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Operative Dentistry publishes articles that advance the practice of operative dentistry. The scope of the journal includes conservation and restoration of teeth; the scientific foundation of operative dental therapy; dental materials; dental education; and the social, political, and economic aspects of dental practice. Review papers, book reviews, letters and classified ads for faculty positions are also published.

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Indiana University School of Dentistry, Room S411
1121 West Michigan Street, Indianapolis, IN 46202-5186
Telephone: (317) 278-4800, Fax: (317) 278-4900
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Editorial

Light Curing Explored in Halifax

Jeffrey A Platt • Richard B Price

I do not remember a time during my dental career when direct resin restorations were not being disparaged by someone. Some of the expressed concerns have validity. Certainly the earlier composite resins offered little wear resistance or ability to predictably create a bonded interface. The chemical activation system used in early materials also limited the ability of a practitioner to form and sculpt restorations.

I remember being taught that the placement of a composite resin restoration should include the same level of care and attention to detail provided to direct gold restorations. I have held the opinion that many of the problems associated with composite resin restorations can be attributed to approaching their placement in a way that mimics the approach taken when placing amalgam, a material that is much more forgiving of less-than-optimal handling.

An important issue in the placement of light-activated composite resin is the amount and type of light energy that is actually being received by the material. Inadequate light curing can easily result in compromised restorative material properties, compromises that likely have a negative influence on restoration longevity. It has been well documented that, worldwide, many offices have been using inadequate amounts of energy and less-than-optimal technique and are delivering inadequate amounts of energy when light curing resins.[1-9] If that is the case, then it should be no surprise to anyone when reading reports about the substandard performance of posterior composite resin restorations.[10-13]

More than 40 key opinion leaders and company scientists met at Dalhousie University in May 2014 to discuss ways to address issues surrounding light curing. Arranged by Dr. Richard B. Price, the symposium participants included:

Bob Angelo, Ahmed Abuelyaman, Suham Alexander, Sibel Antonson, Steve Armstrong, Oliver Benz, Uwe Blunck, Ellen Bruzell, John Burgess, Peter Burtscher, Liang Chen, Ivo Correa, Matt Dailey, Colin Deacon, Omar El-Mowafy, Christopher Felix, Jack Ferracane, Reinhard Hickel, Thomas Hill, Neil Jessop, Hilde Kopperud, Daniel Labrie, Hui Lu, Bernhard Möginger, Lori Moilanen, John O'Keefe, Joe Oxman, Frank Pfefferkorn, Jeffrey Platt, Richard Price, Jean-François Roulet, Fred Rueggeberg, Janine Schweppe, Adrian Shortall, Jeffrey Stansbury, Howard Strassler, Byoung Suh, Andreas Utterodt, David Watts, and Stacy Wyatt.

The symposium received support and active participation from Benco, BISCO, BlueLight Analytics, DENTSPLY, Gigahertz-Optik, Henry Schein, Heraeus-Kulzer, Ivoclar Vivadent, Kerr, Patterson Dental, SDI, 3M-ESPE, and Ultradent.

The group adopted a glossary of terms that are based on the International System of Units (SI) definitions associated with light technology and is encouraging the use of them during communication on the subject (Table 1).[14] In addition, a consensus statement reflecting areas of agreement within the group was drafted and is included here as Figure 1.

Inadequate light curing can easily result in compromised restorative material properties. These compromises will likely have a negative influence on restoration longevity.

The included guidelines are provided for the benefit of your patients and are simultaneously being published here and in the following journals: *Journal of Adhesive Dentistry*, *Dental Materials*, and *Journal of the Canadian Dental Association*.

Table 1: *Glossary of Terms for Light Curing*[a]

Term	Unit Commonly Used in Dentistry	Symbol	Notes
Radiant energy	Joule	J	This describes the energy from the curing light.
Radiant exposure	Joule per square centimeter	J/cm^2	Also referred to as fluence and sometimes incorrectly as "energy density".
Radiant energy density	Joule per cubic centimeter	J/cm^3	This is the correct definition of "energy density".
Radiant flux or radiant power	Watt	W or J/s	Radiant energy per time unit.
Radiant exitance (excitance) or Radiant emittance	milliWatt per square centimeter	mW/cm^2	Radiant power/flux emitted from a surface (eg, a curing light). To be used instead of power density or irradiance when describing the output from a curing light.
Irradiance (incident irradiance)	milliWatt per square centimeter	mW/cm^2	Radiant power/flux incident on a surface. This is what the resin receives.
Spectral radiant power	milliWatt per nanometer	mW/nm	Radiant power per wavelength.
Spectral irradiance	milliWatt per square centimeter per nanometer	$mW/cm^2/nm$	Irradiance received by the resin at each nanometer.

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Light Curing – Guidelines for Practitioners
A Consensus Statement from the 2014 Symposium on Light Curing in Dentistry held at Dalhousie University, Halifax, Canada*

When selecting a light curing unit (LCU):

- **Recognize that all lights are not created equal.** Use a LCU from a manufacturer who provides contact information, a user manual, and service. Preferably the LCU should have received a favorable report or certification from a reputable independent 3rd party.
- **Know** the key performance parameters of your LCU, when new:
  (i) the light output (averaged irradiance over the beam incident area in mW/cm^2 and spectral output from the LCU), (ii) whether the beam has a uniform and effective output (profile) across the light tip, and (iii) the diameter of the light beam.
- **Be cautious** when using high (above 1,500 to 2,000 mW/cm^2) output LCUs that advocate very short (e.g. 1 to 5 seconds) exposure times. When used for such short times, it is critical that the light tip is stabilized over the resin during exposure. Although some resin composites are matched to specific high output curing lights, high output LCUs may not adequately cure all of today's resin-composites to the anticipated depth when used for short exposure times. Seek peer-reviewed literature validating the efficacy and safety of such lights and materials.

Before you light cure, remember to:

- **Regularly monitor** and record the light output over time, with the same measurement device and light guide. Repair or replace the LCU when it no longer meets the manufacturer's specifications.
- **Inspect and clean** the LCU before use to ensure it is on the correct setting, in good working order, and free of defects and debris.
- **Consider that every resin-based material** has a minimum amount of energy that must be provided at the correct wavelengths to achieve satisfactory results. [Energy ($Joules/cm^2$) = output (W/cm^2) x exposure time (seconds)]. However, minimum irradiation times are also required.
- **Follow the recommended light exposure times and increment thickness recommended by the resin manufacturer,** making allowances if you use another manufacturer's light. Increase your curing times for increased distances and darker or opaque shades.
- **Select a LCU tip that** delivers a uniform light output across the light tip and that covers as much of the restoration as possible. Cure each surface independently, using overlapping exposures if the light tip is smaller than the restoration.
- **Position** the light tip as close as possible (without touching) and parallel to the surface of the resin composite being cured.
- **Stabilize and maintain** the tip of the LCU over the resin composite throughout the exposure.
- **Always use** the appropriate "blue blocking" glasses or shield to protect your eyes as you watch and control the position of the curing light.

Precautions:

- **Avoid** conditions that will reduce light delivery to the resin-composite, e.g.:
  - Holding the light tip several millimeters away.
  - Holding the light tip at an angle to the resin surface.
  - Dirty or damaged light-guide optics.
- **Supplementary light exposures** should be considered under circumstances that may limit ideal light access, such as shadows from matrix bands, intervening tooth structure, or from restorative material.
- **Beware of potential thermal damage** to the pulp and soft tissues when delivering high energy exposures or long exposure times.
- **Air-cool** the tooth when exposing for longer times, or when using high output LCUs.
- **Never shine** the LCU into the eyes, and avoid looking at the reflected light, except through an appropriate 'blue-blocking' filter.
- **Testing surface hardness** of the resin-composite in the tooth using a dental explorer provides NO information about adequacy of curing depth.

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Staff Letter

Dear Readers,

We are so excited to announce that in recognition of our 40th volume year which will begin with your next issue, we have undertaken a major redesign of our wonderful journal. Some things have been changed significantly – like our cover and our logo, other things, like our quality content, and commitment to the practicing dentist have not changed at all. We have also noticed that as we progress through the years, we find that our history becomes more and more important to us, not only as a marker of where we have been, but as a foundation of strength, supporting the outstanding research that is taking place today! That strength is one of the many reasons that our social media presence continues to expand. Our growth and quest for excellence doesn't come without a price however, so, in light of these things, we have drafted the following announcements touching on each of these points. We hope that you will agree that these changes will be positive for you as a reader and for the whole dental community as we continue to strive for perfection.

New Cover Photo Opportunity

Dentists are an artistic bunch! If your artistry extends to photography, we welcome submissions from you to be considered as a cover photo in an upcoming issue. We welcome landscapes, cityscapes, and particularly delicious looking fruit in bowls, etc. We would even be willing to entertain some arresting micrographs.

As these photos will constitute advertisement for the journal, we must be very careful of copyright and other property restrictions. Any photos with recognizable people in them, or with corporate logos, or trademarks in them may be submitted, but will have to be accompanied by some significant legal forms (that we have available for you). Photos taken on and/or of private property will also have some legal wrangling to do. As such, we ask our submitters to limit the number of photos submitted that may run afoul of these restrictions. No one likes reviewing legal paperwork – especially our hard working editorial assistant – Kevin!

Submitted photos become the property of Operative Dentistry, Inc. If your photo is chosen for use you will be notified before publication so you can order a copy for all your friends! The copyright for photos that are not used will revert to the author after 24 months. Please see the Non-Manuscript copyright agreement for full details – found at www.jopdent.org under the links journal, and then Cover Photo Copyright.

Photos must be submitted in digital format. Images should be high-resolution – 300 dpi, at least 5x7 inches (1500 × 2100 pixels minimum), and be in high quality JPG or TIFF format. The images will not be returned. Minor digital enhancement is permitted, but images that have been significantly modified or appear unnatural will be rejected.

If you have questions or problems with the submission system, please email the editorial assistant, Kevin Matis, at editor@jopdent.org.

Revised Author Policy

In the past we have sent a complimentary copy of an issue to an author (via the corresponding author) in which their article appears. We have received feedback that this is becoming less and less appreciated as the influence of digital media becomes more and more prevalent at our universities and institutions. Our new policy, effective with the previous issue (39-5) is to send a digital copy of the authors work to them. In the past, this file had to be purchased if a particular author was not an online subscriber of the journal. We are happy to now be able to provide this file, free of charge, to our authors. The restrictions of copyright have not changed however; authors may make unlimited prints of that digital file, but may not share the electronic copy with others. The full policies and guidelines of the journal can be found at https://www.jopdent.com/journal/journal.html.

Legacy Content Available

We are so pleased to announce that after several years of work all of Operative Dentistry is available online at www.jopdentonline.org. We have even included the forerunner of Operative Dentistry, The Journal of the American Academy of Gold Foil Operators. This means that high quality literature from 1958 to the present is now available to all. In celebration of our 40th year, we will also be issuing an electronic special issue, hopefully in early 2015, commemorating the best of Operative Dentistry during the past 40 years. You

will certainly hear more about this special project in future issues!

Social Media Request

Part of the excitement and wonder of the digital age is our access to information – the speed at which we receive new information and updates to previous knowledge is truly exciting. This excitement has truly fueled the social media explosion in our lives. We are pleased to be a part of Twitter and Facebook; these two sites allow us to communicate to you when we have posted brand-new content to our site. We hope that those of you who make use of these sites, will connect with us so that you can share our posts with others who may not have had a chance to familiarize themselves with our excellent content. Find and follow us on Facebook at Operative Dentistry, Inc. or follow our twitter feed at @jopdent.

Revised Policy on Late and Claim fees

Due to the ever increasing costs of postage around the globe, we have been forced to raise our fees for late subscriptions. Our old fee of 15 USD per issue missed has been changed, effective immediately, to 20 USD for subscribers living in the USA and 25 USD for all other subscribers. The sad fact is, that as the internet, and electronic distribution of material, becomes more and more popular, the cost of processing and transporting paper becomes more and more expensive. Please know that Operative Dentistry is doing all within its power to keep these costs to a minimum. For full policies and guidelines of the journal, please see our policy manual at https://www.jopdent.com/journal/journal.html.

As a final note, it is that time of year for most, to verify the subscription term of all periodicals, and memberships that an individual maintains, and start making plans for the renewal of those subscriptions. Please feel free at any time to contact Operative Dentistry at editor@jopdent.org with any questions regarding your subscription, or go to our website at https://www.jopdent.com/shopping/index.html to renew your subscription online. We thank you for having subscribed with us this year and we look forward to keeping you current on the latest clinically relevant research and techniques through 2015. We hope each of you enjoy a wonderful and meaningful Holiday Season where ever in the world you are!

Sincerely,

The Staff of Operative Dentistry, Inc.

Joan Matis, Office Manager

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Clinical Technique/Case Report

Multidisciplinary Approach to Delayed Treatment of Traumatic Teeth Injuries Involving Extrusive Luxation, Avulsion and Crown Fracture

Ü Şermet Elbay • A Baysal • M Elbay
S Sarıdağ

Clinical Relevance

The first dentist to meet a patient with trauma must have sufficient knowledge and equipment for all treatment options to solve traumatic injuries requiring a multidisciplinary approach.

SUMMARY

A 12-year-old boy with extrusion of the maxillary right central incisor, uncomplicated fracture of the left central incisor, avulsion of the mandibular right and left central incisors, and crown fracture of the mandibular right lateral incisor presented to the Kocaeli University Department of Pediatric Dentistry 20 days after sustaining the traumatic injuries.

Orthodontic repositioning of the extrusive maxillary right central incisor was planned. Additionally, this tooth was necrotic and needed root canal treatment. The maxillary left central incisor and right mandibular lateral incisor were necrotic and needed root canal treatment. The orthodontic and endodontic treatments were successfully performed simultaneously.

Restoration of the fractured mandibular right lateral incisor and maxillary left central incisor was completed with resin composite. Subsequent to orthodontic and endodontic treatment, prosthodontic rehabilitation was performed. At the two-year followup, the teeth appeared normal and the patient had no complaints.

Ülkü Şermet Elbay, DDS, PhD, assistant professor, Kocaeli University, Faculty of Dentistry, Department of Pediatric Dentistry Kocaeli, Turkey

Aslı Baysal, DDS, PhD, assistant professor, Katip Çelebi University Faculty of Dentistry, Department of Orthodontics, İzmir, Turkey

Mesut Elbay, DDS, PhD, assistant professor, Kocaeli University, Faculty of Dentistry, Department of Pediatric Dentistry, Kocaeli, Turkey

*Serkan Sarıdağ, DDS, PhD, assistant professor, Kocaeli University, Faculty of Dentistry, Department of Prosthodontics, Kocaeli, Turkey

*Corresponding author: Yuvacık Yerleşkesi, Başiskele, Kocaeli, 41190, Turkey; e-mail: ssaridag@hotmail.com

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INTRODUCTION

Some of the most commonly encountered dental emergencies are dentoalveolar traumatic injuries.[1-5] Traumatic dental injury can result in damage to both dental and periradicular structures. It may also cause injury to the pulp, with or without damage to crown and/or root, or in severe cases, tooth displacement.[1-6]

Most dental injuries involve the anterior teeth and usually affect a single tooth, although certain types of trauma cause multiple injuries.[1-4,7] Studies have shown that most injuries involve the upper front teeth, and fewer involve the lower front teeth. Finally, very few patients have injuries that affect the incisors of the maxillary and mandibular arches simultaneously.[4,7,8]

Traumatic injuries of anterior teeth in children create a psychological effect on the parents and the child, especially if the injury affects the permanent dentition and involves the loss of extensive tooth structure. Untreated fractured teeth have been directly related to the emotional state and appearance of children. Children with fractured teeth experience difficulties with eating and enjoying food. Children with untreated dental issues have also been found to avoid smiling and experience negative social interactions compared with their noninjured peers. Hence, the importance of treating traumatized anterior teeth is increasing day by day.[8,9]

The prognosis of traumatic injuries depends on early intervention to injured teeth. A delay in treatment may influence the diagnostic results.[6,10-13] For instance, the success rate and treatment options of replanted teeth may vary after an hour from the avulsion.[6,13-15]Similarly, the accepted treatment for extrusion is repositioning the extruded tooth at the earliest opportunity and stabilization with a splint for up to three weeks. In cases where the tooth cannot be repositioned because of blood clot blockage or as a result of delayed treatment, a different option, such as orthodontic intrusion for tooth repositioning, may be considered.[6,10-13]

This current report presents a case of a patient in whom treatment was delayed for traumatic dental injuries in both the maxillary and mandibular jaws, including severe extrusive luxation of a maxillary central incisor, crown fracture of the left central incisor, avulsion of the mandibular right and left central incisors, and crown fracture of the right lateral incisor.

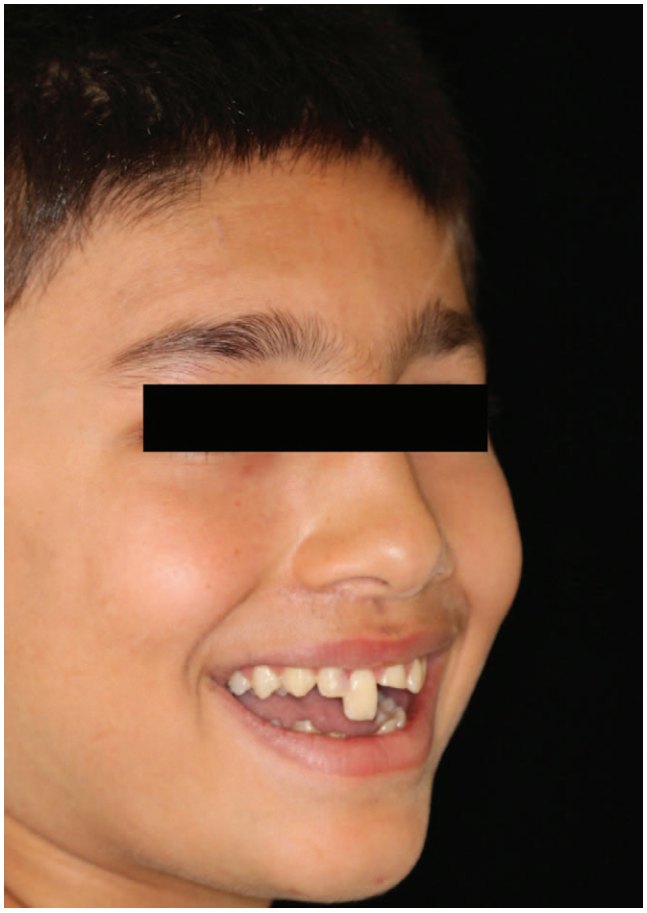

Figure 1. *The smiling appearance of the patient before treatment*.

CASE REPORT

A 12-year-old male patient with dental injury after falling from a bicycle presented to the dental clinic of Kocaeli University Department of Pediatric Dentistry 20 days after the accident.

According to the patient, he was riding with his cousin and sitting on the back seat of a bicycle. The patient sustained trauma to his face involving several skin and mucosa lacerations. He was treated in a medical emergency facility but received no treatment for his dental injuries at that time. The patient was not instructed to present to a dental clinic immediately. Therefore, the patient and his parents postponed going to a dental clinic until the skin and mucosa lacerations healed (Figure 1).

Upon clinical examination of the maxilla, the right maxillary central incisor was found to be severely extruded, and there was an uncomplicated fracture of the left maxillary central incisor (Figures 2 through 4). Both teeth were found to be nonvital according to a sensitivity test. A periapical radiograph showed that both incisors had some degree of

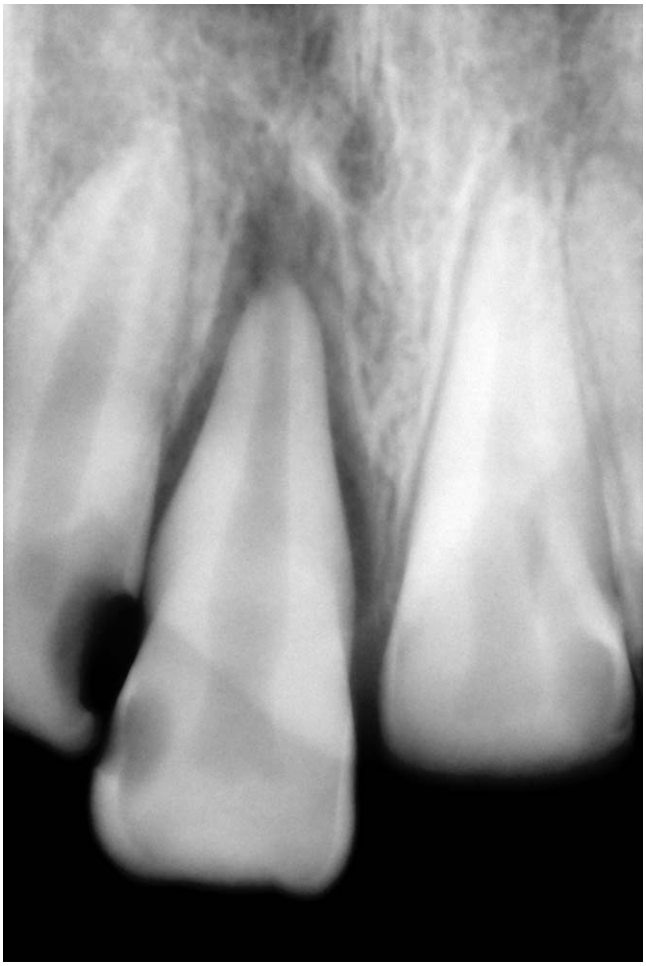

Figure 2. *Periapical radiograph of the upper central incisors before treatment.*

open apices and periapical pathology. The periodontal space around both roots was widened but no root or bone fracture was detected (Figure 2).

Clinical examination of the mandible found a cervical crown fracture of the right lateral mandibular incisor. A periapical radiograph revealed a widened periodontal space and a periapical lesion around the root of that tooth. However, the periapical radiograph showed no evidence of root or bone fracture. The tooth was found to be nonvital according to a sensitivity test. In addition, the patient's right and left mandibular central incisors were avulsed, and unfortunately, he had been unable to locate the avulsed teeth (Figure 5).

After clinical examination, a treatment plan was made. Because of the delay in treatment, repositioning the right maxillary central incisor by conventional digital manipulation was not possible. Therefore, orthodontic repositioning was planned (Figure 6). At the same time, root canal treatment

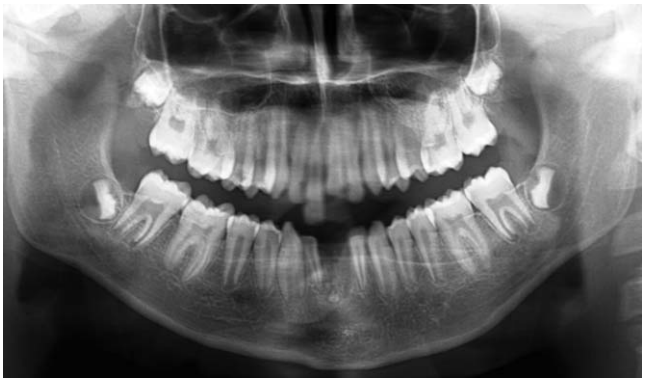

Figure 3. *Orthopantomographic view.*

and apexogenesis of the right and left maxillary central incisors were carried out. In addition, root canal therapy was started for the right lateral mandibular incisor. After accessing and filing the canals, calcium hydroxide was maintained in the canals and changed once a month for six months. Approximately three months after initiating the orthodontic treatment, intrusion of the teeth was completed. Three months after intrusion of the teeth, the root canals were obturated with gutta percha points. Coronal restoration of the teeth was completed using resin composite (Figure 7 through 10).

Prosthodontic rehabilitation was performed using a removable prosthesis for the missing right and left mandibular central incisors after the orthodontic and endodontic treatment was completed (Figure 8).

In the present case, the teeth were stable and the patient was completely asymptomatic for six months after surgery. After a two-year followup, the clinical and radiographic findings demonstrated that the adopted clinical protocol was successful, as the teeth were asymptomatic and there was no gingival inflammation and mobility. Radiographs showed

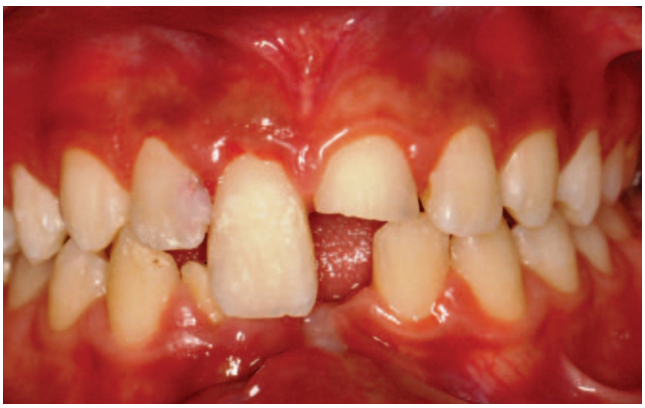

Figure 4. *Intraoral appearance of the patient before treatment.*

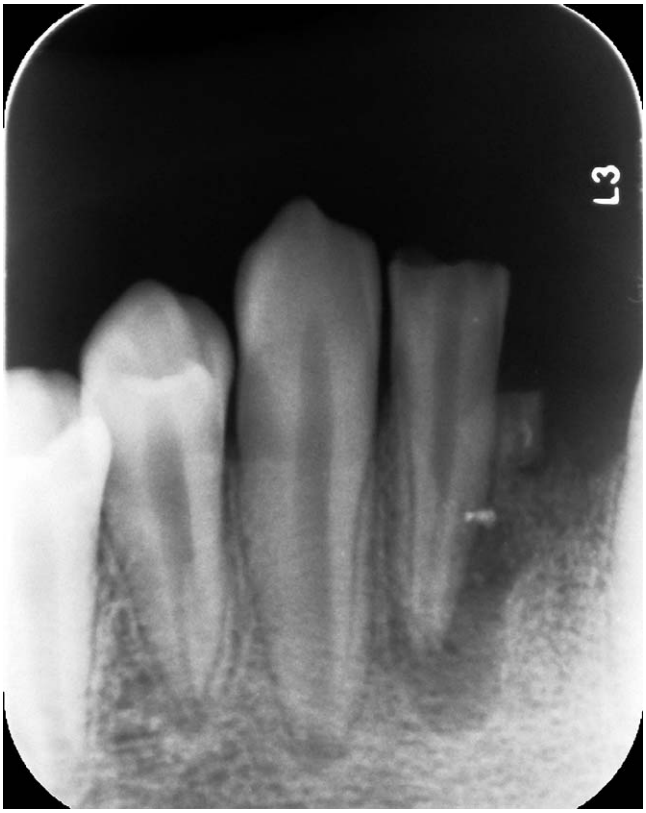

Figure 5. *Periapical radiograph of the lower incisors.*

normal healing of the periapical areas. In addition, the patient was satisfied with his appearance and was smiling well.

DISCUSSION

Facial trauma results in fractured, displaced, or lost teeth[1-16] and can have significantly negative functional, esthetic, and psychological effects.[9,16] In this present case, when the patient presented, he complained of having difficulties eating and was avoiding smiling for esthetic reasons because of his extruded and avulsed teeth.

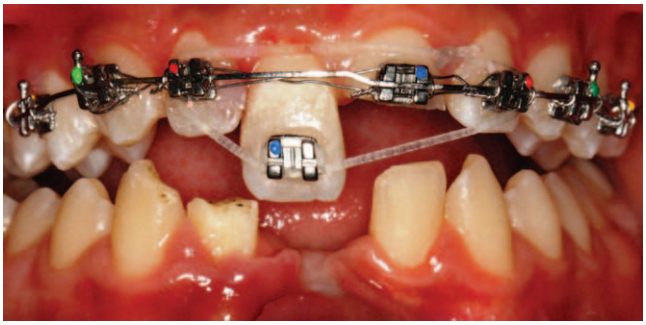

Figure 6. *Orthodontic intrusion of the upper left central incisor.*

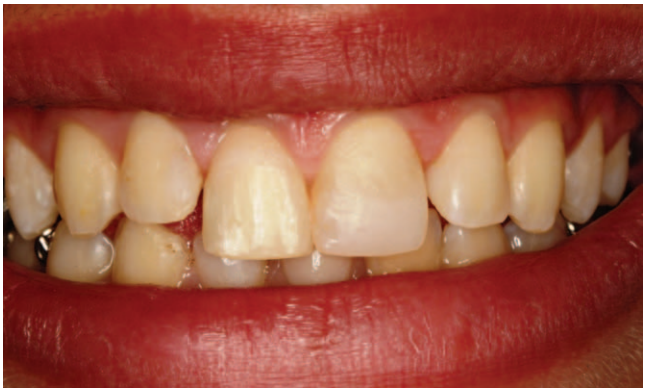

Figure 7. *Intraoral appearance of the patient after treatment.*

The main causes of traumatic dental injuries reported in the literature are violence, collisions, falls, sports, leisure activities, and traffic accidents.[7,16,17] The literature suggests that children seven to twelve years old are the most prone to any form of dental trauma. Furthermore, boys sustain dental trauma almost twice as frequently as girls.[7,8,16] Additionally, the literature suggests that most traumatic dental injures involve the maxillary central incisors, followed by maxillary lateral incisors and mandibular incisors.[7,17,18] The findings of this current case are consistent with the available literature. The current patient was a 12-year-old boy with dental injuries involving extrusion of the maxillary right central incisor, uncomplicated fracture of the maxillary left central incisor, avulsion of the mandibular right and left central incisors, and crown fracture of the right lateral incisor after falling from a bicycle.

The literature shows that maxillary teeth are more frequently traumatized than mandibular teeth.[7,17,18] It has been reported that most dental injuries affect a single anterior tooth and very few patients have injuries affecting incisors of the maxillary and mandibular arches at the same time. The occurrence

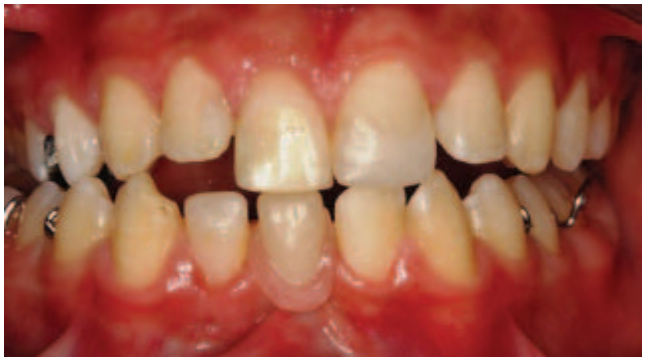

Figure 8. *Intraoral appearance of the patient after treatment.*

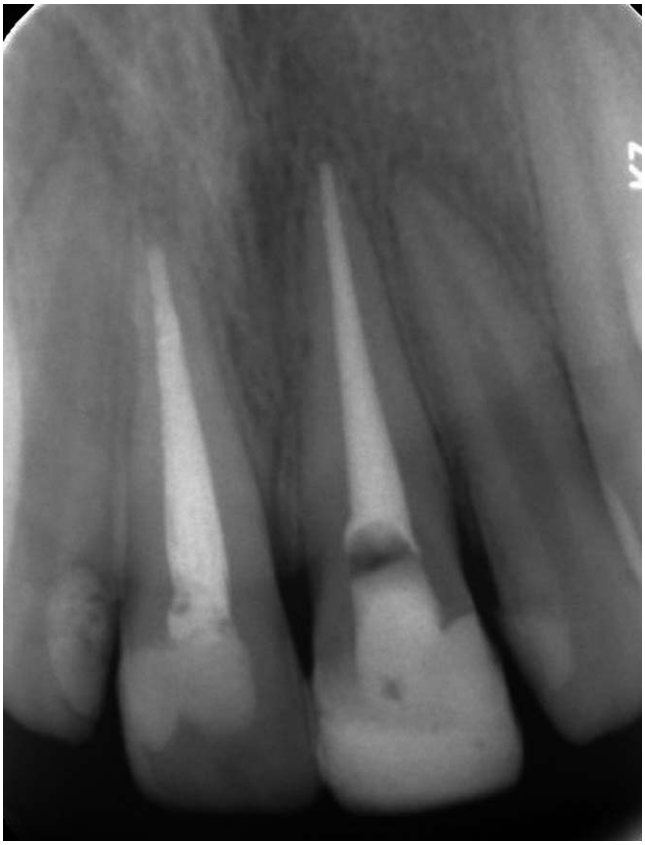

Figure 9. *Periapical radiograph of the upper central incisors after treatment.*

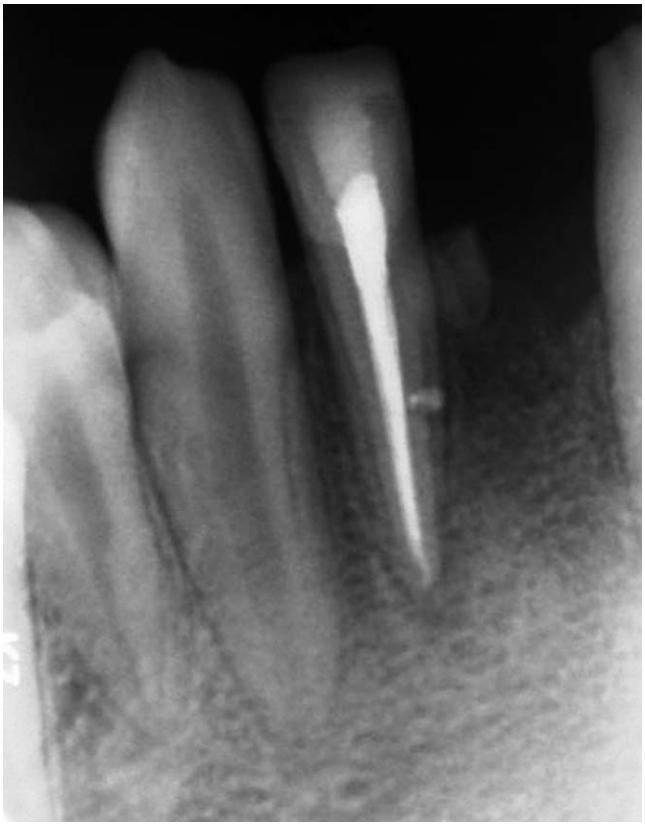

Figure 10. *Periapical radiograph of the lower incisor after treatment.*

of combined injuries of maxillary and mandibular arches is rare.[4,7,8] The reason for this uncommon occurrence may be the different mechanisms of the direction of the sustained force. In the present case, the patient had five injured teeth, two of which were in the mandibular arch and three in the maxillary arch. In the opinion of the authors, the direct impact to the patient's face caused the simultaneous injuries to the maxillary and mandibular arches.

In traumatic injuries, treatment options depend on such factors as injury type, affected teeth, and time elapsed after trauma. In avulsion cases, the current evidence indicates that immediate replantation favors a successful outcome.[6,13,15,19] In the current case, the patient could not locate the avulsed teeth. Therefore, because of the patient's age, the treatment option was a removable prosthesis.

In extrusion cases, the most successful outcomes of extrusion injury occur where the tooth is returned to the original position as soon as possible after trauma. Delays in seeking treatment, poor cooperation, and severity of the traumatic injuries can result in incomplete repositioning of teeth at the time of injury. In cases where the tooth cannot be repositioned because of blood clot blockage or as a result of delayed treatment, a different option, such as orthodontic intrusion for tooth repositioning, may be considered.[6,13] In the present case, orthodontic intrusion with endodontic management was chosen as the treatment option. To reposition the tooth, intrusive orthodontic tooth movement was necessary. Because of the severity of the injury, endodontic treatment was indispensable. If neglected, infection-related root resorption could have been a distinct and dangerous possibility.[12,13,20] In the current case, root canal treatment was carried out at the same time as orthodontic treatment. The reports suggest that extruded, laterally luxated, intruded, and replanted teeth have an increased risk of apical resorption after orthodontic treatment. Therefore, calcium hydroxide was maintained in the canals and changed once a month for six months to reduce the risk of resorption with healing resorptive areas and increase osteoblastic activity.

Pulp necrosis is the most common posttraumatic complication. It occurs mostly in teeth that have had injuries to the periodontal tissues. Complications after uncomplicated crown fractures are rather

uncommon. A concomitant luxation injury has been reported to increase the prevalence of pulp necrosis in teeth with crown fractures.[13,17] A late diagnosis of posttraumatic pulp necrosis can result in the manifestation of additional complications, such as inflammatory root resorption. Root resorption is a serious complication after luxation injuries in permanent teeth.[13,20] In the present case, because of the delay in obtaining treatment, the current authors did not know whether a simultaneous luxation with uncomplicated crown fracture happened when the trauma occurred. However, when the patient presented, the teeth with uncomplicated crown fractures were found to be nonvital. Nevertheless, there was no root resorption. In the opinion of the authors, this lack of resorption was a result of the short time after the trauma occurred.

CONCLUSION

Traumatic injuries may cause functional, esthetic, and psychological problems. A delay in treatment could make these problems more difficult and complicated to treat. These types of injuries can require a multidisciplinary treatment approach. Dentists who first meet a patient with trauma must have sufficient knowledge and equipment for all treatment options to solve traumatic injuries that require a multidisciplinary approach.

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.

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Bleaching Options for Pulp-Calcified Teeth: Case History Reports

DP Lise • C Gutiérrez • TP da Rosa
LCC Vieira

Clinical Relevance

This technique is a conservative approach to recover the esthetics of calcified teeth.

SUMMARY

The aim of this article is to review some essential aspects of anterior tooth calcification and its esthetic treatment. Furthermore, three cases including different and successful bleaching strategies are reported.

