (0.42±0.12 μm) allowed VITA Suprinity to be imperceptible by the tongue. All the other groups fell within the enamel roughness range (0.64-0.90 μm), with the exception of IPS e.max CAD after glazing spray (0.91±0.21 μm).

Unlike for roughness, a clinically accepted threshold for gloss has not been established. However, natural enamel gloss is reported to range between 40 and 52 GU.[58] VITA Suprinity polished for 30 seconds (49±6 GU) and IPS e.max CAD finished with glazing paste (48±10 GU) displayed similar gloss to that of enamel, whereas all the other procedures gave higher values for both materials. The most efficient system was 60 seconds of manual polishing for either VITA Suprinity (85±13 GU) or IPS e.max CAD (66±12 GU). Similar results (57 GU) were observed for IPS e.max CAD finished with 4000-grit silicon carbide paper polishing,[59] whereas the combination of 1200-grit silicon carbide paper polishing and alumina slurry extrapolishing allowed lithium disilicate to become more lustrous (96 GU).[12]

From a clinical perspective it should also be noted that manual polishing systems have the main advantage of completing the in-office restoration in a single session, still ensuring comparable or better performance than glazing systems.[28,60] By manual polishing, the clinician finishes the in-office restoration without any thermal treatment, speeding up and simplifying the overall workflow; this is particularly relevant considering the increasing use of monolithic restorations.

## CONCLUSION

Within the limitations of the present study, the following conclusions can be drawn:

- Manual finishing and polishing for 60 seconds and glazing paste were the most effective procedures at lowering the roughness of CAD-CAM silica-based glass ceramics.
- Manual finishing and polishing for 60 seconds allowed milled silica-based glass ceramics to yield the highest gloss.
- VITA Suprinity displayed higher polishability than IPS e.max CAD.

### 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 14 March 2017)*

### REFERENCES


1. Fasbinder DJ, & Neiva GF (2016) Surface evaluation of polishing techniques for new resilient CAD/CAM restorative materials *Journal of Esthetic and Restorative Dentistry* **28(1)** 56-66.
2. Mörmann WH (2006) The evolution of the CEREC system *Journal of the American Dental Association* **137(Supplement 1)** 7S-13S.
3. Sadowsky SJ (2006) An overview of treatment considerations for esthetic restorations: A review of the literature. *Journal of Prosthetic Dentistry* **96(6)** 433-442.
4. Wittneben JG, Wright RF, Weber HP, & Gallucci GO (2009) A systematic review of the clinical performance of CAD/CAM single-tooth restorations *International Journal of Prosthodontics* **22(5)** 466-471.
5. Vichi A, Sedda M, Del Siena F, Louca C, & Ferrari M (2013) Flexural resistance of CEREC CAD/CAM system ceramic blocks. Part 1: Chairside materials *American Journal of Dentistry* **26(5)** 255-259.
6. Sedda M, Vichi A, Del Siena F, Louca C, & Ferrari M (2014) Flexural resistance of CEREC CAD/CAM system ceramic blocks. Part 2: Outsourcing materials *American Journal of Dentistry* **27(1)** 17-22.
7. Stawarczyk B, Liebermann A, Eichberger M, & Güth JF (2015) Evaluation of mechanical and optical behavior of current esthetic dental restorative CAD/CAM composites *Journal of the Mechanical Behavior of Biomedical Materials* **55(March)** 1-11.
8. Magne P, Carvalho AO, Bruzi G, & Giannini M (2015) Fatigue resistance of ultrathin CAD/CAM complete crowns with a simplified cementation process *Journal of Prosthetic Dentistry* **114(4)** 574-579.
9. Song XF, Ren HT, & Yin L (2015) Machinability of lithium disilicate glass ceramic in *in vitro* dental diamond bur adjusting process *Journal of the Mechanical Behavior of Biomedical Materials* **53(January)** 78-92.
10. Corazza PH, de Castro HL, Feitosa SA, Kimpara ET, & Della Bona A (2015) Influence of CAD-CAM diamond bur deterioration on surface roughness and maximum failure load of Y-TZP-based restorations *American Journal of Dentistry* **28(2)** 95-99.
11. Silva TM, Salvia AC, Carvalho RF, Pagani C, Rocha DM, & Silva EG (2014) Polishing for glass ceramics: Which protocol? *Journal of Prosthodontic Research* **58(3)** 160-170.
12. Lawson NC, & Burgess JO (2015) Gloss and stain resistance of ceramic-polymer CAD/CAM restorative blocks *Journal of Esthetic and Restorative Dentistry* **28(Supplement 1)** S40-S45.
13. Quirynen M, Van der Mei HC, Bollen CML, Schotte A, Marechal M, Doornbusch GI, Naret I, Busscher HJ, & Van Steenberghe D (1993) An *in vivo* study of the influence of surface roughness of implants on the microbiology of supra- and subgingival plaque *Journal of Dental Research* **72(9)** 1304-1309.
14. Bollen CM, Papaioanno W, Van Eldere J, Schepers E, Quirynen M, & van Steenberghe D (1996) The influence of abutment surface roughness on plaque accumulation and peri-implant mucositis *Clinical Oral Implants Research* **7(3)** 201-211.
15. Oh WS, Delong R, &Anusavice KJ (2002) Factors affecting enamel and ceramic wear: A literature review *Journal of Prosthetic Dentistry* **87(4)** 451-459.
16. de Jager N, Feilzer AJ, & Davidson CL (2000) The influence of surface roughness on porcelain strength *Dental Materials* **16(6)** 381-388.
17. Lohbauer U, Müller FA, & Petschelt A (2008) Influence of surface roughness on mechanical strength of resin composite versus glass ceramic materials *Dental Materials* **24(2)** 250-256.
18. Heintze SD, Forjanic M, & Rousson V (2006) Surface roughness and gloss of dental materials as a function of force and polishing time *in vitro Dental Materials* **22(2)** 146-165.
19. Awad D, Stawarczyk B, Liebermann A, & Ilie N (2015) Translucency of esthetic dental restorative CAD/CAM materials and composite resins with respect to thickness and surface roughness *Journal of Prosthetic Dentistry* **113(6)** 534-540.
20. Motro PF, Kursoglu P, & Kazazoglu E (2012) Effects of different surface treatments on stainability of ceramics *Journal of Prosthetic Dentistry* **108(4)** 231-237.
21. Brunot-Gohin C, Duval JL, Azogui EE, Jannetta R, Pezron I, Laurent-Maquin D, Gangloff SC, & Egles C (2013) Soft tissue adhesion of polished versus glazed lithium disilicate ceramic for dental applications *Dental Materials* **29(9)** e205-e212.
22. Odatsu T, Jimbo R, Wennerberg A, Watanabe I, & Sawase T (2013) Effect of polishing and finishing procedures on the surface integrity of restorative ceramics *American Journal of Dentistry* **26(1)** 51-55.
23. Antonson SA, Yazici AR, Kilinc E, Antonson DE, & Hardigan PC (2001) Comparison of different finishing/polishing systems on surface roughness and gloss of resin composites *Journal of Dentistry* **39(Supplement 1)** e9-e17.
24. The European Standard (2004) EN 623-624 Advanced technical ceramics. Monolithic ceramics. General and textures properties. Part 4: Determination of surface roughness. Brussels (B): *European Committee for Standardization*.
25. ISO-Standards (1999) EN ISO 2813 Specular gloss *Geneve: International Organization for Standardization*.
26. Zinelis S, Thomas A, Syres K, Silikas N, & Eliades G (2010). Surface characterization of zirconia dental implants *Dental Materials* **26(4)** 295-305.
27. Wang F, Chen JH, & Wang H (2009) Surface roughness of a novel dental porcelain following different polishing

procedures *International Journal of Prosthodontics* 2009 **22(2)** 178-180.

28. Sasahara RM, Ribeiro Fda C, Cesar PF, & Yoshimura HN (2006) Influence of the finishing technique on surface roughness of dental porcelains with different microstructures *Operative Dentistry* **31(5)** 577-583.
29. Yilmaz K, & Ozkan P (2010) Profilometer evaluation of the effect of various polishing methods on the surface roughness in dental ceramics of different structures subjected to repeated firings *Quintessence International* 2010 **41(7)** e125-e131.
30. Flury S, Lussi A, & Zimmerli B (2010) Performance of different polishing techniques for direct CAD/CAM ceramic restorations *Operative Dentistry* **35(4)** 470-481.
31. Whitehead SA, Shearer AC, Watts DC, & Wilson NH (1995) Comparison of methods for measuring surface roughness of ceramic *Journal of Oral Rehabilitation* **22(6)** 421-427.
32. Vichi A, Louca C, Corciolani G, & Ferrari M (2011) Color related to ceramic and zirconia restorations: A review *Dental Materials* **27(1)** 97-108.
33. Ohara N, Koizumi H, Matsumoto Y, Nakayama D, Ogino T, & Matsumura H (2009) Surface roughness and gloss of indirect composites etched with acidulated phosphate fluoride solution *Acta Odontologica Scandinavica* **67(5)** 313-320.
34. Covey DA, Barnes C, Watanabe H, & Johnson WW (2011) Effects of a paste-free prophylaxis polishing cup and various prophylaxis polishing pastes on tooth enamel and restorative materials *General Dentistry* **59(6)** 466-473.
35. Kakaboura A, Fragouli M, Rahiotis C, & Silikas N (2007) Evaluation of surface characteristics of dental composites using profilometry, scanning electron, atomic force microscopy and gloss-meter *Journal of Materials Science: Materials in Medicine* **18(1)** 155-163.
36. O'Brien WJ, Johnston WM, Fanian F, & Lambert S (1984) The surface roughness and gloss of composites *Journal of Dental Research* **63(5)** 685-688.
37. Tholt de Vasconcellos B, Miranda-Júnior 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.
38. Lawson NC, Janyavula S, Syklawer S, McLaren EA, & Burgess JO (2014) Wear of enamel opposing zirconia and lithium disilicate after adjustment, polishing and glazing *Journal of Dentistry* **42(12)** 1586-1591.
39. Akar GC, Pekkan G, Çal E, Eskitaşçıoğlu G, & Özcan M (2014) Effects of surface-finishing protocols on the roughness, color change, and translucency of different ceramic systems *Journal of Prosthetic Dentistry* **112(2)** 314-321.
40. Amer R, Kürklü D, & Johnston W (2015) Effect of simulated mastication on the surface roughness of three ceramic systems *Journal of Prosthetic Dentistry* **114(2)** 260-265.
41. Preis V, Weiser F, Handel G, & Rosentritt M (2013) Wear performance of monolithic dental ceramics with different surface treatments *Quintessence International* **44(5)** 393-405.
42. Kawai K, Urano M, & Ebisu S (2000) Effect of surface roughness of porcelain on adhesion of bacteria and their synthesizing glucans *Journal of Prosthetic Dentistry* **83(6)** 664-667.
43. Fraga S, Valandro LF, Bottino MA, & May LG (2015) Hard machining, glaze firing and hydrofluoric acid etching: Do these procedures affect the flexural strength of a leucite glass-ceramic? *Dental Materials* **31(7)** e131-e140.
44. Kelly JR, Nishimura I, & Campbell SD (1996) Ceramics in dentistry: Historical roots and current perspectives *Journal of Prosthetic Dentistry* **75(1)** 18-32.
45. Silva TM, Salvia AC, Carvalho RF, Pagani C, Rocha DM, & Silva EG (2014) Polishing for glass ceramics: Which protocol? *Journal of Prosthodontic Research* **58(3)** 160-170.
46. Al-Wahadni AM, & Martin DM (1999) An *in vitro* investigation into the wear effects of glazed, unglazed and refinished dental porcelain on an opposing material *Journal of Oral Rehabilitation* **26(6)** 538-546.
47. Goharian P, Nemati A, Shabanian M & Afshar A (2010) Properties, crystallization mechanism and microstructure of lithium disilicate glass-ceramic *Journal of Non-Crystalline Solids* **365(4-5)** 208-214.
48. Kou W, Molin M, & Sjögren G (2006) Surface roughness of five different dental ceramic core materials after grinding and polishing *Journal of Oral Rehabilitation* **33(2)** 117-124.
49. Vo DT, Arola D, Romberg E, Driscoll CF, Jabra-Rizk MA, & Masri R (2015) Adherence of *Streptococcus mutans* on lithium disilicate porcelain specimens *Journal of Prosthetic Dentistry* **114(5)** 696-701.
50. Asai T, Kazama R, Fukushima M, & Okiji T (2010) Effect of overglazed and polished surface finishes on the compressive fracture strength of machinable ceramic materials *Dental Materials Journal* **29(6)** 661-667.
51. Addison O, Cao X, Sunnar P, & Fleming GJ (2012) Machining variability impacts on the strength of a "chairside" CAD-CAM ceramic *Dental Materials* **28(8)** 880-887.
52. Monaco C, Arena A, & Özcan M (2014) Effect of prophylactic polishing pastes on roughness and translucency of lithium disilicate ceramic *International Journal of Periodontics & Restorative Dentistry* **34(1)** e26-e29.
53. Dalkiz M, Sipahi C, & Beydemir B (2009) Effects of six surface treatment methods on the surface roughness of a low-fusing and an ultra low-fusing feldspathic ceramic material *Journal of Prosthodontics* **18(3)** 217-222.
54. Al-Shammery HA, Bubb NL, Youngson CC, Fasbinder DJ, & Wood DJ (2007) The use of confocal microscopy to assess surface roughness of two milled CAD-CAM ceramics following two polishing techniques *Dental Materials* **23(6)** 36-41.
55. Bollen CM, Papaioanno W, Van Eldere J, Schepers E, Quirynen M, & van Steenberghe D (1996) The influence of abutment surface roughness on plaque accumulation

and peri-implant mucositis *Clinical Oral Implants Research* **7(3)** 201-211.

56. Jones CS, Billington RW, & Pearson GJ (2004) The *in vivo* perception of roughness of restorations *British Dental Journal* **196(1)** 42-45.
57. Willems G, Lambrechts P, Braem M, Vuylsteke-Wauters M, & Vanherle G (1991) The surface roughness of enamel-to-enamel contact areas compared with the intrinsic roughness of dental resin composites *Journal of Dental Research* **70(9)** 1299-1305.
58. Barucci-Pfister N, & Göhring TN (2009) Subjective and objective perceptions of specular gloss and surface roughness of esthetic resin composites before and after artificial aging *American Journal of Dentistry* **22(2)** 102-110.
59. Mörmann 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(Apr)** 113-125.
60. Haywood VB, Heymann HO, & Scurria MS (1989) Effects of water, speed, and experimental instrumentation on finishing and polishing porcelain intra-orally *Dental Materials* **5(3)** 185-188.

©Operative Dentistry, 2018, **43-1,** 101-110

# A Novel Enamel and Dentin Etching Protocol Using α-hydroxy Glycolic Acid: Surface Property, Etching Pattern, and Bond Strength Studies

D Cecchin • AP Farina • CMP Vidal • AK Bedran-Russo



### Clinical Relevance

Glycolic acid is a mild etchant that effectively etches enamel and dentin surfaces, a necessary step to successfully placing dental adhesive restorations.


## SUMMARY

**Objectives: To determine the use of α-hydroxy glycolic acid (GA) as a surface pretreatment for dental restorative applications. The etching pattern of GA pretreatment of dental hard tissues was assessed by surface microhardness and scanning electron microscopy (SEM). The effectiveness of GA surface etching on the enamel and dentin resin bond strengths was assessed using two etchant application modes (rubbing and no rubbing) and three adhesive systems (Single Bond [SB], One Step Plus [OSP], and Scotchbond Universal [SBU]).**

**Methods: Knoop microhardness measurements were carried out on polished enamel and dentin surfaces before and after treatment with 35% GA, 35% phosphoric acid (PA), or distilled water (control group) for 30 seconds. The microtensile bond strength test was carried out on enamel and dentin. Ultrastructural analysis of the surface and interfacial interaction was qualitatively accomplished using SEM.**

**Results: Etching with either PA or GA significantly decreased the enamel microhardness, with GA being significantly less aggressive than PA ($p<0.001$), while both acids showed similar decreases in dentin microhardness ($p=0.810$). SEM revealed similar etching patterns of GA and PA, while apparently a thinner hybrid layer was observed for GA groups. In**


Doglas Cecchin, Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA; and Department of Restorative Dentistry, College of Dentistry, University of Passo Fundo, Passo Fundo, RS, Brazil

Ana Paula Farina, Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA; and Department of Restorative Dentistry, College of Dentistry, University of Passo Fundo, Passo Fundo, RS, Brazil

Cristina MP Vidal, Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA; and Department of Operative Dentistry, College of Dentistry and Dental Clinics, University of Iowa, Iowa City, IA, USA

*Ana K Bedran-Russo, Department of Restorative Dentistry, College of Dentistry, University of Illinois at Chicago, Chicago, IL, USA

*Corresponding author: 801 South Paulina Street, Room 531, Chicago, IL 60612, USA; e-mail: bedran@uic.edu

DOI: 10.2341/16-136-L

**dentin, the bond strengths were statistically similar between PA and GA groups, regardless of the etchant application mode ($p>0.05$). However, rubbing of GA enhanced the bond strength to enamel. PA and GA significantly increased the SBU bond strength to enamel when compared to SB and OSP ($p<0.05$).**

**Conclusions: GA effectively etched enamel and dentin surfaces, resulting in bond strength values similar to those associated with traditional PA. GA is a suitable enamel and dentin surface etchant for adhesive restorative procedures.**

## INTRODUCTION

Resin composite materials are widely used to conservatively restore enamel and dentin after caries or trauma and/or to satisfy esthetic demands. The use of dental adhesive is a necessary step to effectively bond resin composites to dental hard tissues. Dental adhesives rely on micromechanical retention of a resin polymer to enamel and dentin substrates. Such a mechanism occurs through a surface conditioning step, resulting in the superficial tissue demineralization and infiltration of resin monomers, which form resin microtags and a hybrid layer.[1,2] Dental enamel is a highly mineralized tissue composed of 96 wt% mineral, ~3 wt% water, and only ~1 wt% residual biomacromolecules.[3] On the other hand, dentin presents an intricate mineralized organic matrix comprised of ~70 wt% mineral, ~20 wt% organic components (mainly type I collagen), and ~10 wt% water.[4] While clinically successful, the adhesion of hydrophobic and hydrophilic blends of resins to the dentin organic matrix is based on a complex and technique-sensitive mechanism contributing to the short service life of resin composite restorations.[2,5] Because dentin is the bulk tissue, the complex composition and structure of dentin serve as important constraints for technological advances in adhesive dentistry.

Phosphoric acid (PA) in the form of liquid or gel, and with concentrations ranging from 30% to 40%, has been almost exclusively used as a surface conditioner of enamel and dentin prior to the placement of dental adhesives.[1] PA surface treatment increases surface wettability, roughness, and hardness of enamel and dentin.[6] Adequate resin bond strength to PA-etched enamel is highly predictable.[7,8] In dentin, PA demineralizes the peritubular and intertubular dentin, exposing a matrix rich in type I collagen fibrils.[2,9] The high acidity of PA may induce structural changes in dentin collagen[6] and activates the inactive proforms of endogenous dentin proteases associated with resin-dentin degradation.[10] It has been shown that the depth of dentin demineralization does not correlate with bonding effectiveness,[10] leading to strategies such as short application times,[6] lower acid concentrations,[11] and use of alternative dental surface conditioners.[12]

Glycolic acid (GA) is the smallest of a group of naturally occurring organic acids known as α-hydroxy acids. GA is widely used in dermatology to promote skin chemical peeling,[13] while poly(lactic-co-glycolic acid) has been used to promote wound healing in skin and bone.[14] GA is colorless, odorless, and water soluble[15,16] and is reported to elevate collagen synthesis and fibroblast proliferation in *in vivo* and *in vitro* studies.[16-18] Because of such characteristics, GA is potentially attractive for dental applications as a surface conditioner during restorative procedures. Such application is explored for the first time in this study to promote surface demineralization of enamel and dentin for resin adhesion to dental surfaces.

The aim of this study was to determine the effectiveness of GA as a dental surface conditioner for adhesion of commercially available adhesive systems to enamel and dentin. The enamel and dentin surface demineralization pattern was assessed by microhardness measurement and ultrastructural surface morphology using scanning electron microscopy (SEM). Microtensile bond strength (μTBS) to enamel and dentin was evaluated using three different adhesive systems and two application modes (rubbing and no rubbing). The null hypothesis tested was that surface conditioning of enamel and dentin using GA would result in similar surface etching pattern, surface microhardness, and adhesive bond strength to standard phosphoric acid etching.

## METHODS AND MATERIALS

### Study Design

The microhardness test was proposed to determine the demineralization effect of experimental etchants in enamel and dentin. The pattern of surface etching was investigated under SEM. The effectiveness of GA etching for resin adhesion was investigated by μTBS test on enamel and dentin using different etchant application modes (rubbing and no rubbing) and three adhesives (Adper Single Bond [SB; 3M ESPE, St Paul, MN, USA], One Step Plus [OSP; Bisco Inc, Schaumburg, IL, USA], and Scotchbond

Table 1: *Materials, Application Mode, and Manufacturer (Batch No.) of Acid Solutions and Adhesive Systems*

| Material | Application Procedure | Composition[a] | Manufacturer [Batch No.] |
|---|---|---|---|
| 35% Phosphoric acid | Enamel: acid etching for 30 s (rubbing or no rubbing), rinse with distilled water for 30 s and dry. Dentin: acid etching for 30 s (rubbing or no rubbing), rinse with distilled water for 30 s and blot dry. | Dilution in distilled water from a 85% phosphoric acid solution. | Sigma [MKBR6573V] |
| 35% Glycolic acid | Enamel: acid etching for 30 s (rubbing or no rubbing), rinse with distilled water for 30 s and dry. Dentin: acid etching for 30 s (rubbing or no rubbing), rinse with distilled water for 30 s and blot dry. | Preparation in distilled water using 97% Glycolic acid powder. | Sigma [BCBH8450V] |
| Adper Single Bond | 1. Apply the adhesive with the applicator to the entire tooth surface and rub for 15 s.<br>2. Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely.<br>3. Polymerize the adhesive with a curing light for 40 s. | Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, initiators, water, and ethanol. | 3M ESPE (St Paul, MN, USA) [N561025] |
| Scotchbond Universal Adhesive | 1. Apply the adhesive with the applicator to the entire tooth surface and rub for 15 s.<br>2. Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely.<br>3. Polymerize the adhesive with a curing light for 40 s. | MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane. | 3M ESPE (St Paul, MN, USA) [475230] |
| One Step Plus | 1. Apply the adhesive with the applicator to the entire tooth surface and rub for 15 s.<br>2. Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely.<br>3. Polymerize the adhesive with a curing light for 40 s. | Bis-GMA, HEMA, BPDM, acetone. Fillers: 8.5% wt. glass ionomer. | Bisco (Schaumburg, IL, USA) [1400005745] |
| Filtek™ Supreme Ultra | Apply three layers of 1mm each light cured for 40 s. | Matrix: Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA. Filler: Aggregated zirconia /silica clusters (20 nm silica and 4-11nm zirconia particles), average cluster 0.6-1nm; nonagglomerated /non-aggregated 20 nm silica and 4-11 nm zirconia. Filler load: 78.5% wt (63.3% vol). | 3M ESPE (St Paul, MN, USA) [6028A2B] |

*Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, methacryloyloxydecyl dihydrogen phosphate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.*

[a] *Composition of the materials, as provided by the manufacturers.*

Universal Adhesive [SBU; 3M ESPE]). The morphology of the adhesive interfaces was assessed by SEM. Preparation of acid solutions, composition of adhesives, application modes, and manufacturers are detailed in Table 1.

Extracted human central incisors and molars were selected, cleaned, and kept frozen (−20°C) until use. Studies on enamel and dentin were carried out on the polished enamel surface of central incisors and the occlusal dentin surface of third molars, respectively.

## Preparation of Etchant Solutions

A liquid formulation of 35% GA (pH 1.2) was prepared using stock solution (70% GA; Sigma-Aldrich, St Louis, MO, USA). To eliminate potential effects of additives in commercial formulations, a

liquid formulation of 35% PA (pH 0.12) was prepared using a stock solution (85% PA; LabChem Inc, Pittsburgh, PA, USA).

### Microhardness Measurements

Enamel and dentin surfaces were exposed, embedded in epoxy resin, and polished on a water-cooled polishing unit (EcoMet 3000, Buehler, Lake Bluff, IL, USA) with abrasive paper (400-, 600-, and 1200-grit) followed by 0.9, 0.6, 0.3, and 0.1 μm diamond alumina suspensions (Metaldi Supreme, Buehler Ltd). The polished specimens were cleaned ultrasonically in deionized water for 15 minutes to remove residual polishing material. Specimens from the same tooth were etched either with 35% PA or 35% GA for 30 seconds under passive application mode (Table 1) (n=8). An unetched surface was included as a control group. Surface microhardness measurements of dentin and enamel were carried out on a microhardness instrument (Leco, LM700at, St Joseph, MI, USA) using a Knoop hardness tip and 25*g* load force for 15 seconds.[19] The microhardness of each specimen was determined by 20 indentations done after surface treatment.

### Surface Etching Pattern—SEM

The surfaces of enamel and dentin were prepared as described above and etched using either active or passive application modes (n=3). The specimens were dehydrated in ascending concentrations of ethanol, fixed in hexamethyldisilazane (Sigma-Aldrich),[20] mounted on aluminum stubs, and gold sputter-coated (SEM Coating Unit E5150, Polaron Equipment, Hatfield, PA, USA), and the micromorphology of the surfaces was assessed in an SEM (S-3000N Hitachi, Tokyo, Japan).

### μTBS and Adhesive Interface Morphology by SEM

Occlusal dentin of molars and buccal surfaces of upper central incisors were prepared for resin bond strength studies to dentin and enamel, respectively. The buccal surface of upper incisors was flattened with #320 grit. The occlusal enamel of molars was removed with a low-speed diamond saw (Isomet 1000, Buehler Ltd) under water-cooling to expose midcoronal dentin. Both the exposed enamel and dentin surfaces were ground with 600-grit abrasive paper for 60 seconds under wet conditions to produce a standard smear layer.[21] Incisors and molars were randomly divided into 12 groups (n=8) according to the etching protocols and adhesive strategies. Acid etching was done as described in Table 1 using either active or passive mode, and adhesive systems application was performed strictly in accordance with the respective manufacturer's instructions. After etching and bonding procedures, a nanohybrid resin composite (Filtek™ Supreme Ultra, 3M ESPE) was built in three increments of 1 mm each and light-cured for 40 seconds. All adhesive systems and resin composite were light-cured using a halogen light-curing unit operated at 600 mW/cm$^2$ (Optilux, Demetron Res Corp, Danbury, CT, USA).

After bonding procedures, teeth were stored in distilled water for 24 hours at 37°C and then were sectioned longitudinally across the bonded interface with a low-speed diamond saw (Isomet 1000, Buehler Ltd) under water irrigation to obtain resin-enamel or resin-dentin specimens with a cross-sectional area of approximately 0.8 mm$^2$. Four resin-dentin beams were obtained from each tooth, totaling 32 beams tested per group. The specimens were glued to a jig and tested in tension at a crosshead speed of 1 mm/min using a microtensile tester machine (Bisco, Schaumburg, IL, USA). The debonded interfaces were visualized under a stereomicroscope at magnifications up to 40×, and failure mode was classified as adhesive, cohesive in dental substrate (enamel or dentin), cohesive in composite, or mixed failures.[22]

Three additional resin-dentin specimens from each group were randomly selected for interfacial analysis using SEM. Specimens were embedded in epoxy resin and gloss polished using carbide paper and diamond pastes. Processing for SEM was carried out as described above and specimens were visualized in the same microscope.

### Statistical Analysis

Normal distribution of the microhardness data was confirmed by Kolmogorov-Smirnov test. Enamel and dentin microhardness were analyzed by one-way analysis of variance (ANOVA) and post hoc Tukey tests ($p<0.05$). The Levene test shows that the enamel and dentin bond strength data are not normally distributed ($p=0.027$ and $p<0.001$, respectively). The bond strength of each tooth (n = 8 per group) was averaged from 4 tested beams and the enamel and dentin bond strength data were analyzed by three-way ANOVA test. When applicable, a one-way ANOVA followed by post hoc Games Howell test was performed comparing all groups ($p<0.05$). Failure mode distribution was evaluated by Chi-square test ($p<0.05$).

Table 2: *Dentin and Enamel Knoop Microhardness Values (KHN) (Mean [Standard Deviations]) for the Etching Protocols*[a]

| Groups | Microhardness, KHN | |
|---|---|---|
| | Enamel | Dentin |
| Control | 311.89 (18.90) A | 62.20 (3.28) A |
| 35% Phosphoric acid | 200.87 (11.56) C | 49.47 (2.22) B |
| 35% Glycolic acid | 287.35 (4.83) B | 48.99 (1.70) B |

[a] *Means followed by different letters are significantly different at* $p < 0.05$. *Valid comparison only to column values.*

## RESULTS

The microhardness results are shown in Table 2. Statistically significant differences were observed among groups for enamel and dentin ($p<0.001$). Surface treatment with either PA or GA resulted in a statistically significant decrease in the enamel microhardness ($p<0.001$ and $p=0.022$, respectively), while GA was significantly less aggressive than PA ($p<0.001$). Both acids reduced the dentin surface microhardness when compared to the control, with no statistically significant differences between PA and GA ($p=0.810$).

The enamel and dentin surfaces etched with PA and GA revealed a similar etching pattern. It is evident that both etchants exposed enamel prism rods as a result of the preferential interprismatic apatite dissolution (Figure 1a-d). Both etchants removed the smear layer, dissolved intratubular dentin, and exposed collagen fibrils, which is more evident within the dentin tubules (Figure 1e-h). No visual variations could be observed between active and passive applications.

The enamel and dentin μTBS results are depicted in Tables 3 and 4, respectively. On enamel, there were no statistically significant interactions among factors (adhesives vs acids vs application modes, $p>0.05$) and between acids ($p=0.602$). Statistically significant differences were found between application modes ($p=0.003$) and adhesive systems ($p<0.001$). Rubbing the etchant yielded significantly higher bond strength when compared to use of the no rubbing mode ($p=0.0030$). The enamel bond strength of SBU was significantly higher than that of SB and OSP ($p<0.001$), with no statistically significant different between SB and OSP ($p=0.228$). On dentin, significant interactions were observed among factors (adhesives vs acids vs application modes, $p<0.05$), except for adhesive vs acid ($p=0.882$). Overall, SBU presented the highest bond strength values, with no statistical difference compared to OSP ($p=0.110$). The dentin bond strength of the three adhesive systems was differently affected by the acid and application mode, as detailed in Table 4. Most notably, GA significantly increased the bond strength of SB and OSP when compared to PA under rubbing mode ($p>0.05$), and the bond strength of SBU was not significantly affected by the etchant ($p<0.05$).

Statistically significant differences in failure mode distribution were observed for enamel ($p=0.0106$) and dentin ($p<0.0001$) adhesive interfaces. No cohesive failures were observed for enamel. Adhesive failure was predominant for SB and OSP, and there was an increase in mixed failures for SBU, for both GA and PA (Table 3). In dentin interfaces, all failure modes were observed, but mixed and cohesive modes in resin failures were predominant.

Representative SEM images of the resin-enamel interface are shown in Figure 2. The interfaces of all adhesives exhibited similar morphology, with the presence of resin tags and micro-tags.

No visual differences were observed between etchant application modes (rubbing or no rubbing). However, the adhesive layers were thicker for the SBU when compared to the SB and OSP. Representative SEM images of the resin-dentin interfaces are shown in Figure 3. The presence of resin tags in dentin tubules was seen in all groups. An apparently thicker hybrid layer could be noted in PA when compared to GA.

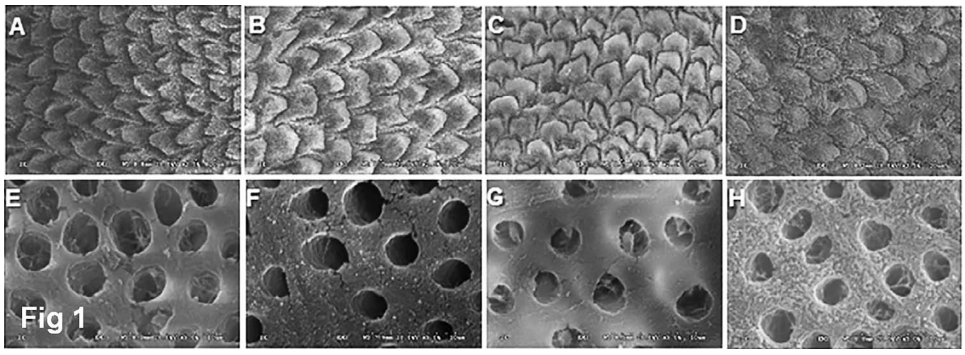



Figure 1. *Micromorphology of enamel and dentin surfaces. (a) PA no rubbing in enamel. (b) PA rubbing in enamel. (c) GA no rubbing in enamel. (d) GA rubbing in enamel. (e) PA no rubbing in dentin. (f) PA rubbing in dentin. (g) GA no rubbing in dentin. (h) GA rubbing in dentin.*

Table 3: *Results of the Microtensile Bond Strength (μTBS) to Enamel (Means [Standard Deviations]) and Failure Mode of the Experimental Groups*[a]

| Adhesive System | Enamel-resin Bond Strength | | | |
|---|---|---|---|---|
| | Phosphoric Acid | | Glycolic Acid | |
| | Rubbing δ | No Rubbing | Rubbing δ | No Rubbing |
| Single Bond[b] | 35.37 (7.98) (15A/15M) | 35.83 (5.54) (17A/13M) | 33.69 (8.49) (17A/13M) | 37.76 (8.33) (15A/15M) |
| One Step Plus[b] | 33.03 (10.42) (20A/10M) | 35.80 (9.92) (18A/12M) | 30.97 (8.35) (21A/9M) | 35.65 (7.46) (20A/10M) |
| Scotch Bond Universal[a] | 48.50 (9.23) (8A/22M) | 50.99 (11.39) (6A/24M) | 47.95 (8.30) (8A/22M) | 50.58 (9.58 (3A/27M) |

[a] *There were no statistically significant interactions among the study factors, adhesive vs application time vs etchant ($p>0.05$). Thus, letters and symbols next indicate statistical differences among pooled data of the study variable. Superscripted lowercase letters indicate statistically significant differences between dental adhesives ($p<0.05$). Symbol (δ) indicates statistical difference between rubbing and non-rubbing ($p<0.003$). Failure mode: A, adhesive; and M, mixed.*

## DISCUSSION

In addition to being simple and clinically acceptable, acid etching is a required step in bonding techniques. The rationale for the current study is based on exploring alternative material with which to etch enamel and dentin using a less aggressive and potentially more biocompatible acid. The treatment with GA etched the surfaces of enamel and dentin, leading to significant changes in the surface microhardness and etching pattern. Such changes favored bonding of the three different commercially available dentin adhesives to enamel and dentin, with bond strength values comparable to those of PA-treated surfaces. Thus, the null hypothesis was partially rejected.