INTRODUCTION

The psychosocial well-being of a person can be influenced by smile attractiveness. Tooth color has particular cosmetic importance and is readily perceived by people. In many cases, just one discolored tooth can compromise the entire smile harmony.[1] The real cause of discoloration should be properly diagnosed in order to conduct a correct treatment.[2]

Trauma to dentition can result in a calcific pulpal response, and in 67% to 79% of affected teeth, the clinical crown exhibits yellow discoloration.[3,4] Pulp canal obliteration, or calcific metamorphosis, is a sequela of tooth trauma characterized by the pronounced deposition of hard tissue (tertiary dentin) within the pulp chamber and root canal space.[5] This decreases the translucency of dentin. Dentin confers the basic hue of the tooth, while enamel just modulates the chroma and the value according to its thickness. The result is a yellowish appearance of the crown in about 3 months or 1 year after the injury.[6-8] The color appearance can be modified in case of any change to the structural composition or thickness of these structures.[9]

It is generally accepted that the frequency of pulp canal obliteration is dependent on the extent of the traumatic injury and the stage of root formation.[10] Partial or total obliteration of the root canal space is reported to develop more often in anterior teeth of young adults with a history of concussion and subluxation injuries.[5] Approximately 4% to 24% of traumatized teeth develop varying degrees of pulp canal obliteration.[11,12]

The exact mechanism of canal obliteration is unknown, but several hypotheses have emerged to

*Diogo Pedrollo Lise, MSD, MSD, PhD student, Universidade Federal de Santa Catarina, Florianópolis, Brazil

Celso Gutiérrez, MSD, Universidade Federal de Santa Catarina, Florianópolis, Brazil

Tiago Pereira da Rosa, MSD, PhD student, Universidade Estadual de Campinas, Piracicaba, Brazil

Luiz Clovis Cardoso Vieira, PhD, professor, Universidade Federal de Santa Catarina, Florianópolis, Brazil

*Corresponding author: Sala 138, Universidade Federal de Santa Catarina, Campus Universitário Trindade, Florianópolis, Santa Catarina 88040-970, Brazil; e-mail: diogolise@hotmail.com

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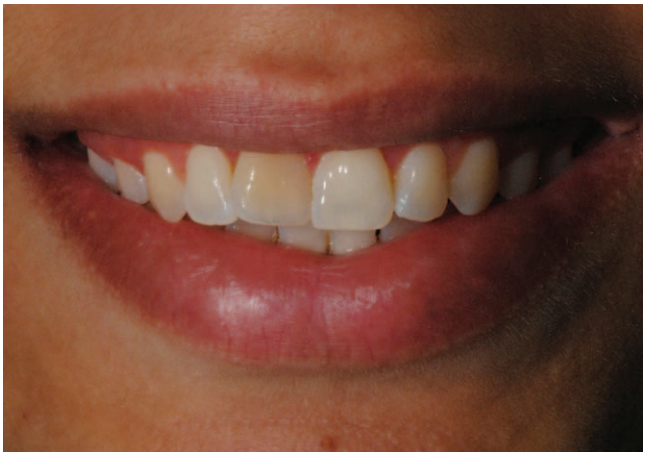

Figure 1. *Discolored anterior tooth No. 8.*

explain the event. Avery[13] suggested that the pronounced deposition of hard tissue could result from a reduced vascular flow in the pulp leading to tissue respiratory depression and subsequent pathological mineralization. Torneck[8] hypothesized that

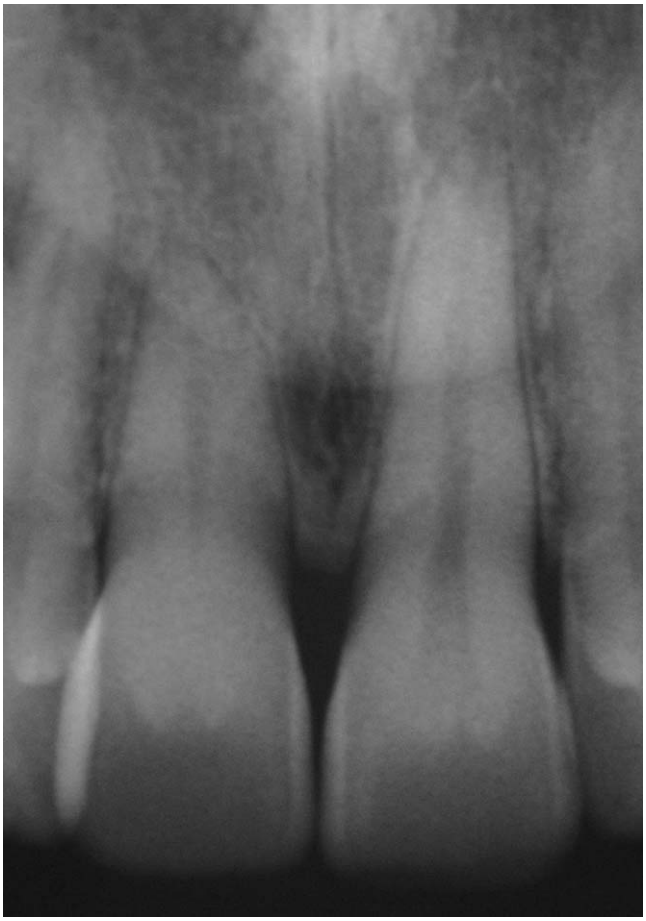

Figure 2. *X-ray shows pulp chamber obliteration.*

Table 1: *Materials Used*

Bleaching product	Manufacturer
Powerbleaching 10%	BM4 Materiais Odontológicos, Palhoça, SC, Brazil
Powerbleaching Office 37%	BM4 Materiais Odontológicos, Palhoça, SC, Brazil

the phenomenon could be either a result of stimulation of the preexisting odontoblasts or loss of their regulatory mechanism. Other authors believe that the calcification occurs in response to a severe injury to the neurovascular supply of the pulp, which, after healing, leads to accelerated dentin deposition.[14-16]

Clinically, there is a progressive decrease in the response to thermal and electrical pulp testing as the deposition of hard tissue becomes more pronounced.[4,17] Nevertheless, it is generally accepted that a negative response to sensitivity tests does not automatically imply pulp necrosis.[4,18]

Although there are different points of view regarding the management of canal-obliterated teeth, studies indicate that the incidence of pulp necrosis varies from 1% to 27% of cases, suggesting that a change in tooth color is not a reliable indication of pulp or periapical pathosis.[4,11,19] Even a complete radiographic obliteration does not necessarily mean the absence of the pulp or canal space; in most of these cases, there is a pulp canal space with pulpal tissue.[8,11,17] Histopathologic studies designed to assess the pulpal status of teeth with pulp canal obliteration have also failed to show any inflammatory component indicative of a pathologic process.[8,20]

The following three case reports describe the management of anterior calcified and discolored teeth, in which different bleaching strategies were used to solve the patients' esthetic discomfort.

CLINICAL CASE REPORTS

Case 1

A 24-year-old woman presented to the Federal University of Santa Catarina complaining of the yellowish appearance of her anterior tooth (No. 8) as a result of a trauma that had occurred 10 years before (Figure 1). Radiographic evaluation revealed pulp chamber obliteration and no apical lesion (Figure 2). The patient manifested no response to thermal pulp testing. She used a home 10% carbamide peroxide gel (Table 1) placed in a customized tray with windows cut out of the tray adjacent to the

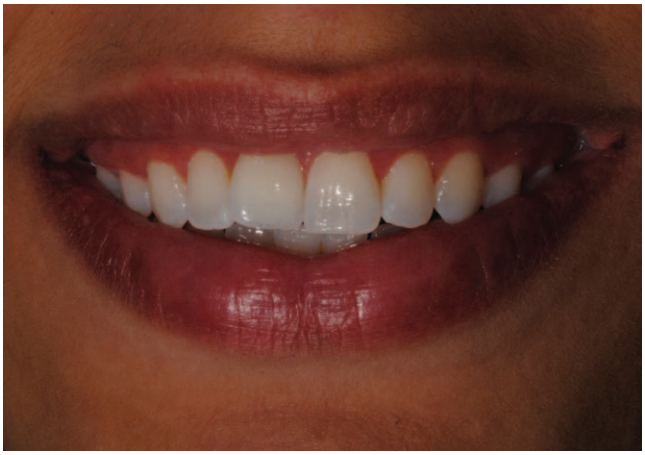

Figure 3. *Final aspect after 21 days of at-home tooth bleaching.*

discolored tooth, on either side. The patient was instructed to use the tray during the daytime (1 hour per day). After 3 weeks, tooth No. 8 presented satisfactorily with the same color as adjacent teeth, and an excellent outcome was obtained (Figure 3).

Case 2

A 35-year-old man presented to the dental office unsatisfied with his tooth No. 9, which had gradually become dark yellow (Figure 4) after labial trauma 20 years before. Radiographic evaluation demonstrated pulp chamber and root canal obliteration without apical lesion (Figure 5) and no response to thermal stimulus. It was proposed to proceed with in-office whitening sessions to assess the color change. The patient was submitted to a 1-hour session with an in-office bleaching gel composed of 37% carbamide peroxide (Table 1) three times per week. Because of its lower concentration of hydrogen peroxide, the gel did not require application of a gingival barrier (Figure 6). After 3 weeks, a pleasant esthetic result was achieved (Figure 7).

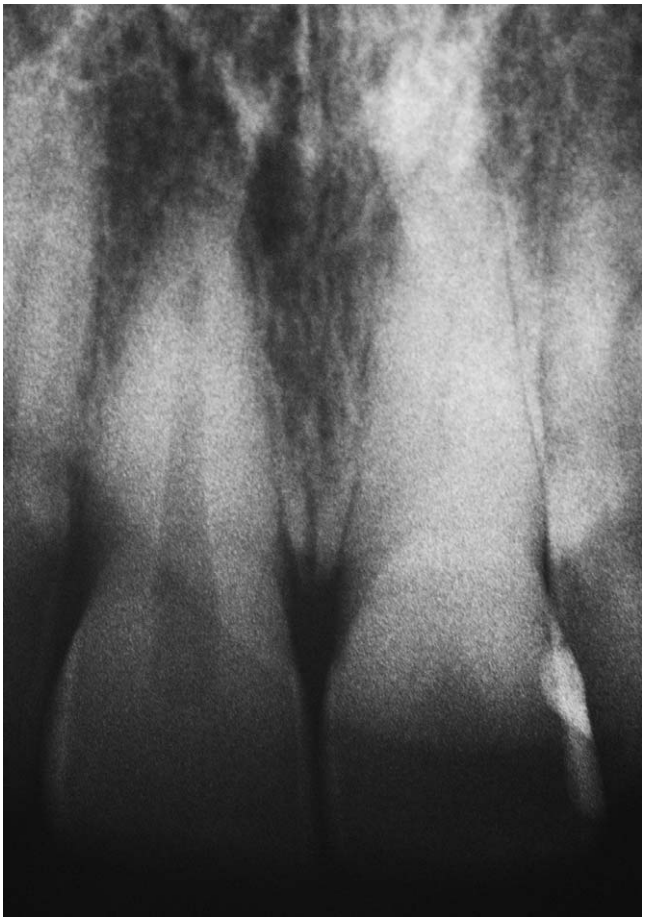

Figure 5. *X-ray reveals complete obliteration of pulp chamber and root canal.*

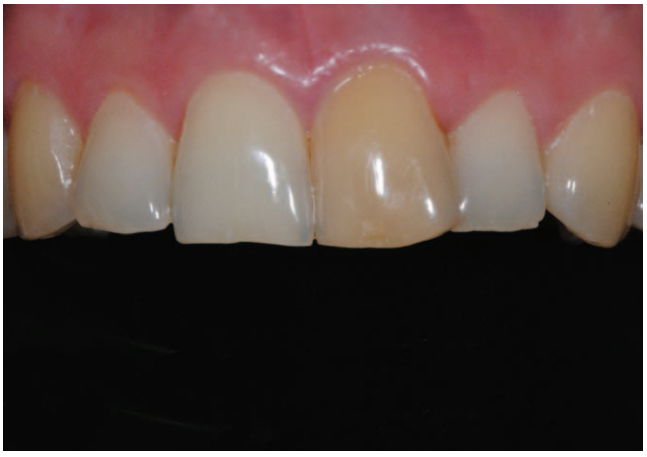

Figure 4. *Intraoral view of darkened tooth No. 9.*

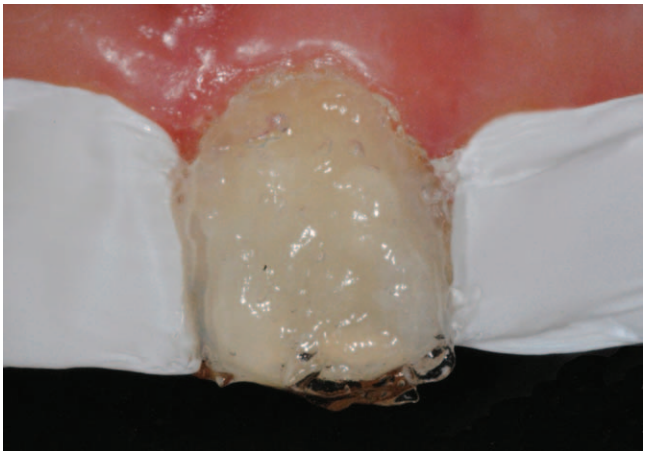

Figure 6. *Thirty-seven percent peroxide carbamide in-office gel applied over the tooth and without gingival barrier.*

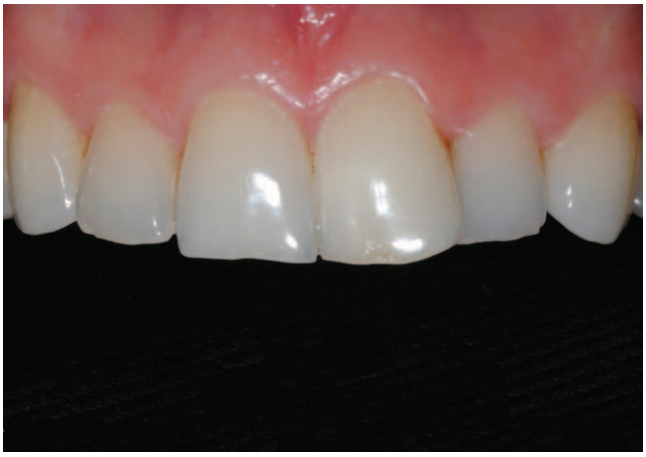

Figure 7. *Aspect after nine in-office whitening sessions.*

Case 3

A 25-year-old woman suffered a fall 11 years before, and since then, her tooth No. 8 had become discolored (Figure 8). Despite radiographic evaluation showing a periapical lesion, the patient related that she never felt pain or any other discomfort since the trauma (Figure 9). Thermal pulp testing was negative. The use of a 37% carbamide peroxide in-office gel was indicated. The patient received a customized at-home bleaching tray and was instructed to use it 1 hour per day (Figure 10). After 9 days, tooth No. 8 reached the same color as the adjacent teeth (Figure 11).

Potential Problems

Because of esthetic concerns, West[21] considered that there are potentially four treatment options for restoring discolored pulpally obliterated teeth to an acceptable color:

- External or vital bleaching that should be considered first as it is the most conservative option
- An intentional root canal treatment followed by intracoronal bleaching
- Internal and external bleaching without root canal treatment
- Extracoronal full or partial coverage restorations

The diagnostic status and treatment-planning decisions regarding teeth with obliterated root canal remain controversial. Based on two clinical parameters, Rock and Grundy[7] suggested that root canal treatment should be performed as soon as narrowing of the pulp chamber shadow is seen radiographically: 1) once the guidance afforded by the pulp canal is lost, it is more difficult to prepare a post hole without penetrating the periodontal ligament, and 2) should necrosis occur in the remaining apical tissue, the only possible access may be surgical intervention. According to Fischer,[22] a pulp undergoing obliteration has a reduction of cellular components, which makes it more susceptible to infection and limits the tissue's ability to heal.

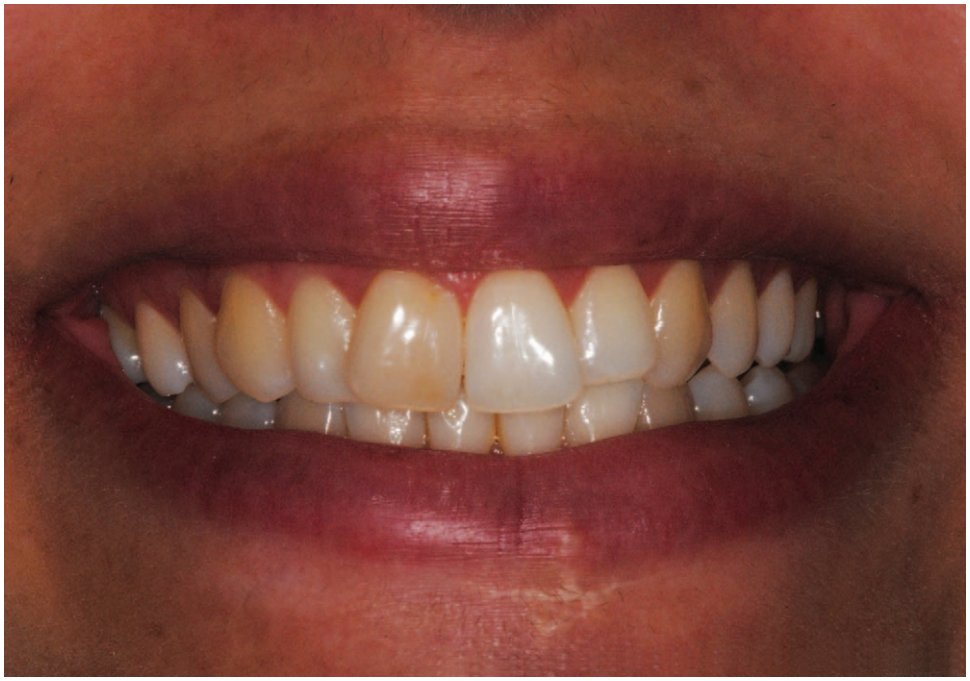

Figure 8. *Initial smile aspect with discolored tooth No. 8.*

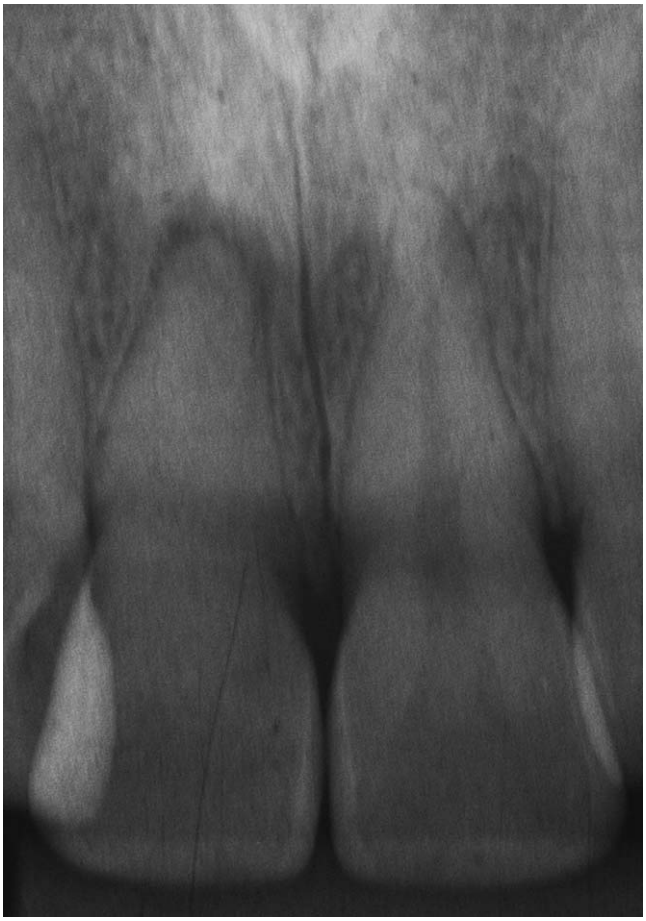

Figure 9. *X-ray demonstrating complete obliteration of pulp chamber and root canal.*

Scientific data produced in the past years demonstrate that peroxide-based tooth-whitening products are safe and effective. When properly indicated, there is no evidence of significant health risks.

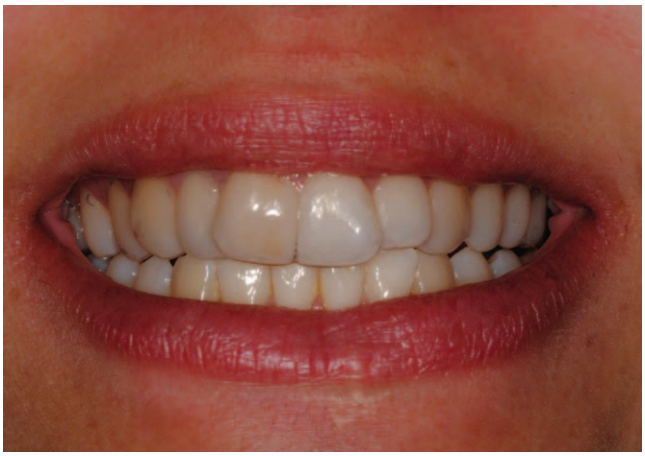

Figure 10. *Patient using a customized bleaching tray filled with 37% carbamide peroxide gel.*

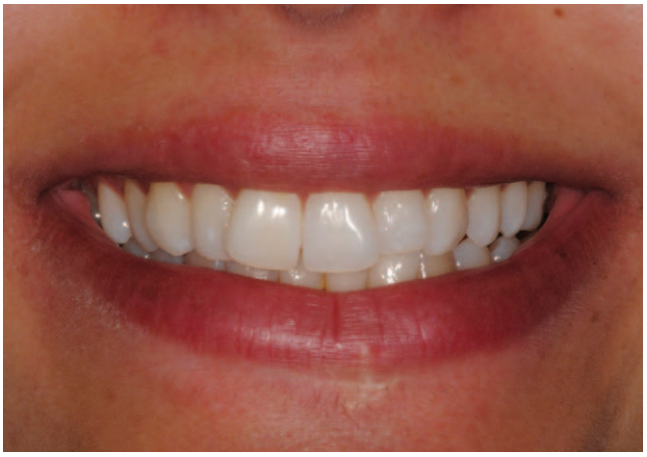

Figure 11. *Final smile aspect after nine days of bleaching.*

Sensitivity and gingival irritation are usually mild and transient.[23]

Most current materials use carbamide peroxide ($CH_6N_2O_3$) and hydrogen peroxide (H_2O_2) as active ingredients for tooth bleaching regardless of in-office or at-home strategies.[24-26] Chemically, carbamide peroxide is composed of approximately 3.5 parts of H_2O_2 and 6.5 parts of urea, so that a bleaching gel of 10% carbamide peroxide provides about 3.5% of H_2O_2.[27] Typically, H_2O_2 concentrations used for in-office bleaching range from 25% to 40%. There is a greater prevalence reported of gingival irritation associated with bleaching products containing higher H_2O_2 concentration.[28,29]

In cases 2 and 3, a 37% peroxide-carbamide gel was used. It contains 12.95% of hydrogen peroxide after its dissociation. In both strategies (in-office and at-home), no gingival irritation was related after its application without any gingival barrier. Furthermore, patients did not report sensitivity of vital adjacent teeth. The use of this whitening product demonstrated a faster result than the traditional 10% carbamide peroxide gel used in case 1.

Advantages

- Conservative treatment not involving tooth anatomy and texture modifications
- No effect on future restorative procedures
- Lower cost than other restorative strategies
- Little or no sensitivity during the whitening treatment

Limitations

- Bleaching progress can be slow because of the lower permeability of tertiary dentin.
- In some cases, the whitening level cannot totally match adjacent teeth.

CONCLUSIONS

- Considering the high incidence of dental trauma, the dentists' working knowledge regarding treatment possibilities of discolored teeth is essential.
- Bleaching of calcified teeth should be performed whenever possible because it is a simple, conservative, and affordable procedure.
- Whitening of calcified teeth using 37% peroxide-carbamide gel provided excellent results.

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.

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Clinical Research

Seven-Year Clinical Performance of Resin Composite Versus Resin-Modified Glass Ionomer Restorations in Noncarious Cervical Lesions

TC Fagundes • TJE Barata • E Bresciani
SL Santiago • EB Franco • JRP Lauris
MF Navarro

Clinical Relevance

Long-term evaluation of material behavior is relevant for class V restorations. The resin-modified glass ionomer studied (Vitremer) showed superior clinical long-term retention rates compared with two-step etch-and-rinse adhesive and resin composite (Excite/Tetric Ceram) restorations.

*Ticiane Cestari Fagundes, DDS, MS, PhD, assistant professor, Department of Restorative Dentistry, Araçatuba School of Dentistry, UNESP - Univ Estadual Paulista, Sao Jose dos Campos, Brazil

Terezinha JE Barata, DDS, MS, PhD, assistant professor, Department of Preventive Dentistry and Oral Rehabilitation, Federal University of Goias–Dental School, Goiania, Brazil

Eduardo Bresciani, PhD, assistant professor, Department of Restorative Dentistry, Institute of Science and Technology, UNESP - Univ Estadual Paulista, Sao Jose dos Campos, Brazil

Sérgio Lima Santiago, PhD, associate professor, Restorative Dentistry, Federal University of Ceará, Fortaleza, Brazil

Eduardo B Franco,DDS, MS, PhD, professor, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Bauru, Brazil

José Roberto P Lauris, MS, PhD, associate professor, Department of Pediatric Dentistry, Orthodontics and Social Dentistry, Bauru School of Dentistry, University of São Paulo, Bauru, Brazil

Maria Fidela Navarro, DDS, PhD, professor, Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Bauru, Brazil

*Corresponding author: Rua José Bonifácio, 1193, Araçatuba, Sao Paulo 16015-050, Brazil; e-mail: ticiane_f@hotmail.com

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SUMMARY

Purpose: The purpose of this study was to comparatively assess the seven-year clinical performance of a one-bottle etch-and-rinse adhesive with resin composite (RC) and resin-modified glass ionomer (RMGI) restorations in noncarious cervical lesions.

Methods and Materials: One operator placed 70 restorations (35 restorations in each group) in 30 patients under rubber dam isolation without mechanical preparation. The restorations were directly assessed by two independent examiners, using modified US Public Health Service criteria at baseline and 6, 12, 24, 60, and 84 months. The obtained data were tabulated and statistically analyzed using the Fisher and McNemar tests. A difference was significant if $p<0.05$.

Results: Twenty patients were available for recall after seven years (66.6%), and 25 RC and 26 RMGI restorations out of 70 restorations were evaluated. Excellent agreement was registered for all criteria between examiners ($\kappa \geq 0.85$). Alfa and bravo scores were classified

as clinically acceptable. The McNemar test detected significant differences within RC restorations between baseline and seven-year evaluations for anatomic form, marginal integrity, and retention ($p<0.05$). For RMGI restorations, a significant difference was identified for marginal integrity ($p<0.05$). As to material comparison, the Fisher exact showed a better retention performance for RMGI restorations than for RC restorations ($p<0.05$). Twelve composite restorations were dislodged (52.0% retention) and three ionomer restorations were lost (88.5% retention). The cumulative success rate for RC and RMGI was 30% and 58.1%, respectively.

Conclusions: After seven years of service, the clinical performance of RMGI restorations was superior to that of the adhesive system/resin composite restorations in this study.

INTRODUCTION

About a quarter of the population has noncarious cervical lesions, and these lesions are significantly more prevalent at older ages, with premolars being the most affected teeth.[1]

Although the dentin characteristics of surfaces from abrasion/abfraction lesions may inhibit dentin etching and the formation of hybrid layers,[2] clinical bonding effectiveness has been shown in noncarious cervical lesions located mainly in dentin.[3] Therefore, it is expected that the clinical performance of restorations placed in noncarious cervical lesions would be more critical, especially in long-term evaluations.

Numerous studies have investigated the clinical behavior of noncarious cervical lesions restored with resin composite and glass ionomer cement. A large number of clinical trials are short-term evaluations. In general, in up to three years of follow-up, both resin composite and glass ionomer restorations have good performance.[4-11] The main causes of failure of this type of restoration are low retention rates for resin composite restorations[5,6,12-14] and poor color stability for glass ionomer restorations.[15,16]

Studies with longer evaluation periods show that resin composite materials tend to fail with time. Retention rates of such restorations are markedly lower when compared with glass ionomer restorations.[17,18] A 13-year follow up of class V noncarious cervical lesions verified a continuous degradation of the bond with a wide variation, independent of the adhesion strategy used, with the best retention for restorations performed with resin-modified glass ionomer cement (RMGI) and with adhesive restorations using four-step etch-and-rinse systems.[3]

A review of prospective clinical trials showed that restorations with glass ionomer resulted in the highest success rate in regard to retention if compared with adhesive systems and resin composite restorations.[18]

Long-term evaluations are especially interesting when considering the behavior of one-bottle or primer-adhesives. Currently, some problems have been identified with this category of adhesive system, such as permeability[19] and hydrolysis. Although a tendency exists toward adhesives with simplified application procedures, simplification appears to induce a loss of effectiveness in restorations of noncarious cervical lesions.[18]

Long-term clinical trials are certainly needed, because they remain the ultimate way to collect scientific evidence on the clinical effectiveness of restorative treatments. Thus, the aim of this study was to assess the seven-year clinical performance of a two-step etch-and-rinse adhesive system and resin composite system in comparison with a RMGI restorative material in noncarious cervical lesions in a prospective study.

METHODS AND MATERIALS

Patient Selection

Thirty volunteers (18 to 50 years of age) were properly instructed about the condition and objectives of the study and signed an informed consent and authorization form in order to participate in this investigation, following the guidelines of the local institutional review board (approved on February 24, 2000). Inclusion criteria were good oral hygiene, no periodontal disease or deleterious habits, and presence of at least two noncarious cervical lesions, following the split-mouth group design.

No lesions were less than 1 mm deep. Each patient randomly received at least one adhesive system/resin composite and one resin-modified glass ionomer restoration, for a total of 70 restorations. All types of class V noncarious lesions were included in the present study. As the association of abrasion, erosion, and abfraction is usually observed, all situations were considered for inclusion. Although the degree of dentin sclerosis was not considered an exclusion criterion, highly mineralized pigmented lesions were not observed.

Table 1: *Composition of the Studied Materials*

Material	Composition
Excite	*Primer/Adhesive:* Phosphono-acid acrylate, hydroxyethyl methacrylate, Bis-GMA, dimethacrylate, highly dispersed silica, ethanol, catalysts, and stabilizers.
Tetric-Ceram	TEGDMA, barium glass, ytterbium trifluoride, barium-aluminum-fluorosilicate glass, highly dispersed silica, additives, catalysts, stabilizers, and pigments
Vitremer	*Primer:* polyacrylic acid modified with grafted pendant HEMA groups, 2-HEMA, ethanol, photocuring initiators.
	Powder: fluoroaluminosilicate glass (70 wt%), potassium persulphate and ascorbic acid (patented catalyst system), benzoyl peroxide.
	Liquid: a copolymer of polymaleic acid and HEMA= polyacrylic acid modified with grafted pendant HEMA groups (15 wt%), 2-HEMA (5 w%), water (8 wt%), camphorquinone, photoinitiator (microencapsulated water-soluble ascorbic acid/potassium persulfate).

Abbreviations: Bis-GMA, bisphenol A diglycidylmethacrylate; TEGDMA, triethyleneglycol-dimethacrylate; 2-HEMA, 2-hydroxyethyl methacrylate.

Restorative Procedures

Each patient received at least two or a multiple of two restorations. Each restorative material was randomly allocated to one of the randomized cervical lesions until the two treatments were present in the same subject and in equal numbers. The randomization process was performed by placing all the selected teeth on a list and assigning treatment according to a predefined sequence: 1) resin composite and 2) RMGI.

Restorative procedures were carried out by an experienced clinician, who was submitted to a calibration process that consisted of performing 10 repeated restorations of each material under direct supervision of the project's coordinator. The operator's questions were addressed and consensus was obtained during the calibration session. All restorations were placed under rubber dam isolation, and no cavity preparation was carried out. Enamel margins were not beveled and no mechanical retention was performed. Twenty-six patients received two restorations, three patients received four restorations, and one patient received six restorations.

Half of the cavities were restored with a two-step etch-and-rinse adhesive system and a resin composite (Excite/Tetric Ceram, Ivoclar Vivadent, Schaan, Liechtenstein) and the other half with the RMGI (Vitremer, 3M ESPE, St Paul, MN, USA) (Table 1), according to the manufacturer's instructions, as follows:

Excite/Tetric Ceram—Enamel was etched for 30 seconds and dentin for 15 seconds with 37% phosphoric acid gel (Total Etch, Ivoclar Vivadent), washed for 30 seconds, and dried gently. Absorbent paper was used to remove the excess water. One coat of the two-step etch-and-rinse adhesive system (Excite, Ivoclar Vivadent) was applied to the visibly moist dentin surface and brushed gently for 10 seconds. An air blast was applied to facilitate the evaporation of the alcohol solvent and the primer adhesive was light-cured for 20 seconds. Resin composite (Tetric Ceram, Ivoclar Vivadent) increments were inserted and light-cured for 40 seconds using a calibrated light-curing unit (XL 3000, 3M ESPE) at 600 mW/cm^2 with a curing radiometer (Demetron/Kerr, Orange, CA, USA). Composite excess was immediately removed using a No. 12 blade. Finishing and polishing was performed one week later using 12-fluted tungsten carbide burs (FG Bur, KG Sorensen, Brazil), abrasive cups (Enhance, Dentsply, USA), and disks (Sof-Lex polishing disks, 3M ESPE);

Vitremer—The RMGI (Vitremer, 3M ESPE) was used according to the manufacturer's instructions. Primer was applied for 30 seconds to the surface of the lesion using a micro-brush. Light-curing was conducted for 20 seconds using the same calibrated device that was used for resin composite restorations. The RMGI was manipulated in a 1:1 powder-to-liquid ratio and was inserted using disposable tips and a syringe (Centrix, Shelton, CT, USA). The restoration was light-cured for 40 seconds and excess material was immediately removed using a No. 12 surgical blade. Finishing and polishing were carried out similarly to resin composite restorations, one week after the restorations were placed.

Clinical Evaluation

Two independent and calibrated examiners other than the operator were responsible for the clinical evaluations. Calibration procedures were carried out using picture slides representing each condition to be assessed in the study. A double-blind design was originally assigned. In cases where the two examiners disagreed on a rating, both reexamined the restoration and arrived at a final joint decision.

Table 2: *Modified US Public Health System Criteria Rating System*

Category	Rating	Criteria
Retention	Alfa (A)	Restoration is present
	Charlie (C)	Restoration is partially or totally lost
Marginal integrity	Alfa (A)	No visible gap in which the explorer will penetrate
	Bravo (B)	There is a visible gap; the explorer will penetrate or catch
	Charlie (C)	The explorer penetrates the gap and dentin or base is exposed
	Delta (D)	The restoration is mobile, partially or totally fractured or lost
Marginal discoloration	Alfa (A)	No discoloration
	Bravo (B)	Discoloration is present but has not penetrated along all the margin
	Charlie (C)	Discoloration has penetrated along all the margin
Anatomic form	Alfa (A)	Restoration is continuous with existing anatomic form
	Bravo (B)	Restoration is discontinuous with existing anatomic form, but dentin or base is not exposed
	Charlie (C)	Sufficient material is lost to expose dentin or base
Secondary caries	Alfa (A)	No caries is present at the margin of the restoration
	Charlie (C)	There is evidence of caries at the margin of the restoration

Modified US Public Health Service criteria[20] were used to evaluate retention, marginal integrity, marginal discoloration, anatomic form, and secondary caries (Table 2) at baseline and 6, 12, 24, 60, and 84 months. The baseline rating was carried out one week after restoration, immediately after the finishing and polishing procedures.

Statistical Methods

Interexaminer agreement was assessed using κ. Excellent agreement was registered between both examiners for all criteria ($\kappa \geq 0.85$). Intragroup comparisons between baseline and other evaluation periods with the same material were performed by the McNemar test ($p<0.05$). Intergroup comparisons to identify differences between restorative materials at each period were conducted by Fisher exact tests ($p<0.05$). The cumulative survival rate was determined following the life table method.

RESULTS

Recall rates registered were 100% for baseline, 6, and 12 months; 93.3% for two years; 73.3% after five years; and 66.6% after seven years. The registered recall rates overcame the minimum requirement of the American Dental Association (ADA) guidelines[21] for sample size in clinical trials for restorative materials, which state that a minimum of 20 patients must be available for the two-year recall and 15 patients must be examined at the four-year evaluation.[21]

Data for retention, marginal integrity, marginal discoloration, anatomic form, and secondary caries of all evaluation periods are presented in Table 3. Numbers in parentheses indicate the total number of restorations classified as clinically acceptable (alfa and bravo ratings) in each evaluation period versus the total number of restorations assessed at that time.

Given that all patients were available for recall at 6 and 12 months, all 70 restorations could be assessed. Although four resin composite restorations were lost at six months and one more was lost at the one-year assessment, no significant differences were detected in all criteria for both materials at these periods.

At the two-year recall, two patients could not be found, so 66 restorations were assessed. Seven composite restorations were lost, whereas no ionomer restoration was lost. When comparing resin composite and RMGI restorations at the two-year recall, the only significant difference was for the retention criterion ($p=0.011$) in favor of RMGI restorations.

At the five-year recall, 22 volunteers returned for evaluation. Of 70 restorations, 55 were evaluated (27 resin composite restorations and 28 resin-modified glass ionomer restorations). Sixteen composite restorations were regarded as failures at this evaluation period (51.5% retention), whereas only one ionomer restoration was lost (96.4% retention). Significant differences were found for resin composite restorations between baseline and the five-year recall for marginal integrity and retention. For RMGI restorations, no significant differences were identified for all criteria. When comparing both materials, significant differences for retention after five years were found.

Table 3: *Clinical Evaluation of Resin Composite and Resin-Modified Glass Ionomer Restorations With Percentages Values of Clinically Acceptable Ratings (Alfa and Bravo)*

Category	Material	Baseline % A+B	6 months % A+B	1 year % A+B	2 years % A+B	5 years % A+B	7 years % A+B
Retention	RC	100% (35/35)[a]	88.2% (30/34)	85.7% (30/35)	78.8% (26/33)	51.5% (17/27)	52.0% (13/25)
	RMGI	100% (35/35)	100% (34/34)	100% (35/35)	100% (33/33) *	96.4% (27/28) *	88.5% (23/26) *
Marginal integrity	RC	97.2% (34/35)	100% (30/30)	100% (30/30)	100% (26/26)	76.5% (13/17)	69.2% (09/13)
	RMGI	100% (35/35)	100% (34/34)	100% (35/35)	100% (33/33)	85.2% (23/27)	87.0% (20/23)
Marginal discoloration	RC	100% (35/35)	100% (30/30)	100% (30/30)	100% (26/26)	100% (17/17)	100% (13/13)
	RMGI	100% (35/35)	100% (34/34)	100% (35/35)	100% (33/33)	100% (27/27)	100% (23/23)
Anatomic form	RC	100% (35/35)	96.6% (29/30)	96.6% (29/30)	96.2% (25/26)	88.2% (15/17)	92.3% (12/13)
	RMGI	100% (35/35)	100% (34/34)	100% (35/35)	100% (33/33)	85.2% (23/27)	91.3% (21/23)
Secondary caries	RC	100% (35/35)	100% (30/30)	100% (30/30)	100% (26/26)	88.2% (15/17)	92.3% (12/13)
	RMGI	100% (35/35)	100% (34/34)	100% (35/35)	100% (33/33)	100% (27/27)	91.3% (23/23)

Abbreviations: RC, resin composite (Excite/Tetric Ceram, Vivadent); RMGI, resin-modified glass ionomer cement (Vitremer, 3M ESPE Dental Products).
[a] *Numbers in parentheses represent restorations evaluated with success/total restorations evaluated.*
* *Indicates significant differences between tested materials for that criterion.*

At the seven-year recall, 20 volunteers returned for evaluation. Of 70 restorations, 51 were evaluated (25 resin composite restorations and 26 RMGI restorations). Twelve composite restorations were considered as failures at this evaluation period due to retention (52.0% retention), whereas three ionomer restorations were lost (88.5% retention). Greater retention was found after seven years if compared with five years for resin composite restorations because one patient who was not evaluated at five years returned after seven years.