In this study, the decrease in enamel microhardness mean values was significantly greater for PA than for GA, while similar decreases in the microhardness of dentin were observed for both acids. The lower pH of PA (pH=0.12) compared to GA (pH=1.2) may have accounted for a more aggressive pattern of demineralization of the highly mineralized enamel. Furthermore, the $pK_a$ of PA is only slightly superior to that of GA (2.16 and 3.83, respectively); thus, the acid dissociation is relatively strong for both solutions. Similar superficial demineralization patterns were observed for both acid solutions (Figure 1), although the interfacial microscopy data support a deeper demineralization of dentin by PA (Figure 3). Moreover, bond strength values for GA etching in enamel and dentin were comparable to those obtained with PA etching, with or without rubbing (Tables 3 and 4), suggesting that the depth of demineralization did not have a significant effect on the bond strength of the investigated dental adhesives. Taken together, these findings indicate that GA can be used to etch enamel and dentin in a clinically relevant condition while being relatively less aggressive than PA.

In addition to removal of minerals, PA can also interfere with dentin organic components. It has the potential to activate endogenous gelatinolytic/collagenolytic enzymes (matrix metalloproteinases and cysteine cathepsins) able to cleave collagen at the bonded interface over time, accelerating the degradation of the adhesive interface.[23-25] Such activation is of particular concern in dentin and the pH of the etching solution.[26] The effect of GA on the activation of endogenous proteases needs to be further investigated.

The findings demonstrate that acid rubbing during the etching procedure can significantly affect

Table 4: *Results of the Microtensile Bond Strength (μTBS) to Dentin (Means [Standard Deviations]) and Failure Mode of the Experimental Groups*[a]

| Adhesive System | Dentin-resin Bond Strength | | | |
|---|---|---|---|---|
| | Phosphoric Acid | | Glycolic Acid | |
| | Rubbing | No Rubbing | Rubbing | No Rubbing |
| Single Bond[b] | 48.55 (6.92) B (1A/16M/ 1CC/11CD) | 47.52 (8.15) B (5A/10M/ 5CC/11CD) | 57.50 (6.90) A (2A/8M/ 7CC/13CD) | 47.84 (8.20) B (7A/11M/ 9CC/3CD) |
| One Step Plus[ab] | 46.15 (7.05) B (7A/8M/ 8CC/7CD) | 52.75 (8.29) A (4A/11M/ 10CC/5CD) | 53.63 (7.97) A (4A/15M/ 6CC/5CD) | 51.77 (12.69) AB (4A/17M/ 7CC/2CD) |
| Scotch Bond Universal[a] | 55.11 (6.18) A (3A/8M/ 8CC11CD) | 48.05 (7.87) B (6A/12M/ 11CC/1CD) | 58.15 (7.99) A (3A/9M/ 12CC/6CD) | 52.51 (9.22) AB (5A/6M/ 11CC/8CD) |

[a] *Different superscripted lowercase letters indicate statistically significant differences among dental adhesives ($p<0.05$). Different uppercase letters indicate statistically significant differences among groups in each row ($p<0.05$). Failure mode: A, adhesive; M, mixed; CC, cohesive in composite; and, CD, cohesive in dentin.*

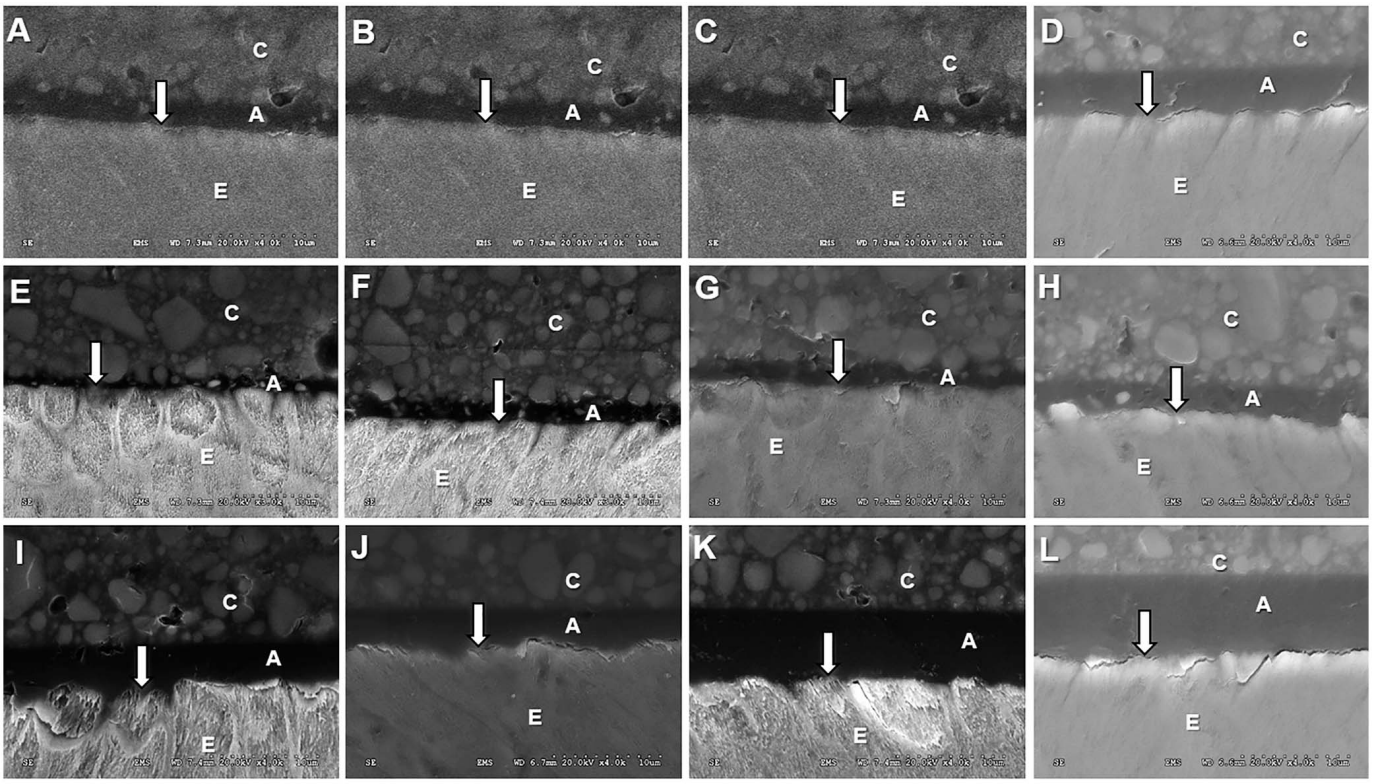



Figure 2. *Representative micrographs of resin-enamel interfaces for the different strategies (PA and GA etching; rubbing and no rubbing) and of three adhesive systems (SB, OSP, and SBU). (a) PA no rubbing and SB. (b) PA rubbing and SB. (c) GA no rubbing and SB. (d) GA rubbing and SB. (e) PA no rubbing and OSP. (f) PA rubbing and OSP. (g) GA no rubbing and OSP. (h) GA rubbing and OSP. (i) PA no rubbing and SBU. (j) PA rubbing and SBU. (k) GA no rubbing and SBU. (l) GA rubbing and SBU. A = adhesive layer; C = composite resin; E = enamel; the adhesive/enamel interface is indicated by arrows.*

the adhesive bond strength to enamel for both PA and GA. Previous studies[27-30] have shown that the active application mode of adhesive itself may improve the immediate and long-term bonding performance of self-etching systems to dentin. Active application of the adhesive improves performance of universal adhesives to enamel when compared to passive mode, with results similar to those associated with the etch-and-rinse technique.[30] In the present study, SEM analyses showed no apparent morphological differences in PA- or GA-etched surfaces with or without rubbing that could explain the outcomes. It may be possible that rubbing of the acid on enamel may have disturbed the exposed hydroxyapatite crystals and chemical composition and weakened the micromechanical retention of the enamel microtags, apparently more significantly than was seen with the GA-etched surfaces. Further characterization of mineral composition at the enamel surfaces and enamel microtag bonding mechanisms of GA to enamel could clarify the effect of application mode.

Hence, adhesive failures were predominant with SB and OSP, implying that the weak link was the bond between the resin and enamel. SBU exhibited predominantly mixed failures (Figure 3), with similar distribution between acid and acid application mode. Although mode of failure in dentin showed variation among the three adhesives, there was no difference in the failure pattern between the GA and PA groups (Table 4).

The enamel and dentin bond strength results indicate that GA is effective in promoting adhesion to dental tissues, regardless of the application mode and adhesive chemistry. It is important to emphasize that the complex composition of dentin warrants a sophisticated bonding mechanism and protocol, in which the water/solvent evaporation after demineralization is crucial and is frequently not done completely.[31] Since GA has lower depth of demineralization than PA, it is plausible that deep areas of residual water/solvent are not as frequent as in PA-treated dentin. In addition, the formation of an apparently thinner hybrid layer was evident when using the GA (Figure 3). However, hybrid layer

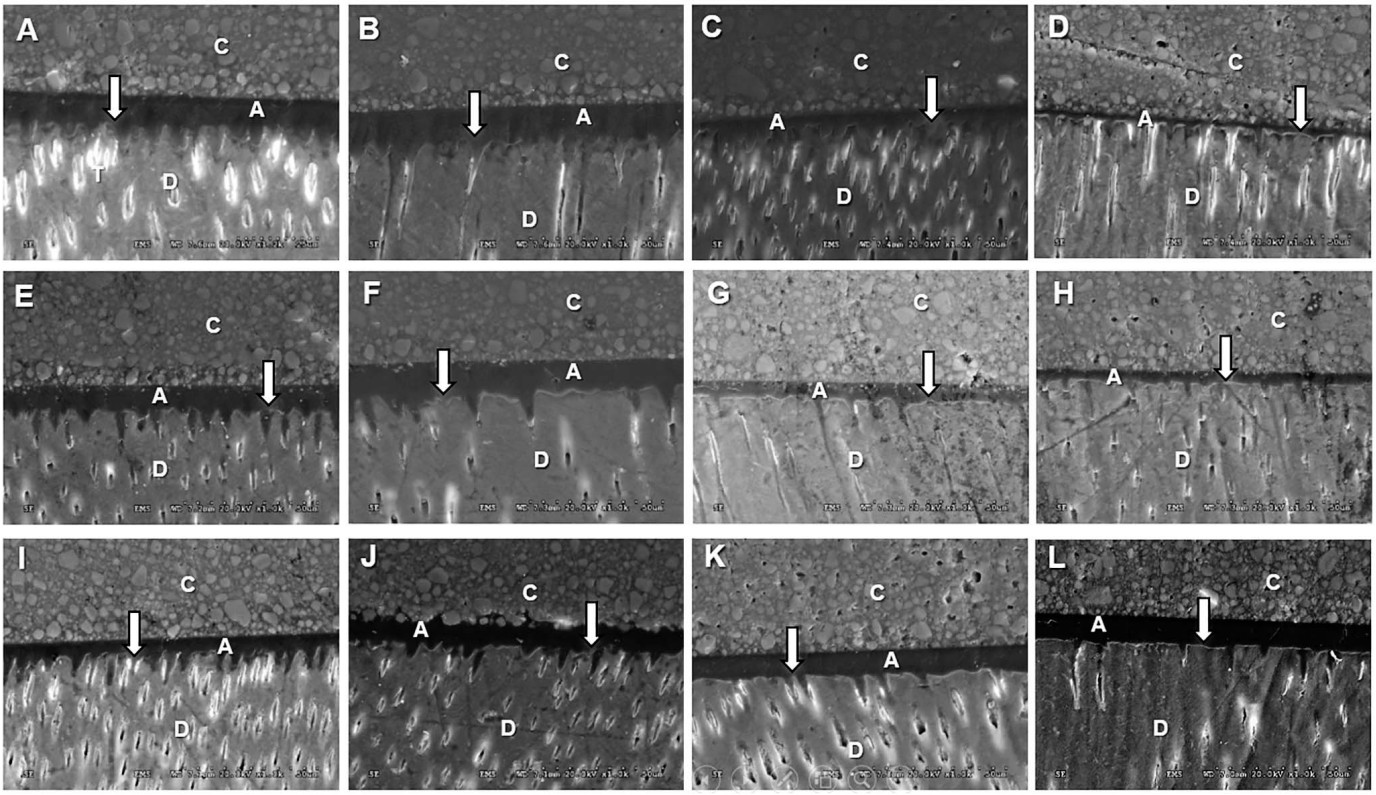



Figure 3. *Micrographs of resin-dentin interfaces for the different strategies (PA and GA etching; rubbing and no rubbing) and of three adhesive systems (SB, OSP, and SBU). (a) PA no rubbing and SB. (b) PA rubbing and SB. (c) GA no rubbing and SB. (d) GA rubbing and SB. (e) PA no rubbing and OSP. (f) PA rubbing and OSP. (g) GA no rubbing and OSP. (h) GA rubbing and OSP. (i) PA no rubbing and SBU. (j) PA rubbing and SBU. (k) GA no rubbing and SBU. (l) GA rubbing and SBU. A = adhesive layer; C = composite resin; D = dentin; H = hybrid layer; T = resin tag.*

thickness does not correlate with bond strength or better bonding quality,[32] as was also observed in the dentin bond strength data. It can be speculated that the less aggressive GA associated with the formation of thinner hybrid layers might reduce the discrepancies between demineralization and adhesive infiltration, potentially resulting in less exposed partially demineralized dentin areas prone to degradation. Long-term data and permeability studies to evaluate the hybrid layer overtime are necessary to confirm such an assumption.

## CONCLUSIONS

The demineralization with either PA or GA significantly decreased enamel and dentin surface microhardness and produced similar surface demineralization morphology. The etching with GA in enamel and dentin resulted in immediate bond strength values comparable to those of PA using three different adhesive systems and adhesion strategies. GA and PA exhibited similar etching effectiveness at clinically relevant application times.

### Acknowledgements

This study was supported by NIH/NIDCR grant DE021040 and CAPES/Brazil grant BEX 17910/12-9 and BEX 17764/12-2. We would like to thank Bisco and 3M ESPE for donation of the restorative materials. The authors declare no potential conflicts of interest with respect to either the authorship or publication of this manuscript.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Illinois at Chicago. The approval code for this study is 2011-0312.

### 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 20 March 2017)*

### REFERENCES

1. 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. Adhe-

sion to enamel and dentin: Current status and future challenges *Operative Dentistry* **28(3)** 215-235.

2. Pashley DH, Tay FR, Breschi L, Tjäderhane L, Carvalho RM, Carrilho M, & Tezvergil-Mutluay A (2011) State of the art etch-and-rinse adhesives *Dental Materials* **27(1)** 1-16.
3. Gordon LM, Cohen MJ, MacRenaris KW, Pasteris JD, Seda T, & Joester D (2015) Dental materials. Amorphous intergranular phases control the properties of rodent tooth enamel. *Science* **13(347)** 746-750.
4. Marshall GW Jr, Marshall SJ, Kinney JH, & Balooch M (1997) The dentin substrate: Structure and properties related to bonding *Journal of Dentistry* **25(6)** 441-458.
5. Agee KA, Prakki A, Abu-Haimed T, Naguib GH, Nawareg MA, Tezvergil-Mutluay A, Scheffel DL, Chen C, Jang SS, Hwang H, Brackett M, Grégoire G, Tay FR, Breschi L, & Pashley DH (2015) Water distribution in dentin matrices: Bound vs. unbound water *Dental Materials* **31(3)** 205-216.
6. Zafar MS, & Ahmed N (2015) The effects of acid etching time on surface mechanical properties of dental hard tissues *Dental Materials Journal* **34(3)** 315-320.
7. 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.
8. 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.
9. Perdigao J, Frankenberger R, Rosa BT, & Breschi L (2000) New trends in dentin/enamel adhesion *American Journal of Dentistry* **13(Spec No)** 25D-30D.
10. Carvalho RM, Manso AP, Geraldeli S, Tay FR, & Pashley DH (2012) Durability of bonds and clinical success of adhesive restorations *Dental Materials* **28(1)** 72-86.
11. Li B, Zhu X, Ma L, Wang F, Liu X, Yang X, Zhou J, Tan J, Pashley DH, & Tay FR (2016) Selective demineralisation of dentine extrafibrillar minerals—A potential method to eliminate water-wet bonding in the etch-and-rinse technique *Journal of Dentistry* **52** 55-62.
12. Imbery TA, Kennedy M, Janus C, & Moon PC (2012) Evaluating EDTA as a substitute for phosphoric acid-etching of enamel and dentin *General Dentistry* **60(2)** e55-e61.
13. Rajaratnam R, Halpern J, Salim A, & Emmett C (2010) Interventions for melasma *Cochrane Database of Systematic Reviews* **7** CD003583.
14. Venkatesan J, & Kim SK (2014) Nano-hydroxyapatite composite biomaterials for bone tissue engineering—A review *Journal of Biomedical Nanotechnology* **10(10)** 3124-3140.
15. Van Scott EJ, & Yu RJ (1989) Alpha hydroxyacids: Therapeutic potentials *Canadian Journal of Dermatology* **1(5)** 108-112.
16. Thibault PK, Wlodarczyk J, & Wenck A (1998) A double-blind randomized clinical trial on the effectiveness of a daily glycolic acid 5% formulation in the treatment of photoaging *Dermatologic Surgery* **24(5)** 573-577.
17. Kim SJ, Park JH, Kim DH, Won YH, & Maibach HI (1998) Increased in vivo collagen synthesis and in vitro cell proliferative effect of glycolic acid *Dermatologic Surgery* **24(10)** 1054-1058.
18. Bernstein EF, Lee J, Brown DB, Yu R, & Van Scott E (2001) Glycolic acid treatment increases type I collagen mRNA and hyaluronic acid content of human skin *Dermatologic Surgery* **27(5)** 429-433.
19. Xie Q, Bedran-Russo AK, & Wu CD (2008) In vitro remineralization effects of grape seed extract on artificial root caries *Journal of Dentistry* **36(11)** 900-906.
20. Gunaydin Z, Yazici AR, & Cehreli ZC (2016) In vivo and in vitro effects of chlorhexidine pretreatment on immediate and aged dentin bond strengths *Operative Dentistry* **26(3)** 258-267.
21. Hu L, Xiao YH, Fang M, Gao Y, Huang L, Jia AQ, & Chen JH (2012) Effects of type I collagen degradation on the durability of three adhesive systems in the early phase of dentin bonding *PLoS One* **17(2)** e0116790.
22. Farina AP, Cecchin D, Barbizam JV, & Carlini-Júnior B (2011) Influence of endodontic irrigants on bond strength of a self-etching adhesive *Australian Endodontic Journal* **37(1)** 26-30.
23. Mazzoni A, Pashley DH, Nishitani Y, Breschi L, Mannello F, Tjäderhane L, Toledano M, Pashley EL, & Tay FR (2006) Reactivation of inactivated endogenous proteolytic activities in phosphoric acid-etched dentine by etch-and-rinse adhesives *Biomaterials* **27(25)** 4470-4476.
24. Carrilho MR, Geraldeli S, Tay F, de Goes MF, Carvalho RM, Tjäderhane L, Reis AF, Hebling J, Mazzoni A, Breschi L, & Pashley D (2007) In vivo preservation of the hybrid layer by chlorhexidine *Journal of Dental Research* **86(6)** 529-533.
25. Tersariol IL, Geraldeli S, Minciotti CL, Nascimento FD, Pääkkönen V, Martins MT, Carrilho MR, Pashley DH, Tay FR, Salo T, & Tjäderhane L (2010) Cysteine cathepsins in human dentin-pulp complex *Journal of Endodontics* **36(3)** 475-481.
26. Nishitani Y, Yoshiyama M, Wadgaonkar B, Breschi L, Mannello F, Mazzoni A, Carvalho RM, Tjäderhane L, Tay FR, & Pashley DH (2006) Activation of gelatinolytic/collagenolytic activity in dentin by self-etching adhesives *European Journal of Oral Science* **114(2)** 160-166.
27. 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.
28. 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.
29. Pucci CR, de Oliveira RS, Caneppele TM, Torres CR, Borges AB, & Tay FR (2013) Effects of surface treatment, hydration and application method on the bond strength of a silorane adhesive and resin system to dentine *Journal of Dentistry* **41(3)** 278-286.

30. Loguercio AD, Muñoz MA, Luque-Martinez I, Hass V, Reis A, & Perdigão J (2015) Does active application of universal adhesives to enamel in self-etch mode improve their performance? *Journal of Dentistry* **43(9)** 1060-1070.

31. Agee KA, Prakki A, Abu-Haimed T, Naguib GH, Nawareg MA, Tezvergil-Mutluay A, Scheffel DL, Chen C, Jang SS, Hwang H, Brackett M, Grégoire G, Tay FR, Breschi L, & Pashley DH (2015) Water distribution in dentin matrices: Bound vs. unbound water *Dental Materials* **31(3)** 205-216.

32. Lodovici E, Reis A, Geraldeli S, Ferracane JL, Ballester RY, & Rodrigues Filho LE (2009) Does adhesive thickness affect resin-dentin bond strength after thermal/load cycling? *Operative Dentistry* **34(1)** 58-64.

# Faculty Positions

**OPERATIVE DENTISTRY GRADUATE PROGRAM DIRECTOR**

The University of North Carolina at Chapel Hill, School of Dentistry seeks an outstanding, experienced academic leader to serve as the Operative Dentistry Graduate Program Director.

This position is a full-time, open rank, tenure-track position or fixed-term position in the Department of Operative Dentistry. Other expectations include teaching operative dentistry in the graduate and DDS programs and conducting research in related areas. Successful applicants should have the ability to rapidly establish a robust intramural practice within the UNC School of Dentistry's Dental Faculty Practice. In addition to excellent clinical credentials, including demonstrable restorative talent, the individual should have experience in organizational and interpersonal skills, mentoring, leadership, and a commitment to diversity, inclusivity and collegiality.

Outstanding opportunities exist for teaching and curriculum development, research in new state of the art facilities, and participation in an active intramural faculty practice. The position will be effective July 1, 2018.

To apply, please go to: http://unc.peopleadmin.com/postings/130149

**Preferred Qualifications and Requirements:** DDS or DMD or equivalent degree and previous teaching and research experience are ***required***; a certificate and/or MS degree from an operative dentistry program is ***preferred***.

Dr. Ryan Cook,

Search Committee Chair

E-mail: ryancookddsms@unc.edu

**The University of North Carolina at Chapel Hill is an Equal Opportunity Employer.**

# Online-Only Articles

Online Only Articles

On occasion we receive manuscripts that we would like to publish, but do not have the page room to include in the print journal. For the full article, please go to www.jopdentonline.org or enter the provided address into your address bar.

### Cuspal Deflection in Premolar Teeth Restored with Bulk-Fill Resin-Based Composite Materials

MM Elsharkasi • JA Platt • NB Cook • GH Yassen • BA Matis

Clinical Relevance: Polymerization shrinkage of conventional resin-based composites can cause cuspal deflection and be associated with enamel cracking, cusp or tooth fracture, and changes in occlusion. High-viscosity bulk-fill resin composites may produce less cuspal deflection than a conventional incrementally placed resin composite.

doi: http://dx/doi.org/10.2341/16-072-L

### Assessing the Appearance and Fluorescence of Resin-Infiltrated White Spot Lesions With Caries Detection Devices

K Markowitz • K Carey

Clinical Relevance: Fluorescent camera caries detectors can be used to assess the effectiveness of resin infiltration in improving the optic properties of white spot lesions. Clinicians can use a fluorescent camera to demonstrate early lesions to patients.

doi: http://dx/doi.org/10.2341/16-153-L

### Real-time Light Transmittance Monitoring for Determining Polymerization Completeness of Conventional and Bulk Fill Dental Composites

M Par • I Repusic • H Skenderovic • E Klaric Sever • D Marovic • Z Tarle

Clinical Relevance: Short curing times of 10-20 seconds may be insufficient for an optimal polymerization, especially under nonideal clinical conditions (eg, variable distance and angulation of the curing unit tip).

doi: http://dx/doi.org/10.2341/17-041-L

### Replacement of a Missing Maxillary Central Incisor Using a Direct Fiber-Reinforced Fixed Dental Prosthesis: A Case Report

MF Romero • FJ Haddock • WW Brackett

Clinical Relevance: Fiber-reinforced fixed dental prosthesis using the direct restorative technique may be accomplished with ideal contours and tooth morphology when a proper material selection and a step-by-step protocol is followed. This option becomes useful as an interim restoration in many clinical situations.

doi: http://dx/doi.org/10.2341/16-279-L

### Does Finishing and Polishing of Restorative Materials Affect Bacterial Adhesion and Biofilm Formation? A Systematic Review

DAM Dutra • GKR Pereira • KZ Kantorski • LF Valandro • FB Zanatta

Clinical Relevance: A polished/smooth surface is mandatory for maintaining clinical health status on restored teeth. However, this review depicts the absence of reliable data that characterize and elucidate the mechanism related to the effect of surface properties on bacterial adhesion/biofilm formation.

doi: http://dx/doi.org/10.2341/17-073-L

©Operative Dentistry, 2018, **43-1,** 1-9

# Cuspal Deflection in Premolar Teeth Restored with Bulk-Fill Resin-Based Composite Materials

MM Elsharkasi • JA Platt • NB Cook • GH Yassen • BA Matis



### Clinical Relevance

Polymerization shrinkage of conventional resin-based composites can cause cuspal deflection and be associated with enamel cracking, cusp or tooth fracture, and changes in occlusion. High-viscosity bulk-fill resin composites may produce less cuspal deflection than a conventional incrementally placed resin composite.


## SUMMARY

**The present study investigated the effect of three high-viscosity bulk-fill resin-based composite materials on cuspal deflection in natural teeth. Thirty-two sound maxillary premolar teeth with large slot mesio-occlusal-distal cavities were distributed into four groups (n=8). Three groups were restored with bulk-fill resin composite materials (Tetric EvoCeram Bulk Fill, Ivoclar Vivadent, Schaan, Liechtenstein; x-tra fil, VOCO, Cuxhaven, Germany; and SonicFill, Kerr, Orange, CA, USA) in a single 4-mm increment. The conventional composite group, Filtek Z100 (3M ESPE, St Paul, MN, USA), was used to restore the cavities in 2-mm increments. Cusp deflection was recorded postirradiation using a Nikon measurescope UM-2 (Nikon, Tokyo, Japan) by measuring the changes in the bucco-palatal widths of the teeth at five minutes, 24 hours, and 48 hours after completion of the restorations. Cuspal deflection was significantly higher in the conventional composite than in the Tetric EvoCeram Bulk Fill ($p=0.0031$), x-tra fil ($p=0.0029$), and SonicFill Bulk ($p=0.0002$) groups. There were no significant differences in cuspal deflection among the three bulk-fill materials (all $p<0.05$). In conclusion, all the investigated bulk-fill resin composites exhibited cuspal deflection values that were smaller than those associated with a conventional incrementally placed resin composite.**



*Marwa M Elsharkasi, BDS, MSD, Benghazi University, School of Dentistry, Department of Conservative Dentistry and Endodontics, Benghazi, Libya

Jeffrey A Platt, DDS, MS, Indiana University, Restorative Dentistry, Indianapolis, IN, USA

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

Ghaeth H Yassen, BDS, MSD, PhD, Indiana University School of Dentistry, Department of Biomedical and Applied Sciences, Indianapolis, IN, USA

Bruce Allan Matis, DDS, MSD, Indiana University School of Dentistry, Restorative Dentistry, Indianapolis, IN, USA

*Corresponding author: 20 street, Benghazi, Libya; e-mail: mmo1391@gmail.com


DOI: 10.2341/16-072-L

## INTRODUCTION

The esthetic and mechanical properties of composite resin have improved over the years. Yet polymerization shrinkage stress remains one of the concerns believed to compromise the clinical performance of

resin-based composite (RBC) materials.[1,2] During polymerization, monomer molecules convert into a polymer network, resulting in a decrease in the distance between monomer molecules as covalent bond formation occurs. This reduction in overall free volume produces a densely cross-linked polymer and results in volumetric shrinkage.[3,4] If this shrinkage occurs when the resin composite materials are inside a cavity preparation and bonded to cavity surfaces, mechanical stresses develop and transmit to the tooth-restoration interface.[5,6]

Methacrylate-based composite materials experience 2% to 5% volumetric shrinkage during polymerization.[2] Polymerization shrinkage is potentially associated with at least two clinical problems. First, if the stress created by polymerization shrinkage exceeds the bond strength of the resin to the tooth, the resin may detach from the tooth structure, leading to marginal microleakage.[7] This failure at the composite-tooth interface may result in postoperative sensitivity and secondary caries.[1,2,8] Second, if the strength of adhesion between the cavity surface and the restorative material exceeds the shrinkage stresses, no detachment occurs, but the restoration maintains internal stresses that pull the cusps together, reducing the intercuspal distance and leading to cuspal deflection. Cuspal deflection can cause changes in occlusion, enamel cracks, and tooth fracture.[2,7,8]

Several techniques have been published in the dental literature for evaluating cuspal deflection in mesio-occlusal-distal (MOD) cavities with resin composite restorations, including strain gauges,[9-11] microscopy,[12] linear variable differential transformers,[13] and flexible ribbons.[14] These techniques have recorded up to 50 μm of mean cuspal deflection. The variations in the cuspal deflection reported are largely due to sensors being used on various positions on the cusps and nonstandardized MOD cavity preparations in teeth of nonstandardized tooth sizes.[9]

The degree of cuspal deflection is affected by many factors, such as the shape and size of the cavity, the amount of polymerization shrinkage, polymerization kinetics, Young's modulus of the composite resin, and placement technique.[8]

Numerous techniques have been used clinically to minimize the impact of shrinkage stresses produced by resin composite restorations, with limited success. Examples include the use of flowable resin liners, indirect resin restorations, control of curing light intensity, and incremental placement techniques, in which the composite materials are placed in 2-mm-thick increments. This last method is advocated to ensure adequate light transmission and to reduce the configuration factor (the ratio between bonded and unbonded restoration surfaces) during polymerization, thereby reducing polymerization stress transfer to surrounding tooth structure.[2,15,16] Although the incremental placement technique has been recommended by many clinicians, the value of reducing polymerization shrinkage stresses with this technique has been questioned in some studies.[17,18] Furthermore, the incremental technique requires increased time for placement and curing of each increment.[9]

Recently marketed bulk-fill resin composite materials have been reported to have lower polymerization shrinkage stresses than do conventional resin-based composites,[6,19,20] consequently reducing cuspal deflection.[21] In addition, these materials can be placed in a single 4-mm increment and still have adequate light polymerization at the depth of the restoration. This simplifies the clinical procedure.[22] An added benefit would be the reduced risk of incorporating air bubbles or contamination between increments.[23]

The primary chemical composition of bulk-fill resin composite materials is similar to that of other methacrylate-based resin composites.[24] Some studies[25,26] mention that the increased depth of cure of bulk-fill composite materials is regulated mainly by increasing the translucency of the material. This translucency is achieved by reducing the concentration of fillers (filler content and translucency correlate linearly)[27] or by minimizing the difference in the refractive indices of the resin matrix and the filler particles.[28,29] In addition, the incorporation of a potent initiator system enhances the polymerization.[26] These materials are classified according to their rheological properties as flowable base materials that require a 2-mm overlay of posterior hybrid composite or as high-viscosity restorative composites that do not require an additional overlying occlusal layer.[19]

There is limited information about the amount of cuspal deflection that occurs from the placement of these bulk-fill materials. Therefore, the objective of this study was to compare cuspal deflection following placement of these newly developed bulk-fill composite materials in a single increment and placement of a conventional incrementally placed composite material. The null hypothesis was that the mean cuspal deflection seen in teeth restored in a single increment with bulk-fill would not be statistically

Table 1: *The Materials Used in this Study*

| *Bulk-Fill Resin-based Composites* | | | | | | |
|---|---|---|---|---|---|---|
| **RBCs** | **Manufacturer Color, Lot No.** | **Resin Matrix** | **Filler** | **Filler Wt%/Vol%** | **Volumetric Shrinkage, %** | **Instruction for Use** |
| Tetric EvoCeram Bulk Fill nanohybrid | Ivoclar Vivadent, (Schaan, Liechtenstein), IVA, T29056 | Bis-GMA, UDMA, Bis-EMA | Barium-aluminum-silica glass, prepolymer filler (monomer, glass filler, ytterbium fluoride), spherical mixed oxide | 79-81/60-61 | 1.7 | 4-mm increment, cure for 10 s. Additional curing from buccal and palatal aspect for proximal resin after removing the matrix |
| x-tra fil hybrid | VOCO (Cuxhaven, Germany), universal 1445489 | Bis-GMA, UDMA, TEGDMA, Bis-EMA | Inorganic fillers | 86/70.1 | 1.7 | 4-mm increment, cure for 20 s. Additional curing from buccal and palatal aspect for proximal resin after removing the matrix |
| SonicFill nanohybrid | Kerr (Orange, CA, USA), A2, 5299375 | Bis-GMA, TEGDMA, EBPDMA, UDMA | $SiO_2$, glass, oxide | 83.5/67 | 1.6 | 4-mm increment, cure for 20 s. Additional curing from buccal and palatal aspect for proximal resin after removing the matrix |
| Traditional universal composite (increments) | | | | | | |
| Filtek Z100 | 3M, ESPE (St Paul, MN, USA), A2, N595515 | Bis-GMA, TEGDMA | Silica/zirconia | 84.5/66 | 2.4 | 2-mm increment, cure for 20 s. Additional curing from buccal and palatal aspect for proximal resin after removing the matrix |

*Abbreviations: Bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA, bisphenol A and glycidyl methacrylate; EBPDMA, ethoxylated bisphenol A–dimethacrylate; RBC, resin-based composite; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.*

different than the mean observed in teeth restored with a traditional incrementally placed resin composite.