The cumulative survival rates of retention for both tested groups were 95.8% and 63.7% for RMGI restorations (Vitremer) and 2-step etch-and-rinse adhesive and resin composite restorations (Excite/Tetric Ceram), respectively (Table 4).

The distribution of restorations regarding location and type of tooth for both tested groups is shown in Table 5. The number of restorations that failed with respect to retention is also cited in Table 5. The ages of the patients whose restorations failed for retention were 30, 35, and 50 for resin-modified glass ionomer restorations; and they were 18, 27, 32, 35, 36, 37, 40, 45, and 50 years for resin composite restorations.

The McNemar test detected significant differences for resin composite restorations between baseline and the seven-year recall for retention ($p=0.002$). For RMGI restorations, a significant difference was identified for marginal integrity ($p<0.05$, Figure 1). When comparing both materials, the Fisher exact test pointed out a significantly better performance for RMGI restorations than for resin composite restorations with respect to retention ($p<0.01$).

DISCUSSION

Noncarious class V clinical trials remain the ultimate testing method for the assessment of bonding

Table 4: *Cumulative Survival Rate of Retention for Both Tested Groups*

Material	Interval	Pair of Restorations at Start of Interval	Dropouts	Pair of Evaluated Restorations	Failures	Success	Survival Rate, %	Cumulative Survival Rate, %
RC	0-6 m	35	1	34	0	34	100.0	100.0
	6 m-1 y	34	3	31	3	28	90.8	90.8
	1-5 y	28	3	25	0	25	100.0	90.8
	5-7 y	25	3	22	7	15	70.2	63.7
RMGI	0-6 m	35	1	34	0	34	100.0	100.0
	6 m-1 y	34	6	28	0	28	100.0	100.0
	1-5 y	28	3	25	0	25	100.0	100.0
	5-7 y	25	2	23	1	22	95.8	95.8

Abbreviations: RC, resin composite (Excite/Tetric Ceram, Vivadent); RMGI, resin-modified glass ionomer cement (Vitremer, 3M ESPE Dental Products).
* *Wilcoxon (Gehan):* $p = 0.008$ *statistically significant difference.*

Table 5: *Distribution of Restorations Regarding Location and Type of Tooth for Both Tested Groups at Baseline. Numbers in Parentheses Represent Restorations Evaluated With Failure of Retention After 7 Years.*

Material	Location		Type			
	Upper	Lower	Premolar	Molar	Incisor	Canine
RC	26 (11)	9 (3)	27 (11)	4 (2)	1	3 (1)
RMGI	27 (4)	8 (1)	31 (5)	1	1	2

Abbreviations: RC, resin composite (Excite/Tetric Ceram, Vivadent); RMGI, resin modified glass ionomer cement (Vitremer, 3M ESPE Dental Products).

effectiveness.[22] Due to a lack of inherent macromechanical retention, adhesion is the most important factor in the retention of restorations in cervical abrasion/erosion lesions.[23] Microtensile adhesion tests have shown a correlation with marginal discoloration of clinical studies on class V nonretentive restorations; however, no significant correlation was found with the clinical index, retention rate, or marginal integrity.[24]

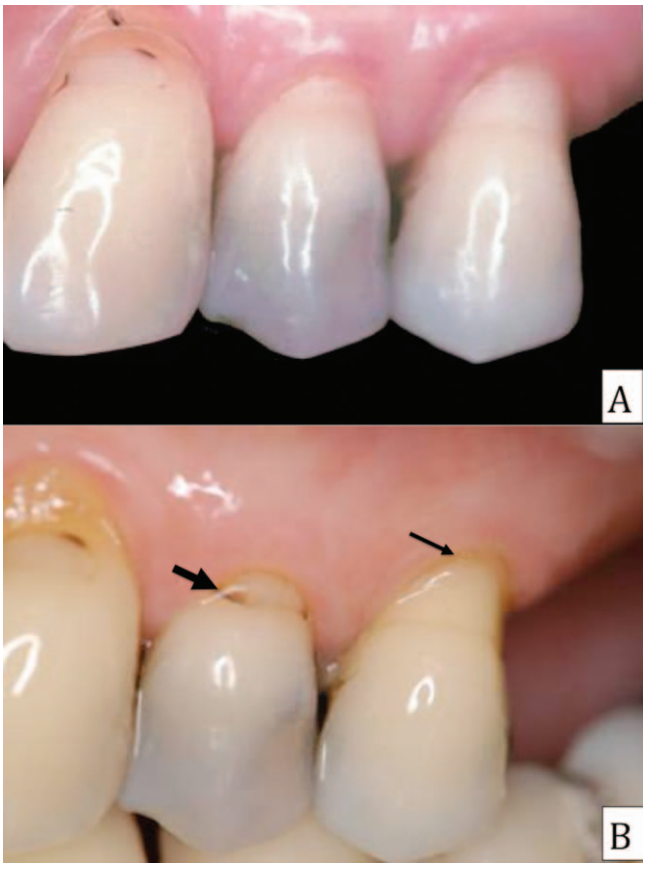

Figure 1. *The cervical lesions on both upper premolars were restored (tooth No. 14, Excite/Tetric; tooth No. 15, Vitremer). (A): Baseline. (B): Seven-year follow-up. At the 7-year recall, the Excite/Tetric restoration showed a small marginal defect and severe marginal discoloration at the cervical enamel margin (big arrow). The restoration was clinically unacceptable and needed to be replaced. The restoration of tooth No. 13 showed only a small marginal defect on the dentin side (small arrow).*

Adhesive materials such as resin composites, RMGI cements, or polyacid-modified resin composites have been mostly used to restore noncarious cervical lesions.[18] This study investigated the clinical performance of a two-step etch-and-rinse/resin composite system and RMGI cement. Retention was considerably lower for resin composite restorations after seven years when compared with RMGI restorations. These results are in accordance with earlier research studies.[3,23,25]

Because no cavity preparation was performed, including no enamel beveling, the true dentin bonding capacity of the restorative systems could be evaluated. Several research studies have used this experimental model to assess the clinical performance of materials in noncarious cervical lesions.[9,12,17]

The loss rate of composite restorations has always been a matter of interest. The low retention rate of resin composites is possibly due to the degradation of the adhesive bond. With former adhesive systems, lost composite restorations composed up to 80% of the total restorations after four years.[17] Other studies confirmed that two-step etch-and-rinse adhesives have shown satisfactory retention rates (80% to 93%) during the first two years of evaluation,[4,26,27] but in the present study the number of lost restorations after seven years of clinical service was considerable. Evidence exists now that two-step etch-and-rinse adhesives are regarded as permeable membranes after polymerization because they lack a comparatively more hydrophobic bonding resin layer.[19] Therefore, they allow the continuous transudation of dentinal fluid and do not provide a hermetic seal of deep dentin,[19] which may accelerate the degradation of the adhesive interface.

Differences of clinical success have been observed according to the type of adhesive system used. High retention rates of etch-and-rinse adhesives after a 24-month evaluation of noncarious cervical lesions were detected, as long as the clinician rubbed the adhesives vigorously onto the dentin surfaces.[7] In noncarious class V lesions, high retention rates were observed in a 13-year clinical evaluation of two

three-step etch-and-rinse adhesives, with values ranging between 85% and 94% depending on the resin composite used.[28] Van Dijken and Pallesen[29] observed that restorations placed with a self-etching primer (Xeno III) and a resin composite (Tetric Ceram) or a modified poly-acid resin composite (Dyract AP) in noncarious cervical lesions showed acceptable clinical rates of retention to dentin surfaces after seven years regardless of the restorative material used. After eight years of clinical service, the clinical effectiveness of a mild two-step self-etch adhesive (Clearfil SE) was excellent.[30] Selective phosphoric acid-etching of the enamel margins had only some minor positive effects in marginal defects/discolorations.[30]

A statistically significant difference was observed in a systematic review[18] of glass ionomer versus two-step etch-and-rinse in noncarious restorations. Glass ionomer restorations showed the highest success rate in regard to retention with a 1.9% mean annual loss of retention; whereas, the mean annual retention loss of two-step etch-and-rinse adhesive systems was 6.2%.[18] Similar observations have been recorded[31] with glass ionomers' retention loss of 2% ± 2%. However, another systematic review[32] about different adhesive systems used for the restoration of noncarious cervical lesions concluded that there is not enough evidence to support one adhesive or bonding strategy over another for the treatment of this type of lesion.

Another challenge is the characteristic of noncarious cervical lesions. Sclerotic dentin and tubule occlusion by mineral crystals are very often present. Additionally, many parts of the wedge-shaped cervical lesion contain a hypermineralized surface that resists acid etching.[2]

The effectiveness of cervical restorations is most often expressed as retention loss relative to the observation time. Hence, retention is one of the most important criteria and is often used to assess the longevity of a restorative material. Retention loss is a robust objective criterion that is affected by a low degree of variability between different examiners in contrast with other clinical criteria. The latest guidelines of the ADA for submission of dentin and enamel adhesive materials require as provisional acceptance a retention rate of at least 95% of the restorations placed at the six-month recall.[21] To obtain full acceptance, retention of 90% after 18 months is required. Unfortunately, the guidelines have no requirements for the long-term durability of adhesive systems. In a systematic review, 96% of glass ionomer and 51% of two-step etch-and-rinse class V restorations got the full ADA acceptance in the observation period of the studies varying between one-half and six years.[18] These results are in accordance with the data obtained in the present study.

High retention rates of Vitremer restorations may be attributed to their adequate mechanical properties and better adhesion to dental tissues.[33,34] Vitremer possesses two adhesion mechanisms: first, an auto-adhesive capacity by forming ionic bonds between the carboxyl groups of polyalkenoic acid and hydroxyapatite[35]; and second, micromechanical interlocking of the polymer.[36] In addition, it is suggested that the clinical retention of an adhesive restoration depends not only on the retention capacity of the adhesive system used but also on the viscoelastic properties of the restorative material tested.[37] It has been reported that the elastic modulus of glass ionomer cements is more similar to enamel and dentin than adhesives are.[38] It has been claimed that flexural deformation of a tooth in the cervical region is at least partly absorbed by restorative material. The materials with a modulus of elasticity similar to that of a tooth when used in cervical restorations tend to bend more like a tooth structure when subjected to a masticatory load and may flex and be retained.[39] The combination of these factors may have been responsible for the higher retention rate of glass ionomer restorations in the present study.

Although RMGI cements have shown good retention rates in the literature, the major problem with this material is poor color stability.[16,23,25,40] Sidhu[41] reviewed some of the existing literature on the clinical performance of RMGI cements and observed an adequate performance in terms of retention. Secondary caries as well as postoperative sensitivity are not a concern for this type of restorative procedure.[41] However, this is not necessarily true for marginal characteristics, surface properties, and color stability.[41] In cases where esthetics is essential, an association of RMGI cement and resin composite has been an optional restorative therapy. The combination of these restorative materials is a good alternative and seems to minimize restoration loss.[12,37,42]

The overall better behavior of ionomer restorations is in accordance with previous papers on short-term[4,15,43-44] or long-term evaluations.[40,45] Few clinical trials suggested a limited longevity for RMGI restorations compared with resin composite restorative materials.[15,46] This difference observed between the present study and other previous studies may be due to cavity or type of lesion. Additionally, the shape

and size of restorations, operator variability, occlusal factors, bonding capacity of the restorative system, application and curing technique used, and other factors during aging of the restoration such as temperature and pH cycles in the mouth[47] could account for the differences between the studies. In a five-year evaluation of class V restorations placed by UK general practitioners, different factors were associated with increased longevity. Greater durability of these restorations could be achieved by improving operator skills, followed by cavity preparation and appropriate material handling.[48] Additionally, Brackett and others[49] observed reduced percentages of clinical success of noncarious class V lesions restored by inexperienced operators due to limitations in the manufacturer's instructions of self-etching adhesives. In the present study, the restorations were performed by only one operator, in order to decrease the effect of the operator factor. This allows ranking of the restorative materials tested but does not give information about variations in durability of the materials due to operator variability.

The cumulative survival rates at the 5- to 7-year follow-up interval in the present study were 95.8% and 63.7% for RMGI restorations and resin composite restorations, respectively. These data are in agreement with long-term studies performed with similar materials, in which better performance of glass ionomer materials was also observed.[3] The comparison of the present data with studies[50] of equivalent evaluation periods revealed similarity to retention rates of resin composite restorations (range, 60% to 74%).

Apparently, light-cured or chemically cured glass ionomer cement continues to be the most retentive material for noncarious cervical lesions.[12-14,18]

CONCLUSION

Despite the limitations of this study, the overall clinical performance of resin-modified glass ionomer restorations (Vitremer) was superior to the two-step etch-and-rinse adhesive and resin composite (Excite/Tetric Ceram) restorations after seven years of evaluation.

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.

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Resin Composite Class I Restorations: A 54-month Randomized Clinical Trial

AKM de Andrade • RM Duarte • FDSC Medeiros e Silva
AUD Batista • KC Lima • GQM Monteiro
MAJR Montes

Clinical Relevance

Restorations in Class I cavities of posterior teeth restored with nanofilled and nanohybrid resin composites showed satisfactory results after 54 months.

Ana Karina Maciel de Andrade, DDS, PhD, Department of Restorative Dentistry, Universidade Federal da Paraíba (UFPB), Cidade Universitária, João Pessoa, Brazil

Rosângela Marques Duarte, DDS, PhD, Department of Restorative Dentistry, Universidade Federal da Paraíba (UFPB), Cidade Universitária, João Pessoa, Brazil

Fábia Danielle Sales Cunha Medeiros e Silva, DDS, PhD, Department of Restorative Dentistry, Universidade Federal da Paraíba (UFPB), Cidade Universitária, João Pessoa, Brazil

André Ulisses Dantas Batista, DDS, PhD, Department of Restorative Dentistry, Universidade Federal da Paraíba (UFPB), Cidade Universitária, João Pessoa, Brazil

Kenio Costa Lima, DDS, PhD, Department of Dentistry, Universidade Federal do Rio Grande do Norte (UFRN), Natal, Brazil

Gabriela Queiroz de Melo Monteiro, DDS, PhD, Dental Materials, School of Dentistry, Universidade de Pernambuco (UPE), Camaragibe, Brazil

*Marcos Antônio Japiassú Resende Montes, DDS, PhD, Dental Materials, School of Dentistry, Universidade de Pernambuco (UPE), Camaragibe, Brazil

*Corresponding author: Faculdade de Odontologia de Pernambuco–UPE, Disciplina de Materiais Dentários, Av. Gen. Newton Cavalcanti, 1650, Camaragibe - PE, 54753-220, Brazil; e-mail: majrm@uol.com.br

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SUMMARY

The objective of this longitudinal clinical randomized trial was to evaluate the clinical performance of a nanofilled and a nanohybrid resin composite in Class I occlusal restorations of posterior teeth over the course of 54 months. Forty-one adolescents participated in the study. The teeth were restored with Adper Single Bond 2 (3M ESPE) and nanofilled (Filtek Z350, 3M ESPE), nanohybrid (Esthet-X, Dentsply) and microhybrid Filtek Z250 (3M ESPE) used as a control. After 54 months, the restorations were evaluated in accordance with the modified United States Public Health Service criteria. The McNemar and Friedman tests were used for statistical analysis, at a level of significance of 5%. Five failed restorations were observed during the follow-up. A change to unacceptable restoration occurred for one Esthet-X, two Filtek Z350, and two Filtek Z250 restorations, which received the clinically unacceptable score, Charlie, for both anatomic form and marginal adaptation. Secondary caries and postoperative sensitivity occurred in one Filtek Z250 and one Filtek Z350 restoration. When the five

evaluation periods (baseline and six, 12, 30, and 54 months) were compared, significant differences were found in the marginal adaptation of Filtek Z250 and Filtek Z350. Significant differences in the roughness criteria (p=0.005) were also observed when the three composites were compared after 54 months (Filtek Z350 > Filtek Z250 > Esthet-X), always within clinically acceptable limits. The materials investigated showed acceptable clinical performance for Class I restoration after 54 months. Long-term reevaluations are necessary for a more detailed analysis of these composites.

INTRODUCTION

Resin-based composites have been used extensively over the past decade to restore posterior teeth.[1] Many clinicians have used this class of materials in posterior stress-bearing areas quite successfully for the last five to 10 years.[2] However, there are some problems associated with resin-based composites in posterior teeth, including occlusal and proximal wear, marginal leakage, discoloration, polymerization shrinkage, and postoperative sensitivity.[3]

Nanotechnology has recently been introduced in dentistry.[4] Nanofillers can be prepared by various techniques, such as flame pyrolysis, flame spray pyrolysis, and sol-gel processes.[5] They can increase the overall filler level as a result of the small size of the particles. In several newer nanocomposites, nanofillers have been included in order to obtain high fracture toughness, longer-lasting polish retention and esthetics, and higher wear resistance.[6,7] As a consequence, manufacturers now recommend the use of nanocomposites for both anterior and posterior restorations.[8]

Nanocomposites comprise nanofilled and nanohybrid materials. While the former is based on nanosized fillers and/or nanofiller clusters, the latter contain the traditional glass filler particles found in hybrid resin composites and smaller concentrations of nanosized fillers and/or nanofiller clusters.[9,10]

Clinical trials are important to verify the performance of composites under actual conditions of use.[11] Focusing solely on the longitudinal clinical performance, this study evaluated the clinical performance of a nanofilled, a nanohybrid, and, as a control, a conventional microhybrid composite in Class I occlusal restorations of posterior teeth over the course of 54 months. The null hypothesis to be tested was that there was no difference in the clinical performance among the three resin composites after 54 months.

METHODS AND MATERIALS

This was a clinical study with a controlled and randomized design and it followed the guidelines published by Consolidated Standards of Reporting Trials.[12] This research was approved by the Research Ethics Committee of the Health Science Center (CEP: # 1252) of the Federal University of Paraíba (Brazil). Written informed consent was obligatory for each patient.

1. Population and Sample

The patients in this study were selected from among male and female students at public schools in the municipality of João Pessoa, Paraíba (Brazil). Our sample was restricted to students in public schools who lived in the suburbs. These patients were adolescents (mean age ± standard deviation, 13.44 ± 2.22 years).

The sample size was calculated based on an expected difference in survival of the three composites of 15%, a power of 0.8, and a significance level of 0.05. In agreement with the recommendations of Hickel and others,[13] there should not be more than one restoration per group per patient, therefore leading to a final sample composed of 123 permanent molars of 41 volunteers, who were divided into three groups (Figure 1).

2. Eligibility Criteria, Randomization, and Blinding

The inclusion criteria were as follows: the presence of three molars requiring replacement of Class I restorations or with primary caries on the occlusal surface; occlusal contact with the antagonist tooth; and a patient who was in a good state of general health.[14] The following were excluded from the study: patients with intense bruxism, observed as abnormal wear patterns of the occlusal surface, abfractions, and fractures in the teeth; molars that presented a carious lesion on a surface other than the occlusal surface and in continuity with the occlusal cavity; pulp exposure during caries removal or cavities with imminent risk of pulp exposure; or spontaneous pain or sensitivity to percussion.

To ensure randomness, a drawing was held using sealed envelopes to establish in which group a certain tooth would be placed, as follows:

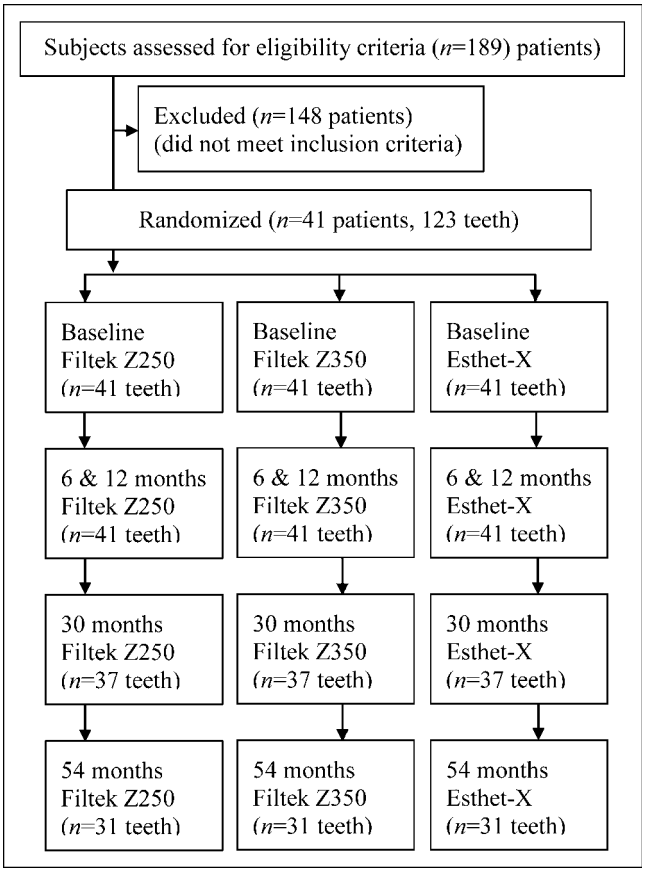

Figure 1. *Flowchart of the trial.*

- Group I: microhybrid resin composite Filtek Z250 (3M ESPE, St Paul, MN, USA), representing the control;
- Group II: nanofilled resin composite Filtek Z350 (3M ESPE); or
- Group III: nanohybrid resin composite Esthet-X (Dentsply/Caulk, Milford, DE, USA).

Neither the patients nor the examiners knew which commercial brand of composite was used in each tooth, thus resulting in a double-blind study.

3. Clinical Procedure

Treated teeth were isolated with rubber dam, and cavity preparations were performed with 245 carbide burs (SS White, Rio de Janeiro, Brazil) using high speed with light intermittent pressure. Tissue removal was limited to that required for access to carious tissue. Residual caries were removed with a spherical bur at low speed. In cases of unsatisfactory restorations, these and any remaining carious tissues were removed. In deep cavities, resin-modified glass ionomer cement (Vitrebond; 3M ESPE) was used. In shallow and medium cavities, only dentin hybridization was performed. One-step total-etch adhesive system, Adper Single Bond 2 (3M ESPE), was applied following the manufacturer's instructions. The composite was inserted using the incremental technique, with a maximum of 2 mm in each layer, and photo-activated with an LED curing light (Optilight LD Max; Gnatus, Ribeirão Preto, São Paulo, Brazil), depending on the manufacturer's recommendations. Irradiance of 600 mW/cm^2 was verified using a radiometer. Occlusion was adjusted with a multibladed bur (FG7714F, KG Sorensen, Brazil) at high speed. The restorations received final finishing and initial polishing with rubber cups and points (Flexicups and Flexipoints, Cosmedent Inc, Chicago, IL, USA). Final polishing was performed using Enamelize paste (Cosmedent Inc) and a diamond felt disk (FGM Joinville, Santa Catarina, Brazil). All of the procedures were performed by the same operator, and all of the patients received individual oral hygiene instructions, tooth brushes, and toothpaste with fluoride.

4. Sample Characteristics

The characteristics of the samples are shown in Table 1. After performing the statistical tests to verify the homogeneity of the sample, it was found that the distribution of the variables was homogeneous in the three groups ($p>0.05$).

5. Evaluations

The restorations were clinically evaluated by two examiners, who had been previously trained and whose performance had been calibrated. Kappa varied from 0.8 to 1. When disagreements arose during the evaluations, consensus among examiners was obtained. The evaluations were made one week after the restorations were performed (baseline) and after six, 12, 30, and 54 months, in accordance with the criteria (Table 2) established by Dresch and others[14] and the modified US Public Health Service criteria (Ryge Criteria).[15] Radiographs (bitewings) and periapicals in teeth with deep cavities were taken and vitality tests were performed. Postoperative sensitivity was evaluated at all sessions by questioning the patients and applying an air spray for three to five seconds from a syringe at a distance of 3-5 mm.

The restorations were classified as Alpha, Bravo, and Charlie. Alpha and Bravo scores corresponded to excellent and clinically acceptable results; a Charlie score corresponded to a clinically unacceptable result and served as an indication to replace the restoration to prevent future damage or to repair the present damage.[11]

6. Statistical Analysis

The Statistical Package for the Social Sciences (version 13.0, SPSS, Chicago, IL, USA) was used for the statistical analysis. The McNemar and Friedman nonparametric tests were used at a level of significance of 5%. The McNemar test was applied to verify the homogeneity of the sample, and the Friedman test was applied to assess and evaluate differences between time periods for each composite and differences between composites at the end of each time period.

RESULTS

In total, 123 restorations were placed in 41 patients. However, after 30 and 54 months, four and 10 patients, respectively, were lost to follow-up because they had moved and could not be located. After 54 months, 31 patients were reevaluated (a total of 93 restorations were available). The results are shown in Table 3. The overall success rate at 54 months was 94.62%. Five failed restorations (5.37%) were observed during the follow-up: two Filtek Z250 (6.45%), two Filtek Z350 (6.45%), and one Esthet X (3.22%). This resulted in annual failure rates of 1.61% for the Filtek Z250 and Filtek Z350 groups and a failure rate of 0.80% for the Esthet X group.

In some restorations, a decline was observed for evaluated criteria in the performance of the restoration from category Alpha to Bravo. An exception occurred for one Esthet-X, two Filtek Z350, and two Filtek Z250 restorations, which received the clinically unacceptable score Charlie for both anatomic form and marginal adaptation. Secondary caries as well as postoperative sensitivity occurred in one Filtek Z250 and one Filtek Z350 restoration. When the five evaluation periods (baseline and six, 12, 30, and 54 months) were compared, there were significant differences in the marginal adaptation of Filtek Z250 and Filtek Z350 ($p<0.05$). Significant differences were also observed in the roughness of Filtek Z250, Filtek Z350, and Esthet-X ($p<0.05$).

There were significant differences in the roughness criteria ($p=0.005$) when the three composites were compared after 54 months. The roughness of Filtek Z350 was greater, followed by that of Filtek Z250 and that of Esthet-X, but all were always within clinically acceptable limits.

Table 1: *Sample Characteristics*

	n	%
Gender		
Male	27	65.9
Female	14	34.1
Dental element		
Maxillary molar	43	35
Mandibular molar	80	65
Dental condition		
Primary caries	93	75.6
Replacement of restoration	30	24.4
Cavity width		
Larger than 1/3	22	17.9
Less than 1/3	101	82.1
Cavity depth		
Shallow	17	13.8
Medium	76	61.8
Deep	30	24.4
Dentin consistency		
Soft	30	24.4
Leathery	93	75.6
Dentin color		
Yellow	43	35.0
Light brown	54	43.9
Brown	26	21.1
Pulp protection		
Adhesive system	93	75.6
Glass ionomer cement and adhesive system	30	24.4

DISCUSSION

In general, early failures, which are encountered after weeks or months, must be distinguished from late failures, which occur after several years of clinical service. Early failures are a result of treatment faults, selection of an incorrect indication for the restorative material, or postoperative symptoms of pain and discomfort. Late failures are predominantly caused by fractures, secondary caries, and wear or deterioration of the materials.[3] After 12 months, all groups presented failures (Charlie rating) for anatomic form and marginal adaptation. At 54 months, the number increased by one for Filtek Z250 and Filtek Z350 restorations. Esthet-X maintained the same number of restoration failures. Secondary caries was first observed at the 36-month evaluation for Filtek Z350. After 54 months, a Filtek Z250 restoration also presented secondary caries.

The loss of marginal adaptation due to fracture and loss of retention and the presence of secondary caries are the main cause of failures of posterior resin-based composites, resulting in restoration replacement. According to Mjör,[16] development of secondary caries is not only due to the material itself. The clinical environment, the patients' history

Table 2: *Modified USPHS Evaluation Criteria*

Criterion	Code	Definition
Anatomic form	Alpha	Restoration continuous with existent anatomic form
	Bravo	Restoration discontinuous with existent anatomic form, but loss of material is not sufficient to expose the dentin base
	Charlie	Loss of material sufficient to expose the dentin or base
Marginal adaptation	Alpha	Restoration completely adapted to the tooth; no visible gap; no explorer catch at the margins or in any direction
	Bravo	Explorer catch; there is no visible evidence of a gap into which the explorer could penetrate
	Charlie	Explorer penetrates into a deep gap that exposes dentin or base
Marginal discoloration	Alpha	No discoloration along the cavo-superficial margin
	Bravo	<50% of the cavo-superficial margin affected by stain
	Charlie	>50% of the cavo-superficial margin affected by stain
Color match	Alpha	Restoration with color and translucency similar to those of the adjacent dental structure
	Bravo	Change in color and translucency within an acceptable standard
	Charlie	Change in color outside the acceptable standard
Surface roughness	Alpha	Restoration surface is smooth
	Bravo	Restoration surface is slightly rough or has scratches but can be refinished
	Charlie	Surface deeply rough, with irregular scratches; cannot be refinished
Secondary caries	Alpha	Absent
	Charlie	Present
Postoperative sensitivity	Alpha	Absent
	Charlie	Present

Table 3: *Results of the Clinical Evaluation of the Restorations*

Evaluation Criteria	Score	Baseline, n (%)			12 mo, n (%)			30 mo, n (%)			54 mo, n (%)		
		Z250 (n=41)	Z350 (n=41)	Esthet-X (n=41)	Z250 (n=41)	Z350 (n=41)	Esthet-X (n=41)	Z250 (n=37)	Z350 (n=37)	Esthet-X (n=37)	Z250 (n=31)	Z350 (n=31)	Esthet-X (n=31)
Anatomic form	A	41 (100)	41 (100)	41 (100)	40 (97.6)	40 (97.6)	40 (97.6)	35 (94.6)	35 (94.6)	35 (94.6)	29 (93.5)	29 (93.5)	29 (93.5)
	B	—	—	—	—	—	—	1 (2.7)	1 (2.7)	1 (2.7)	—	—	1 (3.2)
	C	—	—	—	1 (2.4)	1 (2.4)	1 (2.4)	1 (2.7)	1 (2.7)	1 (2.7)	2 (6.5)	2 (6.5)	1 (3.2)
Marginal adaptation	A	41 (100)	41 (100)	41 (100)	31 (75.6)	32 (78)	35 (85.4)	32 (86.5)	29 (78.4)	30 (81.1)	28 (90.3)	26 (83.9)	26 (83.9)
	B	—	—	—	9 (22)	8 (19.5)	5 (12.2)	4 (10.8)	7 (18.9)	6 (16.2)	1 (3.2)	3 (9.7)	4 (12.9)
	C	—	—	—	1 (2.4)	1 (2.4)	1 (2.4)	1 (2.7)	1 (2.7)	1 (2.7)	2 (6.5)	2 (6.5)	1 (3.2)
Marginal discoloration	A	41 (100)	41 (100)	41 (100)	41 (100)	41 (100)	41 (100)	37 (100)	36 (97.3)	36 (97.3)	30 (96.8)	31 (100)	31 (100)
	B	—	—	—	—	—	—	—	1 (2.7)	1 (2.7)	1 (3.2)	—	—
	C	—	—	—	—	—	—	—	—	—	—	—	—
Color match	A	39 (95.1)	38 (92.7)	39 (95.1)	37 (90.2)	33 (80.5)	35 (85.4)	32 (86.5)	32 (86.5)	32 (86.5)	27 (87.1)	29 (93.5)	26 (83.9)
	B	2 (4.9)	3 (7.3)	2 (4.9)	4 (9.8)	8 (19.5)	6 (14.6)	5 (13.5)	5 (13.5)	5 (13.5)	4 (12.9)	2 (6.5)	5 (16.1)
	C	—	—	—	—	—	—	—	—	—	—	—	—
Surface roughness	A	41 (100)	41 (100)	41 (100)	32 (78)	25 (61)	37 (90.2)	28 (75.7)	22 (59.5)	34 (91.9)	15 (48.4)	12 (38.7)	24 (77.4)
	B	—	—	—	9 (22)	16 (39)	4 (9.8)	9 (24.3)	15 (40.5)	3 (8.1)	16 (51.6)	19 (61.3)	7 (22.6)
	C	—	—	—	—	—	—	—	—	—	—	—	—
Secondary caries	A	41 (100)	41 (100)	41 (100)	41 (100)	41 (100)	41 (100)	37 (100)	36 (97.3)	37 (100)	30 (96.8)	30 (96.8)	31 (100)
	C	—	—	—	—	—	—	—	1 (2.7)	—	1 (3.2)	1 (3.2)	—
Postoperative sensitivity	A	41 (100)	41 (100)	41 (100)	41 (100)	41 (100)	41 (100)	37 (100)	37 (100)	37 (100)	30 (96.8)	30 (96.8)	31 (100)
	C	—	—	—	—	—	—	—	—	—	1 (3.2)	1 (3.2)	—

of previous caries, the criteria for replacements, and different handling characteristics also seemed to affect the clinical results. In addition, Bernardo and others[17] reported that the overall risk of failure due to secondary caries was 3.5 times higher in composite restorations than in amalgam restorations.

In the study of Stefanski and van Dijken,[10] no secondary caries was observed contiguous to the evaluated Class II nanofilled restorations, despite the frequency of high caries risk in the participants, which may indicate a good marginal seal. On the other hand, a two-year evaluation is far too short to observe the formation of secondary caries. This develops mainly after four to six years of intraoral aging, as shown in longer follow-ups.[18]

Marginal discoloration usually results from defects present between the composite restoration and the cavity margins. These defects may be caused by inadequate restoration placement and finishing procedures, by unsatisfactory bonding, and by subsequent stress fatigue.[19,20] The use of the incremental technique with a maximum of 2 mm in each layer likely contributed to a lower incidence of marginal discoloration.

Polymerization shrinkage can generate high stresses at bonded surfaces in confined cavity preparations.[21] The configuration factor of the restoration, the ratio of bonded to nonbonded surfaces in the cavity, has been reported to play an important role during the development of contraction stress. Despite the high C-factor of the cavities, a high durability for the evaluated Class I restorations was observed. Van Dijken[22] observed clinical results with excellent durability after a 12-year evaluation period of incrementally filled cavities. The influence of polymerization shrinkage stress on the longevity of the Class I resin composite restoration was far less than expected and indicated the role of other factors.

Sadeghi and others[21] evaluated bulk filled restorations in small cavities with a high C-factor for 18 months. Despite their good clinical results, the authors highlight that caution should be exercised when restoring such situations and that the use of incremental placement techniques is encouraged where reasonably possible.

The durability of a restoration is multifactorial, and other factors, such as the handling of the material by the operator, the bonding capacity of the restorative system, the application and curing technique used, and several patient-dependant factors during aging (like temperature and pH cycles in the mouth, degree of occlusal loading, and hydrolytic degeneration of the material), may all play a role.[22]

The surface roughness of the resin composites changed over the course of 54 months. However, these changes occurred from the Alpha to Bravo ratings, maintaining restorations as clinically acceptable. This difference was found when comparing the three composites (Filtek Z350 > Filtek Z250 > Esthet-X) and for the same composite over the course of time. Our results are supported by the laboratory investigations of Mayworm and others,[23] who reported that Filtek Supreme has larger particles and/or particle agglomerates and larger interparticle spacing. Moreover, wear tests caused larger and deeper voids on the surface of Filtek Supreme than on the surface of Esthet-X, caused by the removal of particles and possibly of particle agglomerates. However, other clinical trials[24,25] have shown significantly better or equal polishability for Filtek Supreme compared with microhybrid restorations. This divergence of results is not worrisome, because all restorations were classified as clinically acceptable in terms of roughness.

Despite the proposed advantages for nanofilled and nanohybrid composites, their clinical performance was not superior to that of the control group, which was restored with Filtek Z250, a microhybrid composite that has been on the dental market for a longer period of time. The study of Palaniappan and others[26] does not appraise the claimed advantages of nanometer-size particles on the clinical wear performance of resin composites because of the lack of any significant differences between the nanocomposites and microhybrid restoration groups. Similar to this observation, nanocomposites were reported to perform no better than the conventional microfills and microhybrids in recent *in vitro* wear studies.[27] Others studies[9,24] are in agreement with our results. There is a need to evaluate restorations over a longer timescale to determine the long-term clinical performance of these resin composite materials.

CONCLUSIONS

The materials investigated showed acceptable clinical performance in Class I restorations after 54 months. Continuous reevaluations are necessary for a more detailed long-term analysis of these composites. Furthermore, other long-term clinical trials are necessary to confirm our results.

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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.

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Laboratory Research

Influence of No-Ferrule and No-Post Buildup Design on the Fatigue Resistance of Endodontically Treated Molars Restored With Resin Nanoceramic CAD/CAM Crowns

P Magne • AO Carvalho • G Bruzi
RE Anderson • HP Maia • M Giannini

Clinical Relevance

Endodontically treated molars with extensive loss of coronal structure and no ferrule effect could be restored successfully with resin nanoceramic CAD/CAM crowns, with or without underlying composite resin buildup.

*Pascal Magne, PhD, University of Southern California, Restorative Sciences, Los Angeles, CA, USA

Adriana Oliveira Carvalho, PhD, Piracicaba Dental School/ State University of Campinas, Department of Restorative Dentistry, Salvador, Bahia, Brazil

Greciana Bruzi, MSc, Universidade Federal de Santa Catarina, Odontology, Federal University of Santa Catarina (UFSC), Department of Odontology, Santa Catarina, Brazil

Robert E Anderson, DMD, University of Southern California, Graduate Endodontics Department, Los Angeles, CA, USA

Hamilton Pires Maia, PhD, Universidade Federal de Santa Catarina, Operative Dentistry, Department of Odontology, Santa Catarina, Brazil

Marcelo Giannini, DDS, MS, PhD, associate professor, Piracicaba Dental School, University of Campinas, Department of Restorative Dentistry, Piracicaba, Brazil

*Corresponding author: 3305B S. Hoover Street, Bldg. A - Room A-122B, Los Angeles, CA 90089-7001, USA; e-mail: magne@usc.edu

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SUMMARY

Objectives: To evaluate the influence of adhesive core buildup designs—4-mm buildup, 2-mm buildup, and no buildup (endocrown)—on the fatigue resistance and failure mode of endodontically treated molar teeth restored with resin nanoceramic (RNC) CAD/CAM complete crowns placed with self-adhesive resin cement.

Methods and Materials: Forty-five extracted molars were decoronated at the level of the cementoenamel junction, and the roots were endodontically treated. Specimens received different Filtek Z100 adhesive core buildups (4-mm buildup, 2-mm buildup, and no buildup, endocrown preparation) and were restored with Cerec 3 CAD/CAM RNC crowns (Lava Ultimate). Restorations (n=15) and prepared teeth were treated with airborne-particle abrasion, followed by cementation with RelyX

Unicem 2 Automix. Specimens were then subjected to cyclic isometric loading at 10 Hz, beginning with a load of 200 N (for 5000 cycles), followed by stages of 400, 600, 800, 1000, 1200, and 1400 N at a maximum of 30,000 cycles each. Specimens were loaded until failure or to a maximum of 185,000 cycles (10-mm-diameter composite resin sphere antagonist). The failure mode was assessed: "catastrophic" (tooth/root fracture that would require tooth extraction), "possibly reparable" (cohesive/adhesive failure with fragment and minor damage, chip or crack, of underlying tooth structure), or "reparable" fracture (cohesive or cohesive/adhesive fracture of restoration only). Groups were compared using the life table survival analysis. Intact specimens were loaded to failure and compared with one-way analysis of variance.

Results: All specimens survived the fatigue test until the 800 N-step. The survival rates for 4-mm, 2-mm, and no buildup (endocrown) were 53%, 87%, and 87%, respectively, and were not statistically different even though crowns with 2-mm buildups only started to fail at 1200 N. Minor cohesive chips were detected in many samples despite having survived all 185,000 cycles. Postfatigue load-to-failure ranged from 2969 N with 4-mm buildup (eight specimens), 2794 N for 2-mm buildup (13 specimens), and 2606 N for endocrowns (13 specimens) and were also not statistically different. There were only two catastrophic failures during the fatigue test and small subgingival delamination fractures and cracks (only with 4-mm buildup). All specimens in the load-to-failure test exhibited nonrestorable catastrophic fractures.

Conclusions: There was no influence of the buildup design on the performance of endodontically treated molars restored with RNC CAD/CAM complete crowns placed with self-adhesive cement. All restoration designs survived the normal range of masticatory forces. Failure mode tended to be more favorable with the 2-mm buildup or no buildup (endocrown).

INTRODUCTION

The decision of how to rehabilitate endodontically treated molars (ETM) with extensive loss of coronal structure is a challenge for restorative dentistry. Those teeth are considered to have a higher risk of fracture than vital teeth because of their inherently poor structural integrity, with loss of root and coronal dentin resulting from preexisting caries and/or tooth preparation.[1-4] There is controversy regarding which technique would be ideal for ETM restoration.

Although earlier publications have called for stabilization of ETM with intracanal posts and ferrule, other evidence has demonstrated that post reinforcement is not beneficial.[3,5] Even though posts are frequently used to retain coronal buildup materials, they do not reinforce roots and may even weaken them through loss of radicular dentin necessitated by post space preparation.[5,6] In addition, preparing a post space also involves a certain degree of risk of accidental root perforation. The loss of tooth structure during preparation affects tooth stiffness, reduces its resistance to fracture, and consequently limits its prognosis. Other studies[7-9] have confirmed that ETM restored without posts have similar fracture resistances and failure modes compared with those with posts, which suggest that posts are not necessarily required. Lima and others[7] confirmed that the presence of a ferrule (with a composite resin buildup) is more critical than the use of a post. However, there is no consensus about the optimal buildup design necessary to restore ETM in the absence of any ferrule. The endocrown restoration is another alternative restorative treatment for ETM.[10,11] Pissis[10] was a pioneer in proposing this "monobloc" porcelain technique in 1995. This type of restoration preserves root tissue and limits internal preparation of the pulp chamber to its anatomic shape. It constructs both the crown and core build-up as a single unit. Even though the original technique described the use of porcelain, *in vitro* fatigue tests showed that endocrowns made of more flexible composite resin or newer resin nanoceramic (RNC) materials may also have a great potential for this indication.[11-14] Another consideration is the possible use of a core buildup to remove retention from the endodontic preparation, provide some kind of positive geometry, decrease restoration thickness (allowing for the use of light-polymerized luting composites), and facilitate provisionalization. Yet there is a lack of data about the biomechanical behavior of different buildup designs to restore ETM.

Therefore, the aim of this study was to evaluate the influence of a 4-mm buildup, a 2-mm buildup, or no buildup (endocrown) on the mechanical performance and failure mode of ETM restored with RNC CAD/CAM complete crowns placed with self-adhesive cement. The null hypothesis was that there is no significant difference in the fatigue resistance and

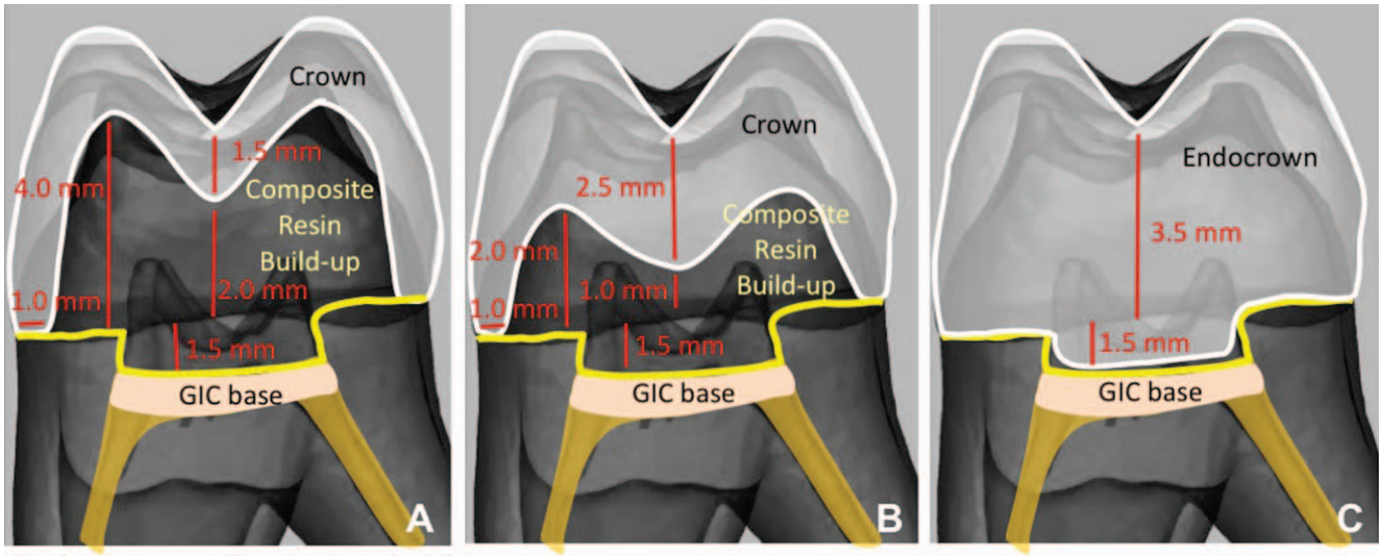

Figure 1. *Restorative techniques. (A): Group I. (B): Group II. (C): Group III.*

failure mode of ETM among the three different designs tested in this *in vitro* study.

METHODS AND MATERIALS

Once approval was obtained from both the Ethical Committee of the Piracicaba Dental School (Campinas State University) and the University of Southern California Review Board, 45 freshly extracted, sound human maxillary molars stored in solution saturated with thymol were used. Teeth were mounted in a special positioning device with acrylic resin (Palapress; Heraeus Kulzer, Armonk, NY, USA) embedding the root up to 3.0 mm below the cementoenamel junction (CEJ).

Tooth Preparation

A standardized tooth preparation was applied to all specimens. The intact crowns were removed by a horizontal section 1 mm above the CEJ using a diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA), under water lubrication. A standard access opening was prepared to simulate root canal treatment in each tooth. Teeth were accessed using slow-speed round and GK269 burs to deroof the pulp chamber and smoothen the internal walls. Canals were located and patency achieved using #10 K-files (Dentsply Tulsa Dental, Johnson City, TN, USA). Coronal flare was created using Gates #3 (Dentsply Tulsa Dental), and canals were chemomechanically debrided using 04 rotary files (Protaper Niti Rotary, Dentsply Tulsa Dental) and NaOCl (5.25%) to within 3 mm of the apex. A final rinse with H_2O was performed, and canals were dried using paper points. Warm vertical obturation of the canals was then performed using gutta percha to the orifice level and condensed. An additional horizontal reduction of 1.0 mm was obtained (flat preparation following the CEJ, no ferrule) with the aid of a coarse round diamond bur (Brasseler, Savannah, GA, USA). Finally, a 1.0- to 1.5-mm-thick glass-ionomer barrier (Ketac Molar, 3M ESPE, St Paul, MN, USA) was applied to the base of the pulp chamber.

The teeth were randomly divided into three groups according to the different restorative techniques (n=15):

- Group I: large buildup (4-mm height from CEJ at cusp tips, 2-mm height from CEJ at central groove) + complete crown restorations (1.5 mm thick; Figure 1A)
- Group II: short buildup (2-mm height from CEJ at cusp tips, 1-mm height from CEJ at central groove) + complete crown restorations (2.5-3.5 mm thick; Figure 1B)
- Group III: endocrown restoration (ca. 5- to 5.5-mm thickness; Figure 1C)

Buildups for groups I and II were made using Optibond FL adhesive system (Kerr Corp, Orange, CA, USA) and Filtek Z100 composite resin (3M ESPE) placed in 1.5-mm increments polymerized for 20 seconds each at 1000 mW/cm^2.

Design and Manufacturing of Restorations

The molars were restored using the Cerec 3 CAD/CAM system (Sirona Dental Systems GmbH, Ben-

Table 1: *Failure Mode During Fatigue Testing*

	Reparable				Not Reparable
	Cohesive Failure at Crown	Cohesive Failure at Crown and Buildup + Adhesive Failure at Dentin Margin	Adhesive Failure Between Crown and Buildup + Adhesive Failure at Dentin Margin	Cohesive Failure at Crown and Buildup + Dentin Chip	Catastrophic Failure
Endocrown	1	—	—	1	—
2-mm Buildup	2	—	—	—	—
4-mm Buildup	1	3	1	—	2

sheim, Germany). The specimens were fitted with a crown or endocrown of standardized thickness and occlusal anatomy (third maxillary molar, Lee Culp Youth database). Using the Crown Master Mode and the Design Tools of the Cerec software (v. 3.6, Sirona Dental Systems), the occlusal surface was moved and rotated to make parallel the cusp tips and the preparation surface as well as to align the central groove. All restorations were milled in RNC (Lava Ultimate blocks, 3M ESPE) using the Endo mode with the sprue located at the lingual surface, then polished mechanically with a diamond ceramic polisher (CeramiPro Dialite W16DM; Brasseler), polishing brush (soft bristle brush) with diamond paste (Diamond Twist SCL, Premier, EC Representative: MDSS GmbH * Schiffgraben, Hannover, Germany), and buffed with a muslin rag wheel.

Crown Placement

All crowns were cemented with a dual-cure self-adhesive resin cement (RelyX Unicem 2 Automix, 3M ESPE). Before cementation, each crown was fitted on its respective tooth to check its marginal adaptation and steam cleaned. The inner surface of LU crowns were sandblasted with 50 μm aluminum oxide (Danville, San Ramon, CA, USA), rinsed, and cleaned in an ultrasonic bath in distilled water for one minute. The prepared teeth were sandblasted with 27 μm aluminum oxide, rinsed, and dried. The cement was applied to the inner surface of the crowns, which were then seated on the tooth with an approximate pressure of 70 N. Cement excesses were removed after a brief light exposure (approximately two seconds) with an LED light (VALO Curing Light, Ultradent Products Inc, South Jordan, UT, USA) and followed by light polymerization for 20 seconds on each surface. Air-blocking barrier (KY Jelly; Johnson & Johnson Inc, Montreal, QC, Canada) was used to cover all margins, and additional polymerization was carried out for 20 seconds per surface. The margins were finished and polished with a diamond ceramic polisher (CeramiPro Dialite W16DM; Brasseler), polishing brush (soft bristle brush) with diamond paste (Diamord Twist SCL, Premier), and buffed with a muslin rag wheel.

Testing

Fatigue Testing—Each specimen was stored in distilled water at room temperature for at least 24 hours following adhesive restoration placement. Masticatory forces were then simulated with an artificial mouth using closed-loop servo hydraulics (Mini Bionix II; MTS Systems, Eden Prairie, MN, USA). Each specimen was placed into the load chamber and situated with a positioning device (sliding table). The chewing cycle was simulated by an isometric contraction (load control) applied through a composite resin sphere (Filtek Z100, 3M ESPE) with a diameter of 10.0 mm. Because of the standardized occlusal anatomy, all specimens could be adjusted (through the positioning device) in the same reproducible position with the sphere contacting the mesiobuccal, distobuccal, and palatal cusps (tripod contact). The load chamber was filled with distilled water to submerge the sample during testing. Cyclic load was applied at a frequency of 10 Hz, starting with a load of 200 N for 5000 cycles (preconditioning phase to guarantee predictable positioning of the sphere with the specimen), followed by stages of 400, 600, 800, 1000, 1200, and 1400 N at a maximum of 30,000 cycles each. Samples were loaded until fracture or to a maximum of 185,000 cycles. The number of endured cycles and failure mode were recorded. Following a two-examiner agreement using optical microscopy, a distinction was made between "catastrophic" failure (crown/root fracture that would require tooth extraction) or "reparable" failure (cohesive or cohesive/adhesive failure with or without fragment and minor damage, chip or crack, of underlying tooth structure; Table 1).

Load-to-Failure Testing of Intact Postfatigue Specimens (in Case of Major Percentage of Specimen Surviving Fatigue)—After the fatigue test, intact specimens were axially loaded until failure or to a maximum load of 4500 N with a 10-mm composite resin sphere. The sphere had the same three-point

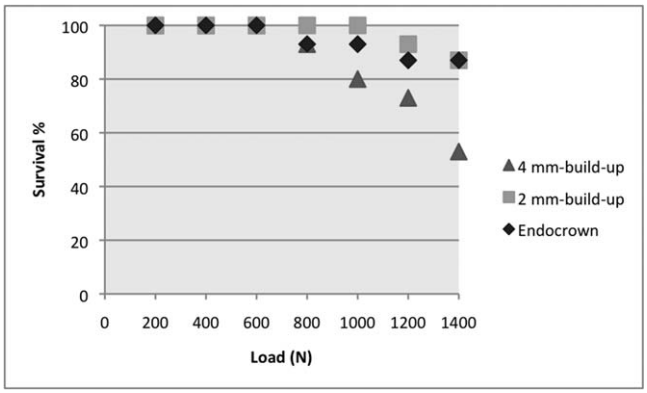

Figure 2. *Life table survival distributions by materials at each load step (n=15).*

occlusal contacts as in the fatigue test. The crosshead speed was 0.5 mm/min. The maximum postfatigue load before failure was recorded in Newtons, and mean values were calculated per group. After load tests, the specimens were analyzed for one of the three failure modes as in the fatigue test.

Statistical Analysis—The fatigue resistance of the three groups was compared using the life table survival analysis. At each time interval (defined by each load step), the number of specimens starting the interval intact and the number of specimens fracturing during the interval were counted, allowing the calculation of survival probability (%) at each interval. The influence of the restorative material on the fracture strength (load step at which failure occurred) was analyzed by using the log-rank test at a significance level of 0.05. The postfatigue load-to-failure resistance of the survived specimens was compared using one-way analysis of variance (ANOVA; data tested normal). For all statistical analyses, the level of significance was set at 95%. The data were analyzed with statistical software (MedCalc, V. 11.0.1; MedCalc Software, Mariakerke, Belgium).

RESULTS

The survival rates after the fatigue test for ETM with 4-mm buildups, 2-mm buildups, and endocrowns were 53% (eight samples), 87% (13 samples), and 87% (13 samples), respectively, and no statistically significant differences were found among them ($p>0.05$; Figure 2). In groups with large buildups and endocrowns, all specimens survived until the 800 N-step, while for specimens with short buildups, samples started to fail only at the 1200 N-step. At the end of the fatigue test, minor surface chips were detected (two specimens with large buildups and nine specimens with short buildups or endocrowns). All specimens demonstrated less wear at the resin sphere antagonist than the restoration itself (Figure 3).

Postfatigue load-to-failure averaged 2969 N with 4-mm buildups (eight specimens), 2794 N for 2-mm buildups (13 specimens), and 2606 N for endocrowns (13 specimens). One-way ANOVA revealed that there were no statistically significant differences among all three restorative techniques. Failure mode analysis showed that there were only two evident catastrophic failures during the fatigue test. All failure modes found at the fatigue test are given in Table 1. All of the specimens after the load-to-failure test exhibited nonrestorable catastrophic fractures.

DISCUSSION

Based on the results of this study, the null hypothesis was accepted for the fatigue resistance but rejected for failure mode analysis of ETM. The failure modes slightly varied, with less favorable outcomes when using a large core buildup.

In an effort to standardize and approximate the clinical situation as much as possible, natural teeth of similar dimensions were selected, the anatomy of the occlusal surface and the thickness of those restorations were standardized by the Cerec ma-

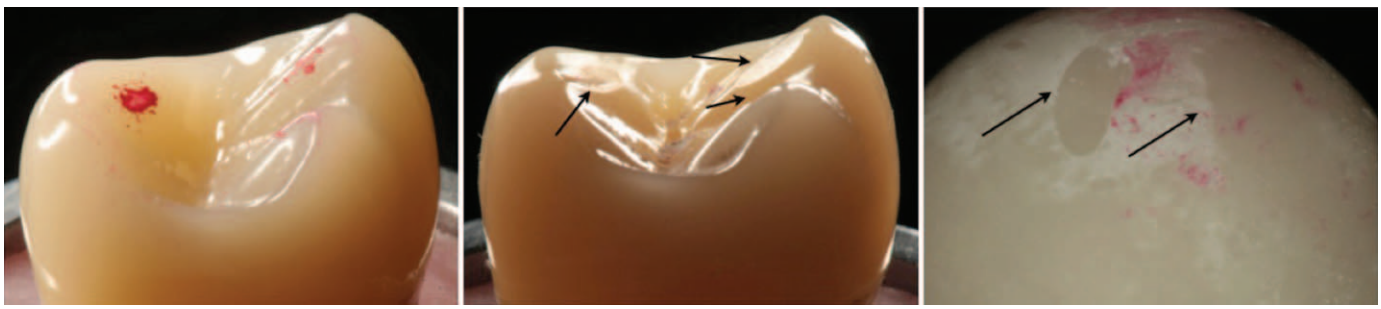

Figure 3. *Photographs of crown and antagonist (resin sphere) wear.*

chine, and the consistent load configuration of all samples was applied. As previously suggested,[13,15] the use of a composite resin sphere antagonist was preferred rather than a stainless-steel one. A more realistic simulation of tooth contacts is enabled by the lower stiffness and higher wear of the composite resin.[16] No failure of the spheres was noted during the test. Because of the simulated natural tooth anatomy of the restorations, a standardized three-point/facet contact could be created and a progressive load protocol generated (from 200 N to 1400 N, maximum 185,000 cycles). Loaded restorations and teeth should show wear facets and not point contacts.[15] Such wear facets both at the restorations and resin sphere antagonists were observed. As was the case in previously published data,[17] the RNC material demonstrated more material wear than the antagonistic wear (resin sphere). This can be explained by the fact that the RNC material has 80% filler content by weight (20% resin matrix) compared with 85%/15% for Filtek Z100 composite resin (antagonist sphere).

The present protocol seems to be the best compromise between available *in vitro* fatigue testing methods and clinical reality and can be called accelerated fatigue. Even though it is not possible to make a direct clinical correlation about the significance of the load range used in this study, this test lies in between load-to-failure (very high single load until failure, not clinically relevant unless during trauma) and fatigue tests (time-consuming low load/high cycles). A true fatigue correlation for one year of clinical service is 250,000 cycles at only 13.6 N.[18] Therefore, given the extended range of load in the present study, the accelerated life cycle of the restored tooth may have been simulated.

A higher frequency (10 Hz) in the cyclic loading test, which was suggested by Kelly and others,[19] was used in this study. It decreases the time of the experiment, allowing testing of three specimens per day. One may wonder whether such high frequency might lead to more heat generation compared with 1 to 2 Hz and possibly exclude time for stress relaxation.[20] Another limitation of this study is that the load was applied only axially, limiting the clinical implication to a vertical loading situation. Biacchi and Basting[21] used an oblique compression force to compare the fracture strength of endocrown and complete crowns retained by glass fiber posts. However, as in the present study, the endocrown restorations performed well, even presenting greater fracture strength than the conventional crowns supported on posts and filling cores.

Another specific element in this study was the use of self-adhesive resin cement. It allows for a convenient, fast, and efficient delivery of complete crowns. This is especially significant when considering excess cement removal in the case of subgingival margins (a common situation when replacing existing complete crowns), for which adhesive luting becomes more technique sensitive. RelyX Unicem 2 was chosen also because of its self-polymerization component, which was desirable for the thick endocrowns. The same cement was used to deliver the other crowns on the different buildups in order not to introduce a new variable.

The results of this study demonstrated that the presence of a buildup does not necessarily enhance the fracture resistance of ETM with extensive loss of coronal structure when using RNC crowns. However, the mean fracture loads for the 4-mm buildup (1171 N), 2-mm buildup (1300 N), and endocrown (1000 N) failed fatigued restorations far exceeded regular masticatory forces. The latter, during normal function, range from 50 N to 250 N and from 500 N to 800 N in bruxism behavior.[22,23] The 4-mm buildup and endocrown restorations started to fail at 800 N, while all short buildups did not show fracture before 1200 N. Those results suggest that it may be possible to use all three types of RNC restorations with self-adhesive cementation for ETM with extensive loss of coronal structure even under high load requirements. It is noteworthy to compare the performance of those restored nonvital teeth with that of crowned vital molars from another study by the same author in strictly identical conditions: 1.5-mm RNC crowns with self-adhesive cementation.[17] Simulated fatigue survival of the crowned vital teeth was 80% (53%-87% in the present study), and the average load-to-failure of intact postfatigue specimens was 3122 N (2606-2969 N in the present study). This indicates that the restorative modalities proposed for ETM in the present study may allow approaching the performance of vital teeth despite the absence of a ferrule effect.

There is evidence that the use of posts does not influence the performance of restored ETM.[3,5,21] In addition, the placement of a post is always associated with a risk of perforation and cracking of the root. Therefore, no posts were used in the present study. No-post endocrown restorations allow for maximum tooth structure preservation, reduce the requirement for macro retentive geometry, provide an efficient and esthetic outcome, and seem clinically

viable.[24-26] From a clinical perspective, the endocrown design seems to have practical advantages over restorations with a core buildup: it is cheaper, it takes less time to complete, and there is no composite resin shrinkage associated with this technique. The endocrown is also a useful option when there is simply no occlusal clearance (extra-short clinical crowns). On the other hand, there are advantages of using an adhesive composite resin core buildup when possible. The buildup is preceded by the use of a dentin adhesive system that safely seals the dentin. It reduces the thickness of the overlaying restoration, allowing for a more efficient light polymerization during cementation. It is known that even for dual-polymerization cements, the light-polymerization component is a determinant in obtaining an acceptable degree of conversion.[27-29] In view of the present results, small composite resin buildups should be preferred. They also induce less polymerization shrinkage than large ones, and they are useful for providing enhanced geometry, removing undercuts from the endodontic preparation, and facilitating provisionalization (by stabilization) when necessary.

Failure modes tended to be more favorable with the 2-mm buildup or no buildup (just cohesive failures). Only one endocrown failed with a small subgingival margin dentin chipping, which was still considered re-restorable because it would be feasible to smooth this margin or, in the worst case scenario, use periodontal surgery or the margin elevation technique.[30] Small superficial chipping of the RNC material around the contact points was frequently observed. Because there was no effect on the integrity of the restoration and stable occlusal contacts were maintained, the fatigue machine did not stop. From a clinical perspective, those defects would be easily corrected and polished, since Lava Ultimate proved extremely polishable, which is another advantage of the RNC material.

Further research should explore the use of different core buildups (such as autocure composite resins), restorative materials, and adhesive luting procedures. Even though more flexible materials seem to be indicated to restore the severely broken down ETM, rather than traditional porcelain,[11-14] the use of ceramics with stronger mechanical properties, such as lithium disilicate, may be a potential alternative.

CONCLUSION

Within the limitations of this *in vitro* study, it can be concluded that there is no influence of the buildup design on the performance of ETM restored with RNC CAD/CAM full crowns placed with self-adhesive resin cement. All restoration designs survived the normal range of masticatory forces. Failure mode tended to be more favorable with the 2-mm buildup or no buildup (endocrown). The endocrown has many practical advantages (simpler, quicker, more economic), while the use of a small composite resin buildup may be useful to provide enhanced geometry, remove undercuts from the endodontic preparation, and facilitate provisionalization when necessary.

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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.

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Incremental Layer Shear Bond Strength of Low-shrinkage Resin Composites Under Different Bonding Conditions

AH Al Musa • HNA Al Nahedh

Clinical Relevance

The chemical compatibility between old and repair composite resins and the use of an appropriate bonding agent are crucial for high incremental bond strength. A silorane-based resin composite should be repaired with a silorane-based composite and silorane adhesive material.

SUMMARY

The purpose of this study was to determine the incremental shear bond strength of a silorane-based composite (Filtek Silorane) repaired with silorane or a methacrylate-based composite (Filtek Z250) under various aging conditions. Also, the incremental bond strength of the silorane-based composite was compared with that of another low-shrinkage methacrylate-based composite (Aelite LS Posterior) under fresh and aged conditions, with and without the use of an adhesive resin between successive layers. The two brands of low-shrinkage composites were compared with a microhybrid, Filtek Z250, which served as the control. Substrate discs were fabricated and second layers were adhered to them immediately, after two weeks of aging, or after four weeks of aging and with and without an adhesive resin. Shear bond strengths were measured and failure modes were evaluated. The incremental bond strength of silorane to the silorane-based composite was not significantly different from that of the methacrylate-based composite. However, repairing a silorane-based composite with a methacrylate-based composite significantly reduced the bond strength. Aelite showed a lower incremental bond strength than Z250 and silorane, but the use of an adhesive significantly improved the bond strength. The absence of an oxygen-inhibited layer did not affect the bond strength of the consecutive layers of the silorane-based composite.

Arwa H. Al Musa, BDS, MSD, King Saud University, Restorative Dental Sciences, Riyadh, Saudi Arabia

*Hend N. A, Al Nahedh, BDS, MSD, King Saud University, Restorative Dental Sciences. Riyadh, Saudi Arabia

*Corresponding author: PO Box 60169, Riyadh, 11545, Saudi Arabia; e-mail: h_nahed@yahoo.com

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INTRODUCTION

Since their introduction to dentistry, resin composite materials have been extensively used by dental professionals. Over the years, these materials have undergone many modifications to improve their

characteristics and performance. Nevertheless, they still have some drawbacks, such as poor water stability, poor color stability, and low wear resistance in high-stress areas.[1]

Polymerization shrinkage is another major drawback that could result in microleakage, recurrent caries, cuspal deflection, and postoperative sensitivity.[2] Attempts to overcome these problems include using an incremental layering technique, varying the curing rate or intensity, using a liner with high elasticity at the tooth-restoration interface, modifying the chemical formulation of the resin composite by increasing the filler content, and using a larger monomer with fewer reactive sites.[3-7]

Manufacturers have tried to minimize polymerization shrinkage by modifying resin composites to develop low-shrinkage materials. One example of this is Aelite LS Posterior (Bisco, Schaumburg, IL, USA), a composite material with a high filler volume (75%). The increase in filler levels results in a decrease in the resin volume, thereby reducing the overall amount of polymerization shrinkage.[8] In 2007, a new low-shrinkage silorane-based resin composite material, Filtek Silorane (3M ESPE, Seefeld, Germany), was developed.[9] The reduced polymerization shrinkage attributed to the silorane monomer is the product of the reaction of oxirane and siloxane molecules. This material polymerizes via a cationic ring-opening polymerization reaction, thereby reducing the polymerization shrinkage.[10]

Siloranes exhibit mechanical properties, such as strength, hardness, and modulus of elasticity, that are comparable to those of methacrylate-based composites.[11] In addition, they show less cuspal deflection than methacrylates, which is attributed to their reduced polymerization shrinkage stresses.[12] Silorane-based composites also demonstrate better color stability and gloss retention than methacrylate-based composites.[13]

In clinical situations, it is sometimes necessary to place a composite in increments in areas where the prepared cavity is deep or when repairing an existing composite restoration after it has been in service for some time. Several studies[14,15] have shown that an oxygen-inhibited layer is needed for optimal bonding between consecutive layers of a methacrylate-based composite. The cationic ring-opening reaction of a silorane-based composite is insensitive to oxygen, and no oxygen-inhibited layer is formed on the surface. Therefore, it has been assumed that this would result in decreased bond strength between increments. Furthermore, this is a different class of resin composites and it may be difficult to bond it to preexisting methacrylate-based composites.[16]

The aims of this study were 1) to determine the shear bond strength of a consecutive layer of a silorane-based composite to a silorane-based composite and a silorane-based composite to a methacrylate-based composite under various aging conditions and with and without the use of an adhesive, 2) to compare the bond strength of a silorane-based composite to a low-shrinkage methacrylate-based composite under the same conditions, and 3) to compare the bond strength values of the silorane-based composite and the low-shrinkage methacrylate-based composite with those of a methacrylate to methacrylate-based composites for both fresh and aged conditions and with and without using an adhesive resin between successive layers.

METHODS AND MATERIALS

In this study, two types of low-shrinkage composite materials were tested, Filtek Silorane, and Aelite LS Posterior, and compared to a hybrid methacrylate-based composite (Filtek Z250, 3M ESPE). The adhesives used (Silorane System Adhesive Bond, One Step Plus, and Adper Single Bond 2) were the ones recommended by the manufacturer of each of the brands (Table 1). Filtek Z250, a microhybrid material, was chosen as the control. Following the manufacturer's instructions, 144 specimens were fabricated. Each of the two low-shrinkage composite materials had five groups of 12 specimens each; groups 11 and 12 were the control groups. The test conditions are summarized in Table 2.

The silorane composite was used as a substrate for groups 1-5. In group 1, the substrate was bonded to the silorane composite without using an adhesive. In group 2, the substrate was aged for two weeks and bonded to the silorane composite without using an adhesive. In group 3, the substrate was bonded to the silorane composite after four weeks of aging and without using an adhesive. In group 4, the substrate was aged for four weeks and bonded to the silorane composite using a silorane adhesive. In group 5, the substrate was aged for four weeks and bonded to the Z250 composite using the Adper Single Bond 2 adhesive.

Groups 6-10 were fabricated using the Aelite LS Posterior resin composite as a substrate. In group 6, the substrate was bonded to the Aelite composite without using an adhesive. In group 7, the substrate was aged for two weeks and bonded to the Aelite

Table 1: *Composition of the Resin Composites and Adhesives Used in the Study*

Material	Lot Number	Manufacturer	Material Composition
Composites			
Filtek Z250 (methacrylate based)	N143944	3M ESPE, St Paul, MN, USA	Bis-GMA, UDMA, Bis-EMA, silicon dioxide, zirconium dioxide, barium glass, ytterbium trifluoride, mixed oxide perpolymer
Aelite LS Posterior (low shrinkage methacrylate based)	100009783	Bisco, Schaumburg, IL, USA	Ethoxylated bis-GMA, glass filler, amorphous silica
Filtek Silorane (low shrinkage Silorane based)	N175795	3M ESPE, St Paul, MN, USA	Silorane (3,4 epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-epoxycyclohexylethylphenylmethylsilane), silicon dioxide, ytterbium triflouride
Adhesives			
Adper Single Bond 2	N176361	3M ESPE, St Paul, MN, USA	Bis-GMA, HEMA, dimethacrylates, ethanol, water, methacrylate functional copolymer of polyacrylic and polyitaconic acids
One Step Plus	1000010182	Bisco, Schaumburg, IL, USA	Biphenyldimethacrylate, hydroxyethyl methacrylate, acetone, glass frit
Silorane System Adhesive Bond	N170406	3M ESPE, St Paul, MN, USA	TEGDMA, phosphoric acid, methacryloxhexylesters, 1,6-hexanediol methacrylate, bis-GMA, UDMA, bis-EMA

Abbreviations: Bis-GMA, bisphenol A glycol dimethacrylate; bis-EMA, bisphenol A ethoxylated dimethacrylate; HEMA, hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

composite without using an adhesive. In group 8, the substrate was bonded to the Aelite composite after four weeks of aging and without using an adhesive. In group 9, the substrate was aged for four weeks and bonded to the Aelite composite using One Step Plus adhesive. In group 10, the substrate was aged for four weeks and bonded to the Z250 composite using the Adper Single Bond 2 adhesive.

Groups 11 and 12 served as positive fresh and aged control groups, respectively. For group 11, the Z250 composite was bonded to the same material immediately and without using an adhesive. For group 12, the Z250 composite was aged for four weeks and bonded to the same material using Adper Single Bond 2 adhesive.

Table 2: *Test Groups and Their Bonding Conditions*

Group	Substrate/Intermediate Layer/Stub	Bonding Condition
1	FS/no adhesive/FS	Fresh bonding
2	FS/no adhesive/FS	Bonding after 2 wk aging
3	FS/no adhesive/FS	Bonding after 4 wk aging
4	FS/Silorane System Adhesive Bond/FS	Bonding after 4 wk aging
5	FS/Adper/FZ250	Bonding after 4 wk aging
6	Aelite/no adhesive/Aelite	Fresh bonding
7	Aelite/no adhesive/Aelite	Bonding after 2 wk aging
8	Aelite/no adhesive/Aelite	Bonding after 4 wk aging
9	Aelite/One Step Plus/Aelite	Bonding after 4 wk aging
10	Aelite/Adper/FZ250	Bonding after 4 wk aging
11	FZ250/no adhesive/FZ250	Fresh bonding
12	FZ250/Adper/FZ250	Bonding after 4 wk aging

Abbreviations: Adper, Adper Single Bond 2; Aelite, Aelite LS Posterior; FS, Filtek Silorane; FZ250, Filtek Z250.

The resin-based substrates were fabricated using disc-shaped stainless steel molds measuring 10 mm in diameter and 3 mm in thickness. Curing was done for 40 seconds using a halogen light curing unit (Elipar 2500 Halogen Curing Light, 3M ESPE) placed 1 mm from the upper surface of the resin for each side. The samples requiring aging were stored in distilled water until the designated time had elapsed.

For the second layer of composite (the stub), the samples were placed in a Teflon alignment mold containing a central cylindrical hole measuring 4 mm in diameter and 2 mm in thickness (Figures 1a and 1b). The substrates were placed in the lower end of the mold and the mold was assembled. Then the designated surface treatment of the composite surface was carried out according to the manufacturer's instructions. The second composite layer (stub) was packed into the upper cylindrical hole and cured for 40 seconds. The samples were carefully removed after two minutes and inspected for defects or any resin flash; defective samples were discarded (Figure 1c).

The samples were stored in 37°C distilled water for one week, during which they were subjected to 5000 cycles of thermal stressing between 5° and 55°C with a dwell time of five seconds. The larger end of the composite sample was embedded in a plastic ring 2.5

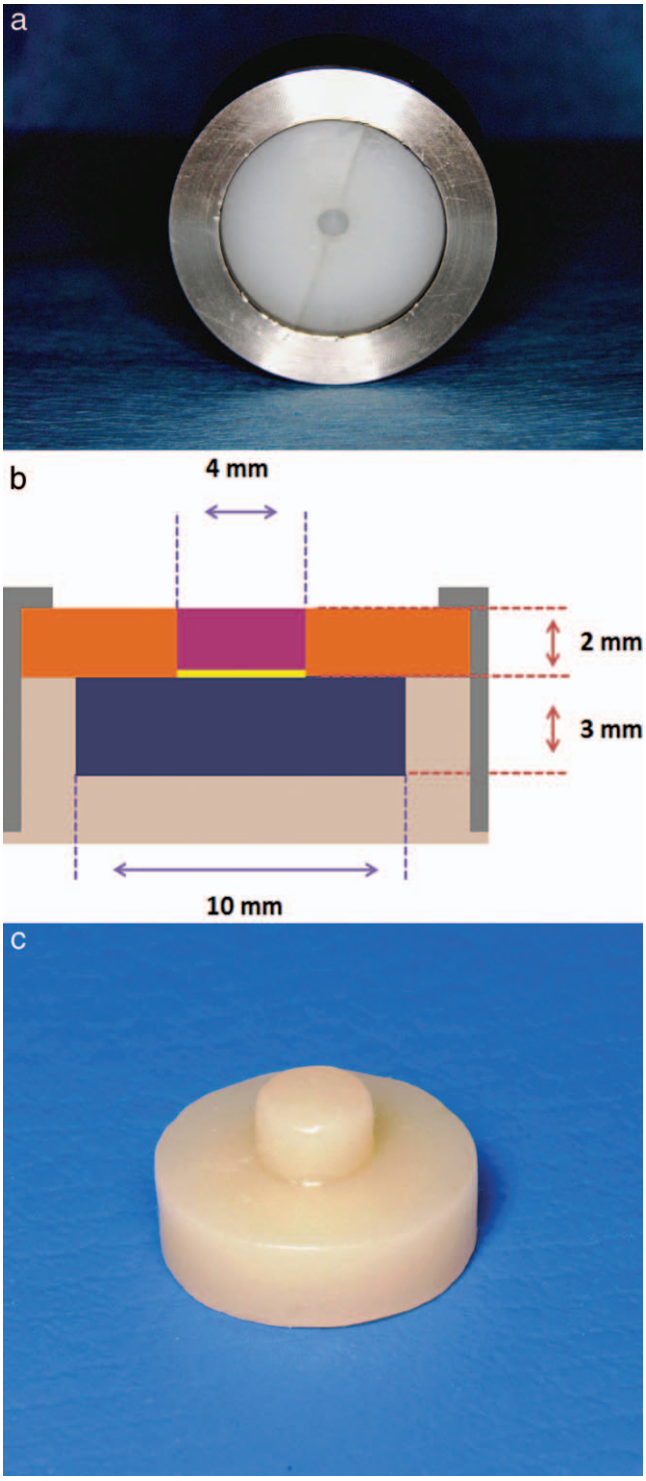

Figure 1. *(a): Teflon alignment mold with metal casing. (b): Schematic diagram of the Teflon alignment mold with metal casing (cross sectional view). (c): Completed sample.*

cm in diameter using Orthoresin (DeguDent GmbH, Hanau, Germany) to fit the specimens to the jig of the Instron machine. Each sample was tested in shear mode using the Instron Universal Testing Machine (Instron 8500, Instron Corp, Norwood, MA, USA) at a cross-head speed of 0.5 mm/min. All results were expressed in megapascal (MPa). All fractured samples were examined using an optical microscope at 50× magnification for failure analysis. Failure modes were categorized as "adhesive" (between the composite substrate and stub), "cohesive" (failure of the composite resin), or "mixed" (cohesive failure of the composite accompanied by adhesive failure at the interface).

The shear bond strength values were analyzed with one-way analysis of variance (ANOVA), and Tukey post hoc analysis at the ($p<0.05$) significance level, using statistical software (SPSS, version 16, SPSS, Chicago, IL, USA)

RESULTS

The mean shear bond strength and standard deviations are presented graphically in Figure 2. One-way ANOVA showed that there was a significant difference among the groups ($p<0.005$) (Table 3).

In the silorane groups, the incremental bond strength values of the fresh specimens tested in group 1 were not statistically significantly different from the fresh Z250 composite, group 11. Aging the silorane discs for two and four weeks (group 2 and 3, respectively) resulted in a significant drop in the incremental bond strength values (20% and 30%, respectively); however, the difference was statistically significant only with group 3 ($p<0.05$).