## METHODS AND MATERIALS

Three high-viscosity bulk-fill resin-based composite materials and one traditional universal composite were included in this study (Table 1). Thirty-two extracted maxillary premolar teeth free from caries, defects, or cracks and received in compliance with local human subjects criteria were used in this *in vitro* study. The selected teeth were cleaned with a hand scaler and then fixed into a cube-shaped mold with acrylic base plate material (Bosworth, Skokie, IL, USA) extending 2 mm cervical to the cemento-enamel junction to simulate the position of the tooth in the alveolar bone and to prevent the reinforcement of the crown by the base. The maximum buccopalatal width (BPW) for each tooth was recorded with a micrometer screw gauge (Moore and Wright, Sheffield, UK) that was accurate to 10 μm. A mean of three measurements per tooth was used to distribute the specimens into four groups (n=8) so that the BPW mean between groups varied by less than 5%. Any tooth that was 5% larger or smaller than the overall mean was excluded from the study. This minimized variation in the buccal-lingual dimension of the cavity preparation.

The repeated measurements of BPW were standardized using an innovative approach that enhanced the ability to determine the amount of deflection at the cusp tip. In summary, small cylinders of flowable composite (Filtek Supreme Ultra, 3M ESPE, St Paul, MN, USA) were constructed, coated with nail polish (Sally Hansen, NY, NY, USA) to minimize water sorption, and attached on both buccal and palatal cusp tips. Then a rhinestone was glued to the upper flat surface of each cylinder to be used as a reference point (Figure 1). Rhinestones have many facets that meet to form sharp line and point angles. Two rhinestone point angles (one on the buccal cusp and one on the palatal cusp) were used as fixed reference points for measurement of the linear intercuspal distance. The mean of three

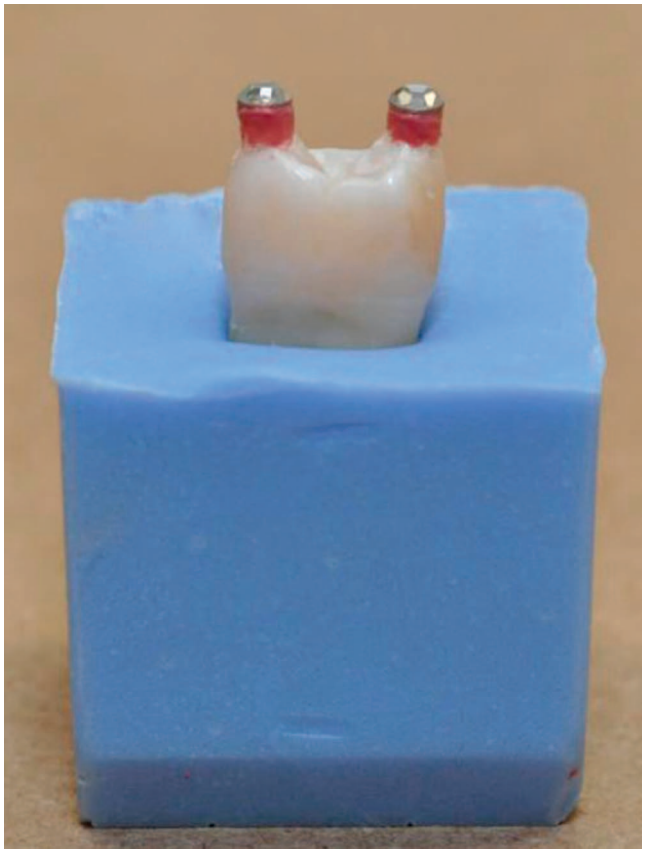


Figure 1. *Tooth with cylindrical composite and rhinestone.*

readings of the intercuspal width was recorded for each maxillary premolar tooth, as described below.

Large slot MOD cavities were prepared on the teeth in order to weaken tooth structure and favor cuspal deflection. The mounted teeth and a high-speed contra-angle air-turbine handpiece were positioned in a dental surveyor (J.M. Ney, Hartford, CT, USA) to ensure proper angulation during tooth preparation. All the teeth were prepared with a straight fissure carbide bur with a rounded end (#1158, SS White, Lakewood, NJ, USA) using a high-speed handpiece with air/water spray. The bur was changed after every five cavity preparations. The width of prepared cavities was two-thirds of the BPW of the tooth. An extrafine Sharpie permanent marker (Sanford Manufacturing Co, Oak Brook, IL, USA) was used on the tooth structure to guide the cavity preparation in the center of the tooth. The cavity depth was 4 mm from the occlusal cavosurface margin to the pulpal floor, and all margins were in enamel. The buccal and lingual walls were prepared to be parallel. The cavities were prepared so that the pulpal floor and mesial and distal gingival walls were at the same level (there was no step going from the pulpal floor to the gingival wall) in order to reduce preparation variation. Any tooth with pulp exposure was excluded from the study. All cavosurface margins were prepared without beveling.[2]

A Tofflemire matrix band was shaped and held snugly in place around the tooth being restored by tightening the retainer to the point at which resistance was initially detected. No further tightening of the matrix was done. A total-etch technique with 37.5% phosphoric acid (Kerr Gel Etchant, Kerr, Orange, CA, USA) was used. The phosphoric acid was applied for 15 seconds and then rinsed with water for 15 seconds. After gentle air-drying with canned air (Whoosh-Duster, Thomas Scientific, Swedesboro, NJ, USA) for one second, a moist dentin surface was maintained by blotting excess moisture from the dentin with a cotton pellet. Two coats of adhesive (OptiBond Solo Plus, Kerr) were actively applied for 15 seconds with a saturated brush tip to the enamel and dentin, until the surface appeared glossy. A gentle stream of compressed canned air was applied for three seconds. Then the adhesive was light-cured for 20 seconds with a visible light unit (DEMI LED light curing system, Kerr) having an irradiance of 1460 mW/cm$^2$, as measured using a MARC Resin Calibrator (BlueLight Analytics, Halifax, NS, Canada). The light irradiance was monitored after every eight specimens.

Three bulk-fill composites (Tetric EvoCeram Bulk Fill, Ivoclar Vivadent, Schaan, Liechtenstein; x-tra fil, VOCO, Cuxhaven, Germany; and SonicFill, Kerr) and one conventional composite (Filtek Z100, 3M ESPE) were used. For each specimen in the bulk-fill groups, a single bulk-fill RBC increment was placed and irradiated for 20 seconds with the LED curing wand touching the inclines of the cusps of the tooth (mesial and distal to the bonded reference cylinders) to achieve maximum curing depth and to maintain fixed distance. SonicFill was placed with sonic energy using an oscillating handpiece, as recommended by the manufacturer. Tetric EvoCeram Bulk Fill and x-tra fil were placed with plastic hand instruments in order to insure proper packing of materials inside the cavity preparation. The conventional composite group was restored incrementally with Filtek Z100 in three triangular-shaped increments with no more than 2-mm thickness for each increment, and each increment was irradiated for 20 seconds with the LED curing wand touching the inclines of the cusps of the tooth, as described above. After that the matrix band and retainer were removed before measurement under the microscope.

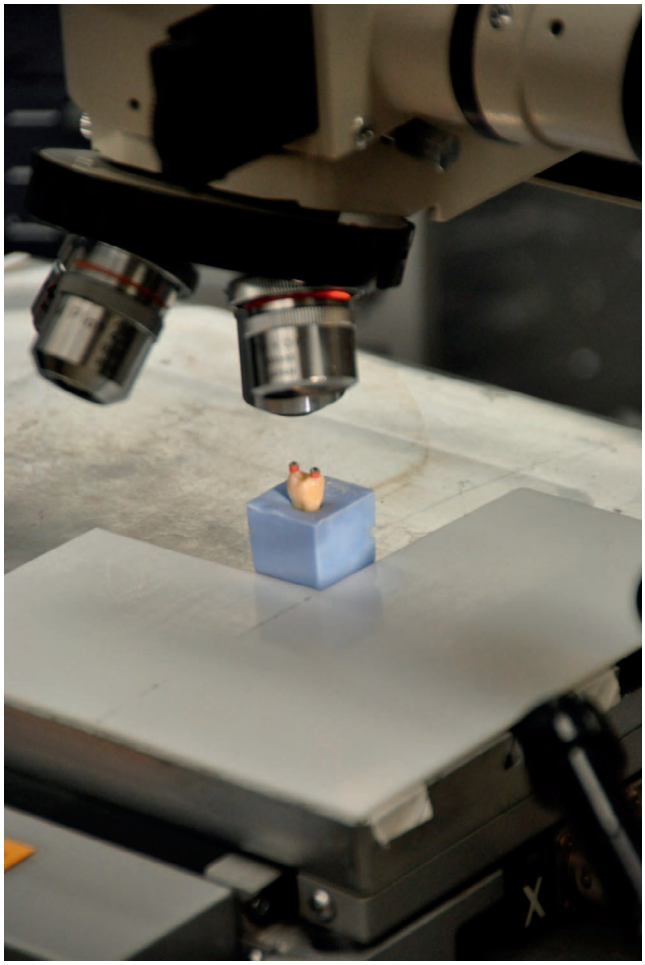


Figure 2. *A custom polymethyl methacrylate (PMMA) sheet and the sample under the microscope.*

### Cuspal Deflection Measurements

A light microscope (Nikon Measurescope UM-2, Nikon, Tokyo, Japan) with 0.001-mm accuracy and a modified microscope stage was used in order to determine the measurements of the cuspal deflection of the teeth. A custom-made polymethyl methacrylate (PMMA) sheet was used to standardize and maintain the horizontal orientation for each specimen during the repeated measurements (Figure 2). Baseline readings were recorded by measuring the linear distance between two point angles on the reference rhinestones prior to tooth preparation. Then the linear measurements were obtained after restoration placement, after five minutes, 24 hours, and 48 hours. The baseline records were subtracted from the all the subsequent measurements to obtain the changes in the positions of the cusps. The mean of three intercuspal width readings was recorded for each tooth at each time point.[7] The teeth were stored in double-distilled deionized water at room temperature (23°C±1°C). All the procedures were performed by the same examiner. Reproducibility of measurements was confirmed by a second evaluator. The entire procedure was performed for four teeth from each group at a time.

Table 2: *Mean (Standard Error, both in μm) for Cuspal Deflection for the Investigated Materials*[a]

| Material | 5 min | 24 h | 48 h |
|---|---|---|---|
| Tetric EvoCeram Bulk Fill | 28 (2) Ba | 19 (3) Bb | 15 (3) Bc |
| x-tra fil | 29 (3) Ba | 18 (3) Bb | 14 (3) Bc |
| SonicFill | 24 (3) Ba | 16 (2) Bb | 12 (2) Bc |
| Conventional composite | 44 (3) Aa | 27 (1) Ab | 23 (1) Ac |

[a] *Different uppercase letters represent significant differences in cuspal deflection between various resin composites within each time point. Different lowercase letters represent significant differences in cuspal deflection within each type of resin composite at various time points.*

### Statistical Methods

The effects of the composite material and time on cuspal deflection were analyzed using mixed-model analysis of variance, which included fixed effect terms for material, time, and their interaction and a repeated-measures effect to account for correlations among the times, as well as the different variances at each time. Pairwise comparisons between groups were made using the Tukey method to adjust for multiple comparisons. An overall 5% significance level was used. With a sample size of eight specimens per group, the study had 80% power to detect a difference of 5 μm between any two groups.

## RESULTS

Mean (±standard error) postrestoration cuspal deflection values are illustrated in Table 2 and Figure 3. Overall, cuspal deflection was significantly greater in the conventional composite group than in the Tetric EvoCeram Bulk Fill ($p$=0.0031), x-tra fil ($p$=0.0029), and SonicFill ($p$=0.0002) groups. There were no significant differences in cuspal deflection among Tetric EvoCeram Bulk Fill, x-tra fil, and SonicFill composites. Cuspal deflection was significantly greater at five minutes than at 24 hours ($p$<0.0001) or 48 hours ($p$<0.0001) and significantly greater at 24 hours than at 48 hours ($p$<0.001) for all the tested materials. At all time points, conventional composite had significantly greater cuspal deflection than did the bulk-fill materials (all $p$<0.05).

## DISCUSSION

This study investigated the effect of three types of high-viscosity bulk-fill composites on cuspal deflec-

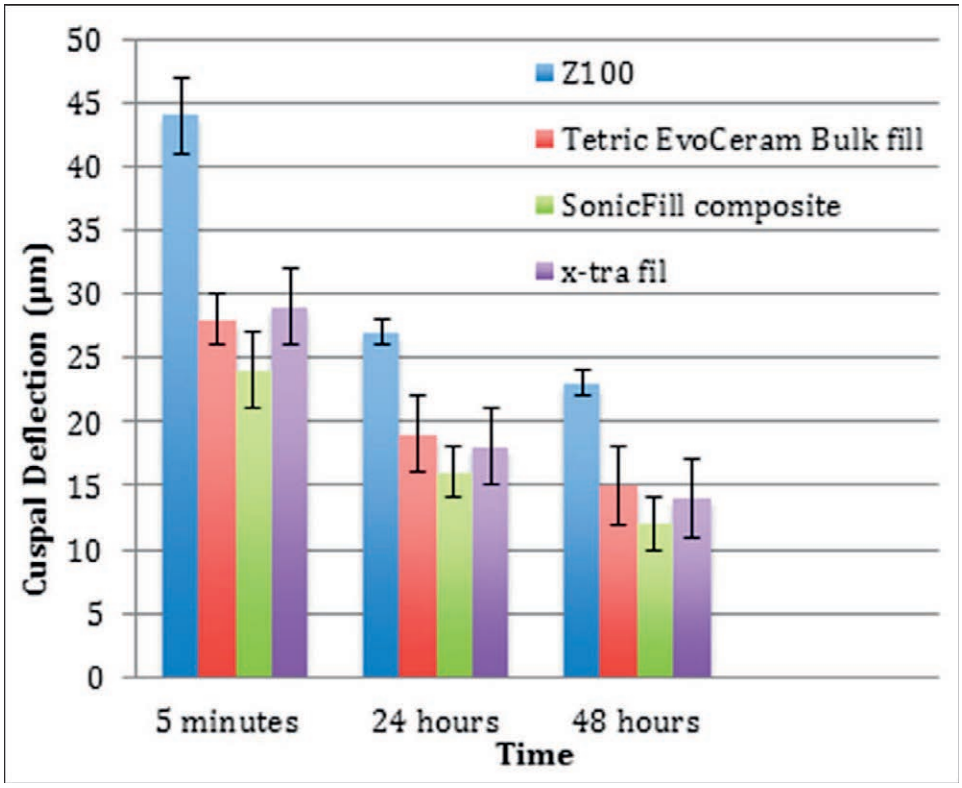



Figure 3. *Mean (standard error) (µm) of cuspal deflection for the investigated materials.*

tion of maxillary premolar teeth compared to an incrementally placed composite. Inward cuspal movement or cuspal deflection represents deformation of tooth structure caused by the effect of polymerization shrinkage stresses.[14,30] In the current study, the mean cuspal deflection varied from 24 µm to 44 µm. Moreover, the inward cuspal movement caused by polymerization shrinkage stresses was observed in each cavity filled with resin composite, as reported by a number of studies,[9,10,30] which means there is an established adhesion at the tooth-restoration interface.

In the present work, a large slot MOD cavity preparation was performed on maxillary premolar teeth in order to weaken tooth structure and favor cuspal deflection. As Gonzalez Lopez and others[31] mentioned, the degree of cuspal deflection is directly related to loss of tooth structure. In addition, as the cavity size increases, more RBC material is required, producing greater shrinkage forces and consequently more cuspal deflection.[31] Although the value of cuspal deflection might theoretically be greater if the baseline measurements were recorded after cavity preparation, it has been reported[7] that there was no significant difference in the cuspal deflection before or after cavity preparation. In addition, reestablishing pre-preparation occlusal relationships should be a goal of restorative treatment. For these reasons, the baseline measurements of the present study were recorded before tooth preparation.[7,32]

Measurement of cuspal deflection using natural teeth can produce many discrepancies among specimens due to the variations in the tooth size, anatomy, and modulus of elasticity. Therefore, to minimize variation among specimens and cavity preparations, our methodology employed the following procedures: 1) specimens were selected, measured, ranked in size, and assigned to treatment groups so that the mean BPWs of all tested groups varied by $\leq$5%; 2) MOD preparations were accomplished so that the pulpal floor and the gingival walls of the mesial and distal boxes were at the same level; and 3) a dental surveyor was utilized during all cavity preparations to facilitate proper alignment of the cavity walls. Moreover, room temperature was selected to allow better comparison with existing studies.[30,33] Future efforts evaluating the impact of 37°C may provide more clinically relevant results.

Our null hypothesis proposed that the mean cuspal deflection caused by bulk-fill resin composites using a single increment would not be statistically different than that of a composite placed in three increments. The study results did not support this hypothesis. Cuspal deflection was significantly greater with the incrementally placed composite

than with each of the three bulk-fill composites, which were not different from each other.

The reduced polymerization shrinkage stresses and subsequent cuspal deformation of bulk-fill resin composite materials could be attributed to optimized resin matrix, initiator chemistry, and filler technology.[34] Both filler technology and monomer content affect the polymerization shrinkage stresses. The incrementally placed (control) composite used in our study contains a bisphenol A–glycidyl methacrylate – triethyleneglycol dimethacrylate (TEGDMA) resin-matrix. TEGDMA-rich matrices create a greater degree of cross-linking and a greater amount of polymerization shrinkage.[35,36] The bulk-fill composites, which incorporate urethane dimethacrylate (UDMA) and bisphenol A polyethylene glycol diether dimethacrylate (Bis-EMA), with lower TEGDMA content, produced less polymerization shrinkage and, consequently, less cuspal deflection. This is in accordance with some studies[10,37] that reported reduced contraction stresses among materials containing UDMA and Bis-EMA. Moreover, the positive correlation between filler load and modulus of elasticity of resin composite materials has been confirmed.[20,24,38] A lower filler content of resin composite is generally associated with a lower modulus of elasticity.[39] It has been reported[19] that the lower filler load, and subsequently the lower modulus of elasticity, is considered another contributing factor to the reduced shrinkage stresses of bulk-fill composite materials. This fact might be one of the causative factors in the reduced polymerization shrinkage stresses of Tetric EvoCeram and SonicFill Bulk Fill, which have elastic moduli measuring around 8.5 and 10 GPa,[40] respectively. The elastic modulus of Z100 resin composite is approximately 21 GPa[41] and, consistent with the theory, demonstrated higher cuspal deflection.

On the other hand, Kim and others[42] reported that bulk-fill composite and conventional composite exhibited similar polymerization shrinkage stress. This could be attributed to a different methodological approach that was used to assess the polymerization shrinkage stresses.

Another potential factor that would contribute to reduced polymerization shrinkage stress and reduced cuspal movement is a lower degree of polymerization of the bulk-fill material. Depth of cure for the restorations placed in this study was not measured and is, therefore, a limitation that should be considered when analyzing the results.

The rationale for starting measurements after five minutes was that the majority of the cuspal movement was reported to occur within five minutes after polymerization.[19,30] Although at five minutes there were no statistically significant differences among the bulk-fill materials, SonicFill exhibited the lowest numeric cuspal deflection. Additionally, the unique advantage of the SonicFill material is its ability to behave like flowable composite during placement, providing better adaptation to cavity walls, when compared to traditional resin composite.[43] In addition, optimizing the filler sizes in SonicFill and x-tra fil around 20 μm[39] might be another contributing factor to the lower polymerization contraction stresses when compared to that of the Z100 resin composite, which contains 0.01-3.5-μm average filler size. Likewise, Abe and others[41] suggested that the smaller filler size causes more polymerization shrinkage stress. In agreement with the present study, Do and others[33] reported that the cuspal deflection of Tetric EvoCeram Bulk Fill was less when compared with that of flowable bulk-fill and conventional composites. Although they did not find a statistically significant difference, the authors mentioned that the results may have been significant if they had used a larger sample size.[33] This also agrees with the work of Zorzin and others,[34] who found that Tetric EvoCeram Bulk Fill produced less polymerization shrinkage than conventional composite. The manufacturer claims that the reduced polymerization shrinkage stresses of Tetric EvoCeram Bulk Fill are achieved by the incorporation of a stress reliever, which keeps the chemical cushion between filler particles intact; this cushion helps to improve the elasticity of the materials and reduces polymerization shrinkage.[42]

Cuspal deflection was significantly greater at five minutes than at 24 hours or 48 hours, and it was significantly greater at 24 hours than at 48 hours for all the tested materials. Although most of the polymerization shrinkage and subsequent cuspal deflection occurs within the first five minutes, the aim of measuring the position of the cusps at 24 and 48 hours was to determine if there was a cuspal relaxation and if it would return to its original position. All specimens tended to recover to their original dimensions, although complete recovery was not achieved during the 48-hour period. This is in agreement with the findings of some other studies,[30,31] whose authors mentioned that the recovery begins after 10 minutes in hydrated teeth and never returns to the original position in large-

or medium-sized cavities. Cusp relaxation or recovery of the cusps is likely to occur as a result of one or more of the following: water sorption, stress relaxation, tooth elasticity, or tooth-restoration gap formation.[30]

## CONCLUSIONS

Within the limits of this *in vitro* study,

- Cuspal deflection was less in teeth restored with bulk-fill resin composites using a single increment compared to teeth filled with a conventional composite placed in three increments;
- Different high-viscosity bulk-fill resin composites produced similar amounts of cuspal deflection; and
- Complete recovery of the cusps to their original positions did not occur during the 48-hour observation period.

### Acknowledgements

This work contributed to the completion of the MSD degree and was supported by the Indiana University School of Dentistry. Sincere thanks to Anderson Hara, DDS, MSD, PhD (Department of Cariology, Operative Dentistry and Dental Public Health, Indiana University, School of Dentistry) and Marco Bottino, DDS, MSc, PhD (Department of Biomedical and Applied Sciences, Indiana University, School of Dentistry) for their valuable assistance in the preparation and completion of this study.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Indiana University. The approval code for this study is IRB 1501282185.

### 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 19 June 2017)*

### REFERENCES

1. Braga RR, Ballester RY, & Ferracane JL (2005) Factors involved in the development of polymerization shrinkage stress in resin-composites: A systematic review *Dental Materials* **21(10)** 962-970.
2. Jafarpour S, El-Badrawy W, Jazi HS, & McComb D (2012) Effect of composite insertion technique on cuspal deflection using an in vitro simulation model *Operative Dentistry* **37(3)** 299-305.
3. Giachetti L, Scaminaci Russo D, Bambi C, & Grandini R (2006) A review of polymerization shrinkage stress: Current techniques for posterior direct resin restorations *Journal of Contemporary Dental Practice* **7(4)** 79-88.
4. Ferracane JL (2008) Buonocore Lecture. Placing dental composites—A stressful experience *Operative Dentistry* **33(3)** 247-257.
5. Davidson CL, de Gee AJ, & Feilzer A (1984) The competition between the composite-dentin bond strength and the polymerization contraction stress *Journal of Dental Research* **63(12)** 1396-1399.
6. Garcia D, Yaman P, Dennison J, & Neiva G (2014) Polymerization shrinkage and depth of cure of bulk fill flowable composite resins *Operative Dentistry* **39(4)** 441-448.
7. Karaman E, & Ozgunaltay G (2013) Cuspal deflection in premolar teeth restored using current composite resins with and without resin-modified glass ionomer liner *Operative Dentistry* **38(3)** 282-289.
8. Kim ME, & Park SH (2011) Comparison of premolar cuspal deflection in bulk or in incremental composite restoration methods *Operative Dentistry* **36(3)** 326-334.
9. 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.
10. Fleming GJ, Hall DP, Shortall AC, & Burke FJ (2005) Cuspal movement and microleakage in premolar teeth restored with posterior filling materials of varying reported volumetric shrinkage values *Journal of Dentistry* **33(2)** 139-146.
11. Sultan A, Moorthy A, & Fleming GJ (2014) The adhesive potential of dentin bonding systems assessed using cuspal deflection measurements and cervical microleakage scores *Dental Materials* **30(10)** 1154-1160.
12. Alomari QD, Reinhardt JW, & Boyer DB (2001) Effect of liners on cusp deflection and gap formation in composite restorations *Operative Dentistry* **26(4)** 406-411.
13. Meredith N, & Setchell DJ (1997) In vitro measurement of cuspal strain and displacement in composite restored teeth *Journal of Dentistry* **25(3-4)** 331-337.
14. McCullock AJ, & Smith BG (1986) In vitro studies of cuspal movement produced by adhesive restorative materials *British Dental Journal* **161(11)** 405-409.
15. 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.
16. Lazarchik DA, Hammond BD, Sikes CL, Looney SW, & Rueggeberg FA (2007) Hardness comparison of bulk-filled/transtooth and incremental-filled/occlusally irradiated composite resins *Journal of Prosthetic Dentistry* **98(2)** 129-140.
17. Versluis A, Douglas WH, Cross M, & Sakaguchi RL (1996) Does an incremental filling technique reduce polymerization shrinkage stresses? *Journal of Dental Research* **75(3)** 871-878.
18. Jedrychowski JR, Bleier RG, & Caputo AA (2001) Shrinkage stresses associated with incremental composite filling techniques in conservative Class II restorations *ASDC Journal of Dental Children* **68(3)** 161-167, 150.

19. El-Damanhoury H, & Platt J (2014) Polymerization shrinkage stress kinetics and related properties of bulk-fill resin composites *Operative Dentistry* **39(4)** 374-382.
20. Ilie N, & Hickel R (2011) Investigations on a methacrylate-based flowable composite based on the SDR technology *Dental Materials* **27(4)** 348-355.
21. Vinagre A, Ramos J, Alves S, Messias A, Alberto N, & Nogueira R (2016) Cuspal displacement induced by bulk fill resin composite polymerization: Biomechanical evaluation using fiber Bragg grating sensors *International Journal of Biomaterials* http://dx.doi.org/10.1155/2016/7134283
22. 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.
23. Margeas RC (2015) Bulk-fill materials: Simplify restorations, reduce chairtime *Compendium of Continuing Education in Dentistry* **36(1)** e1-e4.
24. Ilie N, Rencz A, & Hickel R (2013) Investigations towards nano-hybrid resin-based composites *Clinical Oral Investigations* **17(1)** 185-193.
25. Van Ende A, De Munck J, Van Landuyt KL, Poitevin A, Peumans M, & Van Meerbeek B (2013) Bulk-filling of high C-factor posterior cavities: Effect on adhesion to cavity-bottom dentin *Dental Materials* **29(3)** 269-277.
26. 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.
27. Lee YK (2008) Influence of filler on the difference between the transmitted and reflected colors of experimental resin composites *Dental Materials* **24(9)** 1243-1247.
28. Shortall AC, Palin WM, & Burtscher P (2008) Refractive index mismatch and monomer reactivity influence composite curing depth *Journal of Dental Research* **87(1)** 84-88.
29. Azzopardi N, Moharamzadeh K, Wood DJ, Martin N, & van Noort R (2009) Effect of resin matrix composition on the translucency of experimental dental composite resins *Dental Materials* **25(12)** 1564-1568.
30. Suliman AA, Boyer DB, & Lakes RS (1993) Cusp movement in premolars resulting from composite polymerization shrinkage *Dental Materials* **9(1)** 6-10.
31. Gonzalez Lopez S, Sanz Chinesta MV, Ceballos Garcia L, de Haro Gasquet F, & Gonzalez Rodriguez MP (2006) Influence of cavity type and size of composite restorations on cuspal flexure *Medicina Oral, Patologia Oral y Cirugia Bucal* **11(6)** E536-E540.
32. Güler E, & Karaman E (2014) Cuspal deflection and microleakage in pre molar teeth restored with bulk-fill resin-based composites *Journal of Adhesion Science & Technology* **28(20)** 2089-2099.
33. Do T, Church B, Verissimo C, Tantbirojn D, Tantbirojn D, Simon JF, & Versluis A (2014) Cuspal flexure, depth-of-cure, and bond integrity of bulk-fill composites *Pediatric Dentistry* **36(7)** 468-473.
34. Zorzin J, Maier E, Fey TBR, Lohbauer U, Petschelt A, &Taschner M (2015) Bulk-fill resin composites: Polymerization properties and extended light curing *Dental Materials* **31(3)** 293-301.
35. Goncalves F, Azevedo CL, Ferracane JL, & Braga RR (2011) BisGMA/TEGDMA ratio and filler content effects on shrinkage stress *Dental Materials* **27(6)** 520-526.
36. Barszczewska-Rybarek IM (2014) Characterization of urethane-dimethacrylate derivatives as alternative monomers for the restorative composite matrix *Dental Materials* **30(12)** 1336-1344.
37. Goncalves F, Pfeifer CC, Stansbury JW, Newman SM, & Braga RR (2010) Influence of matrix composition on polymerization stress development of experimental composites *Dental Materials* **26(7)** 697-703.
38. El-Safty SAR, Silikas N, & Watts DC (2012) Nanomechanical properties of dental resin-composites *Dental Materials* **28(12)** 1292-1300.
39. Ilie N, Bucuta S, & Draenert M (2013) Bulk-fill resin-based composites: An in vitro assessment of their mechanical performance *Operative Dentistry* **38(6)** 618-625.
40. Ibarra ETLW, Casey J, Dixon SA, & Vandewalle KS (2015) Physical properties of a new sonically placed composite resin restorative material *General Dentistry* **3(63)** 51-56.
41. Abe YLP, Inoue S, Braem MJ, Takeuchi M, Vanherle G, &Van Meerbeek B (2001) Dynamic elastic modulus of 'packable' composites *Dental Materials* **17(6)** 520-525.
42. Kim RJ, Kim YJ, Choi NS, & Lee IB (2015) Polymerization shrinkage, modulus, and shrinkage stress related to tooth-restoration interfacial debonding in bulk-fill composites *Journal of Dentistry* **43(4)** 430-439.
43. Kapoor N, Bahuguna N, & Anand S (2016) Influence of composite insertion technique on gap formation *Journal of Conservative Dentistry* **19(1)** 77-81.

©Operative Dentistry, 2018, **43-1,** 10-18

# Assessing the Appearance and Fluorescence of Resin-Infiltrated White Spot Lesions With Caries Detection Devices

K Markowitz • K Carey

### Clinical Relevance

Fluorescent camera caries detectors can be used to assess the effectiveness of resin infiltration in improving the optic properties of white spot lesions. Clinicians can use a fluorescent camera to demonstrate early lesions to patients.


## SUMMARY

**Objective: This *in vitro* study examined the effectiveness of caries detector devices in assessing the ability of resin infiltration (RI) (Icon, DMG-Hamburg, Hamburg, Germany) to improve the optical properties of enamel white spot lesions (WSLs).**

**Methods and Materials: Ten caries-free third molars were used. Photographs, a subjective visual assessment of the photographs, fluorescent camera (FC) images using the Spectra (Air Techniques, Melville, NY, USA), and laser fluorescent (LF) readings using the DIAGNOdent (KaVo, Biberach, Germany) were obtained from each tooth's buccal surface. Specimens were coated with nail polish leaving a rectangular window on the buccal surface and placed in pH 4.5 lactic acid gel for two weeks to create a WSL. The WSLs were analyzed by the same methods. RI was applied to half of each WSL; final photographs were then taken, and caries detector assessments were conducted. FC images were converted to grayscale, and the fluorescent image's brightness intensity was measured using ImageJ. Data were analyzed with analysis of variance and Tukey-Kramer honestly significant difference test. Significance was set at $\alpha$=0.05.**

**Results: Subjective assessment of the photographs showed that RI improved the appearance of the WSLs so that they resembled intact enamel. Mean FC-brightness intensities for intact, demineralized, and demineralized RI-treated areas were 159.6 ± 9.2, 123.4 ± 7.2, and 160.9 ± 11.5, respectively. There were no significant differences in fluorescent intensity between the intact and RI areas ($p$=0.58). The demineralized areas had significantly lower fluorescent intensity than both the RI-treated and intact areas ($p$<0.001). LF values did not differ signifi-**


*Kenneth Markowitz, DDS, MSD, Departments of Oral Biology and Restorative Dentistry, Rutgers School of Dental Medicine, Newark, NJ, USA

Kevin Carey, DMD, Department of Community Health, Rutgers School of Dental Medicine, Newark, NJ,

*Corresponding author: 185 S. Orange Ave, Newark, NJ 07103, USA; e-mail: markowkj@sdm.rutgers.edu

DOI: 10.2341/16-153-L

**cantly between intact, demineralized, or RI-treated areas.**

**Conclusions: This study demonstrates the ability of RI to restore artificial WSLs to the esthetics and fluorescence of intact enamel. The FC can be used to assess the optical properties of WSLs and the impact of RI on these properties.**

## INTRODUCTION

The enamel white spot lesion (WSL) is an early manifestation of caries. When exposed to bacterially derived organic acids, subsurface enamel mineral loss occurs and results in porous enamel that appears white owing to its capacity to scatter light.[1] If the process of acid generation and enamel demineralization proceeds unchecked, the enamel surface will give way, resulting in a cavitated lesion.

WSLs can form on any plaque-covered enamel surface. Teeth with orthodontic brackets generally accumulate plaque and are frequently found to have WSLs when the orthodontic treatment is completed.[2] Typically, these lesions are arrested when access to the tooth surface is improved by removal of the orthodontic hardware, which allows for saliva exposure and more effective tooth brushing.[3] Fluoride and various other agents can facilitate WSL remineralization.[4] When WSLs regain mineral, the surface experiences a reduction in porosity, blocking the diffusion of mineral constituents into the subsurface areas of the lesion. Consequently, the use of remineralizing therapies does not ensure complete lesion resolution and restoration of the tooth's esthetics.[5,6]

Resin infiltration (RI) is a professionally applied, microinvasive, treatment for WSLs where a low-viscosity resin-containing triethylene glycol dimethacrylate is applied to the acid-etched surface of the lesion and penetrates the WSL's porous enamel.[7,8] After it penetrates the WSL, the resin is polymerized by light curing. By infiltrating the enamel surface and filling most of the porosities in the WSL, this procedure protects the enamel from further demineralization[9] and improves the mechanical properties of the acid-damaged enamel surface.[10] This procedure has been used to treat both interproximal lesions and WSLs on facial smooth surfaces.[11] Since the resins used in RI have indexes of refraction that match enamel, this procedure reduces the light scattering of the WSL, thereby improving the color and esthetics of the treated tooth.[12-14] To date, assessments of the RI treatment's impact on the appearance and optical properties of teeth with WSLs have been largely limited to the visual evaluation of treated teeth.[15]

Caries detector devices are used to identify caries at various stages of development including WSLs.[16] Quantitative light fluorescence (QLF) (Inspektor Research Systems BV, Amsterdam, The Netherlands) uses a wavelength of light that induces dentin to fluoresce. WSLs scatter the fluorescent light and appear dark in QLF images. The device's software can calculate lesion volume and mineral loss.[1] A laser fluorescence (LF) device, the DIAGNOdent (KaVo, Biberach, Germany), excites bacterially derived pigment to fluoresce. The values on the device's numerical display indicate the intensity of this fluorescence in lesions.[17] Fluorescent camera (FC)–based caries detection systems, such as the VistaProof (Dürr Dental AG, Bietigheim-Bissingen Germany) and the Spectra Caries Detection Aid (Air Techniques, Melville, NY, USA) measure alterations in the tooth's fluorescence as a means of detecting and assessing the severity of WSLs. In this determination, FCs function in a manner like the QLF—identifying WSLs as areas of reduced fluorescence observed as dark areas on fluorescent images. Like the LF device, the FC devices can detect and assess the severity of dentin caries by measuring the light-induced fluorescence of bacterial pigments.[18,19] The FC can present fluorescent images of the tooth, or the device's Visix software can generate false color images of the tooth based on the fluorescent measurements that indicate areas of enamel demineralization and dentin caries as well as numerical values that reflect the severity of these changes.[18,20]

Caries detectors that measure the optical properties of tooth structure can be used in studies comparing the effectiveness of various WSL treatments.[3,21] Caries detectors that produce fluorescent images of teeth should be able to visualize and measure changes in the optical properties of WSLs brought about by RI since this treatment reduces light scattering within the lesion. QLF and spectrophotometer readings have been used in *in vitro* studies to evaluate how RI affects the optical properties of WSLs.[12,22,23] Caries detectors have advantages to visual examination since they allow quantitation of subtle changes in the optical properties of WSLs.

In this *in vitro* study, we examined the effect of RI treatment on the FC and LF devices' assessment of the fluorescence of artificial WSLs created in human teeth by exposure of enamel to a lactic acid gel. Since RI reduces the light scattering of the WSL, we

hypothesized that treatment would restore the fluorescence of treated areas of WSLs to values that were equivalent to that of intact enamel. A subjective assessment of the visual appearance of the surfaces was also conducted to ensure that changes in the caries detector readings caused by demineralization and RI were reflected in the clinically important parameter of WSLs: the esthetics of the affected tooth surface. This study evaluated the potential contribution that the FC and LF devices can make to studies evaluating the impact of RI and other treatments on the appearance and optical properties of teeth with WSLs.

## METHODS AND MATERIALS

### Tooth Selection and Baseline Measurements

This *in vitro* study was performed using extracted third molars collected at the Rutgers School of Dental Medicine's Oral Surgery clinic. Consent to donate third molars for research projects was obtained from adult patients between the ages of 18 and 30 years. The university's Institutional Review Board approved the tooth collection procedure (Protocol number 0120050074).

Following extraction, teeth were debrided of adherent soft tissue and visually inspected. Ten caries-free and restoration-free teeth with intact buccal surfaces that were free of white areas or other discolorations were used in this study. Teeth were stored in 1% phenol solution and used within 1 month of collection. The buccal surfaces of the 10 selected teeth were photographed at eight-power magnification (DP12 Microscope Digital Camera System, Olympus, Tokyo, Japan).

LF readings from each tooth's buccal surface were taken with the DIAGNOdent model 2095 using the instrument's B-tip, as recommended by the manufacturer for smooth surface examinations. The instrument was calibrated per manufacturer's instructions. The B-tip lightly contacted the enamel surface and was gently rocked. The highest reading (instrument's maximum reading = 99) was recorded for each area examined. For each tooth, LF measurements were obtained for four areas near the tooth's height of contour located mesial and distal to the buccal groove. This area would be the future site of the demineralized lesion. Using Microsoft Power Point, LF readings obtained from the various buccal sites were superimposed, at the time that the readings were performed, onto the photograph of those tooth surfaces, creating an LF map of each tooth's buccal surface. In superimposing these readings on the images of the tooth surface, care was taken to insert the readings on the sites examined with the LF device. This procedure was used in a previous study to create LF maps of cut and intact occlusal surfaces.[24]

Fluorescent images from the buccal surfaces of the teeth were obtained using the Spectra Caries Detection Aid. The FC handpiece with an 8-mm spacer was placed over the buccal surface and the images obtained per the manufacturer's instructions. In order to collect data concerning subtle changes in the tooth structure's fluorescent intensity, the images provided by the FC, instead of the false color images and numerical readings generated by the device's software, were used in the subsequent data analysis. To obtain quantitative fluorescent values; the stored FC images were imported into the public domain image analysis program ImageJ (National institute of Health, Bethesda, MD, USA) digitized, and converted to grayscale; the brightness intensity values of selected areas were then recorded.[25] The brightness intensity values of the fluorescent images ranged from 0 to 244 arbitrary units. In preliminary experiments, where we varied the duration of the demineralizing treatment, we observed that these fluorescent intensities correlated better with the visual severity of the buccal surface WSL than did the numerical values obtained with the instrument's software. We also noticed that the instrument's software failed to detect areas of mild demineralization that were detectable on the fluorescent images and observed on visual examination. The buccal areas selected for analysis corresponded to the areas where LF measurements were previously obtained. ImageJ was also used to convert FC images into line graph maps of the tooth surfaces where fluorescent intensity values of all imaged sites are displayed.

In using both caries detectors, measurements were taken in triplicate to ensure reproducibility. All photos and caries detector measurements were taken in dim ambient light and from moist tooth surfaces to simulate intraoral conditions. Following water rinsing, the teeth were exposed to a brief air current to remove excess fluid and leave the tooth surface moist; care was taken to avoid accumulations of fluid on the teeth that would cause reflections. A training and calibration exercise was held where both examiners (KM and KC) obtained FC images and LF measurements from the buccal surfaces of 10 teeth. By following uniform procedures for positioning the handpieces of the two instruments, the LF values

obtained by the two examiners were within ±2 units, and the FC fluorescent intensity values were ±5%.

### Creation of Artificial White Spot Lesions

Following baseline measurements, the 10 teeth were coated with waterproof nail polish except for a 1 × 4 mm window on the buccal surface of each tooth. The teeth were then placed into separate containers with 25 mL of a gel containing 0.1 M lactic acid and 1.5 mM $CaPO_4$ adjusted to pH 4.5 with sodium hydroxide and thickened with ethyl cellulose (Natrosol, Ashland Aqualon Inc, Parlin, NJ, USA).[26] The containers holding the demineralizing teeth were agitated twice daily, and the gel was changed after 7 days. This demineralization treatment was carried out for 14 days at room temperature. Following acid exposure, the teeth were rinsed with deionized water and inspected under eight-power magnification to ensure that the surface of the acid-exposed area was intact and the tooth structure was white when viewed wet. The nail varnish was then removed by agitating the teeth in acetone. After we created the artificial WSLs, the buccal surfaces of the teeth were photographed and evaluated with the LF and FC devices using methods identical to those used to obtain the baseline measurements.

Following the experiment, four of the 10 teeth used were sectioned perpendicular to the long axis of the tooth using a low-speed saw (Isomet, Buehler LTD., Lake Bluff, IL, USA) with diamond blade and water lubrication. Tooth slices 0.25-mm thick were cut through the WSLs and polished with 600-grit silica carbide paper. The areas of the sections containing the WSLs were examined at 50-power magnification. The WSLs were observed to extend to 150 ± 8.2 μm below the enamel surface.

### Application of the Resin Infiltration Treatment and Final Measurements

The mesial or distal half of each tooth's WSL was randomly selected to receive the RI treatment (Icon, DMG-Hamburg, Hamburg, Germany) following the manufacturer's instructions for smooth-surface lesion application. The procedure involved drying the buccal surfaces with air from an air-water syringe and applying the Icon kit's HCl etch. After removing the etchant by copious rinsing and drying with an air-water syringe, the kit's ethanol drying treatment was applied and the tooth surface dried thoroughly. This was followed by application of the RI material and light curing for 30 seconds using an ESPE Elipar light curing unit (3M, St Paul, MN, USA). The etchant, ethanol drying agent, and RI material were applied using the syringes and applicator tips provided in the Icon kit that are intended to be used for treating facial smooth-surface lesions. As recommended by the manufacturer, the RI material was applied and light cured twice. Care was taken to limit application of the etchant and RI to the WSL and to avoid applying excess material. Following RI application, the teeth were stored overnight in a humid atmosphere and then a final set of photographs, FC images, and LF measurements were made.

### Subjective Assessment of WSLs

As an independent method of assessing the severity of the WSLs and the impact of RI, photographs of the teeth used in this study were rated for the presence and intensity of visible WSLs by a panel of five dentists (two orthodontists and three restorative dentists) that did not include the authors. Each evaluator was shown photographs of the teeth used in this study on a flat computer screen. The evaluators were trained to rate the photos using a 0 to 10 visual analog scale (VAS) in which 0 represented no visible WSL and 10 represented an obvious WSL. Following this training the examiners' ratings were found to agree with the ratings of the investigators to within ±1. This type of assessment utilizing photographs and a VAS scale has been used in a clinical study of the effect of RI on WSL esthetics.[15] The photos showed the buccal surfaces prior to demineralization, following the creation of the WSL, and following RI of the mesial or distal half of the WSL. In addition to these 30 photos, each evaluator was shown 10 duplicate images so that the reproducibility of the assessment could be determined. The photos were shown in random order and cropped to show the mesial or distal half of the tooth's buccal surface; hence, separate images showed demineralized or RI-treated areas.

### Data Analysis

LF, FC brightness intensity readings, and subjective VAS ratings are reported as mean ± standard deviation. A repeat measure analysis of variance (ANOVA) with pairwise Tukey-Kramer test was performed using JMP version 11 statistical software (SAS institute, Cary, NC, USA) to determine if significant differences in these parameters were measured from intact, demineralized, and RI enamel.

In conducting the subjective visual assessments of the tooth surfaces, each examiner rated 10 duplicate photographs. A weighted kappa coefficient was

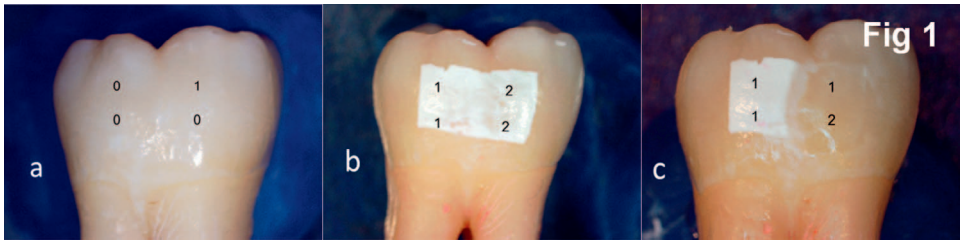



Figure 1. *Photographs with superimposed LF readings of the buccal surface of an extracted third molar (A): before demineralization, (B): after demineralization, and (C): after RI of the right half of the demineralized area. The low LF values recorded from the intact tooth tissue were not affected by demineralization or RI treatment.*

calculated for intraobserver and interobserver agreement using SAS version 9.4 (SAS institute).

Statistical significance was set at $\alpha$=0.05. Based on preliminary experiments, a minimum sample size of eight teeth was necessary to achieve a statistical power of 0.8 in resolving a fluorescent intensity difference of 20% as measured by the FC, with standard deviations of approximately ±10% of the intensity value.

## RESULTS

### Effect of Demineralization and RI on Appearance of the Tooth Structure and LF Readings

Photographs showing the buccal surface of a tooth with LF readings obtained from the areas over which the numbers are superimposed are shown in Figure 1A. Intact tooth structure has LF readings close to zero. A rectangular area of the same tooth's buccal surface was exposed to a pH 4.5 lactic acid gel. This resulted in the white area of demineralization shown in Figure 1B. Despite the marked change in appearance of the tooth surface following acid treatment, LF measurements in the demineralized areas were still close to zero. When half of the demineralized area was treated with RI, there was a marked change in the appearance of the demineralized enamel so that it resembled intact tooth structure (Figure 1C). LF readings obtained from the RI-treated half of the demineralized area continued to be close to zero. As shown in Table 1, intact teeth had a mean LF reading of 1.07 ± 0.9. Following demineralization and RI, the mean LF readings were 1.25 ± 0.9 and 0.9 ± 0.9, respectively. Both demineralization and RI failed to cause significant changes in the LF readings obtained from the buccal surface of the 10 teeth used in this study ($p$=0.69 and $p$=0.67, respectively).

Table 1: *Effect of Demineralization and Resin Infiltration on LF Measurements (Mean ± SD) Obtained from the Buccal Surfaces of Teeth* In Vitro *(N=10)*

| | Intact | Demineralized | RI-Treated Areas |
|---|---|---|---|
| LF readings | 1.07 ± 0.9 | 1.25 ± 0.9* | 0.9 ± 0.9* |

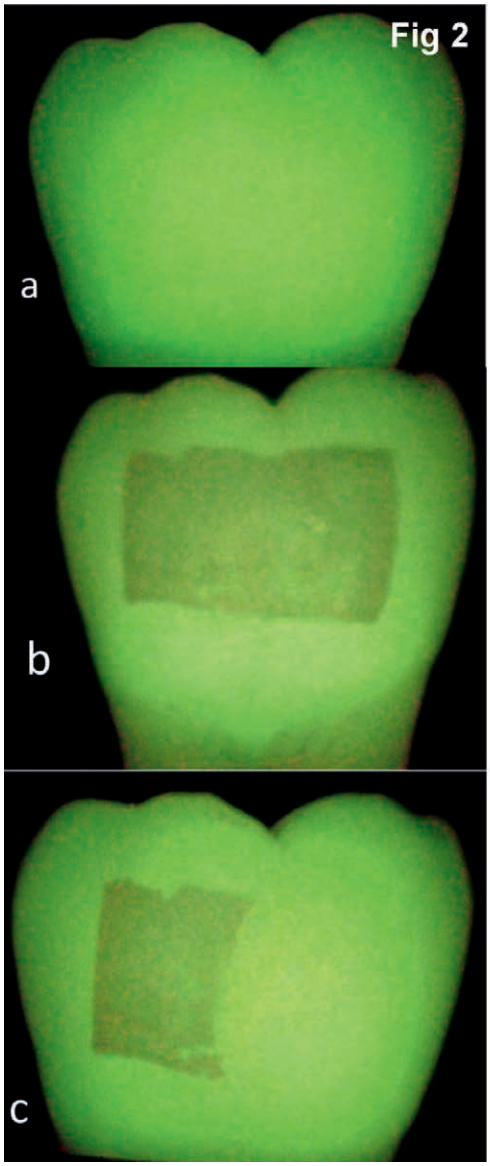
Abbreviation: LF, laser fluorescence; RI, resin infiltration; SD, standard deviation.
* Not statistically different compared with initial measurement ($p$>0.05).

### Effect of Demineralization and RI on Fluorescent Intensity of the Tooth Structure Measured With the FC

FC images of the same tooth shown in Figure 1 are shown in Figure 2. Prior to demineralization, the buccal surface presents a uniform fluorescent intensity (Figure 2A). Following acid treatment of a rectangular portion of the buccal enamel, reduced fluorescence was observed in the acid-exposed area (Figure 2B). Following RI (Figure 2C), the treated portion of the demineralized area showed increased fluorescent intensity and resembled the intact areas of the same tooth.

Figure 3 shows fluorescent brightness intensity readings measured from the buccal surface of one tooth; mesial-distal locations are on the X-axis, gingival-occlusal locations are plotted on the Y-axis, and the Z-axis values for each location on the lines represent image brightness indicating the fluorescent intensity. The intact tooth shown in Figure 3A has a peak fluorescent intensity in the center of the crown near the tooth's height of contour. Following demineralization of a rectangular window, the fluorescent intensity of this demineralized area was reduced (Figure 3B.). Following application of RI to the right portion of the demineralized area, the fluorescent intensity of the treated portion was observed to increase (Figure 3C). The fluorescent intensities measured in the RI-treated area were less uniform than the intensities measured in the same area prior to demineralization.

The mean fluorescent intensity values measured by the FC from the 10 teeth used in this study obtained prior to and following demineralization and from RI-treated areas are shown in Figure 4. Intact enamel had a fluorescent intensity of 159.6 ± 9.2. Demineralization caused a drop in fluorescent intensity to 123.4 ± 7.2, a 22.7% reduction that



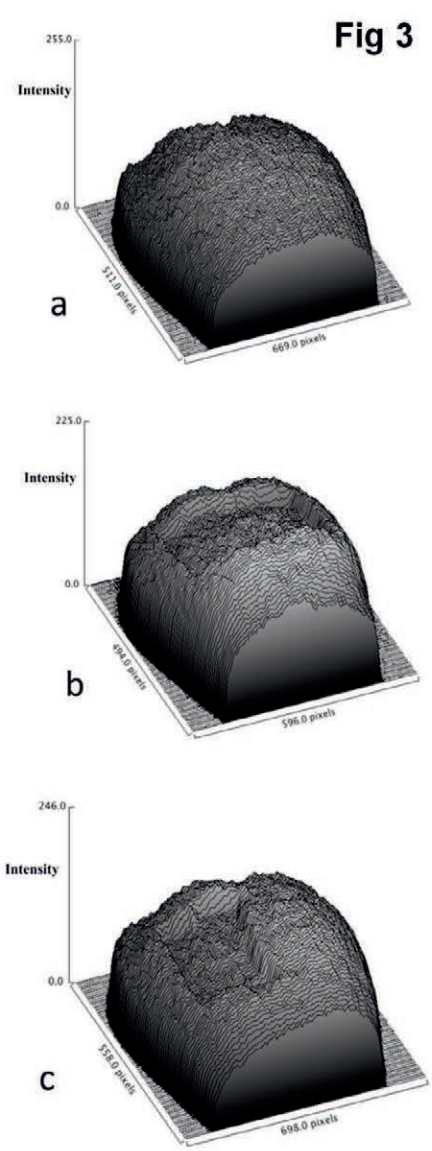




Figure 2. *FC images of the same extracted third molar shown in Figure 1 (A): Before demineralization, (B): after demineralization, and (C): after RI treatment of the right half of the demineralized area. Demineralized enamel appears darker than intact areas due to the scattering of light from the fluorescent dentin.*

Figure 3. *Brightness intensity plots generated from the FC images shown in Figure 2. These images were converted to three-dimensional brightness intensity maps to highlight the effects of demineralization and RI on (A): the intact tooth, (B): demineralized area, and (C): demineralized area where the right half of the demineralized enamel was resin infiltrated.*

was statistically significant ($p<0.001$). Following RI of half of each demineralized area, the mean fluorescent intensity increased to 160.9 ± 11.5, a value that was significantly higher than the value for untreated demineralized areas ($p<0.001$) and not significantly different from the values measured from the intact tooth surfaces ($p=0.58$).

### Subjective Assessment of Demineralized and RI Surfaces

Demineralized enamel surfaces had a distinct chalky white appearance and were readily distinguished by the evaluators and rated highly on the WSL severity scale used in this study. The mean subjective VAS scores for intact, demineralized, and RI surfaces were 1.81 ± 1.32, 9.77 ± 0.58, and 3.77 ± 1.74, respectively (Table 2). Each of these values was significantly different from the other values ($p<0.001$). Weighted kappa coefficients for intra-observer and interexaminer agreement were 0.81 and 0.73, respectively, indicating good agreement.

## DISCUSSION

RI treatment is used to halt the progression of WSLs and improve the appearance of teeth with WSLs.[13] Facial WSLs can be directly observed, but visual examination is difficult to quantify in research studies comparing RI with other methods of WSL treatement.[5,15] Several methods of caries detection have been applied to evaluate the effect of preventive therapies on facial WSLs. The QLF method has been used to monitor the remineralization of WSLs *in*

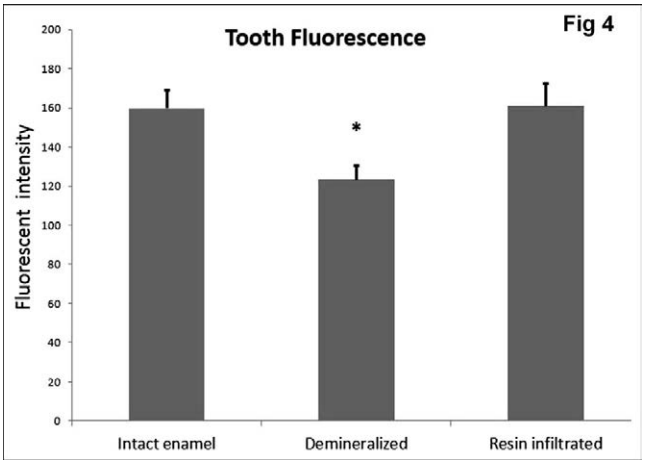



Figure 4. *Mean (± standard deviation) fluorescent intensity readings measured by the FC device from the buccal surface of extracted molars (N=10). Left bar represents intact surfaces, middle bar demineralized surfaces and right bar RI surfaces. The demineralized surfaces (*) had significantly lower brightness intensities than the other two groups (p< 0.01).*

*vitro* and *in vivo* following the removal of fixed orthodontic appliances.[3] In a recent study utilizing the QLF in examining the efficacy of a dentifrice-delivered anti-caries agent, a specially designed apparatus was used to position the patient when measurements were taken from facial WSLs on the anterior teeth. This positioning device was required to ensure the reproducibility of the QLF readings taken at periodic examinations during the treatment period.[27]

The FC and LF devices are simple to use and have been reported to have excellent reliability.[28,29] Since RI alters the optic properties of WSLs, our aim was to characterize the effects of RI on LF and FC readings obtained from teeth with artificial WSLs. The WSLs examined in this study were induced by exposing the enamel to a pH 4.5 lactic acid gel. These WSLs were visible when the tooth surfaces were wet, indicating subsurface mineral loss, and were observed to extend approximately 150 μm into the enamel. This is a typical depth for artificial WSLs on third molars.[26,30]

FC images of the intact buccal surfaces of the extracted third molars used in this study showed characteristic green fluorescence indicative of caries-free tooth structure. In order to quantitatively assess the impact of demineralization and RI on tooth fluorescence, we measured the brightness intensity of FC images rather than the false color images generated by the FC's Visix software. This analysis was accomplished using public domain software and could be used in a variety of clinical research

Table 2: *Subjective Assessment of WSL Severity by a Panel of Five Dentists.*

| Enamel Condition | Subjective WSL VAS Score (Mean ± SD) |
|---|---|
| Intact | 1.81 ± 1.32 |
| Demineralized | 9.77 ± 0.58* |
| Resin infiltrated | 3.77 ± 1.74* |

*Abbreviations: SD, standard deviation; VAS, visual analog scale; WSL, white spot lesion.*
* Significantly different from the VAS score of intact enamel ($p<0.05$).

settings. Following demineralization, we observed a mean 22.7% reduction in the fluorescent brightness intensity of the tooth surface. This value is within the range of QLF-measured fluorescent intensity reductions reported in studies where the fluorescent intensity of natural and artificial WSLs was compared with intact tooth structure.[31] This agreement between the QLF and FC was expected since both devices measure excited dentin fluorescence that is partially blocked by light scattering within WSLs.[1]

Following RI, the fluorescent brightness intensity of the treated demineralized area was restored to values that were not statistically different from those of sound enamel. This supports our hypothesis concerning the effect of RI on WSL fluorescence and agrees with fluorescence measurement results obtained using the QLF.[23] The reason for the small variations in fluorescent intensity observed in the line graph plot of RI enamel (Figure 3C) is not known. Possibly some areas with residual light scattering exist within the RI area.

The finding of this study is consistent with the observed actions of RI on the visual appearance of WSLs as demonstrated by our subjective assessment. In our subjective WSL assessment, the examiners assigned the RI areas significantly higher values than the sound surfaces. These VAS values were well below those given to the demineralized surfaces. It is possible that the smooth and more reflective surface of the RI area or traces of the peripheral aspects of the demineralized area, as observed in Figure 1, affected the examiner rating of the RI surfaces.

Enamel areas with caries-associated WSLs have been reported as having LF readings that are higher than those of sound enamel but lower than those of cavitated lesions.[21] In the current study, LF readings obtained from intact buccal surfaces were close to zero. The LF readings did not significantly change following creation of the WSL or RI of the demineralized enamel, remaining at the low end of the

instrument's range. Since the LF device has been used in clinical studies to assess remineralization, this result was unexpected.[21,32] The LF device measures laser excited fluorescence from bacterial pigments such as porphyrin.[17,24] In naturally occurring WSLs, bacterial pigment absorption by the demineralized enamel has been implicated as being responsible for the LF readings obtained from these lesions.[21] The results of this study and our investigations indicate that LF does not measure demineralization directly.

RI is an evolving technology with expanding indications, where various material or treatment procedure modifications influence the efficacy of the treatment.[33] Improved experimental methods and study designs are needed to determine the most effective method of WSL esthetic management.[34] Quantitative methods such as the FC can be used in clinical studies comparing RI with other methods of WSL esthetic management. In clinical practice, visual observation and patient satisfaction with esthetics are the main goals of facial surface WSL treatment. This is particularly true where RI is concerned since this treatment does not replace mineral lost during the demineralization. Clinicians can use the FC to illustrate areas of dentition with early carious lesions and highlight the need for minimally invasive intervention. The results of this study do not support the use of the LF device in studies of this nature. We plan to use the FC in clinical studies comparing the esthetic impact of RI with remineralization treatments. Maps of the tooth surface's fluorescent brightness intensity (Figure 3) can also be used in future studies to distinguish areas of a WSL that were treated with different RI formulations or procedures.

## CONCLUSIONS

When viewed using the FC, artificial WSLs appear as dark areas of reduced fluorescent intensity. RI restores the fluorescent intensity of demineralized areas to values equivalent to those obtained from intact enamel. These changes in fluorescence accompany changes in appearance of the WSL, where RI improves the esthetics of demineralized enamel. In contrast, values obtained from the tooth structure with an LF caries detector were not significantly altered by WSL formation or resin infiltration. These results indicate that the FC caries detector can be used in studies comparing the ability of RI and other methods of WSL treatment to improve the appearance and optical properties of these lesions.

### Acknowledgements

The authors thank Air Techniques for donating the Spectra Caries Detection Aid and for providing technical support. We thank DMG America for donating samples of the Icon resin infiltration system. We thank David Furgang and Shuying Jiang for assisting with the statistical analysis. We also thank the participants in the panel who performed the subjective evaluation of the white spot lesions. Neither of the authors have any conflict of interest pertinent to this research.

### Regulatory Statement

This *in vitro* study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Rutgers Biomedical and Health Sciences and the university's Institutional Review Board. The approval code for this study is: 0120050074.

### 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 18 June 2017)*

### REFERENCES

1. van der Veen MH, & de Josselin de Jong E (2000) Application of quantitative light-induced fluorescence for assessing early caries lesions *Monograph in Oral Science* **17** 144-162.
2. Ren Y, Jongsma MA, Mei L, van der Mei HC, & Busscher HJ (2014) Orthodontic treatment with fixed appliances and biofilm formation—A potential public health threat? *Clinical Oral Investigations* **18(7)** 1711-1718.
3. Al-Khateeb S, Forsberg CM, de Josselin de Jong E, & Angmar-Mansson B (1998) A longitudinal laser fluorescence study of white spot lesions in orthodontic patients *American Journal of Orthodontics and Dentofacial Orthopedics* **113(6)** 595-602.
4. Fontana M, Young DA, & Wolff MS (2009) Evidence-based caries, risk assessment, and treatment *Dental Clinics of North America* **53**(1) 149-161
5. Bailey DL, Adams GG, Tsao CE, Hyslop A, Escobar K, Manton DJ, Reynolds EC, & Morgan MV (2009) Regression of post-orthodontic lesions by a remineralizing cream *Journal of Dental Research* **88(12)** 1148-1153.
6. Bröchner A, Christensen C, Kristensen B, Tranaeus S, Karlsson L, Sonnesen L, & Twetmen S (2011) Treatment of post-orthodontic white spot lesions with casein phosphopeptide-stabilised amorphous calcium phosphate *Clinical Oral Investigations* **15(3)** 369-373.
7. Phark JH, Duarte S Jr, Meyer-Lueckel H, & Paris S (2009) Caries infiltration with resins: A novel treatment option for interproximal caries *Compendium of Continuing Education in Dentistry* **30**(**Special No 3)** 13-17.
8. Paris S, Schwendicke F, Keltsch J, Dorfer C, & Meyer-Lueckel H (2013) Masking of white spot lesions by resin infiltration in vitro *Journal of Dentistry* **41(Supplement 5)** e28-34.

9. Paris S, & Meyer-Lueckel H (2010) Inhibition of caries progression by resin infiltration in situ *Caries Research* **44(1)** 47-54.
10. Paris S, Schwendicke F, Seddig S, Muller WD, Dorfer C, & Meyer-Lueckel H (2013) Microhardness and mineral loss of enamel lesions after infiltration with various resins: Influence of infiltrant composition and application frequency in vitro *Journal of Dentistry* **41(6)** 543-548.
11. Ekstrand K, Martignon S, Bakhshandeh A, & Ricketts DN (2012) The non-operative resin treatment of proximal caries lesions *Dentistry Update* **39(9)** 614-616, 618-620, 622.
12. Hallgren K, Akyalcin S, English J, Tufekci E, & Paravina RD (2016) Color properties of demineralized enamel surfaces treated with a resin infiltration system *Journal of Esthetic and Restortorative Dententistry* **28(5)** 339-346.
13. Paris S, & Meyer-Lueckel H (2009) Masking of labial enamel white spot lesions by resin infiltration—A clinical report *Quintessence International* **40(9)** 713-718.
14. Rocha Gomes Torres C, Borges AB, Torres LM, Gomes IS, & de Oliveira RS (2011) Effect of caries infiltration technique and fluoride therapy on the colour masking of white spot lesions *Journal of Dentistry* **39(3)** 202-207.
15. Senestraro SV, Crowe JJ, Wang M, Vo A, Huang G, Ferracane J, & Covell DA Jr (2013) Minimally invasive resin infiltration of arrested white-spot lesions: A randomized clinical trial *Journal of the American Dental Association* **144(9)** 997-1005.
16. Pretty IA (2006) Caries detection and diagnosis: Novel technologies *Journal of Dentistry* 408 **34(10)** 727-739.
17. Lussi A, Hibst R, & Paulus R (2004) DIAGNOdent: an optical method for caries detection *Journal of Dental Research* **83(Special No C)** C80-83.
18. Graye M, Markowitz K, Strickland M, Guzy G, Burke M, & Houpt M (2012) In vitro evaluation of the Spectra early caries detection system *Journal of Clinical Dentistry* **23(1)** 1-6.
19. Jablonski-Momeni A1, Heinzel-Gutenbrunner M, & Klein SM (2014) In vivo performance of the VistaProof fluorescence-based camera for detection of occlusal lesions *Clinical Oral Investigations* **18(7)** 1757-1762.
20. Achilleos EE, Rahiotis C, Kakaboura A, & Vougiouklakis G (2013) Evaluation of a new fluorescence-based device in the detection of incipient occlusal caries lesions *Lasers in Medicine and Science* **28(1)** 193-201.
21. Moriyama CM, Rodrigues JA, Lussi A, & Diniz MB (2014) Effectiveness of fluorescence-based methods to detect in situ demineralization and remineralization on smooth surfaces *Caries Reserch* **48(6)** 507-514.
22. Knosel M, Eckstein A, & Helms HJ (2013) Durability of esthetic improvement following Icon resin infiltration of multibracket-induced white spot lesions compared with no therapy over 6 months: A single-center, split-mouth, randomized clinical trial *American Journal of Orthodontics and Dentofacial Orthopedics* **144(1)** 86-96.
23. Yuan H, Li J, Chen L, Cheng L, Cannon RD, & Mei L. (2014) Esthetic comparison of white-spot lesion treatment modalities using spectrometry and fluorescence *Angle Orthodontist* **84(2)** 343-349.
24. Markowitz K, Stenvall RM, & Graye M (2012) The effect of distance and tooth structure on laser fluorescence caries detection *Operative Dentistry* **37(2)** 150-160.
25. Jensen EC (2013) Overview of live-cell imaging: Requirements and methods used *Anatomical Record (Hoboken)* **296(1)** 1-8.
26. Boyle EL, Higham SM, & Edgar WM (1998) The production of subsurface artificial caries lesions on third molar teeth *Caries Research* **32(2)** 154-158.
27. Yin W, Hu DY, Li X, Fan X, Zhang YP, Pretty IA, Mateo LR, Cummins D, & Ellwood RP (2013) The anti-caries efficacy of a dentifrice containing 1.5% arginine and 1450 ppm fluoride as sodium monofluorophosphate assessed using quantitative light-induced fluorescence (QLF) *Journal of Dentistry* **41(Supplement 2)** S22-S28.
28. 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 the American Dental Association* **143(4)** 339-350.
29. 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.
30. Belli R, Rahiotis C, Schubert EW, Baratieri LN, Petschelt A, & Lohbauer U (2011) Wear and morphology of infiltrated white spot lesions *Journal of Dentistry* **39(5)** 376-385.
31. Angmar-Mansson B, & ten Bosch JJ (2001) Quantitative light-induced fluorescence (QLF): A method for assessment of incipient caries lesions *Dentomaxillofacial Radiology* **30(6)** 298-307.
32. Demito CF, Rodrigues GV, Ramos AL, & Bowman SJ (2011) Efficacy of a fluoride varnish in preventing white-spot lesions as measured with laser fluorescence *Journal of Clinical Orthodontics* **45(1)** 25-29.
33. Meyer-Lueckel H, Chatzidakis A, Naumann M, Dorfer CE, & Paris S (2011) Influence of application time on penetration of an infiltrant into natural enamel caries *Journal of Dentistry* **39(7)** 465-469.
34. Sonesson M, Bergstrand F, Gizani S, & Twetman S (2017) Management of post-orthodontic white spot lesions: An updated systematic review *European Journal of Orthodontics* **39(2)** 116-121.

©Operative Dentistry, 2018, **43-1,** 19-31

# Real-time Light Transmittance Monitoring for Determining Polymerization Completeness of Conventional and Bulk Fill Dental Composites

M Par • I Repusic • H Skenderovic • E Klaric Sever • D Marovic • Z Tarle

### Clinical Relevance

Short curing times of 10-20 seconds may be insufficient for an optimal polymerization, especially under nonideal clinical conditions (eg, variable distance and angulation of the curing unit tip).