Group 4 resulted in increased bond strength values that were close to those of group 1 and were not statistically different from those of group 12. On the other hand, group 5 resulted in statistically lower bond strength values compared with groups 1 and 12.

For the Aelite groups, the incremental bond strength values of the fresh specimens (group 6) were slightly lower than those of groups 1 and 11, but the difference was not statistically significant. In addition, aging for two and four weeks (groups 7 and 8, respectively) slightly lowered the bond strength values; however, the difference was not statistically significant compared with group 6.

Bonding the four-week-aged Aelite specimens using One Step Plus adhesive to stubs of the same material (group 9) resulted in bond strength values that were higher than those of group 6; however, the difference was not statistically significant. Furthermore, the incremental bond strength values were not significantly different from those of group 12.

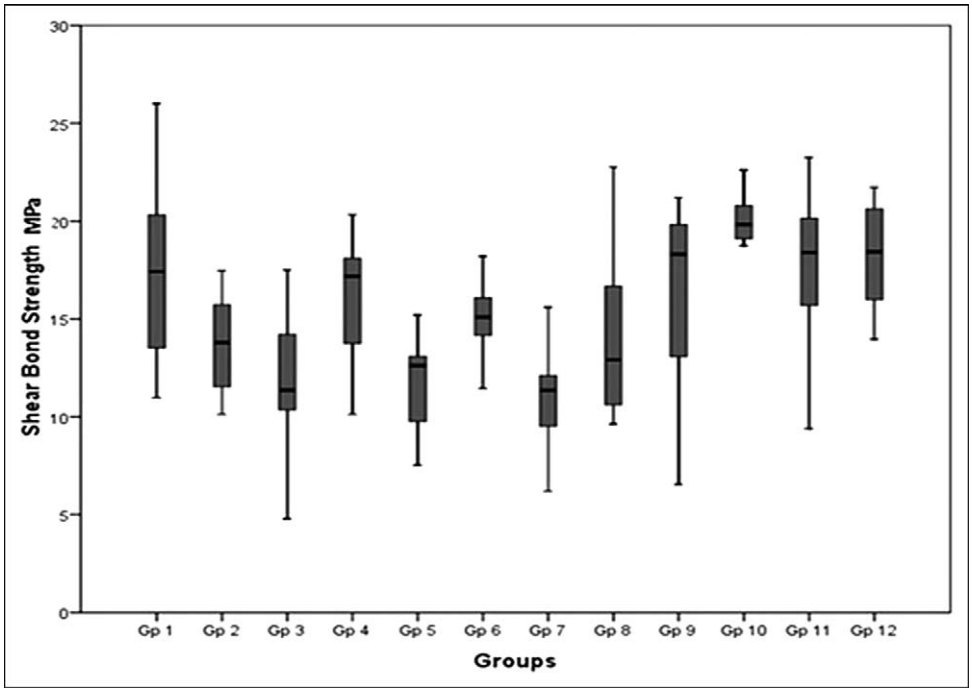

Figure 2. *Box and whisker plot of the shear bond strength values. The upper and lower horizontal lines represent the highest and lowest value, respectively, in each group. The horizontal line within the box represents the median, and the box contains 50% of the values.*

Bonding Z250 specimens to the four-week-aged Aelite stubs using Adper Single Bond 2 adhesive (group 10) resulted in high bond strength values that were not statistically different from those of groups 9 and 1. Similarly, Aelite repaired with Z250 using the Adper adhesive was not significantly different from group 12, but was significantly higher than groups 7 and 8, which were bonded without an adhesive ($p<0.05$).

Mode of Failure

The mode of failure results for the 12 groups are presented in Figure 3. The fresh silorane and Aelite groups bonded without an adhesive showed modes of failure that were either entirely (group 1) or mostly (group 6) cohesive in nature. Group 6 had one specimen fail adhesively.

Aging increased the adhesive mode of failure. After four weeks of aging without using an adhesive,

Table 3: *Homogenous Subsets of Mean Shear Bond Strength Values (MPa)*

Group	Subset for alpha = 0.05			
	1	2	3	4
Group 7: Aelite/no adhesive/Aelite (2 wk)	10.88			
Group 5: FS/Adper/FZ250 (4 wk)	11.52	11.52		
Group 3: FS/no adhesive/FS (4 wk)	11.95	11.95		
Group 2: FS/no adhesive/FS (2 wk)	13.67	13.67	13.67	
Group 8: Aelite/no adhesive/Aelite (4 wk)	14.11	14.11	14.11	
Group 6: Aelite/no adhesive/Aelite (fresh)	14.91	14.91	14.91	
Group 4: FS/Silorane System Adhesive Bond/FS (4 wk)		16.15	16.15	16.15
Group 9: Aelite/One Step Plus/Aelite (4 wk)		16.21	16.21	16.21
Group 1: FS/no adhesive/FS (fresh)			17.23	17.23
Group 11: FZ250/no adhesive/FZ250 (fresh)			17.43	17.43
Group 12: FZ250/Adper/FZ250 (4 wk)			18.23	18.23
Group 10: Aelite/Adper/FZ250 (4 wk)				19.94
Significance between groups	.17	.05	.07	.25

Abbreviations: Adper, Adper Single Bond 2; Aelite, Aelite LS Posterior; FS, Filtek Silorane; FZ250, Filtek Z250.

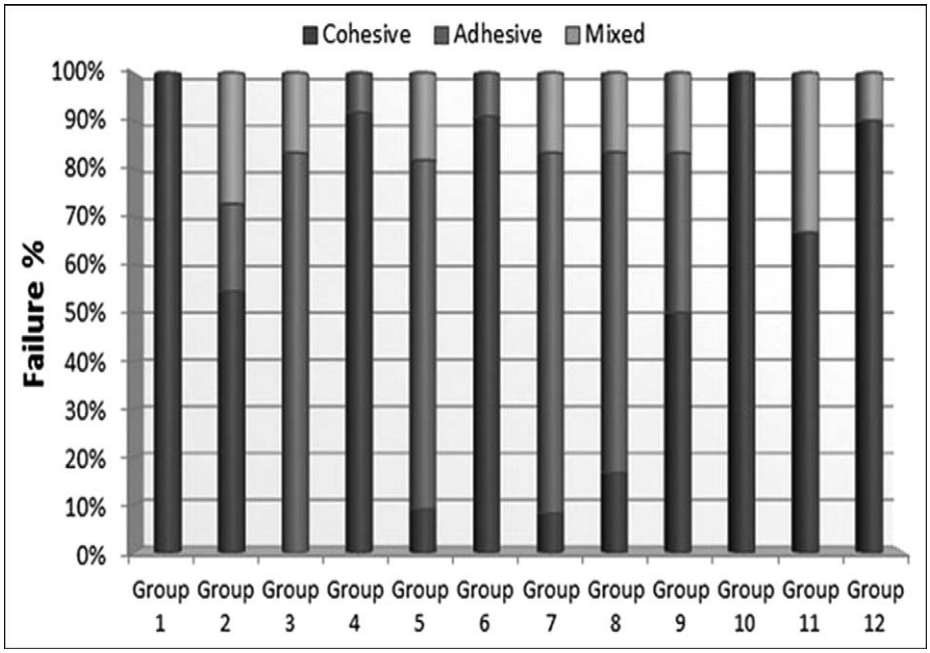

Figure 3. *Diagrammatic representation of the failure modes of the sheared samples for all the tested groups.*

83.3% (10 samples) of the silorane samples and 66.7% (8 samples) of the Aelite samples, groups 3 and 8, respectively, failed adhesively. The use of the silorane adhesive dramatically increased the cohesive type of failure to 91.7% (11 samples) when bonded to the same material after four weeks of aging (group 4), whereas bonding the four-week-aged silorane specimens to the Z250 composite using Adper Single Bond 2 (group 5) resulted in a high percentage of adhesive and mixed failures (91.7%, 11 samples); however, only one specimen failed cohesively.

The use of an adhesive in the Aelite groups increased the cohesive type of failures. When the Aelite substrates were bonded to the same material after four weeks of aging using One Step Plus adhesive (group 9) about half the group failed cohesively, whereas when the Z250 stubs were bonded to the four-week-aged Aelite substrates using the Adper Single Bond 2 adhesive, all the samples failed cohesively.

DISCUSSION

In the current study it was found that the bond strength between immediately placed incremental layers of the silorane composite was not different from that of the methacrylate-based composite, Z250. This shows that the absence of an oxygen-inhibited layer has no effect on the resultant bond strength in consecutive layers. This is in agreement with the findings of Shawkat and others.[16] In their study, they compared the bond strength of incremental layers of silorane composite in oxygen and nitrogen atmospheres. They found no significant difference between the two groups, thus concluding that the absence of an oxygen-inhibited layer did not affect the shear bond strength between two successive layers of silorane-based composites for the fresh conditions. However, within the confines of this study, the effect of this layer on the aged specimens could not be ruled out completely. In addition, there was no difference between the silorane-based composite and the methacrylate-based composite in incremental bond strength values.

The other low-shrinkage material tested in this study was Aelite LS Posterior, which is a methacrylate-based resin composite material with a high filler content (75% vol). The incremental bond strength of this material after immediate placement was lower than that of Silorane and Z250. There is no agreement among researchers on the minimal acceptable limit of composite repair bond strength. However, Teixeria and others[17] suggested that the shear bond strength of repaired composites should range from 15 MPa to 25 MPa. The bond strength of the Aelite group was lower than that limit. This may be the result of the higher viscosity of this material caused by its higher filler level. Composites with a low viscosity are believed to offer better adaptation to the repaired composite resin surface, thereby increasing retention through micromechanical interlocking by flowing into the irregularities of the

material's surface.[18] Furthermore, the high filler content of Aelite LS Posterior increases the material's stiffness. Aelite has a high elastic modulus compared with the Z250 and Silorane composites.[19] As a result, Aelite exhibits more contraction stresses at the adhesive interface even though it has low contraction shrinkage.[20] These stresses may have affected the bond strength of the material. However, this issue is controversial as other studies have found that Aelite demonstrated less stress values compared with the Z250 composite.[19,21]

Aging for two and four weeks decreased the bond strength for the two low-shrinkage composites tested. The decrease was statistically significant for the Silorane groups but not for the Aelite groups. In addition, a greater number of adhesive failures were seen between the incremental layers for both groups. This is in agreement with the findings of Shawkat and others[16] and Tezvergil-Mutluay and others.[22] Aging negatively affects the resin composites by causing water sorption, chemical degradation, and leaching out of some of the constituents of the material. Also, the amount of available unreacted double bonds is reduced, which, in turn, decreases the chemical reactivity of the material. All these changes result in a diminished bond strength between the aged sample and the repair composite material. [23,24]

Surprisingly, the repair strength of the Aelite specimens that were stored for four weeks (group 8) was not statistically different from that of the fresh group. However, when the failure modes were examined, the fresh layering specimens had a 9.1% (1 sample) adhesive failure while 66.7% (8 samples) showed adhesive failure in group 8. This indicates a diminished adhesive potential that could lead to a problem with the reliability and longevity of the repair.

After four weeks of aging, the bond strength between the incremental layers of the silorane composite without using an adhesive (group 3) was significantly lower than that of the fresh Silorane and fresh Z250 groups without using an adhesive (groups 1 and 11, respectively). Furthermore, the mode of failure in group 3 was mostly adhesive, in contrast to all cohesive failure in groups 1 and 11. Because the cationic ring-opening polymerization reaction is insensitive to oxygen, the incremental bond strength is dependent on the chemical reactivity of the material, which decreases over time. The chemical reactivity of silorane-based composite materials seems to undergo a significant drop with aging that could cause a low incremental bond strength in the Silorane restorations.[22]

In a clinical situation, clinicians usually remove the superficial layer of the old composite and roughen the surface before repairing the composite material. In addition, an intermediate layer, such as an adhesive material or a flowable composite material, is used to bind the old composite material to the new.[25,26] Therefore, the effect of the presence or absence of the oxygen-inhibited layer is eliminated. The resultant bond strength would therefore depend on the compatibility of the two types of composites and on the intermediate adhesive layer, if used.

In the present study, all the groups using an adhesive material between the layers showed high bond strength values, which were statistically similar to those of fresh bonding. The only exception was the repair of Silorane with Z250 using Adper Single Bond 2 adhesive (group 5). When the silorane was repaired after four weeks of aging using the silorane composite and adhesive (group 4), the bond strength increased to 93.7% of the value for fresh bonding and the predominant mode of failure was cohesive. In contrast, the four-week-aged group that was repaired without an adhesive (group 3) failed mostly adhesively. However, the use of a methacrylate-based adhesive and restorative material for the repair (group 5) resulted in a bond strength that was significantly lower than that of the fresh group (group 1). This is apparently due to the different chemical compositions of the two materials, which had an adverse effect on the bond strength between them. Shawkat and others[16] and Tezvergil-Mutluay and others[22] reported similar results regarding the incompatibility of the restorative composite resins. Therefore, the chemical compatibility between the old and the repair composite resins and use of the appropriate bonding agent are crucial for high incremental bond strength.

Although the repair of the aged Silorane substrates with Z250 drastically decreased the bond strength values of the specimens, it had a totally opposite effect on Aelite. Repairing the four-week-aged Aelite using the Z250 composite and Adper Single Bond 2 adhesive (group 10) resulted in a bond strength that was higher than that of both the fresh Aelite group (group 6) and the four-week-aged group repaired using the same material and One Step Plus adhesive (group 9). Furthermore, all the samples in group 10 failed cohesively. This result could be due to the lower viscosity of the Z250 composite compared with Aelite, which may have promoted better

adaptation and, therefore, resulted in a higher bond strength. In addition, Z250 has a lower elastic modulus compared with Aelite, which might have relieved the contraction stresses at the interface, thereby increasing the bond strength.

Further research is needed to examine the effect of C-factor on the incremental bond strength of different low-shrinkage composites. In addition, a further examination of the surface preparation procedures, such as roughening, removing the superficial layer, or using an adhesion promoter on the incremental bond strength of low-shrinkage resin-based composites, is warranted.

CONCLUSIONS

Within the limitations of this study, it can be concluded that

- Silorane-based resin composite incremental bond strength is comparable to that of methacrylate-based composite and is not affected by the absence of the oxygen-inhibited layer.
- Silorane-based resin composite should be repaired with the same material and a silorane adhesive should be used to gain adequate bond strength; repairing it with a methacrylate-based resin composite results in a significantly lower bond strength value.
- The low-shrinkage composite Aelite exhibited a lower incremental bond strength than Z250 and the silorane composite.
- Aelite low-shrinkage composite can be repaired with other brands of methacrylate-based resin composites without a deleterious effect on bond strength.

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Note

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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.

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Wear Properties of a Novel Resin Composite Compared to Human Enamel and Other Restorative Materials

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

Clinical Relevance

This *in vitro* study showed that the wear resistance of new resin composites may be similar to that of gold alloys and human enamel, which suggests that they are well suited for use as a restorative solution for the occlusal surfaces of posterior teeth.

SUMMARY

The purpose of this in vitro study was to compare the two-body wear resistance of human enamel, a pressable glass-ceramic (Imagine PressX), a type 3 gold alloy (Aurocast8), three resins composites currently available on the market (Enamel plus HRi, Filtek Supreme XTE, Ceram.X duo), and one recently introduced resin composite (Enamel plus HRi-Function). Resin composites were tested after simple light curing and after a further heat polymerization cycle. Ten cylindrical specimens (7 mm in diameter) were manufactured with each dental material according to standard laboratory procedures. Ten flat enamel specimens were obtained from freshly extracted human molars and included in the control group. All samples were subjected to a two-body wear test in a dual-axis chewing simulator over up to 120,000 loading cycles, against yttria stabilized tetragonal zirconia polycrystal cusps. Wear resistance was analyzed by measuring the vertical substance loss (mm) and the volume loss (mm^3). Antagonist wear (mm) was also recorded. Data were statistically analyzed using one-way analysis of variance

*Camillo D'Arcangelo, DDS, University of Chieti, Department of Operative Dentistry, Dental School, Chieti, Italy

Lorenzo Vanini, DDS, private practitioner, Chiasso, Switzerland

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

Mario Pirani, DDS, University of Chieti, Department of Operative Dentistry, Dental School, Chieti, Italy

Mirco Vadini, DDS, University of Chieti, Department of Operative Dentistry, Dental School, Chieti, Italy

Mariachiara Gattone, DDS, University of Chieti, Department of Operative Dentistry, Dental School, Chieti, Italy

Francesco De Angelis, PhD, DDS, University of Chieti, Department of Operative Dentistry, Dental School, Chieti, Italy

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

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(ANOVA) (wear depth and volume loss) and Kruskal-Wallis one-way ANOVA on ranks (antagonist wear). Heat-cured HRi function and Aurocast8 showed similar mean values for wear depth and volumetric loss, and their results did not statistically differ in comparison with the human enamel.

INTRODUCTION

The term "wear" refers to the net loss of a material from its surface under operational conditions. The phenomenon takes place rather frequently in the oral cavity and is dependent on many different factors that interact almost simultaneously:[1] the abrasive nature of food, neuromuscular force,[2] parafunctional habits,[3] enamel thickness and hardness (which depend on degree of mineralization),[4,5] and pH and nature of the saliva. On the occlusal surfaces of teeth in direct contact, as in parafunctional habits (bruxism and clenching), attrition occurs. Abrasion, on the other hand, takes place in normal function in the presence of a third body, such as food particles, during mastication.

Excessive antagonistic wear may lead to several problems, including hypersensitivity, loss of occlusal contact, destruction of periodontal tissue, loss of masticatory efficiency, faulty tooth relationship, fatigue of masticatory muscles, and changes in the vertical and horizontal jaw relations, which may cause functional and esthetic impairments.[6-10]

As with enamel or dentin, restorative materials are subjected to wear, and the wear mode depends on the type of restorative material. Material loss may occur through microploughing, microcutting, microcracking, and microfatigue.[11]

When dealing with severely compromised teeth, different kinds of metal-free restorations are available as alternatives for metal-supported dental restorations. All-ceramic restorations consist entirely of porcelain or, alternatively, a high-strength ceramic substructure, which is veneered with porcelain. Adhesively luted resin composite indirect restorations[12,13] (onlays and overlays) may also represent a valid[14-16] and conservative approach.

A novel resin-based composite (Enamel plus HRi-Function, Micerium, Avegno, Genova, Italy) has been recently fabricated and proposed as a restorative for direct and indirect posterior restorations and as an esthetic veneering material over metal frameworks for fixed dental and implant supported prostheses. For this new material, the manufacturer claims that the wear characteristics are very close to that of human enamel.

The purpose of this study was to evaluate the *in vitro* two-body wear resistance of the recently introduced resin based composite (HRi-Function) subjected to 120,000 chewing simulation cycles and to compare the results with the wear resistance of human enamel and different dental materials typically used to restore posterior teeth. The null hypothesis tested was that no difference could be detected among the materials under investigation concerning the wear properties.

METHODS AND MATERIALS

Materials used in this study included a silicon oxide (SiO) based pressable glass-ceramic (Imagine PressX, shade A1, Wieland Dental Ceramics, Pforzheim, Germany), a type 3 gold alloy (Aurocast8, Nobil-Metal, Villafranca d'Asti, Asti, Italy), three resin composites currently available on the market (Enamel plus HRi, Micerium, Avegno, Genova, Italy; Filtek Supreme XTE, 3M ESPE, Seefeld, Germany; Ceram.X duo, Dentsply DeTtrey GmbH, Konstanz, Germany), and a recently introduced microhybrid resin composite (Enamel plus HRi-Function, Micerium).

Ten pressable ceramic specimens (n=10) were fabricated according to the conventional lost-wax technique: plexiglass (Plexiglas, Evonik Röhm GmbH, Darmstadt, Germany) discs, 7 mm in diameter and 6 mm thick, were prepared, invested, and burned out by heat. The void was filled with the pressable ceramic material and pressed at a temperature of 930°C for 20 minutes.

Resin composite cylindrical specimens were obtained by placing uncured pastes inside translucent polyethylene cylindrical molds with an inner diameter of 7 mm and height of 6 mm. Each mold was put on a glass surface and then filled with composite resin in three increments of about 2 mm. Each layer was individually light cured for 40 seconds (L.E. Demetron I with a 1200 mW/cm^2 output, Sybron/Kerr, Orange, CA, USA). The distance of the tip from the specimen was maintained at 1 mm. Twenty samples were produced for each one of the three resin composites under investigation: half of them, ten for each material (n=10), were assigned to the light-cured groups. The remaining 40 cylinders (n=10) were subjected to an additional cycle of heat-curing in a composite oven for 10 minutes (LaborLux, Micerium) and assigned to the heat-cured groups. All composite surfaces were ground

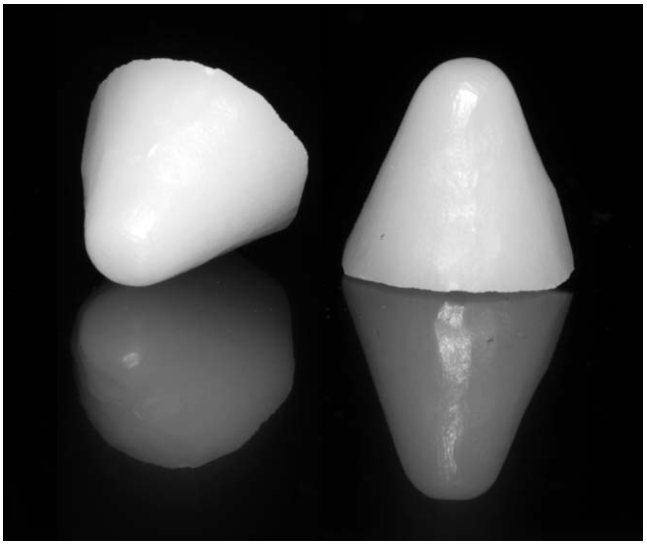

Figure 1. *Standard zirconia cusps used as abraders in the chewing simulation test.*

with 600-grit silicon carbide (SiC) paper under running water for 30 seconds and subjected to finishing and polishing procedures: diamond pastes (Shiny A, 3 μm, and Shiny B, 1 μm;, Micerium) were applied with a soft goat's-hair brush (Micerium) and then polished with aluminum oxide paste (Shiny C, Micerium).

Ten gold alloy samples were made using the traditional lost wax technique, according to the manufacturers' directions.

For the control group, flat human enamel specimens (n=10) were obtained from 10 caries-free, freshly extracted human molars, collected in accordance with the guidelines specified by the local human subjects oversight committee. The teeth were stored in an aqueous solution of 0.5% chloramine T (Delchimica Scientific Glassware, Napoli, Italy) at 4°C until the beginning of the experiment, but no longer than 1 week after extraction. The crown portion of each tooth was sectioned horizontally to the buccal cementoenamel junction, using a cylindrical diamond rotary cutting instrument (Intensiv 314, 1.4 mm in diameter, L.8.0 mm, Intensiv SA, Grancia, Switzerland) mounted on a high-speed handpiece (Bora L, Bien-Air Dental, Bienne, Switzerland) with water-spray cooling. Each crown was then abraded on the buccal aspect with 1200-grit abrasive paper and finished with 4000-grit abrasive paper to a depth of 0.5 mm in order to achieve a flat area of about 5 mm in diameter for loading during the wear test. The enamel surfaces were then examined under a stereomicroscope (Nikon SMZ10, Tokyo, Japan) to ensure that they were free of exposed dentin.

Table 1: *Configuration of the Parameters Set for the Wear Method*

Parameter	Value
Number of cycles	120,000
Force	5 kg
Height	3 mm
Lateral movement	–0.7 mm
Descending speed	60 mm/s
Lifting speed	60 mm/s
Feed speed	40 mm/s
Return speed	40 mm/s
Frequency	1.6 Hz

All specimens were stored in distilled water for 24 hours at 37°C before wear simulation.

Wear Testing and Measurements

Cylindrical specimens were embedded in acrylic resin and then subjected to a two-body wear test in a dual axis chewing simulator (Willytec, Munich, Germany). As antagonists, standard cusps with a slight conical shape and a round tip 3 mm in diameter (Figure 1) were milled from yttria stabilized tetragonal zirconia polycrystalline blocks, using a computer-aided milling machine (Dental CAD/CAM GN-1, GC, Tokyo, Japan). These specimens were finally polished with 6 μm diamond pastes.

Cusps were embedded in autopolymerizing acrylic resin using custom-made copper specimen holders. 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. All other test parameters have been summarized in Table 1. Each sample was loaded at 1.6 Hz for a total of 120,000 chewing cycles.

Subsequently, samples were subjected to a quantitative surface analysis using a CAD/CAM three-dimensional contact scanner (Renishaw Dental Scanner, Renishaw, Wotton-under-Edge, UK). From each worn surface, a three-dimensional mesh was obtained. Vertex position accuracy in the mesh could be approximated to 1 μm, according to the information provided by the scanner manufacturer (Renishaw). The three-dimensional mesh was imported into a computer-aided design software (AutoCAD 2009, Autodesk Inc, San Rafael, CA, USA). A reference plane for the depth measurement was defined, using three points on the mesh, chosen on the flat unworn

Table 2: *Mean Values (and Standard Deviations) for Wear Depth, Volume Loss, and Antagonist Wear Achieved in the Experimental Groups*[a]

	Wear Depth (mm)	Volume Loss (mm^3)	Antagonist Wear (mm)
HRi light-cured	0.485 (0.064)[e]	1.452 (0.245)[d]	0.010 (0.007)[a,b,c]
Filtek XTE light-cured	0.464 (0.069)[e]	0.972 (0.247)[c]	0.004 (0.002)[a,b,c]
Ceram.X duo light-cured	0.416 (0.073)[d,e]	0.894 (0.259)[c]	0.007 (0.007)[a,b,c]
HRi-Function light-cured	0.335 (0.069)[b,c,d]	0.529 (0.139)[a,b]	0.006 (0.001)[a,b,c]
HRi heat-cured	0.463 (0.063)[e]	1.016 (0.198)[c]	0.015 (0.013)[a,b]
Filtek XTE heat-cured	0.459 (0.068)[e]	1.017 (0.239)[c]	0.016 (0.017)[a,b]
Ceram.X duo heat-cured	0.409 (0.118)[c,d,e]	0.806 (0.397)[b,c]	0.015 (0.011)[a,b]
HRi-Function heat-cured	0.276 (0.058)[a,b]	0.464 (0.191)[a]	0.016 (0.003)[a]
Wieland Imagine PressX	0.303 (0.065)[a,b,c]	0.531 (0.143)[a,b]	0.002 (0.002)[c]
Aurocast8 by NobilMetal	0.219 (0.060)[a]	0.328 (0.140)[a]	0.004 (0.005)[b,c]
Human Enamel	0.216 (0.070)[a]	0.404 (0.200)[a]	0.013 (0.004)[a,b]

[a] *Same superscript letters indicate no statistically significant differences (p>.05).*

surface surrounding the abraded area. In this way, the unworn surface was aligned to the XY plane. The most distant vertices from the reference plane were detected. The three-dimensional coordinates of the deepest vertex were measured and its Z coordinate, expressed in millimeters (mm), was assumed as the vertical substance loss for that sample. The volume delimited between the reference plane and the worn surface was calculated and assumed as the volumetric loss (mm^3). Moreover, the height of each zirconia cusp was measured before and after each test using a digital caliper with an accuracy of 1 μm. The height difference between the pretest and posttest measurements of each cusp was recorded as the antagonist wear (mm).

After the three-dimensional evaluation, the wear facets on the abraded samples were also sputter coated (except the gold alloy samples) and subjected to a scanning electron microscope (SEM) analysis (EVO 50 XVP LaB6, Carl Zeiss SMT Ltd, Cambridge, UK) at different magnifications.

Statistical Analysis

Mean and standard deviation values for wear depth, volume loss, and antagonist wear were calculated in each group. Data were statistically analyzed using SPSS 11.0 statistical software (SPSS Inc, Chicago, IL, USA).

After determining that both wear depth and volume loss data were normally distributed, mean values were compared using one-way analysis of variance (ANOVA) tests; multiple comparisons were performed according to the Tukey method.

Data for antagonist wear was not normally distributed. As a consequence, a Kruskal-Wallis one-way ANOVA on ranks was performed to compare the median values; multiple comparisons were executed according to Dunn's method. The significance level was set at $\alpha=0.05$ in all tests.

RESULTS

Table 2 displays the mean values for wear depth and volume loss recorded in each category after 120,000 chewing simulation cycles. The wear recorded for the antagonistic cusps is also shown. Representative SEM images of the wear facets that could be observed in the experimental groups are shown in Figure 2. Samples made of pressable glass-ceramic (Imagine PressX), heat-cured novel resin composite (Enamel plus HRi-function), and type 3 gold alloy (Aurocast8) led to statistically similar wear depth and volumetric loss values, which were generally lower than what was observed for the remaining groups; moreover, these materials were not statistically different in mean wear values compared with human enamel. The highest mean values for wear depth were recorded in the Enamel plus HRi and Filtek Supreme XTE light-cured groups, with no statistically significant differences in comparison with the Ceram.X group.

DISCUSSION

Wear in the oral cavity is a relevant factor that should be carefully considered when selecting the appropriate restorative material in clinical dental practice. An ideal material possesses both high wear resistance and minimum abrasiveness: a restorative material that replaces enamel and/or opposing enamel should have functional characteristics as similar as possible to natural enamel.[17]

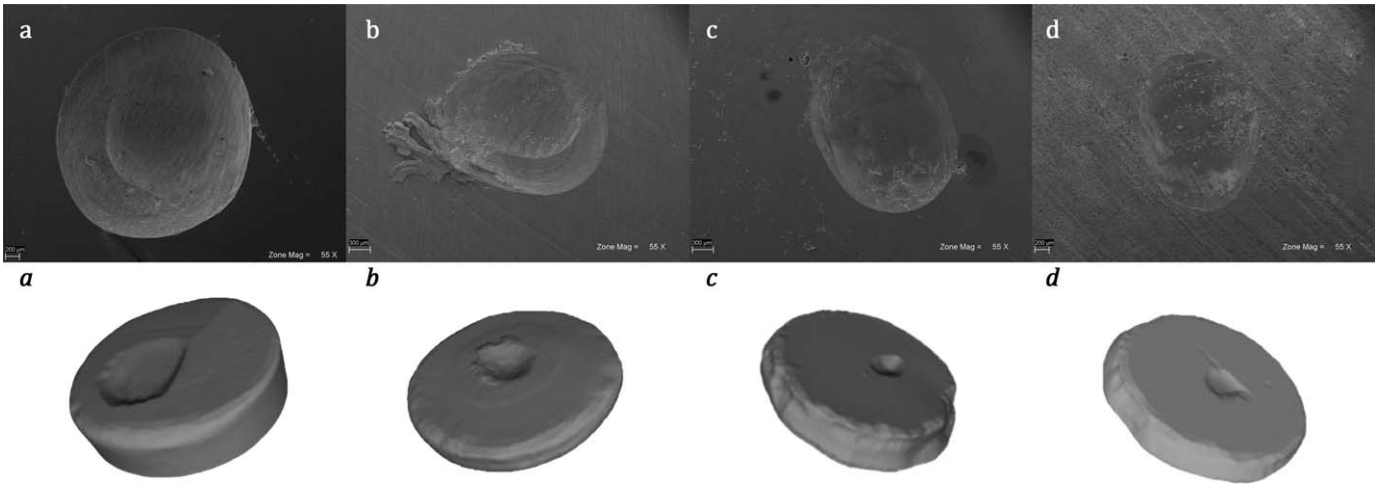

Figure 2. *Scanning electron microphotographs (upper row; original magnification 55×) and three-dimensional meshes (lower row) showing the wear facets of some representative samples from the Filtek XTE heat-cured group (a), Aurocast8 gold alloy group (b), HRi-Function heat-cured group (c), and Imagine PressX glass-ceramic group (d).*

The null hypothesis tested in the present study, which assumed no difference in terms of wear properties among the several restoratives under investigation, has to be rejected.

The type 3 gold alloy (Aurocast8) showed wear behavior very similar to that of human enamel. Over the past decades, the clinical choice of placing metal or gold on the occlusal surfaces proved to be a valid alternative in all cases where the prosthetic occlusion was in contact with enamel, resin composite, porcelain, or a combination of such materials:[18] it has been previously reported as a fairly appropriate solution from a functional point of view,[19] causing minimal or no wear to the opposing occlusal materials.[20] It may be supposed that gold-based alloys hardly interfere with the occlusal balance of patients subjected to prosthetic rehabilitations.[21] The unsightly display of metal is considered one of the main disadvantages of using gold occlusal surfaces.

Also, the new heat-cured resin composite HRi-Function, whose wear depth and volume loss were statistically comparable to those of gold, was not significantly different from human enamel. In our opinion, dental materials that show a wear behavior as close as possible to natural tooth hard tissues should always be preferred in clinical practice, as they are unlikely to interfere strongly with the patient's musculoskeletal equilibrium.

All the other resin composites tested in the present study (Filtek Supreme XTE, Enamel plus HRi, Ceram.X duo) led to increased wear values in the light-cured and the heat-cured groups. In clinical conditions, excessive occlusal wear is not desirable as it may determine loss of occlusal contact, loss of masticatory efficiency, altered tooth and jaw relationships, and muscular fatigue, ultimately compromising both function and esthetics.[6-10] This is clinically quite relevant mainly when dealing with parafunctional patients.

The HRi-Function resin based composite tested in the present study was recently introduced by the manufacturer with the aim of improving mechanical properties and achieving predictable long-term clinical outcomes when used to restore occlusal surfaces on posterior teeth. From this point of view, it can be considered novel, as it is not simply an evolution of Enamel plus HRi: it is basically a different material. The enamel masses of Enamel plus HRi are nanohybrid resin composites, studied to guarantee the best esthetics, mainly on anterior teeth, thanks to the same refractive index (RI) as human enamel. On the other hand, HRi-Function has to be considered as a micro-hybrid resin composite: it lacks the favorable optical properties of Enamel plus HRi (same RI as human enamel) and has been formulated putting the greatest efforts toward optimizing the bond between the filler particles and the resin matrix.

In this study the HRi-Function mean wear values appeared extremely promising. They were the closest to human enamel among the light-cured resin composites under investigation. Furthermore, the heat-cured samples were statistically similar to the wear values achieved with Aurocast8 gold alloy. These results may suggest that the newly proposed HRi-Function is a restorative material functionally

similar to gold alloy in terms of wear behavior, while maintaining all the advantageous peculiarities of a resin composite: a good esthetic appearance, ease of handling and workability, an established predictability of the achievable clinical results, and low cost. Moreover, being a resin composite, this material is primarily suitable for direct use, though it probably exhibits the best functional performances in indirect use for manufacturing adhesively luted indirect partial restorations and as an esthetic veneering material over the metallic frameworks of fixed dental or implant supported prostheses.

In the present study the wear resistance of a SiO-based glass ceramic was also investigated, as glass ceramics are among the most commonly used materials for metal-free single crowns and inlays or onlays; its wear behavior was fairly favorable as it was statistically similar to human enamel in wear depth and volume loss. However, glass-ceramic materials for fixed reconstructions require a certain thickness to have adequate fracture resistance, whereas resin materials are more fracture resistant even in thin reconstructions.[22-24] Moreover, it was previously reported that the wear characteristics of resin-based materials offer some advantages over glass ceramics as they yield less wear in the antagonist as opposed to human enamel.[22,25,26] Finally, dental ceramics cannot be used in a direct technique, making the intraoral repair procedures less reliable and generally leading to a higher overall restoration cost.

As clinical evaluation of wear is expensive, time consuming, and methodologically rather complicated, mastication simulators have been developed in an attempt to simulate the oral environment and produce wear in test specimens.[27] Previous *in vitro* studies[28] used the Willytec chewing simulator[29] and thermal cycling[30] to evaluate two-body[31] or three-body [1] wear resistance of different materials. Antagonists of different shapes[1,32-34] and structures[35,36,37] have also been used in previous research. Heintze and others[38] emphasized the need for using a standardized form of artificial antagonist for comparisons because human enamel requires extensive preparation to achieve an adequate shape of the abrader. Moreover, its natural substrate may show wide variability from one person to another and among different teeth in the same mouth concerning the degree of mineralization and thickness.

Therefore, zirconia ceramic balls have been proposed [38] as antagonistic abraders to properly evaluate the wear of human enamel and dental materials[30] in standardized experimental conditions. In comparison with other artificial abraders (ie, steatite), zirconia ceramic balls retained their shape during the entire test period, so the influence of changes in the antagonist's surface on wear of specimens can be minimized.[35,37]

CONCLUSIONS

The ongoing evolution of dental restoratives is leading to the development of resin composites that are becoming comparable to that of gold alloys in terms of functional qualities, although they maintain their inherent esthetic features, ease of use, and low cost.

The wear properties of the newly introduced HRi-Function resin composite proved to be similar to that of the Aurocast8 type 3 gold alloy and human enamel, indicating that it is a suitable material for the occlusal surfaces of posterior teeth.

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.

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Evaluation of Tensile Retention of Y-TZP Crowns After Long-term Aging: Effect of the Core Substrate and Crown Surface Conditioning

R Amaral • M Rippe • BG Oliveira
PF Cesar • MA Bottino • LF Valandro

Clinical Relevance

The retention of Y-TZP crowns seems to be higher when luted to dentin rather than resin composite. When cementing a Y-TZP crown using an MDP-based resin cement, tribosilicatization and application of a thin layer of low-fusing porcelain glaze might improve the retention.

Regina Amaral, PhD, Sao Paulo State University (UNESP), PhD Graduate Program (Prosthetic Dentistry Unit), São José dos Campos, SP, Brazil

Marilia Rippe, PhD, Sao Paulo State University (UNESP), Graduate Program (Prosthetic Dentistry Unit), São José dos Campos, SP, Brazil

Bruno G Oliveira, DDS (c), Sao Paulo University (USP), Faculty of Dentistry, São Paulo, SP, Brazil

Paulo Francisco Cesar, PhD, Sao Paulo University (USP), Dental Materials Biomaterials and Oral Biology, São Paulo, SP, Brazil

Marco Antonio Bottino, PhD, Sao Paulo State University (UNESP), Dental Materials and Prosthodontics, São José dos Campos, SP, Brazil

*Luiz Felipe Valandro, PhD, Federal University of Santa Maria, Restorative Dentistry (Prosthodontics), Santa Maria, RS, Brazil

*Corresponding author: R. Floriano Peixoto 1184, Santa Maria, RS 97015-372, Brazil; e-mail: lfvalandro@hotmail.com

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SUMMARY

This study evaluated the effect of the core substrate type (dentin and composite resin) on the retention of crowns made of yttrium oxide stabilized tetragonal zirconia polycrystal (Y-TZP), submitted to three inner surface conditionings. For this purpose, 72 freshly extracted molars were embedded in acrylic resin, perpendicular to the long axis, and prepared for full crowns: 36 specimens had crown preparations in dentin; the remaining 36 teeth had the crowns removed, and crown preparations were reconstructed with composite resin plus fiber posts with dimensions identical to the prepared dentin. The preparations were impressed using addition silicone, and 72 Y-TZP copings for the tensile test were produced. Cementation was performed with a dual-cured cement containing phosphate monomers. For cementation, the crown preparation (dentin or resin) was conditioned with the adhesive sys-

tem, and the ceramic was subjected to one of three surface treatments: isopropyl alcohol, tribochemical silica coating, or thin low-fusing glassy porcelain layer application plus silanization. After 24 hours, all specimens were submitted to thermocycling (6000 cycles) and placed in a special tensile testing device in a universal testing machine to determine failure loads. The failure modes of all samples were analyzed under a stereomicroscope. Two-way analysis of variance showed that the surface treatment and substrate type (α=0.05) affected the tensile retention results. The dentin substrate presented the highest tensile retention values, regardless of the surface treatment. When the substrate was resin, the tribochemical silica coating and low-fusing glaze application plus silanization groups showed the higher retention values.

INTRODUCTION

Yttrium oxide stabilized zirconia polycrystal (Y-TZP) ceramic has high flexural strength (around 1000 MPa)[1] and fracture toughness (> 9-10 MPa $m^{1/2}$) in comparison with other dental ceramics.[2] Due to these outstanding mechanical properties, Y-TZP has been successfully used to build the infrastructure for all-ceramic crowns and bridges using computer-aided design – computer-aided manufacture (CAD-CAM) technology. Despite the good mechanical behavior, Y-TZP is a highly crystalline material, without a vitreous phase,[1] which impairs adhesion to resin cements. Therefore, different adhesion strategies have been proposed to improve the bond strength of Y-TZP to resin cements, such as ceramic surface conditioning methods, application of ceramic primers, and/or cements containing phosphate monomers.

Surface treatments on Y-TZP substrates have been extensively explored in the recent literature to improve the bond strength of resin cement to this type of ceramic.[3-10] However, the results of these studies are contradictory. For example, Ernst and others[11] compared 12 different types of cements and showed that the greatest retention value was obtained by a self-cure dental adhesive resin cement based on methyl methacrylate when tribochemical silica coating was used as the ceramic surface treatment, but the reported retention value was not statistically different from that obtained for the group with the same resin cement without the application of tribochemical silica coating.

An alternative adhesive approach has been recently introduced to improve the bond strength between Y-TZP and resin cements without the necessity of using air abrasion procedures, which may result in permanent damage of the inner surface of the crowns.[6,7,9,10,12-14] This novel surface treatment involves the application of a thin layer of low-fusing glass to the cementation surface of Y-TZP crowns in order to enrich that surface with silicon oxides and enable hydrofluoric acid etching, which can create micromechanical retention along the surface, improving bond strength.

Even though there are several laboratory studies showing that both tribochemical sandblasting and the application of a glass film can lead to improved bond strength values, few of those studies evaluated the effect of these surface treatments using an *in vitro* design that takes into account the geometry of prosthetic dental crowns, such as using the retention force test on CAD-CAM machined Y-TZP crowns.[11,15]

Another key factor regarding all-ceramic restorations is the substrate over which it will be cemented. In some clinical situations, there is a lack of remaining dental tissue so that intracanal anchorage is necessary to retain a resin composite core and enhance retention of the prosthetic crown. However, the bond strength between the resin core and the resin cement is different from that observed between a dentin core and the resin cement, which involves the formation of a hybrid layer.

The objective of this current study was to evaluate the retention force of Y-TZP crowns as a function of the cementation substrate (dentin or resin composite) and the surface treatment of the intaglio surface of the Y-TZP crowns. The hypotheses were that: 1) a dentin substrate would promote higher crown retention than a composite substrate; 2) the Y-TZP surface treatment would influence the retention values, regardless of the substrate.

METHODS AND MATERIALS

In order to determine the number of teeth per group, a sample calculation was made based on two other articles.[15,16] In order to achieve a statistical power of 80%, mean standard deviation of 1.7 and a detectable difference of 2.3 MPa, the experimental group size was determined to be 12, for a total of 72 teeth divided into six experimental groups.

The 72 teeth were numbered and, using a computer program (www.randomizer.org), divided randomly into six testing groups (Table 1).

Tooth Embedding

The coronal part of the tooth was bonded to an adapted surveyor for keeping the long axis of the

Table 1: *Testing Groups*

Groups (n=12)	Substrate	Y-TZP Surface Treatment
D-alc	Dentin	Isopropyl alcohol
D-sil		Silicatization + silanization
D-vit		Vitrification
C-alc	Fiber post + composite resin	Isopropyl alcohol
C-sil		Silicatization + silanization
C-vit		Vitrification

tooth perpendicular to the ground (horizontal plane). Self-cured acrylic resin (Dencrilay, Dencril, Caieiras, SP, Brazil) was prepared and poured into a matrix. The tooth was then inserted into the resin up to 3 mm below the cementoenamel junction.

Dental Preparation

The occlusal surface of all teeth was cut by a diamond blade mounted on a cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) 4 mm above the cementoenamel junction. Conical trunk diamond burs (KG 3139 and KG 3139FF, KG Sorensen, Cotia, Brazil) were mounted in a high-speed handpiece and fixed to a modified optical microscope, enabling tooth reduction to be obtained as parallel as possible to the long axis of the tooth. Thus, the axial part was reduced to a depth of 1.5 mm (same as the bur diameter), and a standard angle of convergence was created. The height of the preparation was 4 mm.

Composite Substrate

The 72 prepared teeth were divided into two groups according to the type of coronal substrate: 36 teeth were prepared for a full crown with the exposed dentin; 36 teeth received a fiber post and composite core.

The following procedures were performed for making the resin substrate:

1) An impression of each prepared crown was made using polyvinylsiloxane (Elite Light + Body Normal Set, Zhermack, Badia Polesine, Italy). After producing the master die, a silicone matrix was fabricated to reproduce the preparation. Thus, the future reconstruction in composite had the same characteristics as the full crown preparation.
2) For post cementation, the greatest root canal of each molar received prosthetic preparation with a custom no. 2 drill of a glass fiber post system (White Post DC, FGM, Joinville, Brazil). Afterwards, the coronal and root dentin was prepared using a two-step etch and rinse adhesive system (Ambar, FGM). The dentin was etched with 37% phosphoric acid, rinsed with water for 5 seconds and dried with absorbent papers. The adhesive agent was then applied as recommended by the manufacturer and light-cured for 20 seconds (Radii-cal, SDI, Bayswater, VIC, Australia). The posts received silane (Prosil, FGM) and were cemented with a dual-cure resin cement (Allcem, FGM). The core was built up with a composite resin (Opallis), using the previously fabricated silicone matrix. The specimens were stored in water (37°C) for 24 hours. All preparations were finished with fine conical trunk diamond burs (3139FF, KG Sorensen).

Crown Manufacture

The preparations of the 72 specimens were impressed using polyvinylsiloxane (Elite Light + Body Normal Set, Zhermack), and the dies obtained were taken to the CEREC MC XL inLab (Sirona Dental Systems GmbH, Bensheim, Germany) to manufacture the crowns using the Software inLab 3.60. The copings were designed with retention on the occlusal surface for subsequent tensile testing, and the milling was performed with VITA In-Ceram 2000 YZ CUBES (VITA Zahnfabrik, Bad Säckingen, Germany), followed by sintering the crowns in a furnace (Zircomat, VITA Zahnfabrik).

Cementation

All of the crowns were cemented using a resin cement containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) (Panavia F, Kuraray Medical Inc, Okayama, Japan). The dentin and the composite resin core of all teeth were pretreated with the self-etching adhesive system (Panavia F ED Primer, Kuraray) and were luted with the resin cement according to the manufacturer's recommendations. Each infrastructure was cemented using a device that induced a force of 750 g over the Y-TZP infrastructure, according to the strategies described in Table 1.

The inner surface of the Y-TZP copings of groups 1 and 4 was only cleaned with isopropyl alcohol and silanized with silane agent (ESPE-Sil, 3M ESPE, St Paul, MN, USA), which was left undisturbed for 5 minutes for complete evaporation.

The inner surface of the copings of groups 2 and 5 was air-abraded with 30 μm particles of aluminum

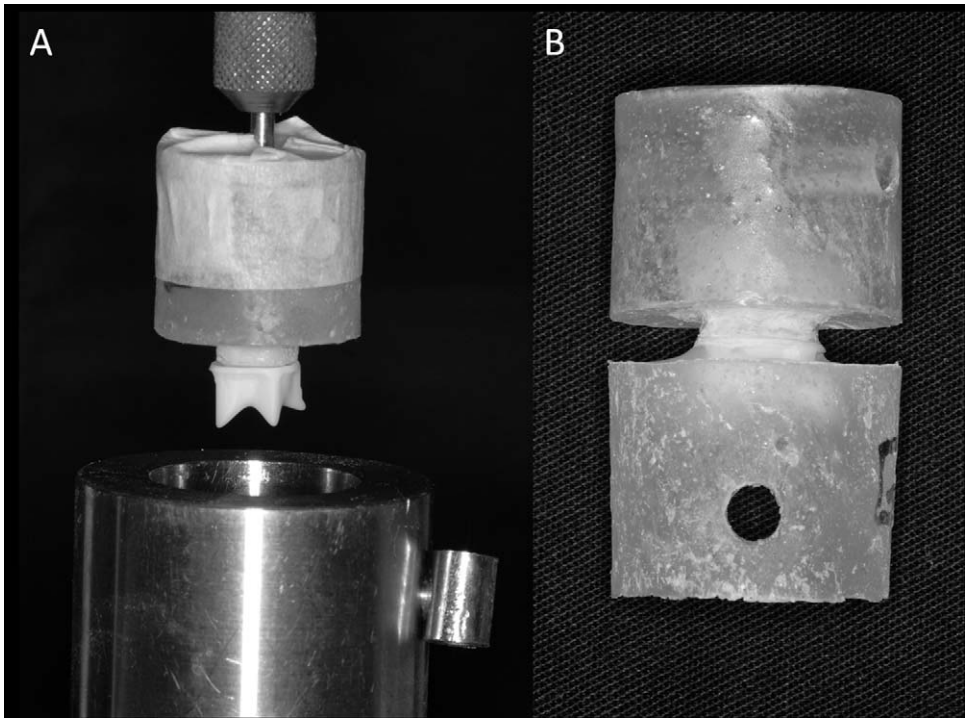

Figure 1. *Preparation of the specimen for tensile testing. (A) Specimen positioned for embedding the Y-TZP crown inside of acrylic resin. (B) Specimen with both tooth and crown embedded in resin to be tested.*

oxide coated with silicon (CoJet sand, 3M ESPE). The sandblasting was performed using an adapted device[17] and a constant pressure of 2.8 bars at a distance of 15 mm from the occlusal infrastructure region, with circular movements. The infrastructure was silanized with a silane agent (ESPE-Sil, 3M ESPE), which was left undisturbed for 5 minutes for complete evaporation.

The inner surface of the copings of groups 3 and 6 received application of a thin layer of a low-fusing porcelain glaze (VITA AKZENT, VITA Zahnfabrik) using a brush. The infrastructure was subjected to a sintering cycle of the glaze material as recommended by the manufacturer. The surface was then etched with hydrofluoric acid for 1 minute, washed with water, and air-dried. Subsequently, the copings were cleaned again in a sonic device (5 minutes in distilled H_2O). The infrastructure was silanized with a silane agent (ESPE-Sil, 3M ESPE), which was left undisturbed for 5 minutes for complete evaporation.

Thermocycling

After cementation, all specimens were stored in distilled water at 37°C for 24 hours and then were submitted to thermocycling (6000 cycles) between baths of 5°C and 55°C, according to Ernst and others[11] and Palacios and others.[15]

Tensile Test

The infrastructure was embedded in acrylic resin (Dencrilay, Dencril) until it covered the retention areas that had been prepared on the crowns (Figure 1). This procedure was performed following the same axis of the root embedding, with the aid of a suitable surveyor.

For testing, the lower base of the assembly was fixed on a universal testing machine (DL-2000, Emic, São José dos Pinhais, PR, Brazil) and the upper base was connected to a mobile device, using a load cell of 1000 N; the tensile strength test was performed at a speed of 0.5 mm/min (Figure 2).

Failure Analysis

The fractured interfacial surface of all the specimens was analyzed using a stereomicroscope (SteREO Discovery V20, Carl Zeiss, Göttingen, Germany). The failure mode was classified as:

- predominantly adhesive at core-cement interface, when over 50% of cement remained on the crown inner surface;
- predominantly adhesive at Y-TZP-cement interface when over 50% of cement remained on the prosthetic preparation; and
- catastrophic failure (post debonding and root fracture).

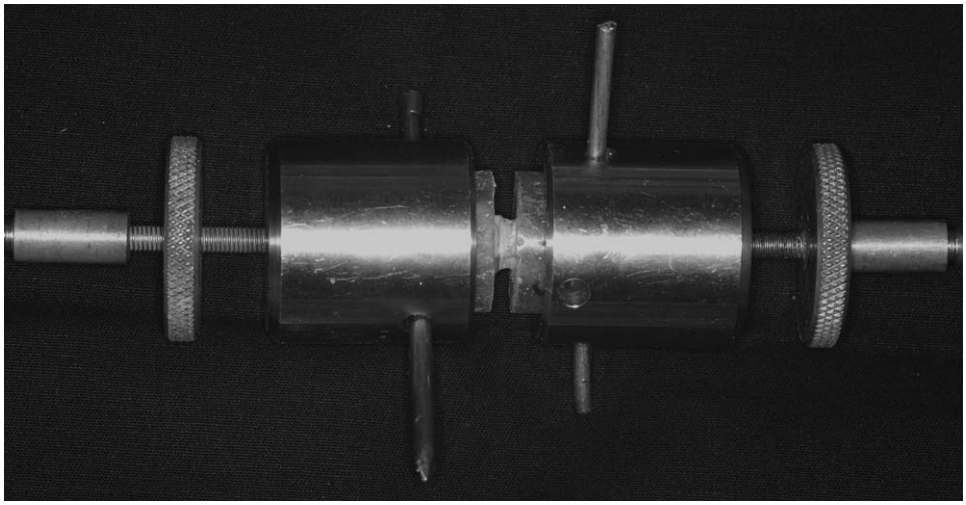

Figure 2. *Specimen placed for tensile test. The universal joints at the two superior and inferior sides can be noted at the ends.*

Statistical Analysis

The tensile retention data were compared using two-way analysis of variance (ANOVA) (substrate and surface treatment factors) and further by Tukey and the *post hoc* Student *t*-tests ($p<0.05$) for pairwise comparisons.

RESULTS

The two-way ANOVA showed that both Y-TZP surface treatment ($p<0.001$) and core substrate type ($p<0.0001$) significantly affected the retention force of the crowns. There was not significant interaction between the surface treatment and substrate ($p>0.105$). Table 2 shows the descriptive analysis and the Tukey test to elucidate the comparisons of the groups.

For crowns cemented on the dentin substrate, no difference was observed among the surface treatments tested ($p>0.05$) (D-alc = D-sil = D-vit) (Table 2). On the other hand, when crowns were cemented on a composite substrate, ANOVA showed that the surface treatment significantly influenced the retention force, with the tribosilicatization and low-fusing porcelain glaze application resulting in significantly higher mean retention values when compared to the control group (alcohol) (Table 2).

When the two core substrates (dentin and composite resin) were compared (Table 2), it is possible to note that bonding to dentin resulted in significantly higher retention mean values when compared to resin composite for both the control and silicatization groups. For the glazed specimens, there was no significant effect of the substrate on retention force.

The failure analysis indicated that for the groups in which the crowns were cemented on dentin, there was a higher percentage of adhesive failure at the Y-TZP-cement interface (cement on the crown inner surface), except for group D-alc (C-alc also), which had failures at the Y-TZP-cement interface (cement on the dental preparation). In general, when conditioning the Y-TZP surface, the failure occurred at the core-cement interface (cement remained on the Y-TZP crown inner surface) (Table 3). Most catastroph-

Table 2: *Mean (and Standard Deviation) of the Results of the Tensile Retention (N) for Different Groups**

Substrates	Y-TZP Treatments		
	Alcohol	Silicatization	Vitrification
Dentin	20.8 ± 8.1^{Aa}	24.7 ± 8^{Aa}	25.4 ± 3.4^{Aa}
Composite resin	5.2 ± 5.3^{Bb}	14.2 ± 6.9^{Ab}	20.5 ± 7.7^{Aa}

Abbreviation: Y-TZP, yttrium oxide stabilized zirconia polycrystal.
** The different lowercase letters indicate a significant difference ($p<0.05$) between the substrate types maintaining the same surface treatment. Different capital letters indicate a significant difference ($p<0.05$) between surface treatment types maintaining the same substrate.*

Table 3: *Number and percentages of failure types of the specimens from different groups.*

Groups	Failure Types		
	Adhesive at Core-Cement Interface[a]	Adhesive at Y-TZP-Cement Interface[b]	Catastrophic Failure[c]
D-alc	2 (16.7%)	10 (83.3%)	0 (0%)
D-sil	5 (41.6%)	2 (16.7%)	5 (41.7%)
D-vit	7 (58.4%)	1 (8.3%)	4 (33.3%)
C-alc	0 (0%)	10 (83.3%)	2 (16.7%)
C-sil	7 (58.3%)	2 (16.7%)	3 (25%)
C-vit	1 (8.3%)	6 (50%)	5 (41.7%)

Abbreviation: Y-TZP, yttrium oxide stabilized zirconia polycrystal.
[a] Over 50% of cement on the crown inner surface.
[b] Over 50% of cement on the prosthetic preparation.
[c] Catastrophic failure: post debonding and root fracture.

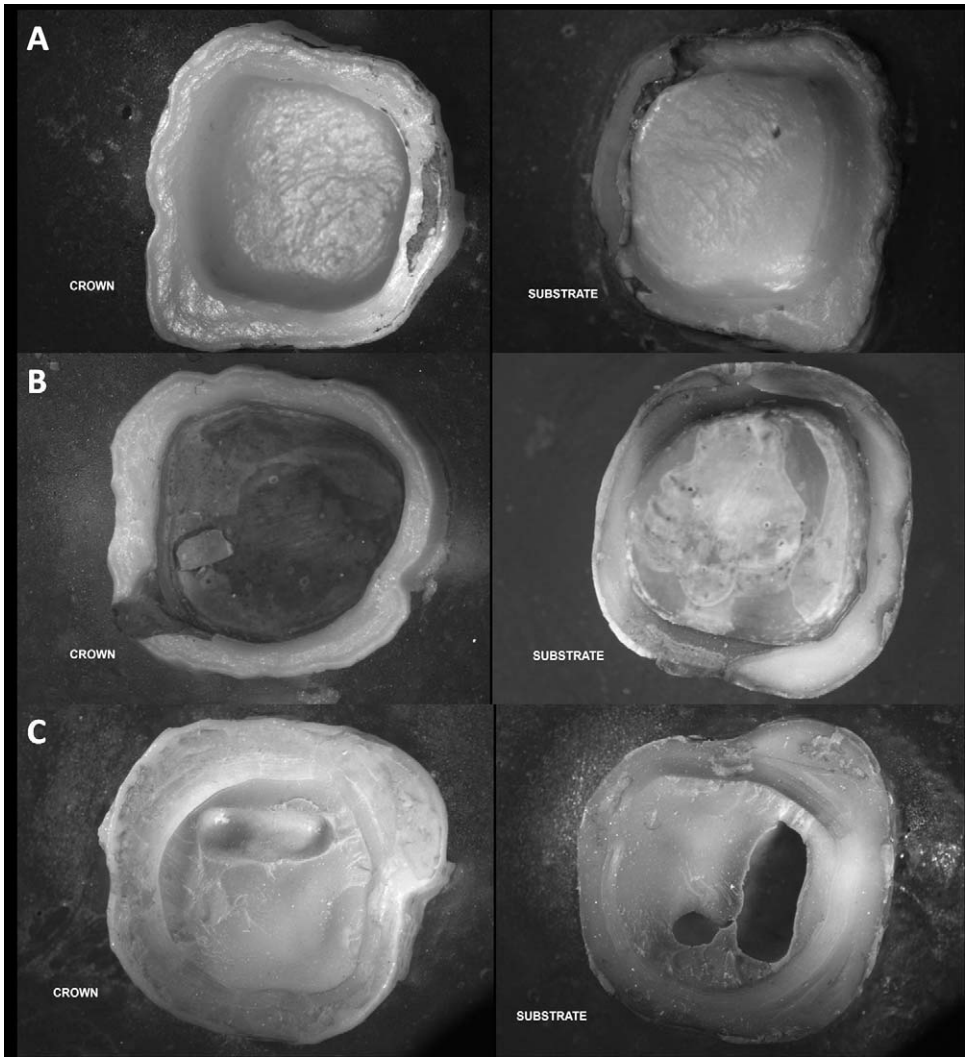

Figure 3. *Photographs from stereomicroscopy of the failure types.*

ic failures (41.7%) occurred in the groups D-sil and C-Vit. Representative stereomicroscope images of fractured specimens are presented in Figure 3.

DISCUSSION

The first hypothesis of this present study was partially accepted because the dentin substrate only resulted in significantly higher retention force for the control and silicatization groups. For the glazed specimens, both substrates resulted in similar retention values. It is possible that the better retention observed for dentin in two of the experimental groups may have occurred because the chemical composition of the resin cement used in the present investigation had a better adhesive interaction with the dentin substrate than with the resin composite. The self-etching adhesive from Panavia forms a hybrid layer with the dentin by dissolving/modifying the smear layer. Therefore, the Panavia cement penetrated into the dentinal tubules, providing micromechanical retention between adhesive and dentin. Additionally, this cement contains the MDP molecule with two ends. One end has vinyl groups that react with the monomers of the resin cement when polymerized. At the other end, hydrophilic phosphate ester groups bond strongly with metal oxides, such as alumina (Al_2O_3) and zirconia (ZrO_2),[18] and with the calcium hydroxyapatite of the dentin and enamel, which does not occur with resin substrate.

In addition, the lower retention values obtained for the resin substrate may be related to a reduction in the number of unreacted methacrylate groups at the resin surface over time, which reduces the potential for bonding to a new composite resin.[19] Another factor that reduces the bonding potential of resin composite cores is the grinding process that takes place during preparation of the core. The

application of a diamond bur to a resin composite surface removes reactive monomers, exposing inorganic filler particles that lose their silane layer and therefore will have reduced bonding ability.[20]

The second hypothesis was partially accepted because a significant effect of the surface treatment on the retention forces was only observed for the resin composite surfaces. For this substrate, the application of a thin layer of a low-fusing glassy porcelain resulted in significantly higher retention values between the cement and ceramic. It is probable that the thin layer on the inner surface (approximately 10 μm of thickness)[9] might have caused increased friction to the preparation walls because the cement space in the software of the CAD-CAM system was 30 μm for all groups, and consequently, an improvement in retention for the two substrates may have resulted. Favorable adhesive behavior of the internal glaze treatment has also been observed in previous studies using other types of adhesion tests.[3,6,7,9,10,21] As a negative issue, the glaze application approach can cause a slight modification in marginal adaptation.[9]

The weak bonding interaction observed between the cement and Y-TZP for the other types of surface treatments (control and silicatization) was also reflected by the great number of failures at the Y-TZP/cement interface (83.3%) (Table 3). This unacceptable bonding behavior of untreated zirconia surfaces was also noticed in previous studies.[4,5,9,10]

With regard to tribosilicatization, though this surface treatment resulted in a very good retention force for the dentin substrate (24.72 N), previous studies have shown this method might create a critical damage zone involving grooves and defects that can act as crack initiators.[22,23] On the other hand, other investigations did not find a negative effect of this method on the Y-TZP material,[24,25] and the clinical failures of Y-TZP crowns appear to have no association with crown inner surface conditioning as well.[26,27] Thus, tribosilicatization appears to be a good option for the treatment of the inner surface of the Y-TZP crown.

One of the limitations of this current study was the occurrence of failures classified as "catastrophic" (post debonding and root fracture), which generates unreal force values. Palacios and others[15] stated that catastrophic failures occur before reaching the maximum load supported by the adhesive interface. In this current study, the incidence of this type of failure type varied from 0% to 41.7%, depending on the experimental group (Table 3). As for the statistical analysis, it is important to point out that the data concerning catastrophic failures were indeed taken into account, because if this type of failure had not occurred, the retention values would probably have been higher. According to Palacios and others,[15] catastrophic failures occur before the maximum load supported by the adhesive interface is reached. Furthermore, had these data been removed from the statistical analysis, the retention values would have been underestimated. Another important limitation of this present study was the small convergence (taper) of the preparation, which made acquiring the image difficult for the CAD-CAM processing. This problem can be overcome in future studies using greater taper of the preparations. This modification will most likely facilitate capturing the preparation image, although the resultant increase in convergence may affect the retention of zirconia crowns.[28]

The clinical relevance of this study is that it simulated different cementation protocols of Y-TZP crowns in an *in vitro* design: freshly extracted human teeth were used and core build-ups with composite resin cores and fiber posts were fabricated to simulate prosthetic cases. To the knowledge of the current authors, no previous investigation has evaluated the factors in the current study and their effect on the retention force of zirconia crowns. Further studies should be conducted to investigate other factors involved in the retention of Y-TZP crowns, such as longitudinal fatigue testing, evaluation of different cementation strategies, and other surface treatments on the inner surface of Y-TZP frameworks.

CONCLUSION

a) The cementation of Y-TZP frameworks on dentin substrates resulted in significantly higher retention values when compared to those cemented on composite resin substrates, except when glazing was applied on the inner surface of the crown.
b) For the dentin substrate, the type of treatment performed on the Y-TZP cementation surface did not affect the retention force of the crown. On the other hand, both silicatization/silanization and glaze application resulted in higher retention values for the composite resin substrate when compared to the control (no surface treatment).

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.

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The Effect of Simplified Adhesives on the Bond Strength to Dentin of Dual-cure Resin Cements

AM Shade • MN Wajdowicz • CW Bailey
KS Vandewalle

Clinical Relevance

Unique initiator systems in resin cements may not sufficiently overcome incompatibility with simplified adhesives.

SUMMARY

The purpose of this study was to compare the shear bond strengths to dentin of two dual-cure resin cements, one with a unique initiator, NX3 (Kerr Corp), and the other with a traditional redox-initiator system, Calibra (Dentsply), when used in combination with simplified or nonsimplified adhesive agents. The two dual-cure resin cements, in either self- or dual-cure activation modes, were bonded to human dentin with four dental adhesives to create 16 subgroups of 10 specimens each. After 24 hours of storage in distilled water at 37°C, the specimens were tested in shear in a universal testing machine. With both NX3 and Calibra, bond strengths significantly increased when the specimens were dual cured. In addition, with either cement in either mode, the nonsimplified adhesives performed significantly better than did the simplified adhesive bonding agents. When used specifically with simplified adhesives in either cure mode, NX3 did not produce significantly higher bond strengths than did Calibra. In general, lower dentin bond strengths were found with simplified adhesives or self-cure activation with either resin cement.

Anita M Shade, BDS, DDS, MS, Scott AFB, IL, USA

Michael N. Wajdowicz, DDS, Keesler AFB, MS, USA

Clifton W. Bailey, DDS, Joint Base San Antonio - Lackland, TX, USA

*Kraig S Vandewalle, DDS, MS, General Dentistry, Joint Base San Antonio - Lackland, TX, USA, and Uniformed Services University of the Health Sciences, Bethesda, MD, USA

*Corresponding author: 1615 Truemper St, Joint Base San Antonio - Lackland, TX 78236, USA; e-mail: kraig.vandewalle@us.af.mil

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INTRODUCTION

Ever since Buonocore[1] described acid etching as a means to increase resin-enamel bond strengths over 50 years ago, we have been in constant pursuit of the ideal dental adhesive. The initial etch-and-rinse adhesives required three steps that included an acidic conditioner, primer, and adhesive monomer. Examples include Optibond FL (Kerr Corp, Orange, CA, USA) and Adper Scotchbond MultiPurpose (3M ESPE, St Paul, MN, USA).

Over the years, the trend has been to develop systems that are "simplified" or, in other words, that involve fewer steps with less procedure time.[2] A simplified adhesive is one in which the adhesive step

is incorporated into the primer. Manufacturers began to combine the primer and resin monomer components to create a two-step or simplified etch-and-rinse agent. Examples include Optibond Solo Plus (Kerr Corp), One-Step (Bisco, Schaumburg, IL, USA), and Prime and Bond NT (Dentsply, Milford, DE, USA).

Self-etch adhesives have been an even more recent introduction in which the use of an acidified primer has eliminated the use of the conditioner. An example of a popular nonsimplified self-etch adhesive is Clearfil SE (Kuraray, Japan). Today, simplified versions of the self-etch adhesives on the market are one-step systems in which the acidified primer and adhesive monomer are mixed together and placed in a single step. Examples include Optibond All-in-One (Kerr Corp), All-Bond SE (Bisco), Xeno 4 (Dentsply), and Adper Prompt L Pop (3M ESPE).

Using restorative systems with simplified adhesives does not necessarily result in reduced bond strength to dentin.[3,4] However, clinicians began to report bonding failures when self-cured "build-up" composites were bonded with simplified adhesive systems.[5] They were alerted to potential incompatibilities between self-cured resins and certain adhesive systems.[6] Simplified systems used with light-cure resins were found to produce bond strengths that were considerably higher than those produced with self-cure resins.[7,8] The process of simplification involves incorporation of acidified resin monomers into the primer-adhesive combination, resulting in a more hydrophilic mix. The concentration of acidic resin monomers is even higher in simplified self-etch adhesives, which serve to etch through the smear layer and enable bonding to the underlying dentin.[7-9] The hydrophilic property improves the wetting of the demineralized collagen matrix.[10] However, this layer acts like a semipermeable membrane, enabling the transudation of water from the underlying dentin across an osmotic gradient toward the oxygen-inhibited adhesive agent–resin cement interface.[9] These are described as water trees and interfacial blisters under transmission electron microscopy and contribute to diminished bond strengths of self-cure composites when compared to their nonsimplified counterparts.[7,8]

More significantly, simplified adhesives can lead to the deactivation of the amine initiators in self- and dual-cure composites. Conventional self-cure composites utilize a binary redox initiator system that consists of benzoyl peroxide with aromatic tertiary amines.[11] The amines react with the acidified monomers present in the superficial oxygen-inhibited layer and are unavailable to initiate the self-cure. This ultimately results in incomplete polymerization and compromised bond strengths along the composite–bonding agent interface.[12-14]

As mentioned earlier, the oxygen-inhibited layer in simplified adhesives acts as a hypertonic medium that triggers osmotic fluid transport through the permeable adhesive layer. It is also a source of acidic resin monomers that deactivate tertiary amines.[15] The combination results in an incompatibility between self- and dual-cure composite resin materials when used with simplified adhesives, as evidenced by lower bond strengths and the presence of water blisters at the interface.[15] The adverse chemical interaction between catalytic components of self-cured composites and simplified adhesives is the major cause of bond strength reduction, whereas permeability of the adhesives to water causes only a minor reduction in bond strength.[7,8] When the permeability component was completely removed, as with the use of neat water-free resins, even low concentrations of acidic monomers were shown to deactivate tertiary amines in self-cured resins.[15]

Overall, the consequences are more acute in simplified self-etch adhesives than in simplified etch-and-rinse adhesives.[7,8] Furthermore, within the simplified etch-and-rinse adhesives, incompatibility was accentuated in some adhesives more than in others.[16] The decrease in tensile bond strengths of self-cure resins to dentin was shown to be inversely proportional to the acidity of the etch-and-rinse system.[17] In both of the above studies, the most acidic simplified etch-and-rinse agent, Prime and Bond NT by Dentsply (pH, 2.68), had the weakest bond strengths when compared to the least acidic, One-Step by Bisco (pH, 4.60).[16,17]

It should be noted that when a dual-cure cement is sufficiently light-cured, the incompatibility does not occur.[7,8] The bond strength to dentin is directly related to the amount of light energy to which it is exposed.[18] Manufacturers of dual- or self-cure cement systems accept this incompatibility and indicate their use exclusively with nonsimplified etch-and-rinse or self-etch adhesives.[19]

If a dual-cure cement is to be used with a simplified adhesive, adequate light-curing of the cement is emphasized. Some manufacturers recommend use of an additional dual-cure activator. However, it has been shown that the use of activators does not completely eliminate this incompatibility.[7,8] A recently released dual-cure resin cement (NX3, Kerr Corp) employs a unique redox

initiator system that the manufacturer claims is acid resistant and that can initiate polymerization in the dark and in the presence of an acidic environment.[20,21] Consequently, the manufacturer proposes that this agent can be used with any adhesive system on the market without compromising bond strength.[22,23] To date, there are no published articles in the literature to verify this claim, and the few recent unpublished abstracts funded by other manufacturing companies that indirectly looked at this agent in their study models have not shown improved dentin bond strengths when employed with simplified agents in self-cure modes.[24-28]

The purpose of this study was to evaluate whether or not the proprietary initiators outlined above are able to circumvent the incompatibility issue with simplified dental adhesives in self-cure mode. If they are, do they exhibit dentin bond strengths similar to those achieved in the dual-cure mode?

It was the aim of this study to substantiate the manufacturer's claim so that clinicians can take advantage of the product's properties with the comfort of knowing it is supported by evidence-based dentistry. This study evaluated the shear bond strengths to dentin of two dual-cure resin cements, NX3 (new initiator system) and Calibra (standard system used as a control), either in self- or dual-cure modes when used in combination with simplified or nonsimplified adhesive agents. The study tested two specific null hypotheses, as follows:

1) There is no difference in the shear bond strength of NX3 or Calibra to dentin based on the choice of adhesive bonding agents, either simplified (Prompt L-Pop, Prime and Bond NT) or nonsimplified (Optibond FL, Clearfil SE); and
2) There is no difference in the shear bond strength to dentin of dual-cure cements, NX3 or Calibra, based on the curing mode, either dual- or self-cure.

METHODS AND MATERIALS

The protocol was approved by the Institutional Review Board at Wilford Hall Ambulatory Surgical Center, Joint Base San Antonio - Lackland, Texas. The resin cements chosen for this study were NX3 (Kerr Corp) and Calibra (Dentsply).

Four dental adhesives were utilized, two nonsimplified (Optibond FL [Kerr Corp] and Clearfil SE [Kuraray]) and two simplified (Prime and Bond NT [Dentsply] and Prompt L Pop [3M ESPE]).

Table 1: *Study Groupings*

Group No.	Adhesive	Subgroup No.	Resin Cement and Curing Mode
1	Prime and Bond NT	1	NX3 + self-cure mode
		2	NX3 + dual-cure mode
		3	Calibra + self-cure mode
		4	Calibra + dual-cure mode
2	Adper Prompt L-Pop	5	NX3 + self-cure mode
		6	NX3 + dual-cure mode
		7	Calibra + self-cure mode
		8	Calibra + dual-cure mode
3	Optibond FL	9	NX3 + self-cure mode
		10	NX3 + dual-cure mode
		11	Calibra + self-cure mode
		12	Calibra + dual-cure mode
4	Clearfil SE	13	NX3 + self-cure mode
		14	NX3 + dual-cure mode
		15	Calibra + self-cure mode
		16	Calibra + dual-cure mode

The following adhesive agent/resin cement combinations were used:

- Prime and Bond NT-NX3/Calibra;
- Prompt L-Pop-NX3/Calibra;
- Optibond FL- NX3/Calibra; and
- Clearfil SE-NX3/Calibra.

The following activation modes, in which each of the above groups were further subdivided based on cure mode, were used:

- Dual-cure and
- Self-cure.

A total of 16 subgroups were created (see Table 1). Ten samples were prepared per subgroup, resulting in 160 total samples. All 160 samples were prepared by a single provider to minimize interoperator differences and to ensure uniformity of fabrication.

One hundred sixty extracted human third molars stored in 0.5% chloramine-T at 4°C were used within six months following extraction. The teeth were mounted in dental stone in polyvinyl chloride pipes with the crown exposed and accessible. A diamond saw (Isomet, Buehler, Lake Forest, IL, USA) was used to remove 2 mm or more of coronal tooth structure to ensure dentin exposure and the proper orientation of the surface relative to the direction of the applied shear force. Each sample was then examined under a stereomicroscope (SMZ-1B, Nikon, Melville, NY, USA) at 10× magnification to ensure complete exposure of the dentin surface with

Table 2: *Application Methods*

Adhesive (Manufacturer)	Type	Manufacturer's Application Instructions
Prime and Bond NT (Dentsply)	Two-step, etch-and-rinse (simplified)	Etchant: apply and leave undisturbed (15 s)
		Water rinse
		Gently air-dry (5 s)
		Bond: apply and leave undisturbed (20 s); gently air-dry
		Light-cure (10 s)
Adper Prompt L-Pop (3M ESPE)	One-step, self-etch (simplified)	Mix the liquids in the red and yellow blister, brush the mixture onto tooth surface (15 s)
		Gently air-dry (5 s)
		Light-cure (10 s)
Optibond FL (Kerr Corp)	Three-step etch-and-rinse (nonsimplified)	Etchant: apply and leave undisturbed (15 s)
		Water rinse
		Gently air-dry (5 s)
		Primer: apply with light scrubbing motion (15 s)
		Gently air-dry (5 s)
		Bond: apply to a thin layer
		Light-cure (30 s)
Clearfil SE (Kuraray)	Two-step self-etch (nonsimplified)	Primer: apply and leave undisturbed (20 s)
		Gently air-dry (5 s)
		Bond: apply to a thin layer
		Light-cure (10 s)
NX3 (Kerr Corp)	Resin cement: dual-cure mode	Dual-cure paste
		2-mm increments
		Light-cure (20 s)
	Resin cement: self-cure mode	Dual-cure paste
		Bulk fill
Calibra (Dentsply)	Resin cement: dual-cure mode	Mix equal lengths of base and catalyst for 20 s
		2-mm increments
		Light-cure (20 s)
	Resin cement: self-cure mode	Mix equal lengths of base and catalyst for 20 s
		Bulk fill

no residual enamel. A uniform smear layer was created on the flat dentin surfaces using 10 passes on 600-grit carbide paper.