## SUMMARY

**Objectives: To monitor the real-time changes in light transmittance during composite curing and to use transmittance data to determine the curing times required for a complete polymerization.**

**Methods: Three conventional and three bulk fill composites were cured with two light-emitting diode curing units at layer thicknesses of 2 mm and 4 mm. The real-time light transmittance data were collected by a UV-Vis spectrometer in the wavelength range of 350-550 nm, plotted against time (t) and fitted to an exponential function f(t), whose first derivative $\Delta T(t) = df(t)/dt$ represented the rate of transmittance change. As the changing transmittance reflects structural changes that occur during polymerization, $\Delta T(t) > 0$ was considered to indicate an ongoing polymerization, whereas $\Delta T(t)$ values approaching zero suggested a complete polymerization. This principle was used to determine times required for a complete polymerization ($t_{complete}$) for each material/thickness/curing unit combination.**

**Results: Light transmittance was significantly influenced by the material type, sample thick-**



*†Matej Par, DMD, private dental practice, Zagreb, Croatia

†Igor Repusic, DMD, Zagreb, Croatia

Hrvoje Skenderovic, PhD, Institute of Physics, Laboratory for Femtosecond Spectroscopy, Zagreb, Croatia

Eva Klaric Sever, PhD, DMD, School of Dental Medicine, Department of Endodontics and Restorative Dentistry, Zagreb, Croatia

Danijela Marovic, PhD, DMD, School of Dental Medicine, University of Zagreb, Department of Endodontics and Restorative Dentistry, Zagreb, Croatia

Zrinka Tarle, PhD, DMD, professor, School of Dental Medicine, Department of Endodontics and Restorative Dentistry, Zagreb, Croatia

*Corresponding author: Dankovecka 9, Zagreb, 10000, Croatia; e-mail: mpar@inet.hr

†These authors contributed equally.


DOI: 10.2341/17-041-L

**ness, and curing unit, amounting to 2.9%-27.0% for the bulk fill and 0.7%-16.7% for the conventional composites. The values of $t_{complete}$ amounted to 15.3-23.3 seconds for the bulk fill composites at 2 mm, 20.2-33.3 seconds for the conventional composites at 2 mm, 26.9-42.1 seconds for the bulk fill composites at 4 mm, and 40.1-59.8 seconds for the conventional composites at 4 mm. Additionally, an exponential relationship was discovered between the light transmittance and $t_{complete}$.**

**Conclusions: Some of the $t_{complete}$ values considerably exceeded the curing times recommended by the manufacturers.**

## INTRODUCTION

The need for simplification of the clinical placement of dental composites was addressed by launching the so-called bulk fill composites, which allow layer thicknesses over 2 mm.[1] This class of materials features larger particles or lower filler loads than do conventional composites, both factors acting to enhance the curing light penetration.[2] Together with other advanced compositional modifications that diminish the negative effect of polymerization shrinkage,[3] the bulk fill composites are suitable for placement in layers of 4 mm or thicker.[4,5] Introduction of bulk fill composites has substantially changed the material application technique, allowing most of the clinically encountered cavities to be filled in a single layer. In line with the efforts to further simplify and shorten the clinical placement of composites, some composite manufacturers recommend using high-irradiance curing units for short curing times of 10-20 seconds.[6] Concerns have been raised regarding the polymerization efficiency of such short curing times, indicating that deeper parts of a thick layer might remain undercured.[6,7] Since the monomer conversion of composite restorations is a fundamental property that underlies mechanical features and biocompatibility,[8,9] the clinical procedures should strive to attain the highest conversion values possible. It is thus unreasonable to compromise the monomer conversion at the restoration bottom just for the sake of saving several tens of seconds of chair time. Hence, the manufacturer's recommendations of short curing times should be critically evaluated by different methods. The curing efficiency at depth is usually assessed through measurements of monomer conversion[10] or microhardness,[11] whereas our article presents an alternative approach by using real-time light transmittance monitoring.

Assessing the polymerization progress through light transmittance monitoring is possible because the monomer conversion rise during curing is reflected as the change in light transmittance.[12] The light transmittance changes during polymerization are due to several processes: photoinitiator consumption, temperature increase, polymerization shrinkage, and change of the refractive index of the polymerizing resin.[13] The latter process is considered to make the greatest contribution to the transmittance change during polymerization.[14] Light attenuation in dental composites is chiefly determined by light scattering,[15] which depends on the refractive index mismatch between the resin and filler.[16] While the refractive index of filler remains constant throughout the polymerization, the refractive index of resin rises with monomer conversion.[17] Thus, the light transmittance can be considered a function of monomer conversion, although the exact mathematical relationship is unknown. However, the real-time light transmittance data can be used to assess the completeness of polymerization using a simple and straightforward rationale: the changing transmittance reflects an ongoing polymerization, whereas the plateau of the transmittance curve indicates a "completed" polymerization.[18] This approach neglects the individual contributions of multiple processes that affect the light transmittance change and simply postulates that the nonchanging transmittance equals a complete polymerization. After plotting the real-time light transmittance data as a function of time, the rate of transmittance change can be represented by calculating the first derivative of the transmittance curves. As the graph of the first derivative approaches zero, the polymerization reaction can be considered to near its completion. This principle is presented as a simple and inexpensive means for evaluating the polymerization completeness of dental composites, contrasting the more expensive and equipment-demanding vibrational spectroscopy, which is the standard method for quantifying the extent of polymerization.[19]

The aim of this study was to monitor the light transmittance of conventional and bulk fill composites during light curing and to use the real-time transmittance data to obtain information on polymerization progress. Furthermore, a method is presented for evaluating the curing times required for a complete polymerization. The hypotheses tested were the following: 1) light transmittance differs among composite materials, layer thicknesses, and curing units; 2) the curing time required for a complete polymerization differs among composite

Table 1: *Information About the Composite Materials, as Provided by Their Respective Manufacturers*

| Material (Abbreviation) | Shade Lot No. EXP | Composition | Filler Load, wt%/vol% | Manufacturer | Minimum Curing Time (Manufacturer's Recommendation)[a], s |
|---|---|---|---|---|---|
| X-tra fil (XF) | U<br>1441587<br>2017-04 | Bis-GMA, UDMA, TEGDMA, Ba-B-Al-Si glass | 86/70 | Voco, Cuxhaven, Germany | 10 s for >800 mW/cm$^2$ |
| Filtek Bulk Fil (FBF) | A2<br>N621319<br>2017-08 | Bis-GMA, Bis-EMA, UDMA, zirconia/silica, ytterbium trifluoride | 65/43 | 3M/ESPE, St Paul, MN, USA | 40 s for <1000 mW/cm$^2$<br>20 s for >1000 mW/cm$^2$ |
| Tetric EvoCeram Bulk Fill (TECBF-IVA) | IVA<br>S21840<br>2017-05 | Dimethacrylate, Ba-Al-Si-glass, prepolymer filler (monomer, glass filler and ytterbium fluoride), spherical mixed oxide | 80/61 (including 17% prepolymers) | Ivoclar Vivadent, Schaan, Liechtenstein | 10 s for >1000 mW/cm$^2$ |
| Tetric EvoCeram Bulk Fill (TECBF-IVB) | IVB<br>R77065<br>2016-10 | | | | |
| Tetric EvoCeram (TEC-A2) | A2<br>T26729<br>2018-07 | UDMA, Bis-GMA, Bis-EMA, ytterbium trifluoride | 76/54 | Ivoclar Vivadent, Schaan, Liechtenstein | 20 s for <1000 mW/cm$^2$<br>10 s for >1000 mW/cm$^2$ |
| Tetric EvoCeram (TEC-A3) | A3<br>S12959<br>2018-07 | | | | |
| Gradia Direct Posterior (GDP) | A3<br>1406252<br>2017-06 | UDMA, dimethacrylate, fluoroaluminosilicate glass | 77/65 | GC Corp, Tokyo, Japan | 20 s for <1200 mW/cm$^2$<br>10 s for >1200 mW/cm$^2$ |
| Grandio (GR) | A3<br>1428354<br>2018-01 | Ba-Al-Borosilicate glass filler, $SiO_2$ nanofillers, Bis-GMA, TEGDMA, Bis-EMA | 87/71 | Voco, Cuxhaven, Germany | 20 s for >500 mW/cm$^2$ |

*Abbreviations: Bis-EMA: ethoxylated bisphenol A dimethacrylate, Bis-GMA: bisphenol A glycidyl methacrylate, TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate.*
[a] *Manufacturers commonly recommend minimal curing time required for complete polymerization of 2- and 4-mm-thick layers for conventional and bulk fill composites, respectively. The recommendation also includes the minimum irradiance of a curing unit for which the listed curing times are advised.*

materials, layer thicknesses, and curing units; 3) the time required for a complete polymerization can be presented as a function of light transmittance; and 4) the curing times recommended by composite manufacturers are sufficient for a complete polymerization of conventional and bulk fill composites when applied in layers of 2 mm and 4 mm, respectively.

## METHODS AND MATERIALS

### Composite Materials and Light-curing Units

Three conventional and three bulk fill composites were investigated (Table 1). The specifications of light-emitting diode (LED)–based curing units are given in Table 2. The reported irradiance values were checked before and after the study to confirm that there was no decline in the curing unit output. The curing units differed by their irradiances and by radiant spectra; Bluephase Style M8 and Bluephase Style, respectively, featured one and two radiant peaks (Figure 1). Dual radiant peaks are characteristic of the latest generation of LED-curing units and are beneficial for curing composites that contain alternative photoinitiators.[20] The time-dependent radiant profiles recorded for the curing units operating for the longest time available (30 seconds) are shown in Figure 2.

Table 2: *Specifications of the Light-curing Units*

| Light-curing Unit | Manufacturer | Emission Maximum, nm[a] | Irradiance: Nominal[b]/True[c], mW/cm$^2$ |
|---|---|---|---|
| Bluephase Style M8 | Ivoclar-Vivadent, Schaan, Liechtenstein | 441 | 800/658 |
| Bluephase Style | Ivoclar-Vivadent, Schaan, Liechtenstein | 450, 405 | 1100/938 |

[a] *Measured with spectrometer HR4000 (Ocean Optics, Dunedin, FL, USA).*
[b] *Provided by the respective manufacturer.*
[c] *Plateau value measured with integrating sphere (IS, Gigahertz Optik GmbH, Puchheim, Germany).*

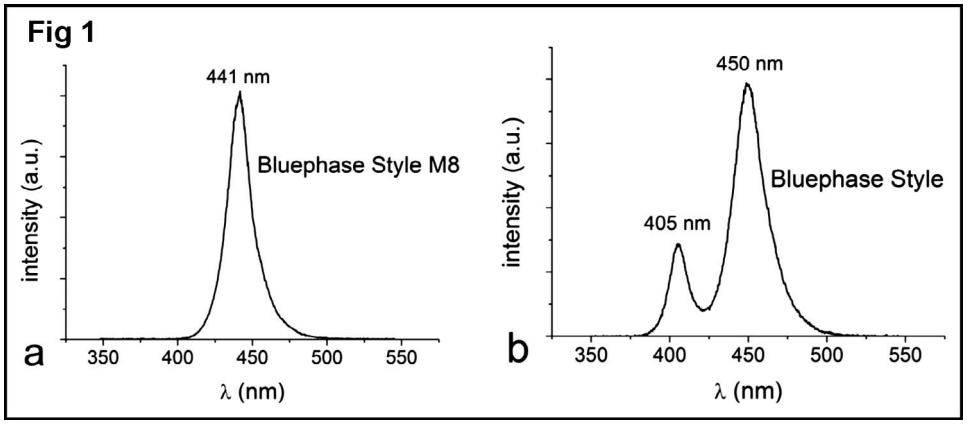



Figure 1. *Emission spectra of the curing units Bluephase Style M8 (a) and Bluephase Style (b).*

### Real-time Light Transmittance Measurements

Cylindrical composite samples (diameter=6 mm, height=2 or 4 mm) were prepared in black Teflon molds, and four repeats were performed (n=4) for each combination of material/thickness/curing unit. The small sample size (n=4 per experimental group) was considered sufficient because of the exploratory nature of this study. The experimental setup is described in a previous work.[13] Briefly, the uncured samples were covered from both sides with a polyethylene terephthalate (PET) film and sandwiched between two glass plates. The curing unit tip was centered to the Teflon ring opening immediately below the glass plate, and the light intensity was monitored from the opposite side of the sample. The spectra of transmitted light were recorded by the charge-coupled device array fiber spectrometer HR4000 (Ocean Optics, Dunedin, FL, USA) in the wavelength range of 350-550 nm during the light-curing period of 30 seconds. The data collection rate was 20 points per second. The intensity of light passing through the empty sample compartment (Teflon ring, two PET films, and two glass plates) was measured in the same manner.

### Analysis of Light Transmittance Data

In order to determine the time of polymerization completeness, the light transmittance data was analyzed through the following steps:

1) Integrated intensity of the light passing through the composite sample ($I_{sample}$) was measured for each time point. The same procedure was done for the measurements obtained from the empty sample compartment ($I_{empty}$) (Figure 3a).
2) Light transmittance was calculated as the ratio $I_{sample}/I_{empty}$. Plotting the transmittance values vs time gives the curve shown in Figure 3b. It is known that the irradiance of dental curing units may vary with time,[21] which was also true for the two curing units in our study (Figure 2). The variability of the curing unit output was compensated for by taking into account the real-time data for $I_{empty}$, rather than a single averaged value.
3) The transmittance curves were trimmed to include only the part during the light curing of 30 seconds. Then the curves were scaled so that the y-values ranged from 0 to 1, the value of 1 representing the maximum transmittance at time (t) = 30 seconds (Figure 3c). This was done to present the change of transmittance on a unified scale, regardless of actual transmittance values, which are different as a result of the material composition and sample thickness.
4) The curves were fitted to the function $f(t) = y_0 + a(1-\exp(-bt)) + c^*(1-\exp(-dt))$ (Figure 3c). This function has been used previously for describing the real-time changes in monomer conversion of dental composites and was demonstrated to be applicable to the light transmittance curves as well.[22] Unlike the conversion curves where the fitting parameters a, b, c, and d reflect certain physical processes,[22] the same parameters obtained from transmittance curves are not directly related to specific physical phenomena. Detailed analysis of the individual fitting parameters in terms of underlying physical processes was not attempted in our study, as the fitting was only performed to facilitate further processing of the transmittance curves.
5) The first derivative of function f(t) from step 4 was calculated as $\Delta T(t) = df(t)/dt$. The function $\Delta T(t)$ represents the rate at which the light transmittance increases during curing (Figure 3d).

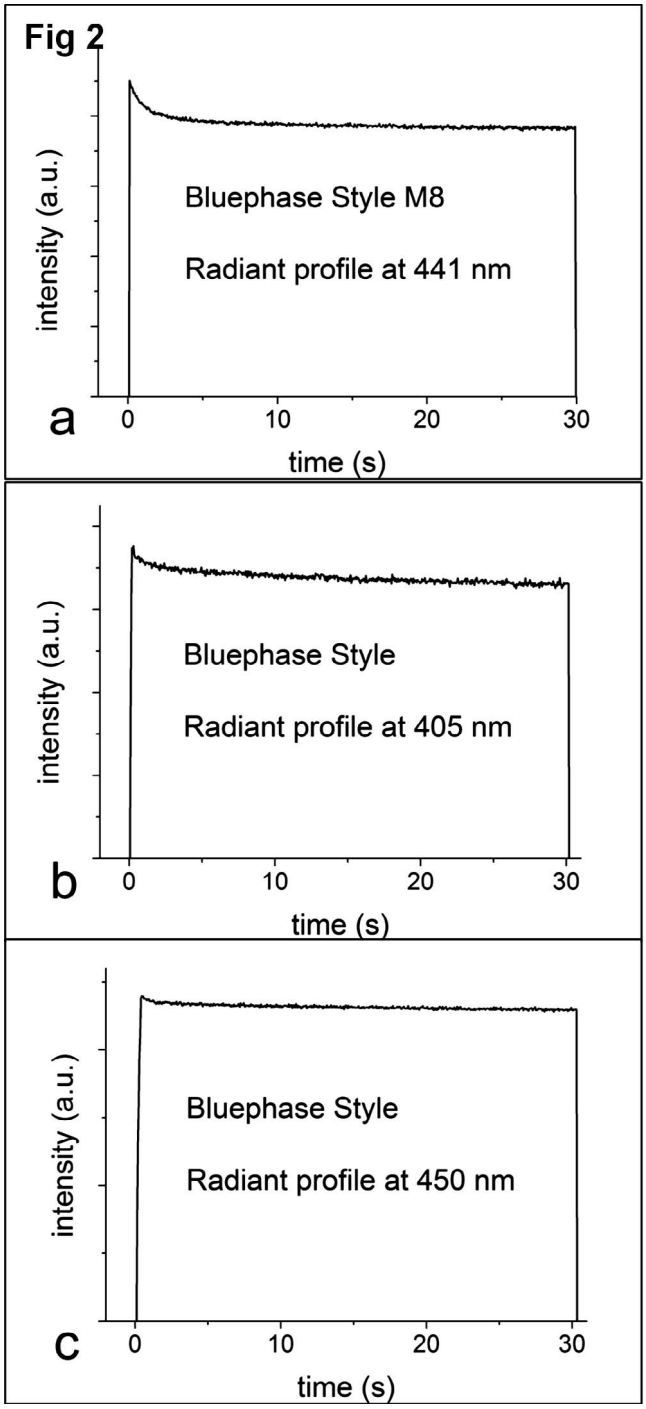



Figure 2. *Radiant profiles of the curing units Bluephase Style M8 (a) and Bluephase Style (b,c). Intensity spikes of different magnitudes are observed immediately on activation, followed by plateaus of a rather stable irradiance.*

6) From $\Delta T(t)$, the time of polymerization completeness ($t_{complete}$) was determined. As this function has no x-intercept, the polymerization was considered completed at the time when $\Delta T(t) = 0.001$ $s^{-1}$. The threshold value of 0.001 $s^{-1}$ was chosen because the preliminary experiments using the approximations of first derivatives obtained from the nonfitted curves showed experimental error of this order of magnitude.
7) For some material/thickness combinations, the $\Delta T(t)$ value of 0.001 $s^{-1}$ was not reached during the light curing of 30 seconds. In such cases, the time of polymerization completeness was determined by extrapolation [ie, plotting the function $\Delta T(t)$ beyond the 30 seconds and then finding the t value for which $\Delta T(t)=0.001$ $s^{-1}$]. This approach was introduced since the maximum continuous curing time supported by the curing units was 30 seconds.

### Statistical Analysis

Normality of distribution was confirmed using the Shapiro-Wilk test. The mean values of light transmittance and $t_{complete}$ were compared using a three-way analysis of variance (ANOVA) with factors "material," "thickness," and "curing unit." The Tukey honestly significantly different (HSD) post hoc test was used for multiple comparisons. Partial eta-squared statistics were used to describe relative influences and interactions of the factors "material," "thickness," and "curing unit." One-way ANOVA was conducted to compare $t_{complete}$ values within a given sample thickness. The Tukey HSD post hoc test was used for the 2-mm thickness (equal variances), while the Games-Howell post hoc test was used for 4-mm thickness (unequal variances). Statistical software SPSS 20 (IBM, Armonk, NY, USA) was used, with level of significance set at 0.05.

## RESULTS

Light transmittance values measured at the start and end of illumination are shown in Table 3. The group of bulk fill composites generally showed higher transmittance (2.9%-27.0%) than the conventional composites (0.7%-16.7%). The transmittance values increased during polymerization for all composites, with considerable differences in the magnitude (minimal increase of 0.2% and maximum increase of 7.0%). For a given material, curing unit, and time point, the transmittance of 4-mm-thick samples was 2 to 7.5 times lower compared to that of the 2-mm samples.

Mean values of $t_{complete}$ are given in Table 4. The shortest times (15.3-23.3 seconds) were observed for the bulk fill composites at sample thickness of 2 mm. Comparatively longer times were noted for the conventional composites at 2 mm (20.2-33.3 sec-

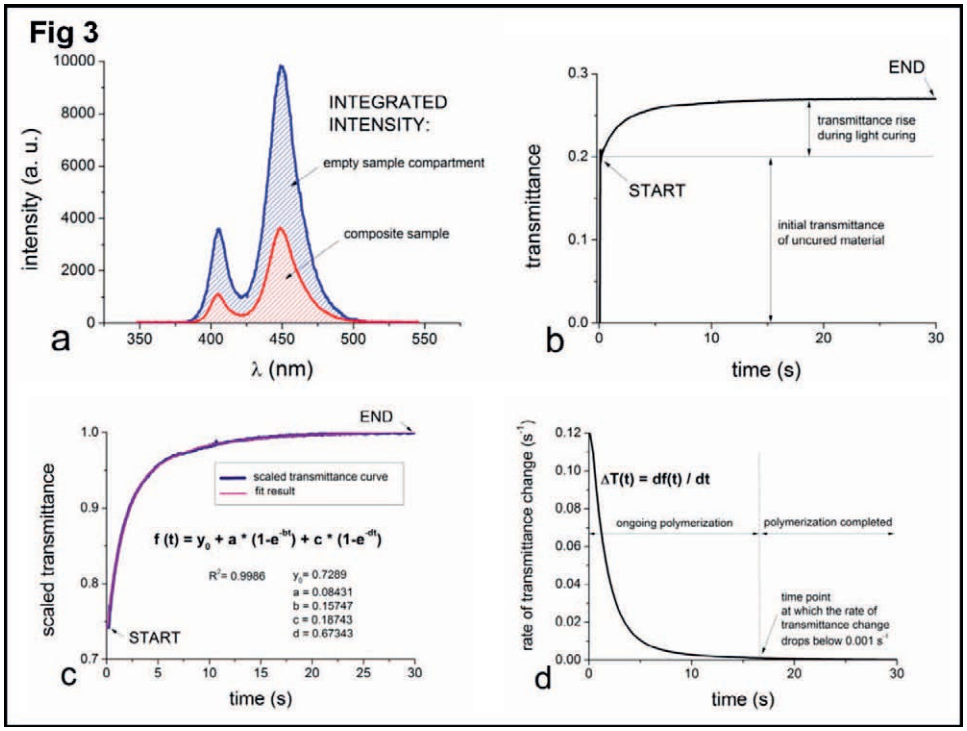



Figure 3. *An example of light transmittance analysis for a 2-mm-thick sample of material X-tra fil cured with Bluephase Style. Integrated intensities of the composite sample and the empty sample compartment (a). Light transmittance through the composite sample as a function of time (b). The points denoted as "start" and "end" are the initial and final marks for trimming the transmittance curve, respectively. The result of trimming and scaling to the y-values between 0 and 1 is shown in (c). This curve was fitted to the exponential function, whose first derivative was calculated to represent the rate of transmittance change (d). The polymerization is considered completed when the y-values of the derivative drop below 0.001 $s^{-1}$.*

Table 3: *Mean Transmittance Values (%). Standard Deviations Are Given in Parentheses. The Initial and Final Transmittance Values Were Determined, Respectively, at the "Start" and "End" Points of the Transmittance Curves, as Shown in Figure 3b*[a]

| Composite Material | 2 mm | | | | 4 mm | | | |
|---|---|---|---|---|---|---|---|---|
| | Bluephase Style | | Bluephase Style M8 | | Bluephase Style | | Bluephase Style M8 | |
| | Initial | Final | Initial | Final | Initial | Final | Initial | Final |
| Bulk Fill | | | | | | | | |
| XF | 20.85 (1.55) Aa | 27.02 (1.49) Aa | 13.11 (0.15) Ab | 15.29 (0.11) ABb | 6.87 (0.45) Ac | 11.08 (0.69) Bc | 5.41 (0.20) Ac | 7.29 (0.19) Ad |
| FBF | 16.26 (0.19) Ba | 22.21 (0.39) Ba | 7.90 (0.26) Db | 9.84 (0.30) Db | 4.95 (0.18) Cc | 8.96 (0.50) Cc | 2.95 (0.10) Ed | 4.34 (0.14) Cd |
| TECBF-IVA | 19.68 (2.19) Aa | 26.70 (2.18) Aa | 11.86 (0.56) Bb | 14.76 (0.52) Bb | 5.47 (0.12) BCc | 10.65 (0.20) Bc | 4.58 (0.30) Bc | 7.31 (0.41) Ad |
| TECBF-IVB | 18.33 (2.18) ABa | 24.73 (1.94) ABa | 12.40 (0.26) ABb | 15.44 (0.28) Ab | 6.06 (0.67) Bc | 12.25 (0.91) Ac | 4.25 (0.04) BCc | 6.86 (0.07) Ad |
| Conventional | | | | | | | | |
| TEC-A2 | 11.26 (1.28) Ca | 15.50 (1.59) Ca | 10.74 (0.31) Ca | 11.85 (0.26) Cb | 2.47 (0.22) Db | 4.47 (0.29) Dc | 3.94 (0.13) CDc | 4.96 (0.26) Bc |
| TEC-A3 | 10.52 (0.23) Ca | 14.80 (0.19) CDa | 10.51 (0.18) Ca | 11.96 (0.16) Cb | 2.15 (0.23) Db | 3.79 (0.43) DEc | 3.59 (0.32) Db | 4.43 (0.33) BCd |
| GDP | 11.51 (0.20) Ca | 16.68 (0.57) Ca | 6.99 (0.41) Eb | 8.51 (0.33) Eb | 2.40 (0.19) Dc | 4.36 (0.39) DEc | 0.93 (0.07) Fd | 1.62 (0.12) Dd |
| GR | 9.81 (0.42) Ca | 11.79 (0.46) Da | 3.05 (0.15) Fb | 3.55 (0.18) Fb | 2.44 (0.15) Dc | 3.25 (0.20) Eb | 0.68 (0.04) Fd | 0.89 (0.04) Ec |

[a] *Same uppercase letters denote statistically similar values within a column. Same lowercase letters denote statistically similar values within a row for a given time point (initial/final).*

Table 4: *Times Required for a Complete Polymerization, Mean (SD)*

| Composite Material | 2 mm | | 4 mm | |
|---|---|---|---|---|
| | Bluephase Style | Bluephase Style M8 | Bluephase Style | Bluephase Style M8 |
| Bulk Fill | | | | |
| XF | 15.29 (1.12) | 16.19 (0.80) | 26.94 (0.73) | 31.78 (1.36) |
| FBF | 17.64 (1.21) | 22.42 (0.74) | 31.54 (1.55) | 38.28 (0.68) |
| TECBF-IVA | 20.85 (1.04) | 23.32 (1.13) | 39.10 (0.53) | 42.05 (1.87) |
| TECBF-IVB | 18.39 (2.31) | 21.28 (0.80) | 36.08 (1.36) | 41.38 (0.66) |
| Conventional | | | | |
| TEC-A2 | 22.09 (2.42) | 22.08 (2.90) | 40.90 (3.80) | 49.03 (4.03) |
| TEC-A3 | 20.15 (0.82) | 24.66 (2.56) | 56.33 (12.88) | 40.05 (8.71) |
| GDP | 26.32 (2.80) | 28.20 (0.80) | 59.78 (4.75) | N/A |
| GR | 33.25 (0.81) | N/A | 56.70 (8.64) | N/A |

*Abbreviations: N/A, not available.*

onds), followed by the bulk fill composites at 4 mm (26.9-42.1 seconds), and finally the conventional composites at 4 mm (40.1-59.8 seconds). As a result of the poor signal/noise ratio and subsequently high data scatter (coefficients of variation up to 100%) for three material/thickness/curing unit combinations, the corresponding data are considered unreliable and are not reported in Table 4. Mean values of $t_{complete}$ are additionally represented graphically and complemented with the results of the statistical analysis in Figure 4. This is convenient for visualizing the differences among the materials, sample thicknesses, and curing units.

Table 5 reports relative contributions of the factors "material," "thickness," and "curing unit" on the light transmittance and $t_{complete}$. All of the factors exerted a highly significant influence on the dependent variables, whereas "thickness" and "material" proved more influential than "curing unit." Also, a significant interaction was observed for all factor combinations.

## DISCUSSION

This work investigated the light transmittance of dental composites and assessed the real-time light transmittance monitoring as a means for determining curing times required for a complete polymerization.

### Effect of Composite Material, Sample Thickness, and Curing Unit on Light Transmittance

The first hypothesis was accepted, as the light transmittance was significantly affected by factors "material," "thickness," and "curing unit" (Table 5). The greatest influence was observed for the factor "thickness," which can be explained by the well-known exponential increase of light attenuation with lengthening of the light path through the composite.[15,16] The values of light transmittance varied greatly among the composites (Table 3). This was caused by differences in filler type, load, and geometry, as well as by different resin compositions

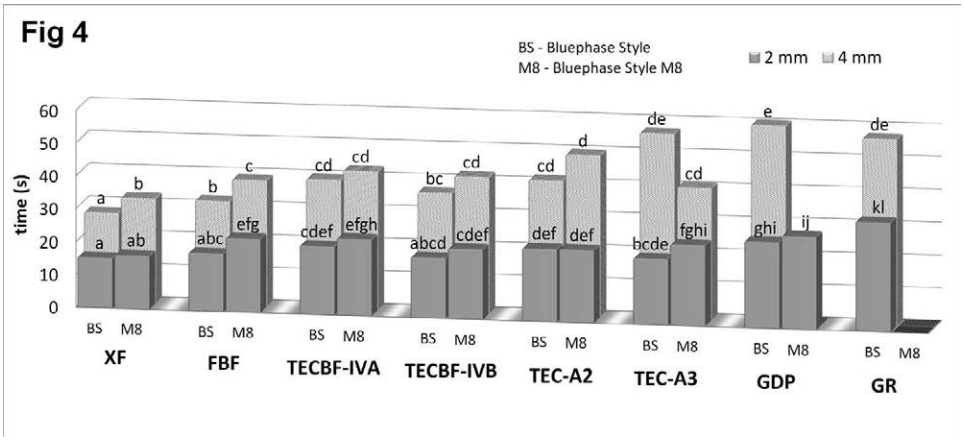



Figure 4. *Time required for a complete polymerization and results of the statistical analysis. Same lowercase letters denote statistically similar groups within an individual layer thickness. The three missing bars represent the data not available.*

Table 5: *Influence of the Factors Thickness, Material, and Curing Unit and Their Interactions on the Initial and Final Transmittance Values and Time of Polymerization Completeness*

| Factor | Initial Transmittance | | Final Transmittance | | Time of Polymerization Completeness | |
|---|---|---|---|---|---|---|
| | *p* | Partial $\eta^2$ | *p* | Partial $\eta^2$ | *p* | Partial $\eta^2$ |
| Thickness | <0.001 | 0.973 | <0.001 | 0.977 | <0.001 | 0.966 |
| Material | <0.001 | 0.925 | <0.001 | 0.960 | <0.001 | 0.918 |
| Curing unit | <0.001 | 0.821 | <0.001 | 0.936 | <0.001 | 0.625 |
| Thickness × material | <0.001 | 0.645 | <0.001 | 0.617 | <0.001 | 0.352 |
| Material × curing unit | <0.001 | 0.702 | <0.001 | 0.768 | 0.005 | 0.231 |
| Thickness × curing unit | <0.001 | 0.709 | <0.001 | 0.802 | 0.004 | 0.112 |

and pigment additives.[23] When considered as a group, the bulk fill composites presented higher transmittance values (2.9%-27.0 %) than did the conventional composites (0.7%-16.7%). This is a common feature of bulk fill composites[2] and is necessary for their bulk fill capability.

As the light scattering at filler particles is the main determinant of light transmittance, one could expect the decrease of transmittance with increasing filler load.[24] However, in our study no relationship between transmittance and filler load was found, since the transmittance was considerably influenced by other factors, mainly filler geometry and filler/resin refractive index mismatch. Thus, filler load alone was not a good predictor of light transmittance. This can be illustrated by the fact that the least translucent material (Grandio) and the most translucent material (X-tra fil) had similar filler loads of 71 vol% and 70 vol%, respectively. This difference in transmittance can be attributed to the Grandio particle size of 0.1-2.5 μm[25] causing much higher scattering of blue light[16] than was caused by the comparatively larger 2-3-μm particles contained in X-tra fil.[25] An additional interesting observation is that X-tra fil showed the highest light transmittance despite being among the most highly filled materials in our study (Table 1), demonstrating that high filler load is not necessarily incompatible with the bulk fill capability.

When comparing the light transmittance values between the two polymerization devices with all other factors being equal, transmittance was generally higher in the case of Bluephase Style (Table 3). The transmittance of dental composites is mainly determined by light scattering,[14] which is in turn dependent upon the wavelength of incident light, geometry of the filler particles, and mismatch of the refractive indices between the filler and resin.[16] As the detailed composition of commercial composites is undisclosed by the manufacturers, insufficient data are available on the particle geometry and refractive indices of individual fillers and monomer blends. Thus, the observed difference in transmittance between two curing units cannot be thoroughly discussed. It can only be speculated that the consistently higher light transmittance for Bluephase Style was due to the differences in the radiant spectra of the curing units: namely, as the intensity of light scattering is inversely related to the wavelength,[15] the light produced by Bluephase Style (major radiant peak at 450 nm) may have been less scattered than that produced by Bluephase Style M8 (single radiant peak at 441 nm) (Figure 1).

All of the composites showed an increase in light transmittance during light curing (Table 3). The individual combinations of material/thickness/curing unit showed large differences in the amount of transmittance increase. For example, transmittance of 2-mm samples of Tetric EvoCeram (shade A2) cured with Bluephase Style M8 increased for a factor of 0.1, while for the 4-mm samples of Tetric EvoCeram Bulk Fill (shade IVB) cured with Bluephase Style, transmittance increased for a factor of 1.0. . The factors, for which the light transmittance was increased in the other composites, ranged between these two extreme values. A practical implication of the transmittance rise during curing is that the gradual improvement of curing light penetration can enhance the radiant energy received by the bottom of the overly thick layers that sometimes occur in clinical practice. These layers are commonly undercured; however, a considerable increase of transmittance during polymerization might help to mitigate the undercuring, provided that sufficiently long curing times are used. In this regard, the composites whose transmittance increases for a larger factor may benefit more from extended curing[7] and be more successful at avoiding the undercuring of accidentally placed layers of excessive thickness.

### Real-time Light Transmittance Monitoring as a Means for Determining Polymerization Completeness

The rate of transmittance change was presented as a function of time, and values above 0.001 $s^{-1}$ were considered to indicate an ongoing polymerization. As the rate of transmittance change fell below this threshold, the polymerization was considered complete, and times at which this occurred were denoted as $t_{complete}$ (Figure 3d). Although the light transmittance increased during curing for all of the tested composites, a different transmittance behavior is possible. For example, one recently launched bulk fill material shows an increase in light transmittance during polymerization.[26] Also, by tuning the filler/resin refractive indices, composites could be formulated with the same initial and final refractive index mismatch, so that their initial and final transmittances correspond.[27] Both mentioned behaviors should be compatible with the presented method of assessing polymerization completeness, as this method relies only on the light transmittance change, regardless of its direction and absolute transmittance values.

The presented method showed some limitations, as observed for the 4-mm samples of conventional composites, the $t_{complete}$ values for which amounted to 40.1-59.8 seconds. Since the curing lasted for 30 seconds, the $t_{complete}$ values longer than this time were estimated by extrapolation. The extrapolation approach is inherently inaccurate, and higher inaccuracy is expected for values that are farther away from the measured range. This explains the highest scatter of $t_{complete}$ data for 4-mm-thick samples of conventional composites among all other material/thickness combinations, as their $t_{complete}$ is rather remote from the measured range of 0-30 seconds. Overall, it appears that our method gives reproducible results for $t_{complete}$ values that are contained within the time interval of curing unit activation, as well as for extrapolated estimates up to about 40 seconds. Estimates of $t_{complete}$ higher than 40 seconds featured high scatter and mostly related to the 4-mm-thick samples of conventional composites. These estimates are, however, of little clinical relevance, as conventional composites are not intended for placement in 4-mm-thick layers.

Another limitation of our method is poor reproducibility when low transmittance rises during polymerization (below 0.7%) were coupled with low transmittance values (0.7%-3.6%), which yielded low signal to noise ratio and consequently high coefficients of variability (above 100%). This was the case for the three cells in Table 4 whose values were denoted as not available. An additional aspect of multifunctional methacrylate polymerization that is not accounted for by our method is the phenomenon of postcure reaction. As a result of an immense increase in viscosity of a reaction medium during polymerization, the mobility of reactive species is impaired and polymerization rate decreases substantially.[28] Thus, a high proportion of monomer remains unreacted and available for further polymerization, which is known to slowly continue for at least 24 hours after light curing.[29] In this regard, our method of determining polymerization times is limited to the changes that occur during the light curing and does not account for the reaction that occurs thereafter. Although the postcure increase in monomer conversion can be rather extensive[30] and may compensate for the initially lower conversions attained during a short period of light curing,[31] it would be beneficial to attain as high a rate of conversion as possible during light curing in order to minimize the toxic potential of unreacted monomer.

The partial eta-squared statistics showed that factors "material," "thickness," and "curing unit" exerted a significant influence on $t_{complete}$, whereas significant interactions between the factors imply that the influence of each individual factor depended on the level of other factors (Table 5). These findings support the second hypothesis and reflect the fact that the polymerization kinetics, and consequently the values of $t_{complete}$, depend upon a complex interplay of multiple factors, some of which are sample geometry, total irradiance and spectral irradiance of the curing unit, monomer viscosity and reactivity, filler load and particle size distribution, photoinitiator type, reactivity, and concentration.[23,32,33] However, it is interesting to note that the $t_{complete}$ could be rather well described as a function of the single parameter (ie, light transmittance). This is demonstrated in Figure 5, which plots $t_{complete}$ as a function of light transmittance and supports our third hypothesis. Although it is obvious that $t_{complete}$ should be inversely related to light transmittance, the fact that their relationship could be well fitted to an exponential function suggests that transmittance was the major determinant of the curing time needed for a complete polymerization, while the other variables appear to play a comparatively smaller role. Since the light transmittance was highly influenced by the factor "thickness," it is understandable that the same factor also exerted the highest influence on $t_{complete}$, as shown by the corresponding partial eta-squared values in Table

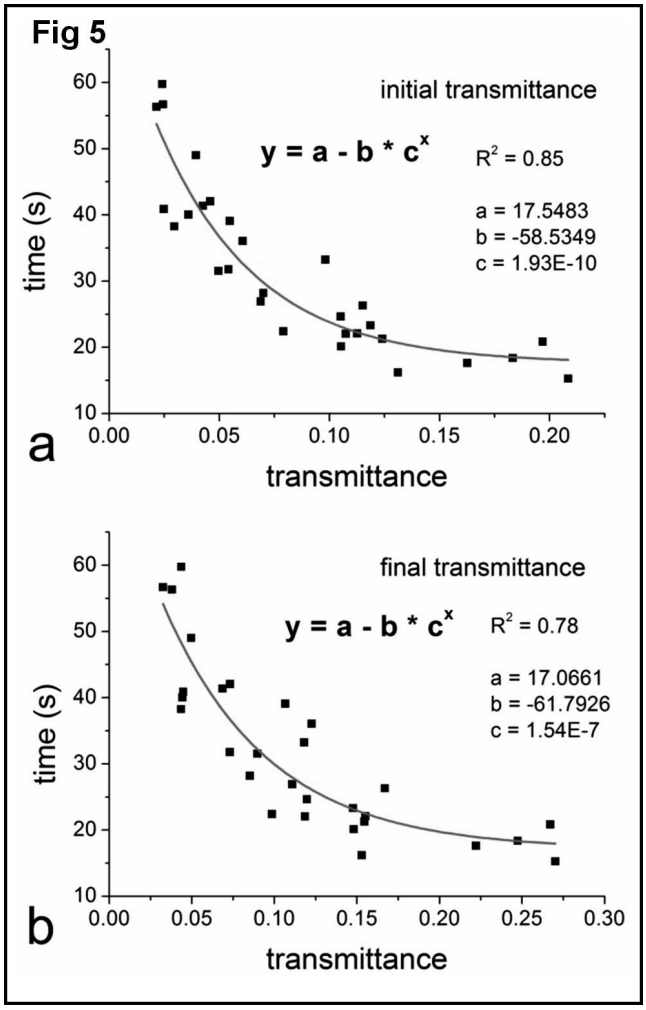



Figure 5. *Relationship between the light transmittance and time of polymerization completeness. The initial ("start" from Figure 3b) and final ("end" from Figure 3b) transmittance values were plotted against the time required for a complete polymerization. Each point represents a single combination of material/thickness/curing unit and is a mean value of n = 4.*

5. Finally, the finding that polymerization completeness highly depends on the layer thickness emphasizes the need to strictly follow recommendations on maximum layer thickness in clinical work.

Curing unit Bluephase Style M8 was recently introduced to the market as an economic alternative to its more expensive counterpart, Bluephase Style. The latter features a wider radiant spectrum (Figure 1), which is favorable for curing composites containing alternative photoinitiators[34]; however, the large majority of available composites are camphorquinone-initiated[35]; hence, such wide spectrum is not necessary. This was the rationale for launching Bluephase Style M8 as a curing unit of a more favorable price/performance ratio.[36] In line with its lower irradiance, Bluephase Style M8 showed curing efficiency that was inferior to that of Bluephase Style, evidenced by the longer $t_{complete}$ values (Table 4; Figure 4). This may be explained by the following characteristics of Bluephase Style: 1) better light penetration through composite materials (Table 3); 2) higher total light irradiance (Table 2), which activates more photoinitiator molecules while also improving resin mobility due to the temperature rise[37]; and 3) major radiant peak closer to the maximum absorption of camphorquinone at 468 nm (Figure 1).[38]

Among the composites in our study, only Tetric EvoCeram Bulk Fill contained an alternative photoinitiator Ivocerin, in addition to the conventional camphorquinone-amine photoinitiator system.[26] Ivocerin is a patented germanium-based compound that is claimed to improve the curing efficiency of thick layers because of its high reactivity at low irradiance.[11] From the data on 4-mm-thick samples of bulk fill composites, the benefit of Ivocerin could not be confirmed, as Tetric EvoCeram Bulk Fill required similar or longer $t_{complete}$ than the camphorquinone-initiated material Filtek Bulk Fill (Figure 4), despite lower light transmittance of the latter (Table 3). The 4-mm values of $t_{complete}$ for Tetric EvoCeram Bulk Fill were also higher than those of another camphorquinone-initiated bulk fill composite, X-tra fil, which can be explained by differences in light transmittance (Table 3; Figure 4).

The composites Tetric EvoCeram and Tetric EvoCeram Bulk Fill were tested in two shades (Table 1). The different shades of the same material are compositionally very similar and differ only in the small amount of pigments. These subtle compositional variations did not cause a statistically significant difference in transmittance values between two shades, nor did they have an impact on the values of $t_{complete}$ (Table 3; Figure 4).

Composite manufacturers commonly provide instructions for use that specify the minimum irradiance and curing time required for curing of an individual layer (Table 1). These data apply to the standard layer thickness of 2 mm for conventional and 4 mm for bulk fill composites. Some manufacturers also suggest that curing times could be shortened if a curing unit of sufficient irradiance is used. Thus, for some of the composites, curing times as short as 10 seconds are recommended. Although certainly attractive from a clinical standpoint, short curing times may yield an incomplete polymerization and compromise multiple restoration properties.

Tarle and others[6] examined five bulk fill composites cured with an LED curing unit with irradiance

of 1170 mW/cm$^2$ and found suboptimal values of monomer conversion and microhardness when 4-mm-thick samples were cured according to the manufacturer's recommendations. They also found that mechanical properties of cured bulk fill composites can be improved by prolonging the curing times up to 30 seconds. A similar investigation was conducted by Zorzin and others,[7] who used irradiance of 1200 mW/cm$^2$ to compare the effect of 30-second curing time with the manufacturer-recommended curing times of 10-20 seconds. In their study, the extended curing for 30 seconds resulted in improved hardness and monomer conversion for several bulk fill composites and the conventional reference material. Moreover, Miletic and others[39] determined optimal curing times by assessing monomer conversion and hardness of bulk fill composites cured with LED unit of 1100 mW/cm$^2$ and showed that for the low-viscosity (flowable) bulk fill composites, a curing time of 10 seconds was sufficient, whereas high-viscosity (sculptable) bulk fill composites required at least 20 seconds. Other studies on bulk fill composites that assessed the effect of various curing protocols on layers up to 6 mm were performed by Ilie and Stark[11,40] and reported the value of 23.5 J/cm$^2$ as the minimum radiant energy required for a sufficient polymerization, which corresponds to 20 seconds of curing with irradiance of 1176 mW/cm$^2$. Our work used curing units of lower irradiances (658 and 938 mW/cm$^2$) than the cited studies,[6,7,11,39,40] which resulted in comparatively longer curing times needed for a complete polymerization (20.2-33.3 seconds for 2-mm layers of conventional composites and 26.9-42.1 seconds for 4-mm layers of bulk fill composites). In summary, all of these results contribute to a considerable amount of evidence suggesting that short curing times of 10-20 seconds, as recommended by some manufacturers, are insufficient for optimal polymerization, even under ideal curing conditions in the laboratory setting. The fourth hypothesis was thus rejected.

Clinicians tend to use longer curing times than those minimally recommended by composite manufacturers as a means of avoiding a possible undercuring.[41] The commonly used curing times are preset in the contemporary curing units and range from 10 to 40 seconds, whereas the longest "program" of 40 seconds exceeds by far the recommendations of composite manufacturers (Table 1). Our results show that the longest curing setting of Bluephase Style and Bluephase Style M8 (30 seconds) ensured an adequate cure of 2-mm layers for all of the tested composites, except for Grandio, whose $t_{complete}$ was 33.3 seconds (Table 4). This can be attributed the fact that Grandio had the lowest transmittance among all of the materials (Table 3). For 4-mm-thick layers, the curing time of 30 seconds ensured a complete polymerization only for material X-tra fil, whereas all other composites required longer curing times. These data suggest that in the clinical work most of the investigated bulk fill composites placed in 4-mm layers would require more than one curing cycle of 30 seconds in order to reach complete polymerization.

It should be noted that the manufacturer recommendations are based on ideal-case laboratory studies, which rarely correspond to a more complex clinical reality. Delivering the sufficient radiant energy to the restorations in clinical practice is often hindered by the distance and angulation of the curing unit tip. Most of the parameters related with the curing effectiveness (true irradiance and its decrease due to the curing unit aging, light beam inhomogeneity, spectral irradiance and its effect on particular photoinitiators) are unknown to clinicians, while some factors (position of the curing unit relative to the restoration) are beyond their control. However, most of these issues could be to some extent mitigated by prolonged curing,[6,7] once again highlighting the benefit of extending the curing times beyond those recommended by the manufacturers.

## CONCLUSIONS

Within the limitations of this study, the following can be concluded:

1) The real-time light transmittance monitoring can be used as a simple and effective means for assessing the time required for a complete polymerization of dental composites;
2) Sample thickness was the most influential factor for the time of polymerization completeness, followed by the factors material and curing unit;
3) Time of polymerization completeness showed an exponential relationship with the light transmittance; and
4) The times required for a complete polymerization under clinically relevant curing conditions (layer thickness of 2 mm for conventional and 4 mm for bulk fill composites) ranged from 15.3 to 42.1 seconds, which considerably exceeds some of the curing times recommended by the manufacturers.

**Acknowledgements**

This investigation was supported by Croatian Science Foundation (Project 08/31 Evaluation of New Bioactive Materials and Procedures in Restorative Dental Medicine). The curing units were kindly provided by Ivoclar Vivadent.

**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 April 2017)*

**REFERENCES**

1. Ilie N, Bucuta S, & Draenert M (2013) Bulk-fill resin-based composites: An in vitro assessment of their mechanical performance *Operative Dentistry* **38(6)** 618-625.
2. Bucuta S, & Ilie N (2014) Light transmittance and micromechanical properties of bulk fill vs. conventional resin based composites *Clinical Oral Investigations* **18(8)** 1991-2000.
3. Marovic D, Taubock TT, Attin T, Panduric V, & Tarle Z (2015) Monomer conversion and shrinkage force kinetics of low-viscosity bulk-fill resin composites *Acta Odontologica Scandinavica* **73(6)** 474-480.
4. 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.
5. Alrahlah A, Silikas N, & Watts DC (2014) Post-cure depth of cure of bulk fill dental resin-composites *Dental Materials* **30(2)** 149-154.
6. Tarle Z, Attin T, Marovic D, Andermatt L, Ristic M, & Taubock TT (2015) Influence of irradiation time on subsurface degree of conversion and microhardness of high-viscosity bulk-fill resin composites *Clinical Oral Investigations* **19(4)** 831-840.
7. Zorzin J, Maier E, Harre S, Fey T, Belli R, & Lohbauer U, Petschelt A & Taschner M (2015) Bulk-fill resin composites: Polymerization properties and extended light curing *Dental Materials* **31(3)** 293-301.
8. Durner J, Obermaier J, Draenert M, & Ilie N (2012) Correlation of the degree of conversion with the amount of elutable substances in nano-hybrid dental composites *Dental Materials* **28(11)** 1146-1153.
9. Rueggeberg FA, & Craig RG (1988) Correlation of parameters used to estimate monomer conversion in a light-cured composite *Journal of Dental Research* **67(6)** 932-937.
10. Par M, Gamulin O, Marovic D, Skenderovic H, Klaric E, & Tarle Z (2016) Conversion and temperature rise of remineralizing composites reinforced with inert fillers *Journal of Dentistry* **48** 26-33.
11. Ilie N, & Stark K (2014) Curing behaviour of high-viscosity bulk-fill composites *Journal of Dentistry* **42(8)** 977-985.
12. Howard B, Wilson ND, Newman SM, Pfeifer CS, & Stansbury JW (2010) Relationships between conversion, temperature and optical properties during composite photopolymerization *Acta Biomaterialia* **6(6)** 2053-2059.
13. Par M, Marovic D, Skenderovic H, Gamulin O, Klaric E, & Tarle Z (2017) Light transmittance and polymerization kinetics of amorphous calcium phosphate composites *Clinical Oral Investigations* **21(4)** 1173-1182.
14. Fujita K, Ikemi T, & Nishiyama N (2011) Effects of particle size of silica filler on polymerization conversion in a light-curing resin composite *Dental Materials* **27(11)** 1079-1085.
15. Ruyter IE, & Oysaed H (1982) Conversion in different depths of ultraviolet and visible light activated composite materials *Acta Odontologica Scandinavica* **40(3)** 179-192.
16. Clewell DH (1941) Scattering of light by pigment particles *Journal of the Optical Society of America* **31** 512-517.
17. Fujita K, Nishiyama N, Nemoto K, Okada T, & Ikemi T (2005) Effect of base monomer's refractive index on curing depth and polymerization conversion of photo-cured resin composites *Dental Materials Journal* **24(3)** 403-408.
18. Harrington E, Wilson HJ, & Shortall AC (1996) Light-activated restorative materials: A method of determining effective radiation times *Journal of Oral Rehabilitation* **23(3)** 210-218.
19. Taubock TT, Tarle Z, Marovic D, & Attin T (2015) Pre-heating of high-viscosity bulk-fill resin composites: Effects on shrinkage force and monomer conversion *Journal of Dentistry* **43(11)** 1358-1364.
20. Miletic V, & Santini A (2012) Micro-Raman spectroscopic analysis of the degree of conversion of composite resins containing different initiators cured by polywave or monowave LED units *Journal of Dentistry* **40(2)** 106-113.
21. Harlow JE, Sullivan B, Shortall AC, Labrie D, & Price RB (2016) Characterizing the output settings of dental curing lights *Journal of Dentistry* **44** 20-26.
22. Ilie N, & Durner J (2014) Polymerization kinetic calculations in dental composites: A method comparison analysis *Clinical Oral Investigations* **18(6)** 1587-1596.
23. Emami N, Sjodahl M, & Soderholm KJ (2005) How filler properties, filler fraction, sample thickness and light source affect light attenuation in particulate filled resin composites *Dental Materials* **21(8)** 721-730.
24. Campbell PM, Johnston WM, & O'Brien WJ (1986) Light scattering and gloss of an experimental quartz-filled composite *Journal of Dental Research* **65(6)** 892-894.
25. Abed YA, Sabry HA, & Alrobeigy NA (2015) Degree of conversion and surface hardness of bulk-fill composite versus incremental-fill composite *Tanta Dental Journal* **12(2)** 71-80.
26. Ivoclar Vivadent AGS, Liechtenstein (2015) Report No. 20; Retrieved online June 10, 2016 from: http://downloads.ivoclarvivadent.com/zoolu-website/media/document/33541
27. Shortall AC, Palin WM, & Burtscher P (2008) Refractive index mismatch and monomer reactivity influence composite curing depth *Journal of Dental Research* **87(1)** 84-88.
28. Andrzejewska E (2001) Photopolymerization kinetics of multifunctional monomers *Progress in Polymer Science* **26(4)** 605-665.

29. Par M, Gamulin O, Marovic D, Klaric E, & Tarle Z (2014) Effect of temperature on post-cure polymerization of bulk-fill composites *Journal of Dentistry* **42(10)** 1255-1260.

30. Par M, Gamulin O, Marovic D, Klaric E, & Tarle Z (2015) Raman spectroscopic assessment of degree of conversion of bulk-fill resin composites—Changes at 24 hours post cure *Operative Dentistry* **40(3)** E92-E101.

31. Par M, Lapas-Barisic M, Gamulin O, Panduric V, Spanovic N, & Tarle Z (2016) Long term degree of conversion of two bulk-fill composites *Acta Stomatologica Croatica* **50(4)** 292-300.

32. Dickens SH, Stansbury JW, Choi KM, & Floyd CJE (2003) Photopolymerization kinetics of methacrylate dental resins *Macromolecules* **36(16)** 6043-6053.

33. Emami N, & Söderholm 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.

34. Stansbury JW (2000) Curing dental resins and composites by photopolymerization *Journal of Esthetic Dentistry* **12(6)** 300-308.

35. Ferracane JL (2011) Resin composite—State of the art *Dental Materials* **27(1)** 29-38.

36. Ivoclar Vivadent AGS, Liechtenstein (2015) Bluephase Style Family—Product brochure; Retrieved online June 10, 2016 from: http://downloads.ivoclarvivadent.com/zoolu-website/media/document/12799/Bluephase+Family

37. Price RB, Whalen JM, Price TB, Felix CM, & Fahey J (2011) The effect of specimen temperature on the polymerization of a resin-composite *Dental Materials* **27(10)** 983-989.

38. Jandt KD, & Mills RW (2013) A brief history of LED photopolymerization *Dental Materials* **29(6)** 605-617.

39. Miletic V, Pongprueksa P, De Munck J, Brooks NR, & Van Meerbeek B (2017) Curing characteristics of flowable and sculptable bulk-fill composites *Clinical Oral Investigations* **21(4)** 1201-1212.

40. Ilie N, & Stark K (2015) Effect of different curing protocols on the mechanical properties of low-viscosity bulk-fill composites *Clinical Oral Investigations* **19(2)** 271-279.

41. Rueggeberg FA (2011) State-of-the-art: Dental photocuring—A review *Dental Materials* **27(1)** 39-52.

©Operative Dentistry, 2018, **43-1,** 32-36

# Replacement of a Missing Maxillary Central Incisor Using a Direct Fiber-Reinforced Fixed Dental Prosthesis: A Case Report

MF Romero • FJ Haddock • WW Brackett

### Clinical Relevance

Fiber-reinforced fixed dental prosthesis using the direct restorative technique may be accomplished with ideal contours and tooth morphology when a proper material selection and a step-by-step protocol is followed. This option becomes useful as an interim restoration in many clinical situations.


## SUMMARY

**The use of the direct fiber-reinforced fixed dental prosthesis (FDP) restorative technique presented in this article will result in an ideal restoration considering both esthetics and function in a single appointment. Although indirect techniques are available and may be used, they are time-consuming, resulting in higher cost; therefore, a simplified approach combining a prebonded fiber-reinforced mesh with a sculptable micro-hybrid composite will deliver an acceptable esthetic result with proper function.**



*Mario F. Romero, DDS, Augusta University, Restorative Sciences, Augusta, GA, USA

Fernando J. Haddock, DDS, Augusta University, Restorative Sciences, Augusta, GA, USA

William W. Brackett, DDS, MSD, Augusta University, Restorative Sciences, Augusta, GA, USA

*Corresponding author: 1120 15th St GC 4328, Augusta, GA 30912, USA; e-mail: maromero@augusta.edu


DOI: 10.2341/16-279-L

## INTRODUCTION

Replacing a single missing tooth in the anterior area is a challenge for the clinician, but a number of restorative options are available. Implant-supported crowns are ideal for mature patients with sufficient alveolar bone, while resin-bonded fixed dental prostheses (FDPs) offer more conservative preparation of abutment teeth than conventional FDPs.[1] However, these options are all costly, and for any indirect FDP, the clinician must deal with either the opacity of a metal framework or with adhesive bonding of lab-processed resin or ceramic.

For patients of limited means, a direct fiber-reinforced composite (FRC) FDP can also be a good alternative. Advantages of this option are minimal or no tooth preparation; optimum adhesion of the enamel-bonded resin composite to the prosthesis, since both have a reactive surface; ease of customizing the esthetics of the restoration; reparability of the restoration if needed; and enhanced strength of resin connectors due to the presence of glass fibers.[2] According to Jiang and others,[3] FRC FDPs are also a good alternative to replace up to three lost anterior teeth with compromised periodontal support on

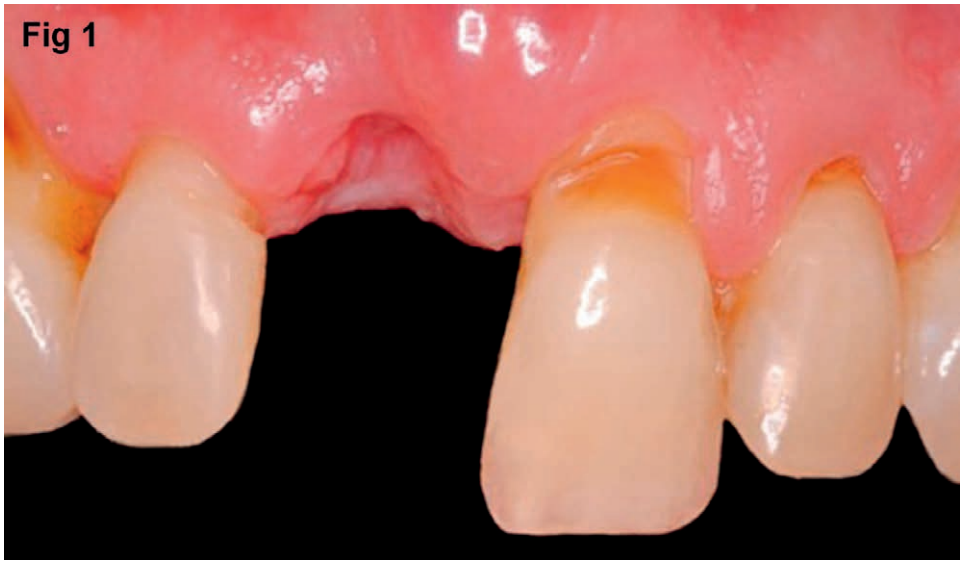



Figure 1. *Preoperative view.*

adjacent teeth, and they reported a survival rate of 89.7% in a four-year clinical study. FRC FDPs can also be made using a denture tooth or the crown of the lost tooth.[4-7] For a resin composite pontic, layering techniques used for direct restorations of teeth can be adapted to produce optimum esthetics. A recent study by Malmstrom and others[8] has concluded that after two years, the cumulative success rate of FRC FDPs was 84.32%, while the survival rate was 92.7%, making this alternative a good, conservative, and cost-effective treatment option.

In this two-year clinical report, the authors describe the use of a direct resin composite FDP, reinforced with polyethylene fibers, for replacement of a maxillary central incisor.

## CASE REPORT

A 57-year-old man was referred to the general dentistry clinic at Eastman Institute for Oral Health after having tooth No. 8 extracted following trauma. He stated at the initial appointment that he had economic concerns and was seeking a "temporary" solution for his problem. After determining from his medical history that he was American Society of Anesthesiologists class I, clinical examination revealed the absence of tooth number 8 with healing gingival tissue, clinical attachment loss on teeth Nos. 7 and 10, and a noncarious cervical lesion on tooth No. 9 (Figure 1), which was considered indicative of occlusal trauma on protrusion. Response to vitality testing of the remaining incisors was normal. Periodontal examination revealed no bleeding on probing and normal sulcular depths, although considerable bone had been lost in the edentulous space of No. 8 due to the tooth's having been traumatized nearly to the point of avulsion. All prosthetic restorative options were presented to the patient, who chose the FRC FDP approach. Benefits and drawbacks of this treatment were discussed.

Local anesthesia was established via infiltration with articaine HCL 4% with epinephrine 1:100,000 (Septocaine, Septodont, Lancaster, PA, USA). The shade needed for the pontic, based on comparison with the adjacent teeth, was determined to be A3. Isolation was achieved with cotton rolls and saliva ejector. Restoration began with minor preparation of the noncarious cervical lesion to remove discolored dentin on tooth No. 9, using a high-speed handpiece and a No. 2 round carbide bur (SS White, Lakewood, NJ, USA) under air/water cooling. Two pieces of prebonded everStickC&B (GC America, Alsip, IL, USA) fiber-reinforced mesh were cut to a length that spanned the edentulous space. Teeth Nos. 7 and 9 were cleaned with Pumice Preppies (Whipmix Corp, Louisville, KY, USA), and facial, lingual, and proximal surfaces were etched for 30 seconds with 32% phosphoric acid (Uni-Etch, BISCO, Schaumburg, IL, USA), followed by rinsing and drying. This was followed by the application of a one-step dental adhesive system, OptiBond Solo, (Kerr Corp, Orange, CA, USA) to all etched areas and light-curing of the adhesive for 20 seconds with an LED light (Valo, Ultradent Products, South Jordan, UT, USA). Restorative procedures for the NCCL involved use of an A3 shade sculptable resin composite (Tetric Ceram, Ivoclar-Vivadent, Amherst, NY, USA) placed in two increments of 2-mm thickness, which were cured independently for 20 seconds each. Fabrication

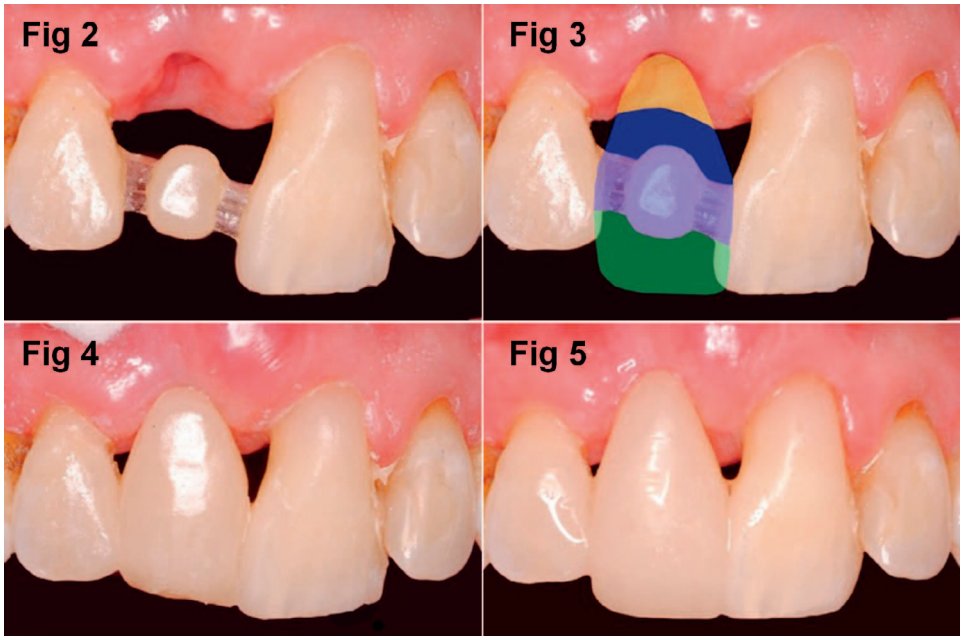



Figure 2. *Completed framework and "button shape" composite increment in place to start fabrication of composite pontic.*
Figure 3. *Color representation of the increments needed to complete the pontic.*
Figure 4. *Pontic after completion of modified ridge lap.*
Figure 5. *Completed restoration.*

of the FRC FDP was begun by bonding of one piece of the fiber mesh from the midlingual of tooth No. 7 to the midlingual of tooth No. 9. This was done using a small amount of A3 flowable resin composite (Tetric Flow, Ivoclar-Vivident) placed on the ends of the mesh. The mesh was then seated against the lingual surface of each abutment tooth and each end cured for 20 seconds. To avoid occlusion on the mesh, clearance of at least 1 mm between the mesh and opposing teeth was verified visually. A second shorter piece of mesh was bonded in the same manner to the facial aspect of the first and to the proximal surfaces of teeth Nos. 7 and 9. The two pieces of mesh were then joined and construction of the pontic initiated with a button-shaped increment of the shade A3 sculptable resin composite (Figure 2). It had been previously determined, since abutment teeth were monochromatic, that no resin layering was needed for the pontic. Gingival and incisal increments were added according to the design depicted in Figure 3. A modified ridge lap pontic design was chosen (Figure 4), and the pontic was restored to match the incisal edge and overall incisal length of tooth No. 9 (Figure 5).

Finishing and contouring were accomplished with an FSD4 diamond (Komet USA, Rock Hill, SC, USA), aluminum oxide finishing strips (Sof-Lex, 3M-ESPE, St Paul, MN, USA), and a No. 12 surgical blade. Occlusion was checked using articulating paper (AccuFilm II Parkell, Edgewood, NY, USA) and adjusted using a football-shaped No. 7406 carbide finishing bur (Komet USA). Stable centric contacts were established on the remaining anterior teeth, with light-centric contacts on the pontic but no excursive contacts. Protrusive guidance was distributed evenly among the remaining anterior teeth, thereby reducing the excursive forces applied to tooth No. 9. Polishing of the lingual surface was done with an aluminum oxide Enhance point (Dentsply Caulk, Milford, DE, USA), with all other surfaces polished using aluminum oxide disks (Sof-Lex XT, 3M-ESPE).

The patient was recalled at six-month intervals, with follow-up photographs taken after two years. The prosthesis remained functional and esthetic over this interval, although the patient did fracture the incisal enamel of tooth No. 7 (Figure 6).

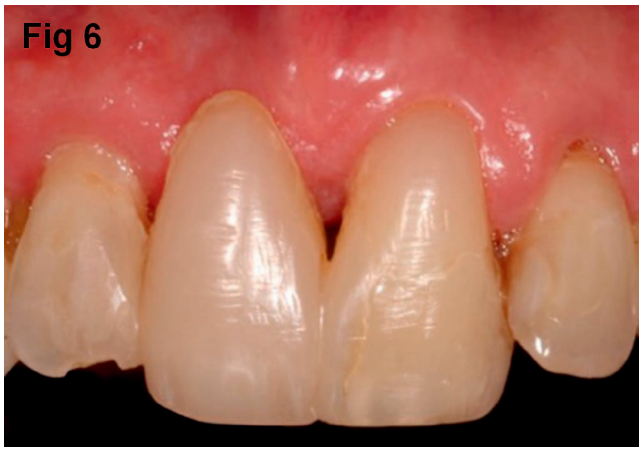



Figure 6. *Two-year follow-up photo.*

## DISCUSSION

The effectiveness of this FDP over two years largely matches the performance of this restoration type previously reported in the literature. To date, there is no evidence in this restoration of the common failure mode of fracture of the connectors, presumably because these have been fiber reinforced. Apparently, the anterior occlusal scheme developed during restoration of this edentulous space has adequately reduced the occlusal trauma that had been previously evident on tooth No. 9. The resin composite restorative material selected effectively mimics the translucency of the abutment teeth and has maintained a smooth, stain-resistant surface, despite its being primarily a posterior material chosen here for its strength and ease of sculpting.

The authors considered an ovate pontic design extended into the residual tooth socket for this restoration but decided that maintenance of the gingival architecture in an area with such bone loss would be unlikely and instead adopted a ridge-lap design. Although this has not yet been necessary, the authors anticipate adding resin to the pontic to retain ridge contact as the ridge resorbs. The FDP was designed to match the patient's adjacent open gingival embrasures, which were not an esthetic concern to him.

Although intended in this case as a definitive restoration, the authors consider this type of prosthesis to be ideal for patients too young to receive an implant, owing to little or no preparation of teeth adjacent to the edentulous space. If the crown of the lost tooth can be retrieved, the authors consider using it as a pontic to be the treatment of choice. The technique for this would be similar to that presented here, except that the lingual surface of the retrieved crown usually must be prepared to allow space for the fiber mesh and encasing resin composite when the fragment is correctly positioned. The authors recommend that for any design, the reinforcing mesh should remain covered with resin composite material following occlusal adjustment, since occlusion on the mesh could weaken it and lessen its reinforcing effect on the FDP connectors.

Fiber reinforcement products are available in two forms, silanated and impregnated with resin by the manufacturer and nonimpregnated, which requires silanation and resin impregnation by the dentist at the time of use. The authors recommend the use of preimpregnated fiber products because they have higher flexural strength and rigidity,[9] they increase the mechanical properties of composite FDPs,[10] and for their ease of use compared with nonimpregnated fiber products. For primarily esthetic reasons, the authors also recommend the technique used for this case of not extending the reinforcement mesh onto the facial surfaces of the abutments, so that it is not necessary to add facial resin to encase the mesh, which could compromise esthetics.