The 160 prepared teeth were randomly distributed to create four groups (40 specimens in each group) based on the four adhesive agents used. The adhesive agents were applied to the dentin surface according to the manufacturer's instructions (see Table 2).[19,29-33] The adhesive was cured as recommended by the manufacturer using the Bluephase 16i (Ivoclar, Amherst, NY, USA) light-curing unit. The irradiance of the curing light was monitored periodically with a radiometer (Bluephase Radiometer, Ivoclar) to verify that irradiance levels remained above 1200 mW/cm^2. Each of the four groups was further divided into four equal subgroups of 10 samples each. Each subgroup tested one of the two resin cements being evaluated in either self- or dual-cure activation mode. The prepared samples were placed in an Ultradent Jig and secured beneath the white, nonstick Delrin insert (Ultradent, South Jordan, UT, USA). The resin cement was then mixed and applied into the mold according to the manufacturer's instructions (see Table 2)[19,21] to a height of 3 mm. The bonding area was limited to the 2.4-mm-diameter circle determined by the Delrin insert. The self-cure subgroups were allowed to self-cure undisturbed for a period of 15 minutes in a light-proof container. The dual-cure subgroups were bulk–light-cured for 20 seconds to simulate the light penetration achieved in a clinical setting. Following the application of the resin cement with the designated curing method, all samples were stored for 24 hours in distilled water at 37°C. After 24 hours, the shear bond strengths of all samples were tested using a universal testing machine (Model 5943, Instron,

Table 3: *Shear Bond Strength Data and Statistical Analysis Based on Adhesive Type*[a]

Cement	Shear Bond Strength (±SD), MPa											
	Adhesive											
	Nonsimplified						Simplified					
	Clearfil SE			Optibond FL			Prime and Bond NT			Prompt L-Pop		
	DC	SC	Two-way ANOVA	DC	SC	Two-way ANOVA	DC	SC	Two-way ANOVA	DC	SC	Two-way ANOVA
NX3	13.2 (4.8)	9.4 (3.2)	A	6.5 (1.6)	6.4 (1.7)	A	2.3 (1.8)	2.8 (2.0)	A	2.5 (2.4)	0.3 (0.3)	A
Calibra	9.5 (3.7)	6.6 (4.2)	A	4.0 (1.7)	2.7 (2.4)	B	3.8 (2.8)	1.1 (1.2)	A	1.1 (1.0)	0.08 (0.04)	A

Abbreviations: ANOVA, analysis of variance; DC, dual cure; SC, self cure; SD, standard deviation.
[a] *Groups with the same capital letter per column within a bond group are not significantly different (p>0.008).*

Norwood, MA, USA) at a crosshead speed of 1 mm/min using the notched blade at a 90° angle. Shear bond strength values in megapascals (MPa) were calculated from the peak load of failure (Newtons) divided by the sample surface area. The mean and standard deviation were determined for each group. The resultant data for the various groups were then analyzed to verify the two null hypotheses.

Following testing, each specimen was examined using a 10× stereomicroscope to determine failure mode as 1) adhesive fracture at the cement/adhesive/dentin interface, 2) cohesive fracture in cement or dentin, or 3) mixed failure (combined adhesive and cohesive).

A mean and standard deviation were determined for each group. The study involved three independent variables—adhesive (four levels), cement (two levels), and cure method (two levels). Consequently, a three-way analysis of variance (ANOVA) was performed to identify differences at the three levels of variability. Alpha was set at 0.05. Though significant differences were detected at all three levels with the three-way ANOVA, global conclusions could not be made from the results as a result of the interactions between the individual parameters. Two-way ANOVAs were then performed, keeping one of the three variables constant each time. Tukey post hoc tests were used to determine differences between groups. The alpha value was adjusted to 0.008 with a Bonferroni correction because multiple comparisons were completed simultaneously.

RESULTS

The statistical analysis was reviewed and approved by the clinical research administrator, Clinical Research Division, JBSA-Lackland (Texas).

Four two-way ANOVA tests were performed for the four levels tested within the adhesives group (Table 3). Within all the samples bonded with simplified adhesives either Prime and Bond NT or Prompt L Pop, regardless of cure mode, no significant difference was noted between NX3 and Calibra. Within all the samples bonded with the nonsimplified Optibond FL, regardless of cure mode, bond strengths with NX3 were significantly higher than those obtained with Calibra.

Two ANOVA (two-way) tests were performed for the two levels tested within the cements group (Table 4). Dual-cure polymerization resulted in an overall higher bond strength than self-cure polymerization using NX3. Within all the groups cemented with NX3, regardless of cure mode, samples bonded with nonsimplified adhesives had significantly high-

Table 4: *Shear Bond Strength Data and Statistical Analysis Based on Cement Type*[a]

Cure Mode	Shear Bond Strength (SD), MPa									
	Cement									
	NX3					Calibra				
	CSE a	OFL b	PB c	PLP c	Two-way ANOVA	CSE a	OFL b	PB bc	PLP c	Two-way ANOVA
Dual cure	13.20 (4.78)	6.49 (1.61)	2.30 (1.76)	2.45 (2.34)	A	9.51 (3.74)	3.97 (1.68)	3.80 (2.85)	1.07 (1.05)	A
Self cure	9.43 (3.15)	6.36 (1.69)	2.81 (2.04)	0.32 (0.34)	B	6.58 (4.22)	2.71 (2.37)	1.13 (1.19)	0.08 (0.04)	B

Abbreviations: ANOVA, analysis of variance; CSE, Clearfil SE; OFL, Optibond FL; PB, Prime&Bond NT; PLP, Prompt L-Pop; SD, standard deviation.
[a] *Groups with the same capital letter per column or lowercase letter per row within a cement group are not significantly different (p>0.008).*

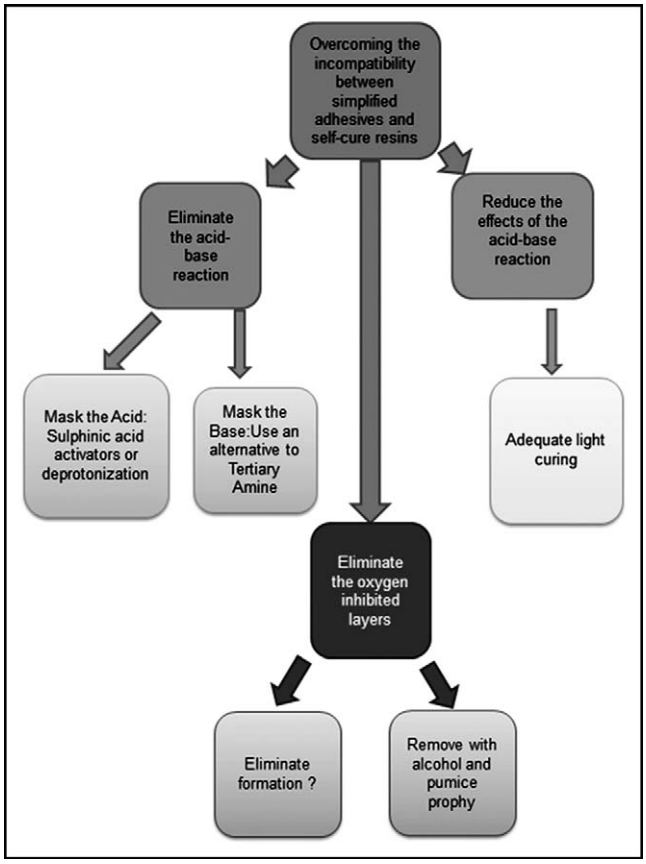

Figure 1. *Treatment strategies flow chart.*

er bond strengths than did those bonded with the simplified adhesives. Among the adhesives, the Clearfil SE samples exhibited the highest bond strengths. Within all the groups cemented with Calibra, regardless of adhesive used, the groups that were dual-cured showed higher bond strengths than did those that were self-cured. Within all the groups cemented with Calibra, regardless of cure mode, samples cemented with the nonsimplified adhesives showed higher bond strengths than did those cemented with the simplified adhesives. It must be noted as an exception that the values shown by Optibond FL were not significantly higher than those bonded with Prime and Bond NT. Samples bonded with Clearfil SE exhibited the highest bond strengths.

Following debonding, all specimens were viewed under a 10× stereomicroscope to determine failure mode. The majority of the failures were adhesive. No cohesive failures were noted. Mixed failures were noted most in Clearfil SE Bond subgroups. More mixed failures were associated with dual-curing.

DISCUSSION

This study showed that dentin bond strengths obtained with NX3 and simplified dental adhesives, regardless of cure mode, are not significantly higher than those obtained with Calibra. This suggests that the proprietary redox initiator in NX3 is not able to sufficiently overcome the incompatibility issues encountered when a simplified dental adhesive is combined with a self-activated dual-cure resin cement. Therefore, like every other dual-cure resin cement, NX3 should be used with a nonsimplified adhesive for optimal results.

The goal of patient-centered care is to provide excellent treatment in an efficient manner at minimum cost. With the development of stronger, yet esthetic, resin materials and adhesives, we are getting closer to achieving this goal. It is no surprise that resin restorative materials and simplified adhesives are major players in the world of restorative dentistry. That being said, it is noteworthy that self- or dual-cure resin cements still maintain their place in any dentist's inventory. They are the norm in limited-light situations such as those involving composite-core buildups and cementation of posts and indirectly fabricated resin or ceramic crowns, inlays, and onlays. However, incompatibilities between simplified dental adhesives and self-cure resins were recognized as early as 1999.[5] Efforts at eliminating this problem can be targeted at three different levels (Figure 1), as follows: 1) Eliminate the acid-base reaction; 2) Reduce the effects of the acid-base reaction; or 3) Eliminate the oxygen-inhibited layer.

The ideal solution would be one that permits continued use of the time-saving simplified dental adhesive with any resin cement without compromising efficiency or bond strength. The remainder of the discussion will explore these levels in detail and correlate them to the results of this study.

Strategy 1—Eliminate the Acid-base Reaction

Elimination of the acid-base reaction can be achieved in two ways: eliminating the acidic component or eliminating the base.

Eliminating the Acidic Component—One could avoid the acid component altogether by exclusive use of nonacidic nonsimplified adhesives. This was reconfirmed by the results of our study, in which we found that with either cement in either cure mode, the nonsimplified adhesives performed the best (Figures 2 and 3).

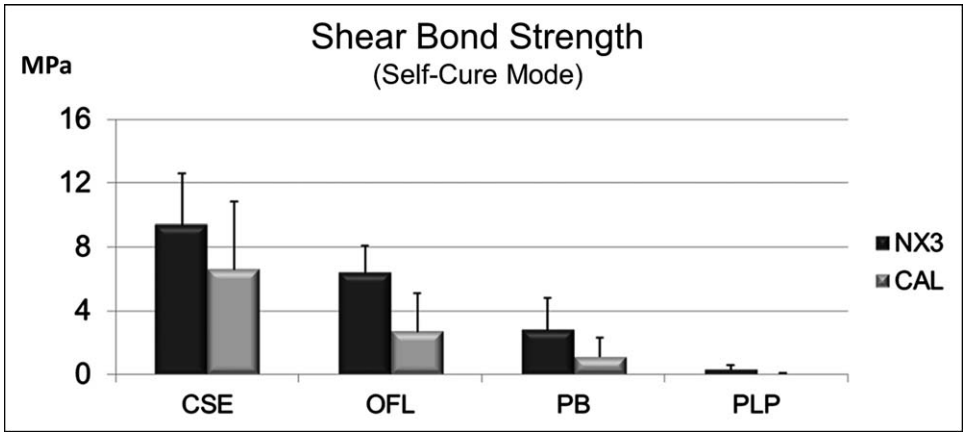

Figure 2. *Shear bond strengths of resin cements (MPa) to dentin in self-cure mode. Error bars represent 1 standard deviation.*

The alternative would be to mask the acid. This has been suggested by use of activators with the simplified adhesives. One commonly used product is the sodium salt of aryl sulfinic acid. This reacts with acidic resin monomers to produce phenyl or benzenesulfonyl free radicals that would then serve to initiate the polymerization of self-cured composites. However, the dentin bond strengths obtained with these activators continue to be suboptimal.[7,8] This is probably because the hydrophilicity of the acidic monomers is not overcome, and osmotic blistering continues to be an issue.[7,8] There is one study[34] that examines the possibility of deprotonization of the acidic adhesive with an anion exchange compound with good results.

Eliminating the Basic Component—Tertiary amines are present in both light- and self-cure resin formulations. In light-cure resins, the light activates the camphorquinone initiator, which is then transformed to its exciplex state by a tertiary amine accelerator. Self-cure systems employ a binary redox catalytic system composed of peroxide and a tertiary amine. However, there is a difference. The tertiary amine in the light-cure formulations is present in far smaller concentrations and is much less nucleophilic than that in self-cure formulations. This, combined with the fact that the light-cure reaction takes place too quickly to allow an acid-base reaction, accounts for the fact that the incompatibilities are restricted to self-cure groups.

The results of our study show that although overall dentin bond strengths with NX3 appear superior to those obtained with Calibra, when we looked at these bond strengths specifically within the realm of simplified adhesives, the bond strengths with NX3 are not statistically higher than those obtained with Calibra. Details on the proprietary redox system are not available in the NX3 Material Safety Data Sheets. Whatever the composition, it is apparent that it does not completely overcome the

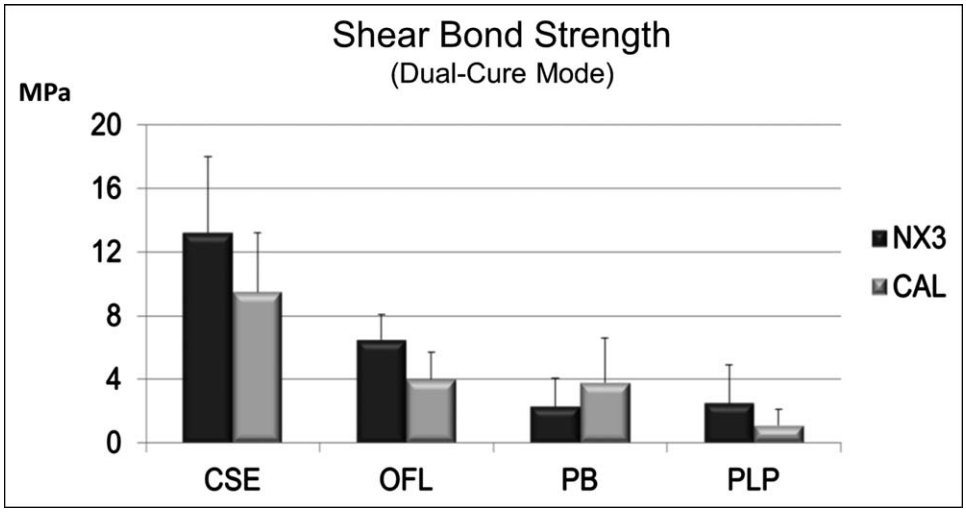

Figure 3. *Shear bond strengths of resin cements (MPa) to dentin in dual-cure mode. Error bars represent 1 standard deviation.*

incompatibilities. NX3 appears no different from other dual-cure resin cements on the market. It is most likely that the numeric bond strength values of NX3 are higher than those of Calibra because the absence of tertiary amines allows for more complete polymerization of the resin. However, the presence of residual uncured hydrophilic acidic monomers at the oxygen-inhibited interface could continue to contribute to interfacial stresses and less-than-optimal bond strengths. Despite manufacturer's claims, we failed to reject the first null hypothesis.

Strategy 2—Reduce the Effects of the Acid-base Reaction: Light-Cure Adequately

When using a dual-cure composite, if clinical conditions allow, adequate light-curing of the cement is sufficient to overcome the acid-base interactions.[7,8] The bond strength to dentin is directly related to the amount of light energy to which the dentin is exposed.[18] Photo-polymerization results in rapid setting of the resin matrix, allowing no time for adverse acid-base reactions to occur. The light energy should also be able to successfully and rapidly cure the acidic monomers in the oxygen-inhibited layer with remnants of the photo-initiator.[35] Results of the two-way ANOVA confirmed that with either resin cement NX3 or Calibra, the dentin bond strengths were significantly higher when the cement is dual-cured. The second null hypothesis was therefore rejected.

Strategy 3—Eliminate the Oxygen-inhibited Layer

Light- and chemically cured dental composite resins leave a soft, sticky superficial layer upon polymerization. When oxygen diffuses through the superficial layer of resin, it forms peroxide radicals with the monomer. The peroxide radicals are poorly reactive and do not participate in the polymerization reaction, creating the sticky oxygen-inhibited layer. This layer has the same composition as the uncured resin except that it has less of the photo-initiator.[36] Additionally, this layer in simplified adhesives serves both as a source of acidic resin monomers that deactivate the tertiary amines and as a hypertonic medium that triggers osmotic fluid transport through the adhesive layer.[15]

How can we eliminate this layer? The answer is twofold. The oxygen-inhibited layer can be prevented or it can be removed after formation. Preventing formation of the oxygen-inhibited layer can be achieved by polymerization in an inert nitrogen environment (not clinically feasible) or by coating the adhesive with glycerol prior to activation. It has been suggested[37] that one can remove the oxygen-inhibited layer by wiping/rubbing with isopropyl alcohol or prophylaxis with a prophy cup with a mixture of flour of pumice and rubbing alcohol. Since dentin adhesive agents are applied in thin layers, unlike restorative composites, the use of the latter technique could remove too much of the bonding agent, creating direct contact of resin with the hybridized dentin.[15] It is noteworthy that contrary to common perception, presence of an oxygen-inhibited layer is not required for higher bond strengths to additional increments of composite.[36,38,39]

In spite of the sound theory behind elimination of the oxygen-inhibited layer, a study[34] evaluating its efficacy in improving bond incompatibilities of self-etch adhesives on self-cure resins showed persistence of low bond strengths regardless of its presence. This study was very limited in sample size (one sample per group) and perhaps warrants additional research.

CONCLUSIONS

In conclusion, within the limitations of this study, the following recommendations can be made. When used specifically with simplified dental adhesives in either cure mode, NX3 does not produce significantly higher bond strengths than does Calibra. In general, lower bond strengths continue to be observed when simplified adhesives are used with the resin cements in self-cure mode. Clinicians should be cautioned to continue to restrict this usage with limited light-cure situations. Dentists should periodically evaluate their curing lights so they consistently provide adequate output to polymerize these dual-cure cements and combat compromised bond strengths. Dentin bond strengths can be maximized when the resin cement is dual-cured. Clearfil SE Bond shows superior bond strengths with the resin cements in any cure mode.

Disclosure

The views expressed in this study are those of the authors and do not reflect the official policy of the US Air Force, the Department of Defense, or the US Government.

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.

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Preheating Impact on the Degree of Conversion and Water Sorption/ Solubility of Selected Single-bottle Adhesive Systems

MRL Vale • FAC Afonso • BCD Borges
AC Freitas Jr • A Farias-Neto • EO Almeida
EJ Souza-Junior • S Geraldeli

Clinical Relevance

The use of preheated ethanol/water-based adhesive systems seems to facilitate solvent evaporation, which translates to improving the degree of conversion and decreasing water sorption/solubility.

Mariana R. L. Vale, undergraduate, Potiguar University (Laureate International Universities), Department of Dentistry, Natal, Brazil

Felipe A. C. Afonso, undergraduate, Potiguar University (Laureate International Universities), Department of Dentistry, Natal, Brazil

*Boniek C. D. Borges, PhD, Federal University of Rio Grande do Norte, Department of Dentistry, Natal, Brazil

Amilcar Chagas Freitas Jr, PhD, Potiguar University (Laureate International Universities), Department of Dentistry, Natal, Brazil

Arcelino Farias-Neto, PhD, Potiguar University (Laureate International Universities), Department of Dentistry, Natal, Brazil

Erika Oliveira Almeida, PhD, Federal University of Rio Grande do Norte, Department of Dentistry, Natal, Brazil

Eduardo José Souza-Junior, DDS, MS, Piracicaba Dental School, State University of Campinas – UNICAMP, Department of Restorative Dentistry, Piracicaba, Brazil

Saulo Geraldeli, PhD, University of Florida, Department of Operative Dentistry, Gainesville, FL, USA

*Corresponding author: Av. Senador Salgado Filho 1787, Natal, 59.056-000, Brazil; e-mail: boniek.castillo@gmail.com

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SUMMARY

Objective: This study evaluated the degree of conversion (DC) and the water sorption/solubility of preheated single-bottle adhesive systems.

Methods and Materials: Five adhesive systems were tested: Adper Easy One and Adper Single Bond 2 (3M ESPE), Excite and Tetric N-Bond (Ivoclar/Vivadent), and XP Bond (Dentsply/Caulk). After storage for two hours at 25°C or 60°C, 50 samples (n=5) were prepared for all adhesive systems and stored dry in lightproof containers at 37°C for 24 hours. Fourier transform infrared/attenuated total reflectance spectroscopy was used to evaluate the DC, and water sorption/solubility was measured by means of mass loss and gain after water storage. The data were analyzed by two-way analysis of variance followed by Tukey's test ($p<0.05$).

Results: Preheated adhesive systems showed statistically significantly higher DC than those kept at 25°C. Except for XP Bond, preheated adhesive systems presented statistically sig-

nificantly lower water sorption/solubility means.

Conclusions: Preheating improved the DC for all tested adhesive systems. Also, it promoted a decrease of water sorption/solubility, except for the XP Bond adhesive system.

INTRODUCTION

The popular expectation for esthetics in dentistry continues to promote the use of direct composite resins for anterior and posterior teeth, which requires the application of an adhesive system to bond to dental structures. Contemporary adhesive systems are referred to as "etch-and-rinse" or "self-etch" (etch-and-dry). These two categories are either multistep (three-step etch-and-rinse and two-step self-etch) or simplified by combining the number of steps required for the clinical application (two-step etch-and-rinse and one-step self-etch).[1] Most clinicians prefer such a combined-process simplification because the reduced number of steps in the adhesive process reduces clinical chair time for the patient.[2,3]

Successful adhesion to enamel and dentin tissue is a fundamental necessity for placement of dental materials, and that requirement is directly dependent on the quality of the dentin hybrid layer. A high degree of conversion (DC) and low water sorption/solubility of the adhesive system is fundamental to improving resistance of material degradation under *in vivo* clinical conditions. In fact, low DC of dental adhesives is associated with high water sorption/solubility as well as low bond strength values, low mechanical properties, increased permeability, and even the occurrence of phase separation.[4-6] Moreover, reduced DC can also account for continuous etching of the tooth substrate due to suboptimally polymerized acidic monomer in self-etch adhesives.[7]

A recently proposed method to increase the DC of resin-based dental materials is preheating before photoactivation.[8] Heating the material to high temperatures decreases viscosity and increases radical mobility, which favors higher DC.[8] It has been shown that preheating some single-bottle adhesive systems can improve the DC.[9]

To date, several commercially available adhesive systems have not been evaluated with regard to the effect on the DC and water sorption/solubility when the adhesive is preheated before application. Therefore, this study aimed to analyze and to compare the DC and the water sorption/solubility of preheated and non-preheated single-bottle adhesive systems. The null hypotheses were that 1) preheating would not affect the DC of the adhesive systems, and 2) preheating would not affect the water sorption/solubility of adhesive systems.

METHODS AND MATERIALS

The single-bottle contemporary adhesive systems used in this study were Adper Easy One (3M ESPE, St Paul, MN, USA), Adper Single Bond 2 (3M ESPE), Excite (Ivoclar-Vivadent, Schaan, Liechtenstein), Tetric N-Bond (Ivoclar Vivadent), and XP Bond (Dentsply/Caulk, Milford, DE, USA). The DC and sorption/solubility were evaluated for each adhesive system at different temperatures (25°C = room temperature; 60°C = preheated) (n=5). The specific compositions, manufacturers, and batch numbers of the tested materials are shown in Table 1.

Preheating of the Adhesive Systems

Each bottle of the adhesive system was kept in an oven (incubator) at 25°C or 60°C for two hours before starting the adhesive procedure.[9] Before specimen preparation, the temperature was checked with a thermometer for each adhesive system.

Specimen Preparation

For DC analysis, 50 bar-shaped samples (6 mm long $\times$ 1 mm deep $\times$ 1 mm wide) were prepared using each adhesive system and polyvinyl siloxane molds. For water sorption/solubility analyses, 50 disks (6-mm diameter by 1-mm thickness) were prepared using cylindrical polyvinyl siloxane molds. One drop of each adhesive was placed into the mold followed by solvent evaporation for 10 seconds using an uncontaminated air stream. Then, a Mylar strip was placed over the mold, and the adhesive was photoactivated for 20 seconds with a light-emitting diode (LED) unit (Coltolux, Coltène/Whaledent, Allstätten, Switzerland). The 8-mm-diameter tip of the LED covered the entire sample during photoactivation. An irradiance of 1264 mW/cm^2 was distributed over the top surface of each sample. After photoactivation, the sample was removed from the mold and stored dry in lightproof containers at 37°C for 24 hours.

DC Analysis

The DC was analyzed using Fourier transform infrared/attenuated total reflectance spectroscopy (Spectrum 100, PerkinElmer, Shelton, CT, USA) at 24°C under 64% relative humidity.[3] The absorption spectra of nonpolymerized and polymerized adhesives were obtained from the wavenumber region

Table 1: *Composition, Manufacturer, and Lots of the Adhesive Systems Used in the Study*

Adhesive System	Composition (% by Weight)	Manufacturer	Lot No.
Adper Easy One	Bis-GMA (15-25), HEMA (15-25), ethanol (10-15), water (10-15), phosphoric acid-6-methacryloxy-hexylesters (5-15), silane treated silica (8-12), 1,6-hexanediol dimethacrylate (5-10), copolymer of acrylic and itaconic acid (1-5), (dimethylamino)ethyl methacrylate (1-5), camphorquinone (1-3), 2,4,6- trimethylbenzoyldiphenylphosphine oxide (1-3)	3M ESPE, St Paul, MN, USA	436286
Excite	Phosphonic acid acrylate (<11), HEMA (<15), dimethacrylates (<53), alcohol (<20)	Ivoclar-Vivadent, Schaan, Liechtenstein	N11163
Adper Single Bond 2	Ethyl alcohol (25-30), silane treated silica (nanofiller) (10-20), bis-GMA (10-20), HEMA (5-10), glycerol 1,3-dimethacrylate (5-10), copolymer of acrylic and itaconic acids (5-10), water (5), diurethane dimethacrylate (1-5)	3M ESPE, St Paul, MN, USA	N300770BR
Tetric N-Bond	Phosphonic acid acrylate (<11), HEMA (<15), dimethacrylates (<53), alcohol (<20)	Ivoclar-Vivadent, Schaan, Liechtenstein	N76256
XP Bond	HEMA (25-50), methacrylate (10-25), tert-butyl alcohol (10-25), acrylates (10-25)	Dentsply/Caulk, Milford, DE, USA	1011000650

between 4000 cm^{-1} and 650 cm^{-1}, with 32 scans at 4 cm^{-1}. The DC (%) was calculated using the following equation:

$$DC(\%) = 100 \times (1 - [\text{R polymerized}/\text{R nonpolymerized}]) \quad (1)$$

where R represents the ratio between the absorbance peaks at 1638 and 1608 cm^{-1}. The final DC values were analyzed using two-way analysis of variance (ANOVA) considering adhesive system × temperature and Tukey's test ($p<0.05$).

Water Sorption and Solubility Measurement

Water sorption and solubility were determined according to the ISO specification 4049, except for specimen dimensions. The disks were stored at 37°C in desiccators that contained silica gel desiccant and weighed daily (24-hour intervals) on an analytical balance (Chyo Balance JK 180, Chyo Corp., Tokyo, Japan). The weighing was conducted until a constant mass (m1) (48 hours with no weight variance) was obtained. Thickness (four measurements at four equidistant points on the circumference) and diameter (two measurements at the right angles) of each specimen were measured using a digital electronic caliper (Mitutoyo Corporation, Tokyo, Japan). These mean values of the multiple measurements per specimen per dimension were used to calculate the volume (V) for each individual specimen (mm^3). Thereafter, the samples were stored in 6 mL of distilled water at 37°C for 7 days. After the immersion period, samples were weighed (24-hour intervals) after being carefully wiped with an absorbent paper. When constant weight was obtained (48 hours with no weight variance), it was recorded as m2. After this weighing, the samples were returned to the desiccators, the entire mass reconditioning cycle was repeated, and the third constant mass process (48 hours with no weight variance) was recorded as m3. The values for water sorption (WS) and solubility (WSB), in micrograms per cubic millimeters, were calculated using the following equations:

$$WS = (m2 - m3)/V \quad (2)$$

$$WSB = (m1 - m3)/V \quad (3)$$

RESULTS

Degree of Conversion

Two-way ANOVA showed statistically significant differences between materials and temperatures ($p<0.05$). Multiple comparisons among the groups are listed in Table 2. All five adhesive systems exhibited higher mean DC values at 60°C than they did at 25°C. The Adper Easy One had the highest mean value, and XP Bond provided the lowest mean value, regardless of the adhesive temperature condition.

Water Sorption

Two-way ANOVA showed statistically significant differences in the interaction for materials × temperatures ($p<0.05$). Multiple comparisons among the groups are listed in Table 3. With the exception of XP Bond, the remaining four adhesive materials

Table 2: *DC Mean Percentages (Standard Deviations) for the Adhesive Systems at Different Temperatures*[a]

Materials	Temperature	
	25°C	60°C
Adper Easy One	83.8 (3.4) aB	89.3 (2.7) aA
Excite	72.4 (1.9) bB	79.9 (2.0) bA
Adper Single Bond 2	65.1 (2.0) cB	81.8 (2.2) bA
Tetric N-Bond	71.8 (3.6) bB	79.9 (4.0) bA
XP Bond	42.7 (3.3) dB	63.0 (2.1) cA

[a] *Different uppercase letters in rows and lowercase letters in columns indicate statistically significant differences* ($p<0.05$).

demonstrated lower water sorption at 60°C than at 25°C. Adper Easy One and Adper Single Bond 2 provided the highest mean values at 25°C. XP Bond produced the highest values at 60°C.

Solubility

Two-way ANOVA showed statistically significant differences in the interaction materials × temperatures ($p<0.05$). Multiple comparisons among the groups are listed in Table 4. Except for XP Bond, all materials showed lower solubility at 60°C than at 25°C. Adper Easy One showed the highest mean at 25°C. On the other hand, XP Bond showed the highest mean at 60°C.

DISCUSSION

The first null hypothesis tested in this study, that higher temperature would not affect the DC of adhesive systems, was rejected as increased temperature provided higher DC to all adhesive systems tested (See Table 2). As a matter of fact, the DC of resin-based materials is known to be incomplete at room temperature.[10] Higher temperatures are known to reduce viscosity and enhance radical mobility, hence resulting in additional polymerization and higher conversion.[8] The collision frequency of unreacted active groups and radicals increases at higher curing temperatures that are below the glass transition temperature.[10] Further, as the material temperature rises, additional free volume builds up. This, in turn, provides increased mobility to trapped radicals, resulting in further conversion.[10] Therefore, the results obtained in the present investigation appear to be relevant for improving the clinical performance of single-bottle adhesive systems, as optimal physical and mechanical properties of adhesive systems are dependent on a high DC.[5,6] It has been shown in a previous study[9] that preheated Single Bond did not enhance DC. It is possible to argue that the 50°C temperature may have been the reason as explained earlier. In addition, the 10-second air application used to evaporate the solvent can substantially decrease the temperature of the adhesive, leading to reduction in DC. For this reason, the temperature of 60°C was used in the present study. However, further analyses are needed to confirm these assumptions.

Table 3: *Water Sorption Means (Standard Deviations) (μg/mm) for the Adhesive Systems at Different Temperatures*[a]

Materials	Temperature	
	25°C	60°C
Adper Easy One	336.3 (36.0) Aa	238.1 (45.5) Bb
Excite	254.2 (25.6) Ab	198.6 (65.9) Bbc
Adper Single Bond 2	370.7 (55.9) Aa	146.2 (27.1) Bc
Tetric N-Bond	221.2 (26.6) Ab	144.7 (21.6) Bc
XP Bond	244.5 (25.5) Bb	460.5 (27.2) Aa

[a] *Different uppercase letters in rows and lowercase letters in columns indicate statistically significant differences* ($p<0.05$).

Table 4: *Water Solubility Means (Standard Deviations) ($\mu g/mm^3$) for the Adhesive Systems at Different Temperatures*[a]

Materials	Temperature	
	25°C	60°C
Adper Easy One	270.2 (9.6) Aa	159.2 (37.6) Bb
Excite	130.0 (11.8) Ac	74.6 (7.5) Bc
Adper Single Bond 2	133.5 (16.3) Ac	60.7 (4.1) Bc
Tetric N-Bond	82.7 (7.4) Ad	65.4 (3.8) Bc
XP Bond	184.3 (49.5) Bb	324.9 (13.7) Aa

[a] *Different uppercase letters in rows and lowercase letters in columns indicate statistically significant differences* ($p<0.05$).

Simplified dental bonding agents are composed of a mixture of hydrophilic primers and hydrophobic adhesive resins dissolved in acetone, ethanol, water, or some combination of these solvents.[3] Acetone has a lower boiling temperature (56.5°C) and a higher vapor pressure (200 mm Hg) than ethanol (78.3°C and 43.9 mm Hg) and water (100°C and 17.5 mm Hg).[11] This means that when adhesive systems are heated to high temperatures, such as 60°C, faster solvent evaporation might occur with acetone-based systems because this temperature is higher than the boiling temperature of acetone (56.5°C).[9] The faster solvent evaporation can impair ideal monomer infiltration to dentin leaving a thin adhesive layer that is more susceptible to polymerization inhibition by oxygen. As a result this can be expected to result in decreased bond strength values. For these

reasons, only alcohol-based adhesive systems were tested in this study.

Adper Easy One had the highest DC among the adhesive systems in this study, regardless of the temperature. This finding can be explained by its photoinitiator species, which differs from those in other adhesive systems. The formulation of current adhesive systems is usually based on hydrophobic initiators (eg, camphorquinone [CQ]) and co-initiators (eg, ethyl-4-[dimethylamino]benzoate).[12] Most likely they are distributed preferentially to the hydrophobic domains, jeopardizing the overall polymerization of the adhesive.[5] Thus, the inclusion of an alternative hydrophilic photoinitiator, such as trimethylbenzoyldiphenyl phosphine oxide (TPO), with higher reactivity to hydrophilic domains than CQ, has been shown to improve the overall DC of experimental solvent-based adhesive resins.[6] Because Adper Easy One contains CQ and TPO, it is likely that such a combination accounted for the observed higher DC. As an assumption and based on the 350-425 nm absorption range of TPO,[13] the use of a polywave LED with an additional peak around 400 nm could improve the DC for Adper Easy One, although the present study used monowave LED (350-425 nm band). Nevertheless, further analyses should be performed to investigate this possibility.

The concomitant study of physical properties of adhesive systems can contribute to meaningful interpretation of the results. In this study, the benefits of preheating single-bottle adhesive systems on DC, water sorption, and solubility were investigated. Although an increased temperature of single-bottle alcohol-based adhesive systems could enhance the DC for the five materials tested, it decreased the water sorption and solubility for XP Bond alone. This finding requires rejection of the second null hypothesis that preheating would not affect the water sorption and solubility of adhesive systems tested in this study. The presence of residual solvent after air-drying solvated monomer mixtures and the degree of hydrophilicity play a great role in their water sorption and solubility behavior.[14] Fast solvent evaporation is facilitated by its high vapor pressure,[15] but complete evaporation is difficult to achieve and is hampered by the short clinical air-blowing time (10 seconds).[16] XP Bond has tert-butyl alcohol (2-methyl-2-propanol) as a solvent, which consists of a C4 body with an alcohol group surrounded by three methyl groups. This makes it totally miscible with water and polymerizable resins.[17] Thus, although ethanol and tert-butyl alcohol have similar vapor pressures, the latter presents better stability toward chemical reaction with monomers.[16] This may have caused a higher solvent retention in XP Bond samples, so that a higher adhesive temperature was able to increase the DC but not to facilitate the solvent evaporation. Higher amounts of remaining solvent in the adhesive layer may result in more voids, and hence, increased permeability and nanoleakage.[18] In the presence of high water sorption, macromolecular polymer chains undergo a relaxation process as they swell to absorb the water. Initially, the presence of water softens the polymer by swelling the network, and reducing the frictional forces between the polymer chains.[19] After the relaxation process, unreacted monomers trapped in the polymer network are released at a rate that is controlled by the swelling and relaxation capacities of the polymer.[20] Thus, a high amount of permeability will facilitate fluid transport in and out of the network, leading to enhanced water uptake and elution.[21] These affirmations may explain why XP Bond presented the lowest DC, regardless of the temperature, and increased water sorption and solubility when it was preheated. To improve the DC of XP Bond, the use of prolonged evaporation times (60 seconds), with or without air stream, has been observed to be effective.[3]

On the other hand, it is possible that the temperature of 60°C could facilitate ethanol evaporation, which would explain the decreased water sorption and solubility obtained for all other preheated ethanol-based adhesive systems. In fact, Adper Easy One showed the highest water sorption and solubility when it was maintained at room temperature (25°C). It is possible that a higher amount of solvent remained in this adhesive at 25°C. It should also be considered that the extent of solvent retention in polymer networks depends on resin polarity. The resin polarity influences the number of hydrogen bonding sites, and the attraction between the polymer and the solvent. The higher the formation of hydrogen bonds between solvent and monomers, the harder it is to volatilize the solvent.[22] It is likely that a high number of hydrogen bonds between water and self-etching monomers of Adper Easy One could have remained at room temperature, favoring solvent retention. In addition, inclusion of relatively high concentrations of acidic monomers and water permits ionization of those monomers and solubilization of calcium and phosphate, but at the same time it makes the polymers of one-bottle self-etching adhesive systems very hydrophilic.[23] These observations may justify the high sorption and solubility of Adper Easy One at room temperature.

The method used in this study to warm the adhesive systems may not be feasible clinically. However, single-dose adhesive systems can be properly warmed in a few minutes by means of a device used for resin-based composite application, that is, Calset Compule Heater (AdDent Inc, Danbury, CT, USA).

The use of preheated ethanol/water-based adhesive systems seems to facilitate solvent evaporation, which translates to improving the DC and decreasing water sorption/solubility. However, further studies evaluating the impact of preheating other etch-and-rinse and self-etching systems on the DC, water sorption, and solubility are necessary. Also, physical properties and bond strength tests of preheated adhesive systems to tooth or composite should be performed.

CONCLUSION

Preheating single-bottle adhesive systems to 60°C improved the DC of the five adhesive materials tested in this study. Therefore, the first hypothesis that preheating would not affect the DC adhesive system was rejected. The water sorption and solubility for four of the five preheated adhesive materials was also improved, with the exception of the adhesive system containing tert-butyl alcohol. Thus, the second hypothesis that the preheating would not affect the water sorption/solubility adhesive systems was also rejected.

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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.

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In Vitro Evaluation of Midwest Caries ID: A Novel Light-emitting Diode for Caries Detection

SA Patel • WD Shepard • JA Barros
CF Streckfus • RL Quock

Clinical Relevance

Recent technology, such as Midwest Caries ID, has become available to assist clinicians with caries diagnosis. However, in light of the limited sensitivity of the Midwest Caries ID, it should only be used as an adjunctive tool until further evidence supports its diagnostic accuracy.

SUMMARY

Introduction: Traditional detection techniques have limits in diagnosing occlusal caries. Thus, more accurate methods are needed. This study evaluates the ability of the Midwest Caries ID (Midwest) to detect caries.