The authors acknowledge that this type of FDP could be fabricated in the laboratory but do not recommend this because of the risk of the resin used to secure the prosthesis to the abutments not adequately copolymerizing with the lab-processed pontic, thereby weakening retention. It is recommended that traumatic occlusion be eliminated on any type of resin-bonded prosthesis, as was done here for tooth No. 9. Given the two years of effective service already rendered by this restoration, the authors anticipate several more years of survival. Finally, the authors wish to point out that the techniques used for this type of restoration, such as direct shade matching or use of a lingual matrix, are very similar to those employed for layered resin composite restorations and will be familiar to any experienced clinician.

### 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 18 June 2017)*

## REFERENCES

1. Piovesan EM, Demarco FF, & Piva E (2006) Fiber-reinforced fixed partial dentures: a preliminary retrospective clinical study *Journal of Applied Oral Science* **14(2)** 100-104.
2. Belli S, & Ozer F (2000) A simple method for single anterior tooth replacement *Journal of Adhesive Dentistry* **2(1)** 67-70.
3. Li J, Jiang T, Lv P, Fang X, Xiao Z, & Jia L (2016) Four-year clinical evaluation of GFRC-RBFPDs as periodontal splints to replace lost anterior teeth *International Journal of Prosthodontics* **29(5)** 522-527.
4. Van Heumen CM, van Dijken J, Tanner J, Pikaar R, Lassila L, Creugers N, Vallitu P, & Kreulen C (2009) Five-year survival of 3-unit fiber-reinforced composite fixed partial dentures in the anterior area *Dental Materials* **25(6)** 820-827.
5. Nixon RL, & Weinstock A (1998) An immediate-extraction anterior single-tooth replacement utilizing fiber-reinforced dual-component bridge *Practical Periodontics and Aesthetic Dentistry* **10(1)** 17-26.

6. Feinman RA, & Smidt A (1997) A combination porcelain/fiber-reinforced composite bridge: a case report. *Practical Periodontics and Aesthetic Dentistry* **9(8)** 925-929.

7. Miller MB (1993) Aesthetic anterior reconstruction using a combined periodontal/restorative approach *Practical Periodontics and Aesthetic Dentistry* **5(8)** 33-40.

8. Malmstrom H, Dellanzo-Savu A, Xiao J, Feng A, Jabeen A, Romero M, Huang J, Ren Y, & Yunker MA (2015) Success, clinical performance and patient satisfaction of a direct fibre-reinforced composite fixed partial dentures: a two-year clinical study. *Journal of Oral Rehabilitation* **42(12)** 906-913.

9. Vallittu P, & Sevelius C (2000) Resin bonded glass fiber-reinforced composite fixed partial dentures: a clinical study *Journal of Prosthetic Dentistry* **84(4)** 413-418.

10. BaSaran E, Ayna E, Uctasli S, Vallittu P, & Lassila L (2013) Load-bearing capacity of fiber reinforced fixed composite bridges *Acta Odontologica Scandinavica* **71(1)** 65-71.

©Operative Dentistry, 2018, **43-1,** 37-52

# Does Finishing and Polishing of Restorative Materials Affect Bacterial Adhesion and Biofilm Formation? A Systematic Review

DAM Dutra • GKR Pereira • KZ Kantorski • LF Valandro • FB Zanatta



### Clinical Relevance

A polished/smooth surface is mandatory for maintaining clinical health of restored teeth. However, this review depicts the absence of reliable data that characterize and elucidate the mechanism related to the effect of surface properties on bacterial adhesion/biofilm formation.


## SUMMARY

**Biofilm (bacterial plaque) accumulation on the surface of restorative materials favors the occurrence of secondary caries and periodontal inflammation. Surface characteristics of restorations can be modified by finishing and/or polishing procedures and may affect bacterial adhesion. The aim of this systematic review was to characterize how finishing and polishing methods affect the surface properties of different restorative materials with regard to bacterial adhesion and biofilm formation. Searches were carried out in MEDLINE-PubMed, EMBASE, Cochrane-CENTRAL, and LILACS databases. From 2882 potential articles found in the initial searches, only 18 met the eligible criteria and were included in this review (12 with *in vitro* design, four with *in situ* design, and two clinical trials). However, they presented high heterogeneity regarding materials considered and methodology for evaluating the desired outcome. Risk bias analysis showed that only two studies presented low risk (whereas 11 showed high and five showed medium risk). Thus, only descriptive analyses considering study design, materials,**



Danilo Antonio Milbradt Dutra, DDS, MSc, PhD, Post-Graduate Program in Oral Science, Periodontology Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, Brazil

Gabriel KR Pereira, DDS, MSc, PhD, assistant professor, MSc Post-Graduate Program in Dentistry, School of Dentistry, Meridional Faculty – IMED, Passo Fundo, RS, Brazil

Karla Zanini Kantorski, DDS, MSc, PhD, associate professor, Post-Graduate Program in Oral Science, Periodontology Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, RS, Brazil

*Luiz Felipe Valandro (LF Valandro), DDS, MSc, PhD, associate professor, Post-Graduate Program in Oral Science, Prosthodontics Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, RS, Brazil

Fabrício Batistin Zanatta, DDS, MSc, PhD, associate professor, Post-Graduate Program in Oral Science, Periodontology Unit, Faculty of Odontology, Federal University of Santa Maria, Santa Maria, RS, Brazil

*Corresponding author: R. Floriano Peixoto 1184, Santa Maria, Rio Grande do Sul State (RS), 97015-372, Brazil; e-mail lfvalandro@hotmail.com


DOI: 10.2341/17-073-L

**intervention (finishing/polishing), surface characteristics (roughness and surface free energy), and protocol for biofilm formation (bacterial adhesion) could be performed. Some conclusions could be drawn: the impact of roughness on bacterial adhesion seems to be related not to a roughness threshold (as previously believed) but rather to a range, the range of surface roughness among different polishing methods is wide and material dependent, finishing invariably creates a rougher surface and should always be followed by a polishing method, each dental material requires its own treatment modality to obtain and maintain as smooth a surface as possible, and *in vitro* designs do not seem to be powerful tools to draw relevant conclusions, so *in vivo* and *in situ* designs become strongly recommended.**

## INTRODUCTION

Biofilm (bacterial plaque) accumulation on the surface of restorative materials favors the occurrence of secondary caries and periodontal inflammation,[1] which is an important aspect related to the longevity of restorations. Greater bacterial adhesion to dental abutments also favors the development of peri-implant diseases,[2] especially in individuals who are susceptible to periodontal disease. Therefore, restorative materials with low susceptibility to bacterial adhesion are desirable.

*In vivo* and *in vitro* studies evaluating microbial adhesion to restorative materials have shown differences in biofilm formation.[3-5] The variation in microbial adherence among different materials is related to the properties of the material, such as chemical composition and its surface characteristics.[6-8] Substrates with high surface free energy (SFE; ie, hydrophilic surface) exhibit more biofilm than substrates with low SFE (hydrophobic). Moreover, rough surfaces provide niches in which microorganisms are protected from brushing, muscle action, and salivary flow. While both SFE and roughness influence microbial adherence and the formation of biofilm, roughness seems to be more important to the accumulation and composition of biofilm, whereas the impact of SFE is greater when comparing surfaces with a similar pattern of roughness.[9]

From a clinical standpoint, dentists sometimes need to carry out clinical adjustments of the restoration (eg, occlusal adjustments, contouring of the restoration or cementation areas) with the use of finishing procedures. The aim of finishing is to obtain the desired anatomic shape and adaptation by contouring the restoration (eg, emergence profile, restoration marginal fit). Such adjustments are usually performed with fine-grained diamond rotary cutting instruments that break the polished layer and modify the surface characteristics of the restoration, changing the surface topography and causing an increase in surface roughness.[6]

A poorly finished restoration can therefore favor the adherence of biofilm to the surface and adjoining areas in the oral cavity. To minimize this effect, several polishing kits are available to eliminate the grooves and achieve a smoother surface (polishing procedures). Sandpaper discs, rubber wheels, and wheels with diamond paste are commonly used. Literature reviews have been conducted to evaluate the impact of these procedures on the surface characteristics of restorations as well as biofilm formation.[7,8]

Therefore, finishing and polishing procedures can modify roughness characteristics of restorations, thereby either promoting or inhibiting/decreasing the formation of biofilm. The aim of the present systematic review was to characterize how these methods affect the surface properties of different restorative materials with regard to bacterial adhesion and biofilm formation.

## METHODS

### Focused Question

This systematic review was conducted to answer the following question: based on clinical, *in vitro* or *in situ* studies, do restorative finishing and/or polishing procedures decrease bacterial adherence to the surface of dental materials?

This study was conducted in accordance with the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA).[10] The protocol is registered with the Prospective Register of Systematic Reviews (PROSPERO: CRD42016036234).

### Search Strategy

Four Internet sources were searched for eligible articles published by November 4, 2016: the MEDLINE-PubMed, EMBASE, Cochrane-CENTRAL, and LILACS databases. The structured search was performed using a combination of controlled vocabulary and key words (Table 1), and a similar search strategy was adapted for the other databases. Searches for relevant ongoing trials from the US clinical trials register (http://www.clinicaltrials.gov) and grey literature (OpenGrey repository) were also

| Table 1: *Search Keyword: <Intervention AND Control AND Outcome>* |
|---|
| Intervention: surface treatment (472,425 titles—07/03) |
| #1 Population: dental restorations |
| *[MeSH Terms]:* "Dental Prosthesis" OR "Dental Restoration, Permanent" OR "Crowns" OR "Dental Abutments" OR "Ceramics" OR "Metal Ceramic Alloys" OR "Dental Porcelain" OR "Dental Materials" OR "Composite Resins" OR "Compomers" OR "Glass Ionomer Cements" OR "Dental Amalgam"<br>*[text/words]:* dental crown OR dental crowns OR dental restoration OR dental restorations OR dental filling OR dental ceramic OR metal ceramic alloys OR porcelain-metal alloys OR metallo ceramic alloys OR metalloceramic OR metal ceramic restorations OR dental porcelain OR dental material OR composite OR resin OR resin composite OR compomers OR glass-ionomer OR dental amalgam OR abutments OR lithium disilicate OR lithium dislocate glass-ceramic OR feldspathic ceramic OR feldspathic porcelain OR feldspathic veneers OR glass ceramic OR porcelain OR alumina OR alumina ceramic OR alumina zirconia OR zirconia OR Y-TZP or zirconium |
| #2 Intervention: finishing/polishing (299,283 titles) |
| *[MeSH Terms]:* "dental polishing" OR "Prosthesis Fitting" OR "Restore polishing paste" [Supplementary Concept]<br>*[text/words]:* prosthesis fitting OR prosthesis adjustment OR grinding OR gross OR glaze OR texture OR abrasive OR abrasives OR polish* OR finish* OR burnish* |
| #3 Outcome: Dental Plaque OR Gingival parameters |
| *[MeSH Terms]:* "biofilms" OR "dental plaque" OR "dental plaque index" OR "dental plaque indexes" OR "dental plaque indices" OR "bacterial adhesion" |

performed. Manual searches of all references of the selected studies were performed in an attempt to find further relevant reports.

### Screening and Study Selection

Two reviewers (DAMD and GKRP) independently screened the articles. Study selection was performed in two steps: 1) evaluation of title and abstract and 2) full-text analysis. Titles and abstracts were evaluated for the preselection of *in vitro*, *in situ*, or clinical studies published in the English language that evaluated bacterial adhesion to the surface of dental restorative materials. The reviewers then performed the full-text analysis of the selected studies using the following inclusion criteria:

- Intervention: Finishing/polishing procedures on the surface of dental restorative materials.
- Comparison: Unmodified or treated surfaces with the same material as the intervention group. Thus, studies that evaluated only finishing/polishing procedures among different restorative materials were not considered eligible.
- Quantitative assessment of bacterial adhesion to the surface of restorations.
- Surface characteristics (eg, roughness, free energy) determined using profilometry, scanning electron microscopy, or atomic force microscopy.

Articles that fulfilled all selection criteria were considered eligible for this investigation and submitted to the data extraction process. The concordance between the reviewers for full-text analysis was statistically assessed showing a 0.9 kappa score. Divergences between the reviewers were discussed and resolved by consensus. If a disagreement persisted, the judgment of a third reviewer (FBZ) was decisive.

### Data Collection

Both reviewers independently collected the following data from eligible studies: study identification (authors, year of publication, country in which study was conducted), study design, description of methods (restorative material evaluated and description of finishing/polishing procedures), and type of biofilm formation (eg, type of microorganism). The main results and conclusions of the studies were recorded. For cases in which the article did not provide enough data for inclusion in the analysis, the first or corresponding authors were contacted to determine whether additional data could be provided. If contact with the authors was not achieved after three attempts, the article was excluded.

### Risk of Bias (Quality Assessment)

The quality assessment of the selected studies was adapted from previous investigations.[11,12] The evaluation of the risk of bias involved the use of a chart considering the following aspects for each study design: description of sample size calculation, randomization of the sample, untreated control group, materials used according to the manufacturer's instructions, description of finishing/polishing standardization, blinding of the examiner of the outcome, and repetition of biofilm experiment (*in vitro*). If the authors reported the parameter, the article had a Y (yes) on that specific parameter; if it was not possible to find the information, the article received an N (no). Articles that reported zero to two items were

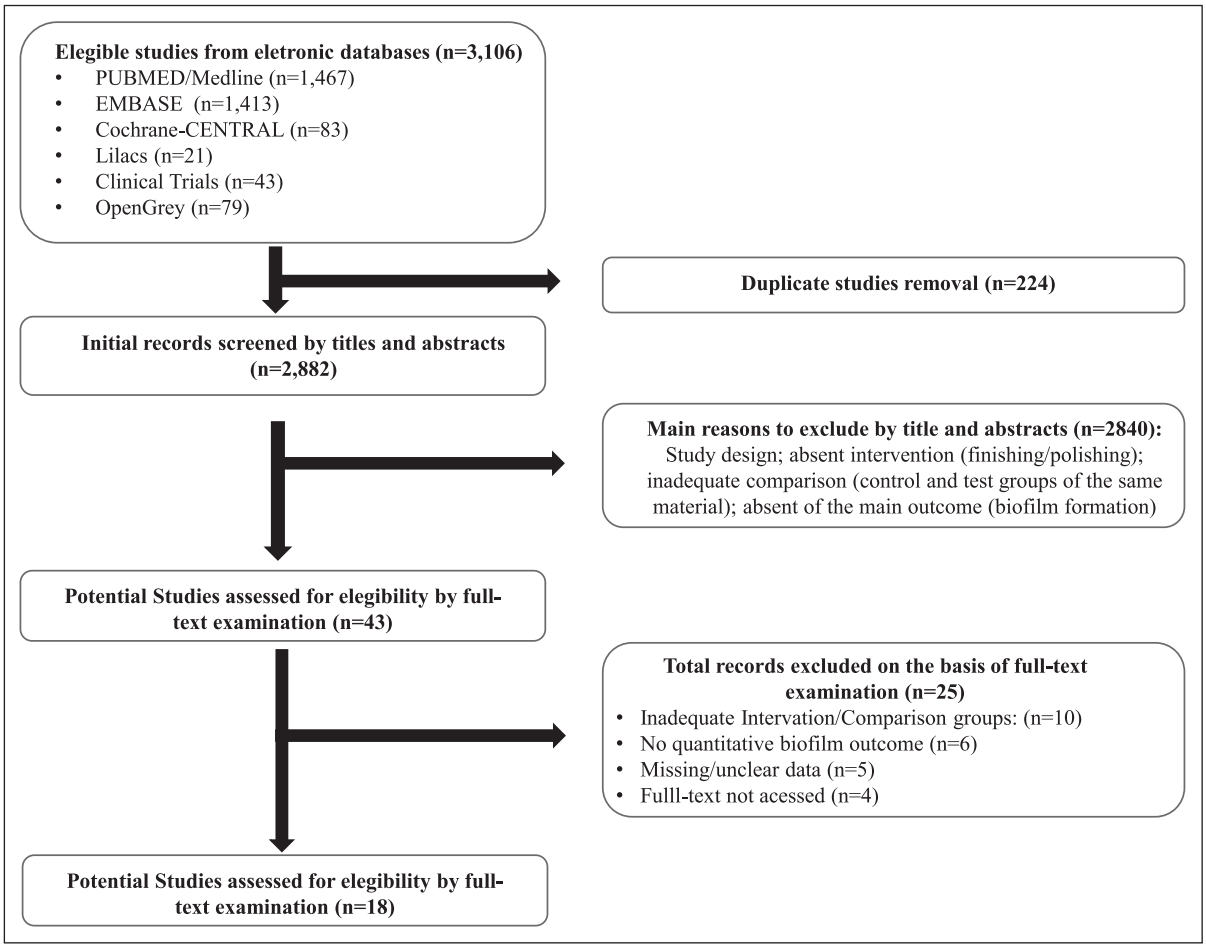



Fig. 1. *Selection of studies for systematic review*

classified as at high risk of bias, three or four as medium risk, and five to seven as low risk.

### Data Analysis

Due to the considerable variability in the methodologies used to evaluate the effect of the finishing/polishing of different restorative materials on biofilm formation and the consequent heterogeneity of the results, meta-analysis was not possible. Thus, descriptive analysis was performed considering study design, materials, intervention on the surface of the restoration, surface characteristics (roughness and SFE), and the evaluation protocol for biofilm formation and bacterial adhesion.

## RESULTS

Figure 1 shows the flowchart of the systematic review as well as the reasons for the exclusion of studies. A total of 2882 articles were found in the initial searches of the electronic databases. After reading the titles and abstracts, 2840 were excluded, and 42 articles were submitted to full-text analysis. The manual search yielded no additional studies, and one study was selected by direct contact with the authors. Among the 43 articles, 18 met the eligible criteria and were included in the review. Table 2 displays the characteristics of the studies selected.

All studies evaluated the effect of finishing/polishing methods on the surface properties of restorative materials as well as the impact on bacterial adhesion and biofilm formation. Most studies were developed using an *in vitro* design[2,13-23] (n=12), four studies used an *in situ* model,[5,24-26] and two studies were clinical trials.[27,28]

Different materials were evaluated in the studies analyzed. Eighteen experimental groups were used to test direct and indirect resin composites.[14,16-20,22,26] Four experimental groups were used to evaluate glass ionomer cements.[14,22,26] Five experimental groups were used to evaluate dental ceramics (four feldspar ceramic,[5,13,19,25] and one Y-

TZP ceramic[23]), and six experimental groups were used to evaluate titanium samples.[2,15,21,24,27,28]

The studies demonstrated considerable variability regarding the biofilm formation model. Two clinical studies evaluated supragingival and subgingival biofilm formation.[27,28] Four studies used natural human biofilm formed on dental appliances *in situ*.[5,24,25] Synthesized biofilm was used in 11 studies,[2,13-22] and biofilm was cultivated from human saliva in one study.[23] Moreover, different methods were used to quantify biofilm formation, such as the percentage of area covered,[2,15,22,25,26,28] total counts of colony-forming units,[13,18,19,23,27] counts per minute,[14] hemocytometer,[29] optical density,[16,17] and the quantification of viable biomass and biovolume.[5,20,24]

Table 3 displays the descriptive analyses of the effect of finishing/polishing methods on the surface roughness of different materials and the impact on bacterial adhesion. A wide variety was found regarding the finishing or polishing method for each material evaluated, with varied results. In all 13 experimental groups evaluating a finishing method, an increase in surface roughness was found in comparison to polished and control groups.[5,17,23,26,28,30] Fifty experimental polishing groups were evaluated and exhibited a tendency toward a smoother surface compared to the control.[2,6,13-18,20,22-26,29,30] However, some studies found no difference between polishing and control groups.[2,13,14,18,20,26,27,30] The impact of roughness on bacterial adhesion seems to be related not to a roughness threshold but rather to a range.

In the analysis of the risk of bias (Table 4), 11 studies presented high risk,[13-18,20,22,28,30] five studies presented medium risk,[2,5,24,25,29] and two studies presented low risk.[23,27] The main aspects related to a higher risk of bias were the description of sample size calculation, randomization of the samples, and the blinding of the operator.

## DISCUSSION

The present systematic review offers a summary of data regarding the effect of finishing and polishing methods on different materials as well as the impact on bacterial adhesion and biofilm formation. Several restorative materials and finishing/polishing methods have been evaluated and exhibit different degrees of surface roughness. There was a tendency for polishing protocols to produce a similar pattern of surface roughness in comparison to untreated or glazed (control) surfaces, whereas finishing methods seem to increase the surface roughness significantly. The impact of surface roughness on bacterial adhesion differs depending on the type of material, study design, and range of surface roughness but does not seem to be strongly related to a preestablished roughness threshold.

### Effect of Finishing and Polishing on Surface Roughness

The studies included in the present systematic review tested a large variety of finishing and polishing methods, including diamond finishing burs, abrasive paper discs, silicon carbide (SiC) and silicone points, abrasive-impregnated rubber, felt wheels, and polishing pastes. The effect of each method on the restoration surface is reported to be material dependent, and its effectiveness is mainly system dependent.[31] Thus, the effect of finishing and polishing systems in each material was explored individually as follows.

*Resin Composite*—Eight studies evaluated the surface of resin composites.[14,16-18,20,22,26,30] Only one study[14] tested an untreated surface (Ra=0.15 μm) as the control group. In two other studies,[20,26] the control group was a resin composite compressed against a Mylar matrix to create a smooth surface (Ra values up to 0.2 μm). When finishing and polishing groups were considered,[17,26] finishing by grinding with diamond burs was found to promote a drastic increase in surface roughness, with Ra values ranging from 2.0 μm (Grandio, Voco)[17] to 4.5 μm (Grandio, Voco).[26] In the study conducted by Ono and others,[17] polishing was performed using a diamond paste that reduced surface irregularities, with Ra values of approximately 0.2 μm. In the study by Perez,[26] polishing was performed with the use of a BisCove resin polisher (Bisco), leading to a decrease in surface roughness, with Ra values up to 0.43 μm.

Most studies compared two different polishing protocols. When polishing was performed with SiC sandpaper, the surface roughness pattern was directly related to grit size, and the range of Ra values was varied among studies. Dezelic and others[18] found the smoothest surface using a sequence of 1200-grit, 2400-grit, and 4000-grit SiC sandpaper (Ra values of 0.04 μm) compared to 320-grit SiC sandpaper (Ra value of 0.5; Tetric, Ivoclar; and Ra value of 0.6; Tetric Flow, Ivoclar).[18] Carlén and others[14] compared a 1000-grit SiC sandpaper to an untreated surface and found a rougher surface in the test group (Ra=0.5 μm vs 0.15 μm). Yuan and others[22] reported similar results using a 1200-grit wet abrasive sandpaper (Z250 and Z350, 3M ESPE;

Table 2: *Summary of the Description of the Included Studies*

| Study Design | Author | Material | Intervention | Sample/Biofilm | Bacterial Adhesion Outcome |
|---|---|---|---|---|---|
| *Clinical* | | | | | |
| | Elter and others[28] | ▪ Titanium abutments (Nobel Biocare, Kloten, Switzerland) | ▪ Control: untreated surface<br>▪ Finishing: grinding with a smooth diamond bur | Supra- and subgingival natural human biofilm adhered to the abutments surface (n=15) | Percentage of biofilm covering the abutment surface analyzed by SEM |
| | Quirynen and others[9] | ▪ Titanium (Nobelpharma AB, Göteborg, Sweden) | ▪ Control: untreated<br>▪ Polishing (machined): machine polished using diamond material<br>▪ Polishing (manual): manually polished using diamond material | Supra- and subgingival natural human biofilm adhered to the abutments surfaces (six volunteers) | Mean CFUs of supra- and subgingival biofilm formation around the abutment surface |
| *In situ* | | | | | |
| | Haralur[25] | ▪ Porcelain (Vita VMK, Vita Zahnfabrik, Bad Säckingen, Germany) | ▪ Control: autoglazed and overglazed<br>▪ Polishing (Shofu): Shofu kit<br>▪ Polishing (DFS): DFS kit<br>▪ Polishing (Eve): Eve kit | Natural human biofilm adhered to the samples using oral appliances *in situ* (12 volunteers) | Percentage of biofilm covering the surface area calculated by placing the OHP graph sheet on the test specimen |
| | Brentel and others[5] | ▪ Feldspar ceramic (VM7, Vita Zahnfabrik) | ▪ Control: glazed surface<br>▪ Finishing (grind): coarse diamond bur<br>▪ F&P (1): coarse diamond bur + silicon rubber tips<br>▪ F&P (2): coarse diamond bur + silicon rubber tips + felt disk impregnated with a fine-diamond particle-based paste | Natural human biofilm adhered to the samples using oral appliances *in situ* (10 volunteers) | Mean biovolume analyzed by CLSM and CONSTAT software |
| | Perez[26] | ▪ Glass ionomer (Ionofil plus, Voco, Cuxhaven, Germany)<br>▪ Glass ionomer (Vitremer, 3M ESPE, Seefeld, Germany)<br>▪ Resin composite (Filtek Supreme, 3M ESPE)<br>▪ Resin composite (Grandio, Voco) | ▪ Control: Mylar strips<br>▪ Finishing (grind): extra-fine diamond burs<br>▪ Polishing: BisCover resin polisher (Bisco) | *Streptococcus mutans*<br>Natural human biofilm adhered to the samples using oral appliances *in situ* (one volunteer) | Percentage area coverage by adherent bacteria |
| | Rimondini and others[24] | ▪ Titanium discs | ▪ Polishing (1): grinding paper + diamond paste (3 μm) + suspension of $SiO_2$ (0.04 μm)<br>▪ Polishing (2): grinding paper + diamond paste (6 μm) | Natural human biofilm adhered to the samples using oral appliances *in situ* (eight volunteers) | Total bacteria amount (biomass – optical density) by spectroscopy |
| *In vitro* | | | | | |
| | Dutra and others[23] | ▪ Y-TZP (In-Ceram YZ, Vita Zahnfabrik) | ▪ Control: untreated<br>▪ Finishing: grinding with a fine diamond bur<br>▪ Finishing: grinding with a coarse diamond bur | Polymicrobial biofilm formed from human saliva (single donor) | Mean CFU of biofilm formation |
| | Yuan and others[22] | ▪ Nanoparticle resin composite (Filtek Z350, 3M ESPE)<br>▪ Nano-hybrid resin composite (Filtek Z250 XT, 3M ESPE)<br>▪ Low-shrink resin composite (Filtek P90, 3M ESPE)<br>▪ Polymer-based pre-reacted glass ionomer (Beautifil II, Shofu, Kyoto, Japan) | ▪ Polished: polished with 11-μm grit (1200-grain wet abrasive paper disc)<br>▪ Polished: polished with nano–silicon dioxide fabric | *S. mutans*<br>Single-species synthesized biofilm | Area of bacterial adhesion (A%) by CLSM images |

Table 2: *Summary of the Description of the Included Studies (cont.)*

| Study Design | Author | Material | Intervention | Sample/Biofilm | Bacterial Adhesion Outcome |
|---|---|---|---|---|---|
| | Li and others[21] | ▪ Titanium (Chinese National, GB/T 3623-1998) | ▪ Control: untreated surface<br>▪ Polishing (manual): manual polishing (carborundum point + silicon points + hard rubber wheel)<br>▪ Polishing (electrolytic): electrolytic polishing<br>▪ Polishing (centrifugal): centrifugal mill polishing | *Candida albicans* Single-species synthesized biofilm | Total counts by a hemocytometer under the objective (40×) of an optical microscope |
| | Ionescu and others[20] | ▪ Resin composite (Filtek Supreme XT, 3M ESPE)<br>▪ Resin composite (Filtek Silorano, 3M ESPE) | ▪ Control: Mylar strips<br>▪ Polishing (4000 Sic): 1000- + 4000-grit SiC paper | *S, mutans* Single-species synthesized biofilm | Viable biomass assessment by MTT assay |
| | Aykent and others[19] | ▪ Direct resin composite (Tetric Evo-Ceram, Ivoclar-Vivadent, Schaan, Liechtenstein)<br>▪ Indirect resin composite Estenia (KL 100; Kuraray, Kurashiki, Japan)<br>▪ Indirect resin composite (SR Adoro, Ivoclar-Vivadent)<br>▪ Feldspar ceramic (VITABLOCS Mark II, Vita Zahnfabrik) | ▪ Finishing (grind): fine (46 μm) and extra-fine (25 μm) diamond rotatory cutting instruments<br>▪ Polishing (Sof-Lex): sequence of three sandpaper discs (Sof-Lex coarse: 100 μm; medium: 29 μm; fine: 14 μm)<br>▪ Polishing (diamond paste): felt wheel with diamond paste | *S. mutans* Single-species synthesized biofilm | Total counts of vital adhered bacteria analyzed by CLSM |
| | | | ▪ F&P: finished with a white stone + polished with a sequence of 3-SiC rubber points (Ceramiste Standard: 48 μm; Ultra: 28 μm; Ultra II: 6.3 μm) | | |
| | Barbour and others[2] | ▪ Standard titanium abutments (Nobel Biocare) | ▪ Control: untreated surface<br>▪ Polishing: fine diamond grit rotary bur + green carborundum stone rotary point + brown impregnated silicon rubber point + green impregnated silicon rubber point + cloth mop and polishing compound containing amorphous silica/silicon carbide<br>▪ Polishing: brown impregnated silicon rubber point + green impregnated silicon rubber point + cloth mop and polishing compound containing amorphous silica/silicon carbide | *S. mutans* Single-species synthesized biofilm | Median values of percentage coverage area analyzed by AFM |
| | Dezelic and others[18] | ▪ Resin composite (Tetric, Ivoclar-Vivadent)<br>▪ Flowable resin composite (Tetric Flow, Ivoclar-Vivadent)<br>▪ Unfilled resin composite (Heliobond, Ivoclar-Vivadent) | ▪ Control (320 SiC): 320-grit SiC sandpaper.<br>▪ Polishing (4000 SiC): sequence of 1200-grit, 2400-grit, and 4000- grit SiC sandpaper. | *Actinomyces naeslundii*, *Veillonella dispar*, *Fusobacterium nucleatum*, *Streptococcus sobrinus*, *Streptococcus oralis*, and *C. albicans* Single-species synthesized biofilm | Mean CFUs (log10) of biofilm formation |

Table 2: *Summary of the Description of the Included Studies (cont.)*

| Study Design | Author | Material | Intervention | Sample/Biofilm | Bacterial Adhesion Outcome |
|---|---|---|---|---|---|
| | Ono and others[17] | ▪ Resin composite (Clearfil AP-X, Kuraray)<br>▪ Resin composite (Grandio, Voco) | ▪ Polishing (800 SiC): polished with 800-grit SiC sandpaper<br>▪ Polishing (diamond paste): diamond paste up to 1 μm particle size | *S. mutans* Single-species synthesized biofilm | Total count of bacteria/ $mm^3$ quantified by turbidimetric analysis ($OD_{550nm}$) |
| | Ikeda and others[16] | ▪ Resin composite (Estenia C&B, Kuraray)<br>▪ Resin composite (Gradia, GC America, Alsip, USA) | ▪ Polishing: ground with 800-grit SiC paper<br>▪ Polishing: diamond pastes up to 1 μm | *S. mutans* Single-species synthesized biofilm | Amount of bacteria (optical density) by infrared spectroscopy |
| | Pier-Francesco and others[15] | ▪ Titanium (Goodfellow Cambridge Limited) | ▪ Polishing (brushes): hand polishing with rotary brushes<br>▪ Polishing (machine): Eco Mini dry-polishing machine | *Porphyromonas gingivalis* Single-species synthesized biofilm | Median values of percentage coverage area analyzed by fluorescent microscopy |
| | Carlén and others[14] | ▪ Glass ionomer (KetacFilt Aplicapt; 3M ESPE)<br>▪ Resin composite (TPH Spectrum, Dentsply DeTrey, York, USA) | ▪ Control: untreated surface<br>▪ Polishing (1000 SiC): 1000-grit SiC sandpaper | *S. mutans*, *Streptococcus sanguis*, and *A. naeslundii* Single-species synthesized biofilm | Total number of cells were calculated from the CPM values |
| | Kawai and others[13] | ▪ Porcelain (Vita Celay blanks, A3M-9, Vita Zahnfabrik) | ▪ Control: glazed<br>▪ Polishing (120 EP): 120-grit emery paper<br>▪ Polishing (600 EP): 200-grit emery paper<br>▪ Polishing (diamond paste): felt wheel with diamond paste | *S. sobrinus* Single-species synthesized biofilm | Total counts of adhered cells measured by using a liquid scintillation method |

*Abbreviations: AFM, atomic force microscopy; CFUs, colony-forming units; CLSM, confocal laser scanning microscopy; CPM, count per minute; OHP, overhead projection; SEM, scanning electric microscopy.*

Ra=0.4 μm; Filtek P90, 3M ESPE; Ra=0.5 μm); the authors also tested a polishing method using a nano–silicon dioxide fabric (polishing pad) that achieved a smoother surface (Ra=0.02 μm) in comparison to the sandpaper group.[22]

*Glass Ionomer Cement*—Three studies assessed a glass ionomer experimental group.[14,22,26] Perez[26] compared three surface treatment methods: 1) compression against a Mylar matrix (control), 2) finishing with fine-grain diamond points, and 3) polishing with the application of the BisCover resin polisher (Bisco) after finishing. The results showed that finishing led to a significant increase in surface roughness (up to Ra=4.39 μm), whereas polishing reestablished a degree of roughness similar to that in the control group (Ra values=0.2 to 0.8 μm). Carlén and others[14] found that polishing with 1000-grit SiC sandpaper created a rougher surface (Ra=1.05 μm) in comparison to an untreated group (Ra=0.86 μm). Recently, Yuan and others[22] achieved a very smooth surface (Ra=0.03 μm) polishing with a nano–silicon dioxide fabric (polishing pad).

*Ceramics*—Different dental ceramics were evaluated in four studies.[5,13,25] Dutra and others[23] evaluated the effect of finishing by grinding with diamond burs on a Y-TZP surface and found an increase in surface roughness with the increase in bur grit size (up to Ra=1.16 μm) compared to an untreated control group (Ra=0.13 μm). Two studies[5,13] that used a glazed group (Ra=0.5 μm) as control found that no polishing method tested was fully effective at reestablishing the surface roughness pattern of the control group after finishing. Likewise, Aykent and others[19] tested three different polishing methods on feldspar ceramic after finishing (Ra=1.1 μm) and found a surface roughness pattern with the Ra value ranging from 0.6 to 0.9 μm.

*Titanium*—Six studies evaluated titanium samples.[2,15,21,24,27,28] In a clinical trial, Quirynen and others[9] tested machined and manual polishing methods on titanium abutments and found that both methods created a smoother surface (Ra=0.11 and 0.06 μm, respectively) in comparison to the control group (Ra=0.12 μm). In another clinical trial, Elter and others[28] evaluated the effect of a finishing method on implant abutments and found a slightly rougher surface (Ra=0.4 μm) in comparison to the control (Ra=0.2 μm). In an *in situ* study, Rimondini and others[24] evaluated the effect of polishing with grinding paper and diamond paste with and without $SiO_2$ suspension and found very smooth surface

patterns (Ra=0.09 and 0.2 μm, respectively). In an *in vitro* study, Li and others[21] evaluated three different polishing protocols (manual, electrolytic, and centrifugal) and found similar surface roughness patterns (Ra=0.35, 0.19, and 0.18 μm, respectively). Likewise, Pier-Francesco and others[15] compared manual and machined polishing methods and found Ra values of 0.03 and 0.16 μm, respectively.

### Impact of Surface Roughness on Bacterial Adhesion

The data collected in this systematic review showed that finishing and polishing affect the surface roughness and promote a heterogeneous impact to bacterial adhesion considering each material evaluated and the method of evaluation of bacterial adhesion outcome (thickness, covered area, biomass, and colony-forming units).

In general, smoother surfaces are less likely to lead to the formation of biofilm regardless of restorative material and are therefore desirable. Based on the present findings, it may be concluded that 1) finishing procedures when not followed by a polishing system provide greater adhesion and retention of bacteria, 2) some studies showed that polishing successfully reestablished the level of biofilm formation observed on untreated or glazed control groups regardless of whether the same pattern of surface roughness was achieved, and 3) other studies showed significant differences of biofilm formation among polishing groups even when similar patterns of surface roughness were compared.

The impact of finishing and polishing methods to titanium abutments was evaluated in two clinical studies included in this review.[27,28] Quirynen and others[9] evaluated the influence of the surface smoothing on supra- and subgingival biofilm formation comparing titanium abutments with different surface roughnesses (untreated, machined, and manually polishing protocols) in six partially edentulous patients. The data showed no significant differences on colony-forming unit counts between the control (Ra=0.2 μm) and polished groups (manual, Ra=0.06 μm; machined, Ra=0.11 μm). These results indicated that a reduction in surface roughness (a roughness less than 0.2 μm) had no major effect on the microbiologic composition either supra- or subgingivally. Based on these observations, the authors suggested an existence of a threshold roughness (Ra=0.2 μm) below which no further impact on the bacterial adhesion and/or colonization should be expected. This threshold roughness has been extensively used in the literature. Later, Elter and others[28] evaluated supra- and subgingival natural human biofilm formation to finishing and untreated titanium abutment surface. Their results showed that finishing the surfaces (Ra=0.4 μm) retained more supragingival biofilm compared to the control (Ra=0.2 μm) analyzed using scanning electron microscopy, while no differences were observed in the subgingival biofilm. These results corroborated the threshold roughness, especially when supragingival biofilm was considered. The greater impact of roughness on supra- than on subgingival biofilm may be explained because the clinical impact of surface roughness becomes especially important when larger shear forces are active.[32]

In agreement with the previous studies, Rimondini and others[24] evaluated the surface roughness necessary to reduce early (24 hours) *in vivo* biofilm colonization on titanium disks assigned to different polishing groups. The results showed no significant differences in bacteria biomass among the polishing groups below the threshold roughness. All other *in situ* studies included in this systematic review compared finished and polished surfaces with roughness above the threshold roughness.[5,25,276] Brentel and others[5] assessed the *in situ* biofilm formation on feldspar ceramic (VM7, Vita). The biomass assessment showed greater bacterial adhesion when the ceramic surface was ground (finished) only by diamond burs (Ra=2.0 μm) compared to the glazed group (Ra=0.5 μm). On the other hand, when the feldspar ceramic was polished after being ground (F&P (2) Ra=0.8 μm), it successfully reestablished the bacterial adhesion level to the control groups even with a slightly rougher surface. Controversially, results were related by Haralur and others[25] evaluating the percentage of covered area by natural biofilm to porcelain (Vita VMK) ceramics. Their results showed that polished groups (Ra=0.6 and 0.9 μm) failed to achieve a similar percentage of bacteria accumulation compared to the smoother groups (autoglazed: Ra=0.4; overglazed: Ra=0.3 μm).

Perez[26] evaluated *in situ* bacterial adhesion to glass ionomer and resin composite specimens submitted to finishing and polishing protocols. The authors found that the ground surfaces (finishing group) always showed drastically rougher surfaces and presented higher biofilm formation compared to the control group, while polished surfaces presented no differences in the control regarding the biofilm accumulation. Based on the data from these *in situ* studies, it may be stated that mild differences of

Table 3: *Summary of the Results of Roughness and Bacterial Adhesion*

| Author | Material | Intervention | Surface Roughness (μm) | Bacterial Adhesion Outcome | Statement |
|---|---|---|---|---|---|
| Clinical studies | | | | | |
| Elter and others[28] | Titanium (Nobel Biocare) | Control | 0.2 | ▪ Supragingival biofilm: finishing > control<br>▪ Subgingival biofilm: no significant difference | ▪ Finished surfaces (rougher surface) retained more supragingival biofilm compared to the control<br>▪ Regardless of the roughness differences, finishing surfaces did not retained more subgingival biofilm compared to the control |
| | | Finishing (grind) | 0.4 | | |
| Quirynen and others[9] | Titanium (Nobelpharma) | Control | 0.21 | ▪ No significant differences were observed between control and polishing groups | ▪ Roughness values below the threshold of 0.2 μm did not present a significant amount of bacterial adhesion<br>▪ Both the polishing protocols were effective to establish the level of bacterial adhesion observed on the control |
| | | Polishing (machined) | 0.11 | | |
| | | Polishing (manual) | 0.06 | | |
| *In situ* studies | | | | | |
| Haralur[25] | Feldspar ceramic (Vita VMK, Vita Zahnfabrik) | Control (autoglazed) | 0.42 (0.06) | ▪ A significantly smaller percentage of plaque accumulation was found on surfaces of the control groups (auto- and overglazed) than polished surfaces | ▪ All polishing protocols failed to prevent the bacterial adhesion when compared to the control groups |
| | | Control (overglazed) | 0.34 (0.07) | | |
| | | Polishing (Shofu) | 0.62 (0.01) | | |
| | | Polishing (DFS) | 0.91 (0.02) | | |
| Brentel and others[5] | Feldspar ceramic (VM7, Vita Zahnfabrik) | Control (glazed) | 0.53 (0.11) | ▪ Finishing and F&P (1) > control<br>▪ No significant differences were observed between F&P (2) and control | ▪ Differences between 0.53 and 0.88 μm did not result in increased bacterial adhesion<br>▪ F&P (2) protocol successfully reestablished the level observed on the control |
| | | Finishing (grind) | 2.02 (0.12) | | |
| | | F&P (1) | 1.27 (0.14) | | |
| | | F&P (2) | 0.88 (0.11) | | |
| Perez[26] | Glass ionomer (Ionofil plus, Voco) | Control | 0.78 | ▪ Finishing > polishing > control | ▪ The increase of biofilm formation was related to the increase of surface roughness<br>▪ No finishing/ polishing protocol successfully reestablished the level of bacterial adhesion observed on the control |
| | | Polishing | 0.88 | | |
| | | Finishing (grind) | 4.39 | | |
| | Glass ionomer (Vitremer, 3M ESPE) | Control | 0.23 | ▪ Finishing > control<br>▪ Polishing presented no statistical differences to the control | ▪ Differences between 0.2 and 0.8 μm did not result in increased bacterial adhesion<br>▪ Polishing protocol successfully reestablished the level observed on the control |
| | | Polishing | 0.79 | | |
| | | Finishing (grind) | 1.67 | | |
| | Resin composite (Filtek Supreme, 3M ESPE) | Control | 0.19 | ▪ Finishing > control<br>▪ Polishing presented no statistical differences to the control | ▪ Differences between 0.2 and 0.6 μm did not result in increased bacterial adhesion<br>▪ Polishing protocol successfully reestablished the level observed on the control |
| | | Polishing | 0.61 | | |
| | | Finishing (grind) | 3.20 | | |
| | Resin composite (Grandio, Voco) | Control | 0.04 | ▪ Finishing > control<br>▪ Polishing presented no statistical differences to the control | ▪ Differences between 0.04 and 0.4 μm did not result in increased bacterial adhesion<br>▪ Polishing protocol successfully reestablished the level observed on the control |
| | | Polishing | 0.43 (0.07) | | |
| | | Finishing (grind) | 4.54 (0.23) | | |

Table 3: *Summary of the Results of Roughness and Bacterial Adhesion (cont.)*

| Author | Material | Intervention | Surface Roughness (μm) | Bacterial Adhesion Outcome | Statement |
|---|---|---|---|---|---|
| Rimondini and others[24] | Titanium discs | Polishing (1) | 0.09 (0.01) | ▪ No significant differences were observed between polishing (1) and polishing (2) groups | ▪ Roughness values below the threshold of 0.2 μm did not present a significant amount of bacterial adhesion |
| | | Polishing (2) | 0.2 (0.06) | | |
| *In vitro* | | | | | |
| Dutra and others[23] | Y-TZP (In Ceram YZ, Vita Zahnfabrik) | Control | 0.13 (0.02) | ▪ No significant differences were observed between control and finishing groups | ▪ Differences between 0.13 and 1.16 μm did not result in increased bacterial adhesion |
| | | Finishing (fine bur) | 0.70 (0.21) | | |
| | | Finishing (coarse bur) | 1.16 (0.14) | | |
| Yuan and others[22] | Nanoparticle restorative (Filtek Z350, 3M ESPE) | Polished (1200 grit) | 0.44 | ▪ Polished (1200 grit) > polished (nanosilicon) | ▪ Results showed a significant increase in bacterial adhesion and an increase in surface roughness<br>▪ Polishing (nanosilicon) protocol was more effective in preventing bacterial adhesion in comparison to polishing (1200 grit) protocol |
| | | Polished (nanosilicon) | 0.02 | | |
| | Nanohybrid universal restorative (Filtek Z250 XT, 3M ESPE) | Polished (1200 grit) | 0.43 | ▪ Polished (1200 grit) > polished (nanosilicon) | ▪ Results showed a significant increase in bacterial adhesion and an increase in surface roughness<br>▪ Polishing (nano silicon) protocol was more effective in preventing bacterial adhesion in comparison to polishing (1200 grit) protocol |
| | | Polished (nanosilicon) | 0.02 | | |
| | Low-shrink posterior restorative based on siloxane and oxirane (Filtek P90, 3M ESPE) | Polished (1200 grit) | 0.53 | ▪ Polished (1200 grit) > polished (nano silicon) | ▪ Results showed a significant increase in bacterial adhesion and an increase in surface roughness<br>▪ Polishing (nano silicon) protocol was more effective in preventing bacterial adhesion in comparison to polishing (1200 grit) protocol |
| | | Polished (nanosilicon) | 0.02 | | |
| | Polymer-based pre-reacted glass ionomer (Beautifil II, Shofu) | Polished (1200 grit) | 0.67 | ▪ Polished (1200 grit) > polished (nano silicon) | ▪ Results showed a significant increase in bacterial adhesion and an increase in surface roughness<br>▪ Polishing (nano silicon) protocol was more effective in preventing bacterial adhesion in comparison to polishing (1200 grit) protocol |
| | | Polished (nanosilicon) | 0.03 | | |
| Li and others[29] | Titanium (Chinese National, GB/T 3623-1998) | Control | 2.04 (0.42) | ▪ Control > polishing (manual and electrolytic) > polishing (centrifugal) | ▪ Roughness values below the threshold of 0.2 μm presented s significant amount of bacterial adhesion<br>▪ Polishing using the centrifugal mill protocol was effective in preventing *Candida albicans* adhesion |
| | | Polishing (manual) | 0.35 (0.12) | | |
| | | Polishing (electrolytic) | 0.19 (0.09) | | |
| | | Polishing (centrifugal) | 0.18 (0.08) | | |
| Ionescu and others[20] | Resin composite (Filtek Supreme XT, 3M ESPE) | Control (Mylar strips) | 0.05 (0.02) | ▪ 48-h incubation: no significant difference<br>▪ 96-h incubation: polishing > control | ▪ Polishing protocol was not successful in establishing the level observed on control considering 96-h biofilm incubation |
| | | Polishing (4000 SiC) | 0.06 (0.02) | | |
| | Resin composite (Filtek Silorano, 3M ESPE) | Control (Mylar strips) | 0.06 (0.03) | ▪ 48-h incubation: no significant difference<br>▪ 96-h incubation: no significant difference | ▪ Similar roughness values resulted in no significant difference in bacterial adhesion regardless the incubation time |
| | | Polishing (4000 SiC) | 0.07 (0.03) | | |

Table 3: *Summary of the Results of Roughness and Bacterial Adhesion (cont.)*

| Author | Material | Intervention | Surface Roughness (μm) | Bacterial Adhesion Outcome | Statement |
|---|---|---|---|---|---|
| Aykent and others[30] | Direct composite (Tetric Evo-Ceram, Ivoclar-Vivadent) | Finishing (grind) | 1 (0.4) | ▪ No significant differences among surface treatments and no significant interactions between restorative materials and surface treatments | ▪ Differences between 0.58 and 1.0 μm did not result in increased bacterial adhesion |
| | | Polishing (Sof-Lex) | 0.58 (0.4) | | |
| | | Polishing (diamond paste) | 0.9 (0.2) | | |
| | | F&P | 0.7 (0.1) | | |
| | Indirect composite Estenia (KL 100, Kuraray) | Finishing (grind) | 1.2 (0.4) | ▪ No significant differences among surface treatments and no significant interactions between restorative materials and surface treatments | ▪ Differences between 0.58 and 1.2 μm did not result in increased bacterial adhesion |
| | | Polishing (Sof-Lex) | 0.6 (0.4) | | |
| | | Polishing (diamond paste) | 1 (0.4) | | |
| | | F&P | 0.58 (0.4) | | |
| | Indirect composite (SR Adoro, Ivoclar-Vivadent) | Finishing (grind) | 1.5 (0.2) | ▪ No significant differences among surface treatments and no significant interactions between restorative materials and surface treatments | ▪ Differences between 0.7 and 1.5 μm did not result in increased bacterial adhesion |
| | | Polishing (Sof-Lex) | 0.7 (0.4) | | |
| | | Polishing (diamond paste) | 1.1 (0.4) | | |
| | | F&P | 0.9 (0.4) | | |
| | Ceramic material (VITABLOCS Mark II, Vita Zahnfabrik) | Finishing (grind) | 1.1 (0.8) | ▪ No significant differences among surface treatments and no significant interactions between restorative materials and surface treatments | ▪ Differences between 0.6 and 1.1 μm did not result in increased bacterial adhesion |
| | | Polishing (Sof-Lex) | 0.6 (0.2) | | |
| | | Polishing (diamond paste) | 0.9 (0.6) | | |
| | | F&P | 0.7 (0.4) | | |
| Barbour and others[2] | Standard titanium abutments (Nobel Biocare) | Control | 0.25 (0.42) | ▪ *Streptococcus mutans:* polishing (2) < control No significant differences were observed between polishing (1) and control<br>▪ *Actinomyces naeslundii:* polishing (1) and (2) > control | ▪ Similar roughness values presented significant differences in bacterial adhesion<br>▪ Polishing (2) was effective in g preventing bacterial adhesion for both the *S. mutans* and the *A. naeslundii* strains |
| | | Polishing (1) | 0.27 (0.04) | | |
| | | Polishing (2) | 0.25 (0.04) | | |
| Dezelic and others[18] | Resin composite (Tetric, Ivoclar-Vivadent) | Control (320 SiC) | 0.49 | ▪ 15-min incubation: polishing < control<br>▪ 15-h incubation: no significant difference | ▪ Polishing protocol successfully reestablished the level observed in the control using a very early biofilm formation (15 min)<br>▪ After 15-h biofilm formation, polishing procedure was not effective in preventing bacterial adhesion |
| | | Polishing (4000 SiC) | 0.04 | | |
| | Flowable composite (Tetric Flow, Ivoclar-Vivadent) | Control (320 SiC) | 0.61 | ▪ 15-min incubation: no significant differences<br>▪ 15-h incubation: no significant difference | ▪ Regardless of the roughness values and incubation time, the polishing procedure did not differ from the control condition |
| | | Polishing (4000 SiC) | 0.04 | | |
| | Unfilled resin (Heliobond, Ivoclar-Vivadent) | Control (320 SiC) | 0.82 | ▪ 15-min incubation: no significant difference<br>▪ 15-h incubation: no significant difference | ▪ Regardless of the roughness values and incubation time, the polishing procedure did not differ from the control condition |
| | | Polishing (4000 SiC) | 0.07 | | |
| Ono and others[17] | Resin composite (Clearfil AP-X, Kuraray) | Finishing (grind) | 2.22 (0.13) | ▪ Finishing > polishing | ▪ Results showed a significant increase in bacterial adhesion and an increase in surface roughness<br>▪ Polishing protocol was more effective in preventing bacterial adhesion in comparison to finishing procedures |
| | | Polishing (diamond paste) | 0.25 (0.66) | | |

Table 3: *Summary of the Results of Roughness and Bacterial Adhesion (cont.)*

| Author | Material | Intervention | Surface Roughness (μm) | Bacterial Adhesion Outcome | Statement |
|---|---|---|---|---|---|
| | Resin composite (Grandio, Voco) | Finishing (grind) | 2.01 (1.12) | ▪ Finishing > polishing | ▪ Results showed a significant increase in bacterial adhesion and an increase in surface roughness<br>▪ Polishing protocol was more effective in preventing bacterial adhesion in comparison to finishing procedures |
| | | Polishing (diamond paste) | 0.22 (0.01) | | |
| Ikeda and others[16] | Resin composite Estenia (KL 100, Kuraray) | Polishing (600 SiC) | 11.7 (0.3) | ▪ Polishing (SiC) > polishing (diamond paste) | ▪ Results showed a significant increase in bacterial adhesion and an increase in surface roughness |
| | | Polishing (diamond paste) | 6.4 (0.2) | | |
| | Gradia (GC America) | Polishing (600 SiC) | 11.2 (0.4) | ▪ Polishing (SiC) > polishing (diamond paste) | ▪ Results showed a significant increase in bacterial adhesion and an increase in surface roughness |
| | | Polishing (diamond paste) | 7.3 (0.5) | | |
| Pier-Francesco and others[15] | Titanium (Goodfellow Cambridge Ltd) | Polishing (brushes) | 0.03 | ▪ Polishing (machined) > polishing (brushes) | ▪ Results showed a significant increase in bacterial adhesion and an increase in surface roughness<br>▪ Roughness values below the threshold of 0.2 μm presented a significant amount of bacterial adhesion |
| | | Polishing (machined) | 0.16 | | |
| Crlén and others[14] | Glass ionomer (Ketac Aplicap, 3M ESPE) | Control | 0.86 (0.06) | ▪ No significant differences were observed between control and polishing groups | ▪ Differences between 0.86 and 1.05 μm did not result in increased bacterial adhesion<br>▪ Polishing protocol successfully reestablished the level observed on the control |
| | | Polishing (1000 SiC) | 1.05 (0.12) | | |
| | Composite resin (TPH Spectrum, Dentsply DeTrey) | Control | 0.15 (0.05) | ▪ Polishing > control | ▪ Polishing protocol was not successful in reestablishing the level observed on the control |
| | | Polishing (1000 SiC) | 0.56 (0.06) | | |
| Kawai and others[13] | Feldspar ceramic (Vita Celay blanks, A3M-9, Vita Zahnfabrik) | Control (glazed) | 0.15 (0.04) | ▪ 3-h incubation: no significant difference<br>▪ 8-h incubation: no significant difference<br>▪ 12-h incubation: no significant difference<br>▪ 24-h incubation: polishing (diamond paste) < control (glazed) | ▪ Considering short periods of incubation (3, 8, and 12 h), roughness values ranging from 0.12 to 0.53 μm did not result in a significant increase of bacterial adhesion<br>▪ Polishing with diamond paste was successful in preventing bacterial adhesion compared to the control |
| | | Polishing (120 EP) | 0.53 (0.09) | | |
| | | Polishing (600 EP) | 0.25 (0.07) | | |
| | | Polishing (diamond paste) | 0.12 (0.02) | | |

roughness are not enough to affect the amount of biofilm accumulation, even when comparing surfaces above the threshold roughness, once polishing surfaces achieved similar results of biofilm accumulation compared to the pretreatment surfaces without presenting the same level of surface roughness.

In addition to the data from clinical and *in situ* studies, most of the articles included in this systematic review used *in vitro* experiments. In brief, it was observed that several articles found no differences in biofilm formation when surfaces with Ra values above the threshold of 0.2 μm were compared,[2,13,14,18,23,30] whereas significant differences in biofilm formation were found in other studies in which only smooth surfaces (Ra values up to 0.2 μm) were evaluated.[15,20] Thus, based on data from these laboratory studies, the threshold roughness of 0.2 μm was not fully corroborated, and it should be used cautiously among the different materials evaluated. This divergence may be explained by the intrinsic limitations of laboratory studies, which do not offer the strongest evidence.[33]

Only one *in vitro* study used a polymicrobial biofilm model formed from human saliva,[23] while all other studies used synthesized biofilm. In addition, even though studies of monospecimens

Table 4: *Risk of Bias of the Studies Included on Systematic Review Considering the Aspects Reported in the Materials and Methods Section*[a]

| Author | Sample | Random | Control | Materials | Treatment | Blinding | Repetition | Risk of Bias |
|---|---|---|---|---|---|---|---|---|
| Clinical studies | | | | | | | | |
| Elter and others[28] | N | N | Y | Y | N | N | NA | High |
| Quirynen and others[9] | N | Y | Y | Y | Y | Y | NA | Low |
| *In situ* studies | | | | | | | | |
| Haralur[25] | N | N | Y | Y | Y | N | NA | Medium |
| Brentel and others[5] | N | Y | Y | Y | Y | N | NA | Medium |
| Perez[26] | N | N | Y | N | Y | N | NA | High |
| Rimondini and others[24] | N | Y | N | Y | Y | N | NA | Medium |
| *In vitro* studies | | | | | | | | |
| Dutra and others[23] | N | Y | Y | Y | Y | N | Y | Low |
| Yuan and others[22] | N | N | N | N | Y | N | N | High |
| Li and others[21] | N | Y | Y | N | Y | N | N | Medium |
| Ionescu and others[20] | N | Y | Y | N | N | N | N | High |
| Aykent and others[19] | N | N | N | N | N | N | N | High |
| Barbour and others[2] | N | N | Y | Y | N | N | Y | Medium |
| Dezelic and others[18] | N | N | Y | N | Y | N | N | High |
| Ono and others[17] | N | N | N | N | N | N | Y | High |
| Ikeda and others[16] | N | N | N | Y | N | N | Y | High |
| Pier-Francesco and others[15] | N | N | N | Y | N | N | N | High |
| Carlén and others[14] | N | N | Y | Y | N | N | N | High |
| Kawai and others[13] | N | N | Y | N | N | N | N | High |

*Abbreviations: N, no; NA, not applicable; Y, yes.*
[a] *Sample size calculation; randomization of the sample; untreated control group; materials used according to manufacturer's instructions; description of finishing/polishing standardization; blinding of the examiner of the outcome and repetition of biofilm experiment (*in vitro*).*

have enhanced knowledge of the mechanisms of bacterial adhesion to surfaces and differentiation into multicellular biofilms, the use of polymicrobial biofilm models should be incentivized once the majority of chronic infections harboring polymicrobial communities. Although *in vitro* models have been extensively used to study dental biofilm, there are limitations when trying to simulate the oral environment and *in vivo* conditions. It has to be highlighted that during *in vivo* chronic infection, there is a complex interplay between host and pathogen, with species not directly mixing but rather residing within their own ecological space, something that is not easily replicated *in vitro* and that leads to observable differences between *in vitro* and *in vivo* "chronic infections."[33]

It is well accepted that hard tissues with rougher surfaces in the oral cavity contribute to microorganism retention since rougher surfaces have a greater area for the development of biofilm as well as topographical irregularities that produce niches in which microorganisms are protected from shear forces and salivary flow. Such factors affect microorganism retention only in clinical and *in vivo* studies, as these factors are rarely simulated in laboratory studies. Therefore, the impact of topographical irregularities on bacterial retention in *in vitro* studies appears to be limited, and the amount of biofilm in such studies may be strongly related to other factors linked to the biofilm protocol, such as the type of inoculum (bacterial strain and human saliva) and culture conditions (temperature, pH, nutritional status and nutrient flow, presence of salivary pellicle, and incubation time).

## Limitations of the Study

The results of the present review should be interpreted cautiously since most of the included studies were carried out using laboratory studies that do not represent the same evidence as clinical studies. Roberts and others[33] stated that "whilst there is no 'gold-standard' for the study of *in vivo* and *in vitro* biofilm formation, it is crucial to know the limiting factors of selected models so as to not over-extrapolate data, and generate assumptions beyond the capabilities of the model." For this reason, we discussed the results from each study design individually.

Moreover, it must be mentioned that the assessment of the risk of bias showed that most studies had high risk (61%). It was especially critical for *in vitro* studies, as nine of the 12 articles had high risk while only one had low risk. This result highlights that *in vitro* studies had poor control regarding the methodological variables that could influence the results, directly affecting the validity of the studies and explaining in part the resulting heterogeneity.

## CONCLUSIONS

Based on the findings of this systematic review, the following conclusions may be drawn:

- Finishing invariably creates a rougher surface and should always be followed by a polishing method.
- The range of surface roughness among different polishing methods is wide and material dependent.
- Each dental material requires its own treatment modality to obtain and maintain as smooth a surface as possible.
- A surface roughness threshold of Ra = 0.2 μm did not properly predict biofilm formation in nonclinical studies.
- Topographical irregularities of restorative surfaces played a limited effect on *in vitro* bacterial retention, while a higher impact was observed in *in vivo* studies.

Additionally, wide methodological heterogeneity and poor bias control in the majority of studies included in this review became evident. These study limitations made interstudy comparison and the summarization of related evidence difficult. Future investigations characterizing bacterial adhesion on restorative materials and evaluating the effect of surface treatments and topographical irregularities on bacterial adhesion and biofilm formation must be planned considering each study design restriction and predicting the validity and relevance of the evidence to be generated. Thus, in order to better standardize the studies in this area and to produce evidence of greater clinical relevance, well-designed *in vivo* studies are strongly recommended.

### 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 10 May 2017)*

## REFERENCES


1. Axelsson P, & Lindhe J (1978) Effect of controlled oral hygiene procedures on caries and periodontal disease in adults *Journal of Clinical Periodontology* **5(2)** 133-151.
2. Barbour ME, O'Sullivan DJ, Jenkinson HF, & Jagger DC (2007) The effects of polishing methods on surface morphology, roughness and bacterial colonisation of titanium abutments *Journal of Materials Science: Materials in Medicine* **18(7)** 1439-1447.
3. Scotti R, Kantorski KZ, Monaco C, Valandro LF, Ciocca L, & Bottino MA (2007) SEM evaluation of in situ early bacterial colonization on a Y-TZP ceramic: A pilot study *International Journal Prosthodontics* **20(4)** 419-422.
4. Kantorski KZ, Scotti R, Valandro LF, Bottino MA, Koga-Ito CY, & Jorge AO (2009) Surface roughness and bacterial adherence to resin composites and ceramics *Oral Health and Preventive Dentistry* **7(1)** 29-32.
5. Brentel AS, Kantorski KZ, Valandro LF, Fúcio SB, Puppin-Rontani RM, & Bottino M (2011) Confocal laser microscopic analysis of biofilm on newer feldspar ceramic *Operative Dentistry* **36(1)** 43-51.
6. Bollen CML, Lambrechts P, & Quirynen M (1997) Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: A review of the literature *Dental Materials* **13(4)** 258-269.
7. Teughels W, Assche N, Sliepen I, & Quirynen M (2006) Effect of material characteristics and/or surface topography on biofilm development *Clinical Oral Implants Research* **17(Supplement 2)** 68-81.
8. Song F, Koo H, & Ren D (2015) Effects of material properties on bacterial adhesion and biofilm formation *Journal of Dental Research* **94(8)** 1-8.
9. Quirynen M, Marechal M, Busscher HJ, Weerkamp AH, Darius PL, & van Steenberghe D (1990) The influence of surface free energy and surface roughness on early plaque formation: An in vivo study in man *Journal of Clinical Periodontology* **17(3)** 138-144.
10. Moher D, Liberati A, Tetzlaff J, & Altman DG (2009) Reprint—Preferred reporting items for systematic reviews and meta-analyses: The PRISMA statement *Physical Therapy* **89(9)** 873-880.
11. Sarkis-Onofre R, Skupien JA, Cenci MS, Moraes RR, & Pereira-Cenci T (2014) The role of resin cement on bond strength of glass-fiber posts luted into root canals: A systematic review and meta-analysis of in vitro studies *Operative Dentistry* **39(1)** E31-E44.
12. Montagner AF, Sarkis-Onofre R, Pereira-Cenci T, & Cenci MS (2014) MMP inhibitors on dentin stability: A systematic review and meta-analysis. *Journal of Dental Research* **93(8)** 733-743.
13. Kawai K, Urano M, & Ebisu S (2000) Effect of surface roughness of porcelain on adhesion of bacteria and their synthesizing glucans *Journal of Prosthetic Dentistry* **83(6)** 664-667.
14. Carlén A, Nikdel K, Wennerberg A, Holmberg K, & Olsson J (2001) Surface characteristics and in vitro

biofilm formation on glass ionomer and composite resin *Biomaterials* **22(5)** 481-487.

15. Pier-Francesco A, Adams RJ, Waters MGJ, & Williams DW (2006) Titanium surface modification and its effect on the adherence of Porphyromonas gingivalis: An in vitro study *Clinical Oral Implants Research* **17(6)** 633-637.
16. Ikeda M, Matin K, Nikaido T, Foxton RM, & Tagami J (2007) Effect of surface characteristics on adherence of S. mutans biofilms to indirect resin composites *Dental Materials Journal* **26(6)** 915-923.
17. Ono M, Nikaido T, Ikeda M, Imai S, Hanada N, Tagami J, Matin K (2007) Surface properties of resin composite materials relative to biofilm formation *Dental Materials Journal* **26(5)** 613-622.
18. Dezelic T, Guggenheim B, & Schmidlin PR (2009) Multi-species biofilm formation on dental materials and an adhesive patch *Oral Health and Preventive Dentistry* **7(1)** 47-53.
19. Aykent F, Yondem I, Ozyesil AG, Gunal SK, Avunduk MC, & Ozkan S (2010) Effect of different finishing techniques for restorative materials on surface roughness and bacterial adhesion *Journal of Prosthetic Dentistry* **103(4)** 221-227.
20. Ionescu A, Wutscher E, Brambilla E, Schneider-Feyrer S, Giessibl FJ, & Hahnel S (2012) Influence of surface properties of resin-based composites on in vitro Streptococcus mutans biofilm development *European Journal of Oral Science* **120(5)** 458-465.
21. Li Y, Guo T, Zhao J, & Wang J (2013) Effects of polishing methods on Candida albicans adhesion on cast pure titanium surfaces *Implant Dentistry* **22(5)** 546-551.
22. Yuan C, Wang X, Gao X, Chen F, Liang X, & Li D (2016) Effects of surface properties of polymer-based restorative materials on early adhesion of Streptococcus mutans in vitro *Journal of Dentistry* **54** 33-40.
23. Dutra DA, Pereira GK, Kantorski KZ, Exterkate RAM, Kleverlaan CJ, Valandro LF, & Zanatta FB (2017) Grinding with diamond burs and hydrothermal aging of a Y-TZP material: Effect on the material surface characteristics and bacterial adhesion on Y-TZP surface *Operative Dentistry* In Press http://dx.doi.org/10.2341/16-108-L
24. Rimondini L, Farè S, Brambilla E, Felloni A, Consonni C, Brossa F, Carrassi A (1997) The effect of surface roughness on early in vivo plaque colonization on titanium *Journal of Periodontology* **68(6)** 556-562.
25. Haralur SB (2012) Evaluation of efficiency of manual polishing over autoglazed and overglazed porcelain and its effect on plaque accumulation. *Journal of Advanced Prosthodontics* **4(4)** 179-186.
26. Perez C (2008) Effect of a resin polishing system on the Streptococus mutans adhesion in situ PhD thesis, Rio de Janeiro State University; Available from: http://pesquisa.bvsalud.org/ghl/resourse/en/bbo-34453
27. Quirynen M, Bollen CM, Papaioannou W, Van Eldere J, & van Steenberghe D (1997) The influence of titanium abutment surface roughness on plaque accumulation and gingivitis: Short-term observations *International Journal of Oral and Maxillofacial Implants* **11(2)** 169-178.
28. Elter C, Heuer W, Demling A, Hannig M, Heidenblut T, Bach FW, Stiesch-Scholz M (2008) Supra- and subgingival biofilm formation on implant abutments with different surface characteristics *International Journal of Oral and Maxillofacial Implants* **23(2)** 327-334.
29. Li RWK, Chow TW, & Matinlinna JP (2014) Ceramic dental biomaterials and CAD/CAM technology: State of the art *Journal of Prosthodontic Research* **58(4)** 1-9.
30. Aykent F, Yondem I, Ozyesil AG, Gunal SK, Avunduk MC, & Ozkan S (2010) Effect of different finishing techniques for restorative materials on surface roughness and bacterial adhesion *Journal of Prosthetic Dentistry* **103(4)** 221-227.
31. Yadav RD, Raisingani D, Jindal D, & Mahur R (2016) A comparative analysis of different finishing and polishing devices on nanofilled, microfilled, and hybrid composite: A scanning electron microscopy and profilometric study *International Journal of Clinical Pediatric Dentistry* **9(3)** 201-208.
32. Hannig M (1999) Ultrastructural investigation of pellicle morphogenesis at two different intraoral sites during a 24-h period *Clinical Oral Investigations* **3(2)** 88-95.
33. Roberts AEL, Kragh KN, Bjarnsholt T, & Diggle SP (2015) The limitations of in vitro experimentation in understanding biofilms and chronic infection *Journal of Molecular Biology* **427(23)** 3646-3661.

# 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 Sirona

ivoclar vivadent

KAVO Kerr

BISCO

AMERICAN BOARD OF OPERATIVE DENTISTRY
IN PURSUIT OF EXCELLENCE
FOUNDED 1980

# OPERATIVE DENTISTRY

Volume 43 / Number 1
January/February 2018

www.jopdent.org
1–112

## Guest Editorial



## Clinical Technique/Case Report



## Clinical Research



## Laboratory Research



## Departments



## Online Only Articles

Vol 43/No 1 January/February 2018 Pages 1–112 OPERATIVE DENTISTRY ISSN 0361-7734