Methods: Two hundred sixty-four extracted, nonrestored premolars and molars were cleaned and stored in 0.2% sodium azide. Teeth were divided into three groups of 88. One examination site on each occlusal surface was chosen. Each site was inspected by a calibrated examiner via visual, Midwest, and histologic exams. First, a visual exam was performed following the International Caries Detection and Assessment guidelines. Next, the same site was inspected using the Midwest device. Finally, the tooth was sectioned mesiodistally through the site. The half with greater caries progression was visualized under a stereomicroscope (64×). Histologic appearance was scored based on the Downer system. Data were analyzed using Kendall tau-b, partial correlation coefficients, and the receiver operating characteristics curve.

Results: Overall, the Midwest scoring assessment correlated with histologic assessments (tau = 0.32; $p<0.0001$), but the visual exam had a stronger correlation (tau = 0.53; $p<0.0001$) with the histologic exam. The sensitivity and specificity of the Midwest was also reported at

*Shalizeh A Patel, DDS, University of Texas School of Dentistry at Houston, Restorative Dentistry and Prosthetics, Houston, TX

William D Shepard, BS, University of Texas School of Dentistry at Houston, Houston, TX

Juliana A Barros, DDS, MS, University of Texas-Houston School of Dentistry, Restorative Dentistry & Biomaterials, Houston, TX

Charles F. Streckfus, DDS, MA, University of Texas-Houston School of Dentistry, Houston, TX

Ryan L Quock, DDS, University of Texas-Houston School of Dentistry, Restorative Dentistry & Biomaterials, Houston, TX

*Corresponding author: University of Texas School of Dentistry at Houston, Restorative Dentistry and Prosthetics, 7500 Cambridge St., Suite 5350, Houston, TX 77054, USA; e-mail: Shalizeh.Patel@uth.tmc.edu

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0.56 and 0.84, compared with 0.92 and 0.43, respectively, for the visual exam.

Conclusions: Midwest Caries ID is a novel caries detection device that has limitations and should not be used as the sole means to detect occlusal caries.

INTRODUCTION

Conventional pit-and-fissure caries detection methods include visual, modified tactile, and radiographic examinations. Visual inspection is the dentist's primary method of caries diagnosis, but it is limited by its subjective nature.[1-4] To standardize caries diagnosis, the International and Caries Detection Assessment System (ICDAS) was created. It offers set guidelines for the quantitative and qualitative determination of caries progression and has been shown to be effective for both novice and expert practitioners.[2,5,6] Furthermore, the recent increase in the use of dental loupes has improved the practitioner's ability to diagnose pit-and-fissure caries.[7] In the past, traditional tactile exams were simple to perform but posed several risks. Pressure from a sharp explorer may cause a potentially remineralizable lesion to cavitate.[8] In addition, bacteria may be transported to other sites in the oral cavity via the explorer.[9,10] For these reasons, a modified tactile technique is now recommended, where an explorer can be used to remove plaque and lightly assess the hardness of involved tooth surfaces.[11] Radiographic methods include the use of bitewings and have become the standard of care in effectively identifying proximal lesions, which may remain undetected during visual exams. However, bitewings pose a radiation exposure to the patient and have a low sensitivity (30%) in detecting early-stage lesions in enamel (occlusal and proximal).[9,10,12,13] Furthermore, tooth structure must demineralize by at least 40% to appear as a lesion on radiographic film.[14] Therefore, radiographs have limited capabilities for early detection.

Despite the multitude of methods of caries detection, there are still considerable limitations regarding subjectivity. A more objective, reproducible, and accurate assessment method is needed. Recent studies have shown that bacteria in carious lesions release metabolites during growth, many of which are ringed structures, such as porphyrins. These structures demonstrate the ability to absorb energy at a given wavelength (650 nm, red spectrum) and release it at a higher level (680 nm), thereby fluorescing.[3,9-10,15-18] A previously designed system (DIAGNOdent, KaVo Dental GmbH, Biberach/Riβ, Germany) uses this physical property to find and detect caries. However, this system is recommended as an adjunct and not as the sole means of diagnosis.[10,16] A new product, Midwest Caries ID (Dentsply, York, PA, USA), has recently been introduced as a caries detector, but limited studies concerning its efficacy have been conducted.[19] Because of the proprietary nature of the device technology, the only technical information regarding the Midwest Caries ID (Midwest) was obtained through personal communication (Marie D. George, RDH, MS, oral communication, January 23, 2012). According to the manufacturer, Midwest detects hypocalcification by analyzing the structural integrity of enamel rods. Translucency is determined in part by the mineral content of the rods; therefore, a difference in the reflectance characteristics of sound versus carious tooth structure can theoretically be used to calculate the degree of caries progression present. This is accomplished via light-emitting diodes, and caries progression is determined based on manufacturer's studies concerning the absorption characteristics of carious material. This is in contrast to DIAGNOdent, where the detection method is based on the amount of carious metabolites present in infected tooth structure.

The practical purpose of this study was to compare the diagnostic ability (sensitivity and specificity) of the Midwest technology to a current standard visual assessment system (ICDAS). Both methods were confirmed for caries detection with histologic sections as this study's gold standard. To provide quality care, the Midwest needs to be able to identify caries (sensitivity) and differentiate it from surrounding healthy tooth structure (specificity). Our null hypothesis was that the Midwest device does not differ in sensitivity and specificity from a visual exam, using histologic exam as the gold standard.

METHODS AND MATERIALS

Two hundred sixty-four extracted permanent premolars and molars (nonrestored) were selected from a pool of extracted, noncavitated (ie, ICDAS scores of 0-4) teeth collected according to human subjects regulations at the University of Texas Health Science Center in Houston. Teeth were stored in 0.9% sodium chloride/0.2% sodium azide. Teeth were cleaned with prophylactic paste (Enamel Pro with ACP, Premier, Hannover, Germany) and a rotating brush. Next, teeth were thoroughly rinsed to ensure that no residual paste was left and then dried and photographed (Canon Digital Rebel XSi, Canon Macro Lens EF 100mm 1:2.8 USM, Lake Success,

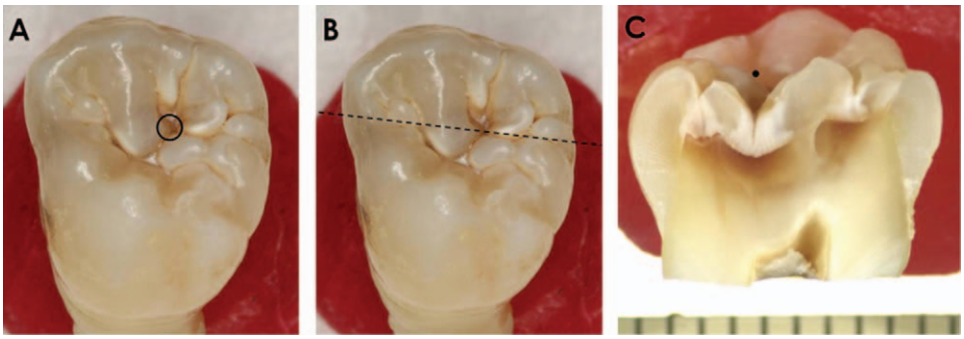

Figure 1. *Site selection and sectioning. (A) Each tooth was photographed, and the examination site was chosen and recorded on the occlusal surface. Visual exam and Midwest evaluation were completed following methodology previously described. (B) The examination site was sectioned for histologic examination and (C) imaged by stereomicroscope (64×).*

NY, USA) before examination.[20] One random site on each tooth's occlusal surface within the pit-and-fissure system was selected and marked on the photographed image for ease of relocation and consistency among the involved examiners (Figure 1A). It has been noted that one site can accurately approximate the entire occlusal surface when using laser reflectance for caries measurement.[21] Each tooth was subjected to three tests: visual, Midwest, and histologic. Three calibrated examiners were recruited to perform the tests. Before the study, examiners were trained in the ICDAS and Downer histologic classification systems.[22,23] Examiners also watched the DVD supplied by Dentsply for the Midwest calibration (provided to clinician upon purchase of the device).[24,25] Approximately 20 digital photographs of various teeth were used in these calibration sessions, which were not included in the main study. To reduce systematic error due to observer bias,[26] teeth were divided into three groups of equal size, with each examiner assigned to perform one test per group. No examiner performed the same test for more than one group (Table 1).

The following protocol was used during the visual exam. Each tooth was air dried via air syringe (5 seconds) and observed under standard dental operating light. Examiners used 2.5× magnification dental loupes. Caries presence and progression were based on ICDAS scoring (Table 2).[2,3,5,6,16,20]

Table 1: *Examiners and Assigned Teeth*[a]

Tooth No.	Examiner		
	1	2	3
1-88	A	B	C
89-176	C	A	B
177-264	B	C	A

[a] *To reduce the score bias, the study recruited three calibrated examiners. The 264 teeth were divided into three groups of 88. Each examiner was assigned to perform one examination (A, visual; B, Midwest; C, histologic) per group.*

The reflectance technology exam used the Midwest. Per manufacturer's instructions, the occlusal surface was not dried. Manufacturer guidelines provided a qualitative scoring system, which was used to determine caries presence and progression (Table 2).

The histologic exam was performed to determine the true extent of caries progression (gold standard). Roots of teeth were removed apical to the cemento-enamel junction via diamond bur (D849, Komet USA, Rock Hill, SC, USA). The crown was cut mesiodistally through the investigation site via a diamond saw with continuous water irrigation (Isomet 11-1180 Low Speed Saw, Buehler Ltd, Lake Bluff, IL, USA) (Figure 1B). Sections were histologically examined at 64× under a stereomicroscope (Leica MZ9.5, Wetzlar, Germany) (Figure 1C). Caries presence and progression was based on the Downer system (Table 2).[1,3,16,20]

For each test, data from the three examiners were compiled and analyzed using the IBM SPSS Statistics 20 software package (Armonk, NY, USA). The first level of analysis was to perform descriptive analyses. Tables with frequency and expected observations were also constructed. The data were ordinal and therefore required nonparametric statistical evaluation. The categoric scores for each evaluation (ICDAS 0-6, Downer 0-4, and Midwest 0-3) differed in their carious assessment criteria and could not undergo meaningful recategorization or be collapsed to common categories across the varying assessments. Weighted kappas were also difficult to perform as categories 5 and 6 of the ICDAS scoring scale were excluded from the scoring. As shown in Table 1, each examiner scored 264 teeth; however, to control for examiner bias, each examiner scored

Table 2: *Criteria for Examinations*[a]

Visual Exam		Midwest Exam		Histology Exam	
Score	**Criteria**	**Score**	**Criteria**	**Score**	**Criteria**
VE-0	No change in enamel translucency after 5 seconds of air drying	ME-0	No caries	HE-0	No enamel demineralization or a narrow surface zone of opacity
VE-1	First visual change in enamel after 5 seconds of air drying; limited to the confines of the pit and fissure area	ME-1	Demineralization close to the surface: caries in enamel	HE-1	Enamel demineralization limited to the outer 50% of the enamel layer
VE-2	Distinctly visible changes on a wet surface and/or and area wider than the fissure area	ME-2	Medium-size demineralization in area possibly past the dentoenamel junction	HE-2	Demineralization involving the inner 50% of the enamel, up to the dentoenamel junction
VE-3	Localized enamel breakdown due to caries, with no visible dentin or underlying shadow	ME-3	Small/deep lesion into dentin	HE-3	Demineralization involving the outer 50% of the dentin
VE-4	Underlying dark shadow from dentin	—	—	HE-4	Demineralization involving the inner 50% of the dentin

[a] *These values represent the characteristics used to quantify the extent of a carious lesion. Visual exam used ICDAS.[6] ICDAS levels 5 and 6 were preferentially excluded from this study (ie, gross cavitation of coronal surface). Midwest exam used the manufacturer's guidelines for detecting carious involvement.[24,25] Histologic exam used the Downer classification system.[23]*

different teeth for each assessment. Because each examiner did not score the same teeth with the same assessment technique, the interrater kappa statistic is not applicable. As a consequence, a nonparametric assessment comparing rank order was determined by using the Kendall tau-b statistic. A partial correlation coefficient was also applied controlling for tooth number or which tooth was scored.

A receiver operating characteristics (ROC) curve was constructed comparing the sensitivity and specificity of the visual exam with the Midwest and histologic assessments. The histologic scores were transformed from an ordinal score to a dichotomous score (0 or 1). A zero represented no caries; a score of one, the disease state, represented the histologic presence of caries.

RESULTS

Two hundred sixty four teeth were examined. Each examiner evaluated 88 of the 264 teeth using each of the three caries assessment techniques. The descriptive analyses are shown in Table 3.

Table 4 shows that the visual exam correlated with both the Midwest (tau = 0.32; $p<0.0001$) and histologic (tau = 0.53; $p<0.0001$) assessments. Furthermore, the results demonstrated that the Midwest evaluation correlated with the histologic (tau = 0.33; $p<0.0001$) assessment. If the histologic assessment were considered the gold-standard, then the Kendall tau-b statistic suggests a stronger association between the visual exam and the histologic assessment compared with the Midwest evaluation.

Because the examiners did not inspect the same teeth with the same assessment technique, a partial correlation coefficient was also performed on the data controlling for tooth number. This further confirmed the Kendall tau-b statistical analysis. Table 5 shows that the visual exam correlated with both the Midwest (tau = 0.34; $p<0.0001$) and histologic (tau = 0.63; $p<0.0001$) assessments. The results also indicated that the Midwest evaluation correlated with both the visual (tau = 0.34; $p<0.0001$) and histologic (tau = 0.35; $p<0.0001$) assessments. However, the partial correlation analysis suggests that the visual exam has a stronger association with the histologic exam than the Midwest exam.

The results of the sensitivity and specificity are similarly illustrated by the graphic ROC curve shown in Figure 2. The area under the curve for the visual exam was 0.84, with sensitivity and specificity of 0.92 and 0.43, respectively; the area under the curve for the Midwest was 0.68, with a sensitivity and specificity of 0.56 and 0.84, respectively.

DISCUSSION

Midwest Caries ID is a small, battery-operated, and easily autoclavable device that is available to oral health care providers for detecting and quantifying caries. Because of the limited data concerning its effectiveness, this investigation was conducted. Although this study supported the finding that the Midwest has the capability to detect caries, the Kendall tau-b and partial correlation coefficients

Table 3: *Assessed Teeth Using Three Modes of Examination Techniques*[a]

Examination Technique	Examiner			Total
	1	2	3	
Visual Exam				
0				
Count	14	13	11	38
Expected Count	12.7	12.7	12.7	38.0
1				
Count	27	10	42	79
Expected Count	26.3	26.3	26.3	79.0
2				
Count	22	11	17	50
Expected Count	16.7	16.7	16.7	50.0
3				
Count	15	38	15	68
Expected Count	22.7	22.7	22.7	68.0
4				
Count	8	8	2	18
Expected Count	6.0	6.0	6.0	18.0
5				
Count	2	8	1	11
Expected Count	3.7	3.7	3.7	11.0
Total				
Count	88	88	88	264
Expected Count	88.0	88.0	88.0	264.0
Midwest Exam				
0				
Count	50	45	41	136
Expected Count	45.3	45.3	45.3	136.0
1				
Count	6	8	9	23
Expected Count	7.7	7.7	7.7	23.0
2				
Count	5	9	3	17
Expected Count	5.7	5.7	5.7	17.0
3				
Count	27	26	35	88
Expected Count	29.3	29.3	29.3	88.0
Total				
Count	88	88	88	264
Expected Count	88.0	88.0	88.0	264.0
Histological Exam				
0				
Count	12	31	6	49
Expected Count	16.3	16.3	16.3	49.0
1				
Count	8	17	12	37
Expected Count	12.3	12.3	12.3	37.0
2				
Count	25	13	19	57
Expected Count	19.0	19.0	19.0	57.0
3				
Count	31	24	40	95
Expected Count	31.7	31.7	31.7	95.0
4				
Count	12	3	11	26
Expected Count	8.7	8.7	8.7	26.0
Total				
Count	88	88	88	264
Expected Count	88.0	88.0	88.0	264.0

[a] *Descriptive analyses of the 264 teeth examined by three examiners evaluating 88 teeth per group using the three caries assessment techniques.*

illustrated that the visual exam correlated more with the histologic findings, the gold standard, than the Midwest. The sensitivity and specificity for this detection device was also reported at 0.56 and 0.84, respectively.

At the time of experimental design, the authors only encountered two studies regarding the Midwest.[27,28] Krause and others[28] reported sensitivity of 100%, which was calculated by comparing the Midwest to radiographic findings. As mentioned earlier, radiographs have 30% sensitivity in detecting incipient caries.[12,13] Additionally, radiographic film can only detect a lesion after 40% demineralization.[14] However, this present study did not support the numbers reported by Krause and others.[28] Such a discrepancy can be explained by the fact that in the study by Krause and colleagues, histology, and not radiology, was used as the standard of comparison. Because radiographs are inherently less sensitive, the reported values of Midwest may be higher in the preceding study. It has been established that ground sections examined by stereomicroscopy can be validated against the true diagnosis and therefore can be used as the gold standard where accuracy of caries diagnosis are being tested.[29] We also used a visual system, ICDAS, which is a standardized approach to caries detection and management. Such visual examination criteria help clinicians to identify caries in all stages, in particular at an early phase, which is a known limitation of radiographs.

One study has subsequently been published that examined the efficacy of the Midwest compared with DiagnoDENT and ICDAS.[19] Sensitivity and specificity of the device were reported as 56% and 84%, respectively, which is identical to the values reported

Table 4: *Kendall tau-b Correlations*[a]

Kendall tau-b	Tooth No.	Visual Exam	Midwest Exam	Histologic Exam	Examiner
Tooth number					
Correlation coefficient	1.000	–.077*	–.048	–.230**	.818**
Significance (one-tailed)	—	.045	.157	.000	.000
N	264	264	264	264	264
Visual exam					
Correlation coefficient	–.077*	1.000	**.321****	**.531****	–.069
Significance (one-tailed)	.045	—	.000	.000	.094
N	264	264	264	264	264
Midwest exam					
Correlation coefficient	–.048	**.321****	1.000	**.333****	–.024
Significance (one-tailed)	.157	.000	—	.000	.333
N	264	264	264	264	264
Histologic exam					
Correlation coefficient	–.230**	**.531****	.333**	1.000	–.257**
Significance (one-tailed)	.000	.000	.000	—	.000
N	264	264	264	264	264
Examiner					
Correlation coefficient	.818**	–.069	–.024	–.257**	1.000
Significance (one-tailed)	.000	.094	.333	.000	.
N	264	264	264	264	264

[a] *Kendall tau-b statistic suggests a stronger association between the ICDAS scoring assessment and the histologic exam than with the Midwest exam.*
** Correlation is significant at the .05 level (one-tailed).*
*** Correlation is significant at the .01 level (one-tailed).*

Table 5: *Partial Statistical Correlations*[a]

Control Variables	Visual Exam	Midwest Exam	Histologic Exam	Examiner
Tooth no.				
Visual exam				
Correlation	1.000	**.341**	.629	.037
Significance (one-tailed)	—	.000	.000	.273
df	0	261	261	261
Midwest exam				
Correlation	**.341**	1.000	.351	.092
Significance (one-tailed)	.000	—	.000	.068
df	261	0	261	261
Histologic exam				
Correlation	**.629**	**.351**	1.000	–.041
Significance (one-tailed)	.000	.000	—	.253
df	261	261	0	261
Examiner				
Correlation	.037	.092	–.041	1.000
Significance (one-tailed)	.273	.068	.253	—
df	261	261	261	0

[a] *Partial correlation analysis confirms Kendall tau-b statistics, suggesting a stronger association between the ICDAS scoring assessment and the histologic exam than with the Midwest exam.*

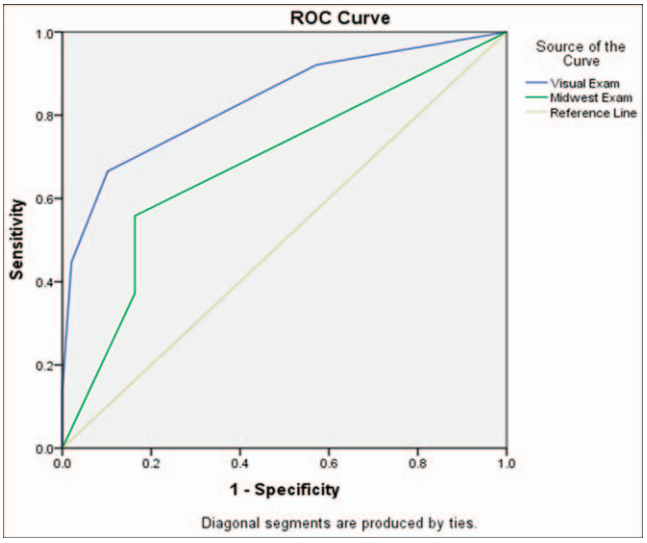

Figure 2. *Area under the ROC curve. The areas under the curve for the visual and Midwest exams suggest that the visual exam is more sensitive in detecting caries (better at identifying caries: true positive), whereas, Midwest is more specific (better in differentiating caries against sound tooth structure: true negative).*

in this study. This suggests that the Midwest is not optimal at detecting caries but is fairly reliable at determining healthy tooth structure. ICDAS, by contrast, demonstrated sensitivity and specificity of 0.92 and 0.43, respectively.

To review, sensitivity is defined as the identification of caries (true positive), and specificity is the differentiation of caries versus fully mineralized tooth structure (true negative).[3,15,16] A sensitivity of 0.92 suggests that a visual system rarely misses caries when it is present; specificity of 0.43 suggests, however, that it can lead to some overdiagnosis (false positive). We believe this phenomenon is due to the nature of ICDAS—a quantitative system that can inherently make a practitioner more aware of initial signs of incipient lesions. Although the overdiagnosis of caries is undesirable, therapies directed at addressing incipient lesions are typically noninvasive.[5] In contrast, detection with Midwest can mean caries is most likely present, but it may also fail to detect early-stage lesions (sensitivity 0.56), leaving the site susceptible to further caries progression.

It is important to acknowledge the difference between detection and diagnosis.[13] Detection of caries is the recognition of a significant clinical finding. Diagnosis is both an art and a science: it requires a keen eye and a comprehensive knowledge of the disease process. The practitioner must be able to critically appraise such clinical findings rather than solely relying on data obtained from a particular detection method. Based on the results of this study, the null hypothesis that the Midwest device does not differ in sensitivity or specificity from histologic examination is rejected.

CONCLUSION

Light-emitting diodereflectance technology, like the Midwest, may only be useful in detecting caries when coupled with visual exam, modified tactile exam, and radiographs. Such a full spectrum of exams will enable the examiner to identify caries in all stages. However, a full understanding of the equipment can lead to appreciation for the device's strengths and shortcomings. When used appropriately, it may prove to be a functional addition to the practitioner's armamentarium.

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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.

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A Comprehensive Laboratory Screening of Three-Step Etch-and-Rinse Adhesives

AD Loguercio • I Luque-Martinez • MA Muñoz
AL Szesz • J Cuadros-Sánchez • A Reis

Clinical Relevance

The evaluation of several bonding and mechanical properties in short-term laboratory screening tests allowed better screening of the adhesives available on the market without requiring long-term studies.

SUMMARY

Objectives: This study evaluated several bonding (microtensile bond strengths [μTBS], nanoleakage [NL], and *in situ* degree of conversion [ISDC] on dentin) and mechanical properties (ultimate tensile strength [UTS], degree of conversion [DC], water sorption [WS], and solubility [SL] in water) of four three-step etch-and-rinse adhesives in the short term.

Methods: A total of 28 molars were used in this study. The dentin surfaces were bonded with the following adhesives: All-Bond 3 (ALB3); Fusion Duralink (FSDL); Optibond FL (OBFL), and Scotchbond Multi-Purpose (SBMP). After each adhesive-system application, composite resin build-ups were added. For bonding tests, specimens were sectioned in order to obtain bonded sticks. The sticks were divided to be tested for μTBS (0.5 mm/min), for NL (n=2), and ISDC (n=2). For NL, they were immersed in 50% silver nitrate and analyzed by scanning election microscopy. For ISDC, the hybrid layer was evaluated by micro-Raman spectroscopy. An hourglass-shaped matrix (UTS) or disk-shaped matrix (WS and SL) was filled with primer and adhesive (1:1 ratio) and light-polymerized. For UTS evaluation, the specimens were tested under tension. For WS and SL, specimens were desiccated and stored

*Alessandro D Loguercio, DDS, MS, PhD, professor, Restorative Dentistry, Universidade Estadual de Ponta Grossa, Ponta Grossa, Brazil

Issis Luque-Martinez, DDS, MS, PhD student, Restorative Dentistry, Universidade Estadual de Ponta Grossa, Ponta Grossa, Brazil

Miguel Angel Muñoz, DDS, MS, PhD student, Restorative Dentistry, Universidade Estadual de Ponta Grossa, Ponta Grossa, Brazil; professor, Faculty of Dentistry, Universidad de Valparaíso, Valparaíso, Chile

Anna Luiza Szesz, DDS, MS student, Restorative Dentistry, Universidade Estadual de Ponta Grossa, Ponta Grossa, Brazil

Johanna Cuadros-Sánchez, DDS, MS student, Restorative Dentistry, Universidade Estadual de Ponta Grossa, Ponta Grossa, Brazil

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

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

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in distilled water to evaluate water diffusion kinetics over a 28-day period. The DC of the adhesives was evaluated by Fourier transformed infrared spectroscopy. The data from each test were analyzed by appropriate statistical methods.

Results: OBFL resulted in the highest μTBS, lower NL, higher ISDC and DC, and higher UTS than other adhesives ($p<0.05$), as well as lower WS (similar to ALB3 and FSDL) and SL (similar to ALB3 and SBMP) ($p>0.05$). ALB3 showed a higher NL and the lowest DC value. FSDL showed the highest NL and SL and the lowest ISDC. SBMP showed the lowest pattern of WS ($p<0.05$).

Conclusion: OBFL showed the best results in all the properties evaluated, and it can be considered the gold standard of the three-step etch-and-rinse adhesive systems.

INTRODUCTION

The use of 85% phosphoric acid to improve the infiltration of an acrylic resin into enamel was the genesis of all dental adhesive systems used in dentistry.[1] The interaction of dental adhesives with enamel and dentin results from resin monomers permeating the microporosities created by acidic agents and polymerized monomers subsequently becoming wrapped around the exposed hydroxyapatite crystals and/or collagen fibers.[2]

In spite of the different classifications that have appeared within the last two decades, dental adhesives are currently classified according to how they interact with the dental substrate and thus are divided into etch-and-rinse (ER) and self-etch (SE) adhesives.[2] The number of application steps categorizes adhesive systems within each of the two strategies. For example, ER adhesive systems can be available in a three-step and a two-step protocol, depending on whether the primer and bonding resin are separate or combined in one bottle.

Although three-step ER adhesives have been considered the clinical gold standard in dental bonding,[3-6] this issue seems to be controversial, according to recent systematic reviews of the literature.[3,5,7-9] The retention rates of three-step ER systems are quite variable, with an annual failure rate varying from 0% to 16%.[3] Furthermore, Peumans and others[3] in their systematic review reported that at least three out of 10 three-step ER adhesives did not meet the requirements of the American Dental Association guidelines for provisional and full acceptance of the restorations, and in some of the clinical studies reviewed, they did not gain full acceptance.

It is claimed that the use of a hydrophobic resin coating in the three-step ER is responsible for better *in vitro* and *in vivo* performance than that of their 2-step counterparts[10]; however, one cannot rule out the role of the chemistry of three-step ER systems, which varies considerably depending on the brand.

For instance, the type of solvent presented in the formulation of the primer has been mentioned as a factor affecting the performance of the materials, with the ethanol-based systems performing better than the acetone-based types.[4] Moreover, the bonding resin usually claimed to be a hydrophobic resin coating can contain hydrophilic monomers, such as hydroxyl-ethyl-methacrylate (HEMA), which may ultimately influence the bond performance.[11] Other differences such as filler loading may also impact the material's performance.

Based on the foregoing, the aim of this study was to compare several bonding and mechanical properties of three-step ER adhesives available on the market. The microtensile bond strengths (μTBS), nanoleakage (NL), *in situ* degree of conversion (ISDC) on dentin and ultimate tensile strength (UTS), degree of conversion (DC), water sorption (WS), and solubility in water (SL) for all the adhesives were also evaluated.

METHODS AND MATERIALS

Tooth Selection and Preparation

A total of 28 extracted, caries-free human third molars were used. The teeth were collected after obtaining the patients' informed consent under a protocol approved by the local Ethics Committee Review Board. The teeth were disinfected in 0.5% chloramine, stored in distilled water, and used within six months after extraction.

A flat occlusal dentin surface was exposed after wet grinding the occlusal enamel with 180-grit silicone-carbide (SiC) paper for 60 seconds. The exposed dentin surfaces were further polished with wet 600-grit SiC paper for 60 seconds to standardize the smear layer. These teeth were used for testing resin-dentin μTBS, NL, and measurement of ISDC.

Adhesive and Restorative Procedure

Teeth were randomly assigned for bonding with four different materials (n=7): All-Bond 3 (ALB3; Bisco Inc, Schaumburg, IL, USA); Fusion Duralink (FSDL;

Table 1: *Adhesive Materials (Manufacturer), Composition (Batch No.), and Application Mode of the Adhesive Systems Used*

Adhesives (Manufacturer)	Composition (Batch No.)	Mode of Application
All-Bond 3 (Bisco)	Etchant: UNI-ETCH-37: 37% phosphoric acid, polymer thickener (1100005635). Adhesive: Part A ethanol, sodium benzene sulfinate dehydrate, NTG-GMA (1100005448); Part B: Bis(glyceryl 1,3 dimethacrylate) phosphate, HEMA, biphenyl dimethacrylate (1100005448). Resin: Bis-GMA, urethane dimethacrylate, Sr glass (1100001528).	1. Application of 37% phosphoric acid (15 s) 2. Rinse (15 s). 3. Air-dry, leaving the preparation visibly moist. 4. Mix equal amounts of part A and B in mixing well with a microbrush for 5 s. 5. Apply one coat under finger pressure for 15-20 s. 6. Air-dry under maximum pressure for 15 s. 7. Light-polymerize for 10 s at 1200 mW/ cm^2. 8. Apply a thin layer of All-Bond Resin. 9. Light-polymerize for 10 s at 1200 mW/cm^2.
Fusion Duralink (Angelus)	Etchant: 37% orthophosphoric acid, water, thickener, pigments (22458). Primer: methacrylate esthers, ethanol, water (14545). Adhesive: methacrylate esthers, photoinitiators, polymerization accelerators (14549).	1. Apply the phosphoric acid gel on dentin for 15 s. 2. Rinse the acid gel with a water spray for 15 s. 3. Remove the excess water with absorbent paper or a clean brush, leaving the surfaces slightly wet (shiny). 4. With a disposable brush, apply a layer of primer on dentin and rub it slightly for 30 s. 5. Apply a light air jet from a distance of 10 cm for 10 s; the dentin should be shiny and without excess primer. 6. With another disposable brush, apply a thin and uniform layer of adhesive on dentin. 7. Light-polymerize for 10 s at 1200 mW/cm^2.
Optibond FL (Kerr)	Etchant: 37.5% phosphoric acid, water, silica thickener (3676600). FL Prime: HEMA, GPDM, MMEP, water, ethanol, CQ, BHT (3539622). FL Adhesive: Bis-GMA, HEMA, GDMA, CQ, ODMAB, filler (fumed SiO_2, barium aluminoborosilicate, Na_2SiF_6), coupling factor A174 (approximately 48 wt% filled) (3538016).	1. Apply etchant on dentin for 15 s. 2. Rinse thoroughly for 15 s. 3. Air-dry for 3 s (do not desiccate). 4. Apply primer with brushing motion for 15 s. 5. Air-dry for 5 s. 6. Using same applicator, apply adhesive with light brushing motion for 15 s. 7. Air-thin for 3 s. 8. Light-polymerize for 20 s at 1200 mW/cm^2.
Scotchbond Multi-Purpose (3M ESPE)	Etchant: 35% phosphoric acid, silica thickener (N261433). Primer: HEMA, polyalkenoic acid polymer, water (N322814). Adhesive: Bis-GMA, HEMA, tertiary amines, and photo-initiator (N342538).	1. Apply etchant for 15 s on dentin. 2. Rinse thoroughly for 15 s. 3. Dry for 5 s. 4. Apply primer on dentin for 10 s. 5. Dry gently for 5 s. 6. Apply adhesive. 7. Light-polymerize for 10 s at 1200 mW/cm^2.

Abbreviations: BHT, butylated hydroxytoluene; Bis-GMA, bisphenol A diglycidyl methacrylate; CQ, camphorquinone; GPDM, glycerol phosphate dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; MMEP: mono-2-methacryloyloxyethyl phthalate; NTG-GMA: N-tolyglycine glycidyl methacrylate; ODMAB: 2-(Ethylhexyl)-4-(dimethylamino)benzoate.

Angelus, Londrina, Brazil); Optibond FL (OBFL; Kerr, Orange, CA, USA), and Scotchbond Multi-Purpose (SBMP; 3M ESPE, St Paul, MN, USA). Each adhesive system was applied on the flat dentin surfaces, according to the manufacturers' instructions (Table 1).

On each prepared surface a composite restoration (Filtek Z350, 3M ESPE) was built up in two increments of 2 mm and was light-polymerized for 40 seconds using a LED light-curing unit set at 1200 mW/cm^2 (Radii-cal, SDI Limited, Bayswater, Australia). The same light-curing unit was used throughout this study.

Microtensile Bond Strength

After the restored teeth had been stored in distilled water at 37°C for 24 hours, they were longitudinally sectioned in the mesio-to-distal and buccal-to-lingual directions across the bonded interface, using a low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to obtain resin-dentin sticks with a cross-sectional area of approximately 0.8 mm^2, measured with a digital caliper (Digimatic caliper, Mitutoyo, Tokyo, Japan). Four resin-dentin bonded sticks from each tooth were not tested in the tensile mode, because they were used for the ISDC and NL evaluations.

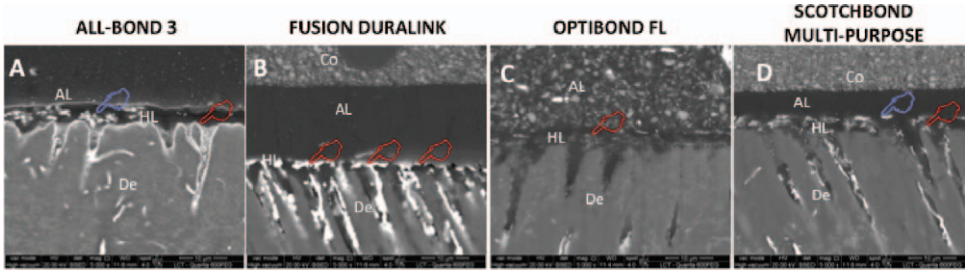

Figure 1. *Representative backscatter scanning electron microscope images of the resin-dentin interfaces bonded to each one of the three-step etch-and-rinse adhesive systems tested. The amount of nanoleakage was lower and practically occurred within the hybrid layer for Optibond FL (red hand). For Fusion Duralink, the amount of nanoleakage was higher than other materials; with most silver nitrate uptake occurring throughout the entire thickness of the HL (red hands). Nevertheless, All-Bond 3 and Scotchbond Multi-Purpose showed an intermediate amount of silver nitrate uptake, which occurred in the same points of the HL (red hands) and the AL, forming so-called water trees (blue hands) (Co, composite; De, dentin; HL, hybrid layer; AL, adhesive layer).*

All the remaining bonded sticks were attached to a Geraldeli jig[12] (Odeme Biotechnology, Joaçaba, Brazil) with cyanoacrylate adhesive and tested under tensile forces (Model 5565, Instron, Canton, OH, USA) at 0.5 mm/min until failure. The μTBS values were calculated by dividing the load at failure by the cross-sectional bonding area. The failure mode was classified as cohesive (failure exclusively within dentin or resin composite), adhesive (failure at the resin/dentin interface), or mixed (failure at the resin/dentin interface that included cohesive failure of the neighboring substrates). The failure mode was analyzed under a stereomicroscope at 100× magnification (Olympus SZ40, Tokyo, Japan). Because few specimens presented premature failures, they were included in the bond-strength mean.

Nanoleakage Evaluation

Two resin-dentin bonded sticks were placed in ammoniacal silver nitrate[13] in darkness (24 hours), rinsed thoroughly in distilled water, and immersed in photo-developing solution (8 hours) under a fluorescent light to reduce silver ions to metallic silver grains. Specimens were polished down with 600-, 1000-, 1200-, 1500-, 2000-, and 2500-grit SiC paper and 1 and 0.25 μm diamond paste (Buehler Ltd) using a polishing cloth. They were ultrasonically cleaned, air-dried, mounted on stubs, and sputter-coated with carbon-gold (MED 010, Balzers Union, Balzers, Liechtenstein). Resin-dentin interfaces were analyzed in a scanning electron microscope operated in the backscattered mode (LEO 435 VP, LEO Electron Microscopy Ltd, Cambridge, UK) (Figure 1).

Three pictures from each bonded stick were taken.[14] The relative percentage of NL in each image was measured by a blinded researcher, using the UTHSCSA ImageTool 3.0 software program (Department of Dental Diagnostic Science at The University of Texas Health Science Center, San Antonio, TX, USA).

In Situ Degree of Conversion

The adhesive interfaces of the two other resin-dentin bonded sticks were wet polished with 1500-, 2000-, and 2500-grit SiC paper for 15 seconds each. They were ultrasonically cleaned for 20 minutes in distilled water and stored in water at 37°C for 24 hours prior to performing the DC readings. The micro-Raman spectrophotometer (Bruker Optik GmbH, Ettlingen, Baden-Württemberg, Germany) was first calibrated for zero and then for coefficient values using a silicon specimen. Specimens were analyzed using the following micro-Raman parameters: 20 mW neon laser with 532 nm wavelength, spatial resolution of ≈3 μm, spectral resolution ≈5 cm^{-1}, accumulation time of 30 seconds with six coadditions, and magnification of 100× (Olympus UK, London, UK) to a ≈1 μm beam diameter. Spectra were taken at the dentin-adhesive interface, at three different sites for each specimen.

Spectra of unpolymerized adhesives were taken as reference. Postprocessing of spectra was performed using the Opus Spectroscopy Software Program version 6.5 (Bruker Optik GmbH, Ettlingen, Baden-Württemberg, Germany). The ratio of the double-bond content of the monomer to the polymer in the adhesive was calculated according to the following formula:

$$DC(\%) = \left(1 - \frac{R_{(cured)}}{R_{(uncured)}}\right) \times 100$$

where R is the ratio of aliphatic and aromatic peak areas at 1639 cm^{-1} and 1609 cm^{-1} in polymerized and unpolymerized adhesives.

Ultimate Tensile Strength

An hourglass-shaped metal matrix (10 mm long, 2 mm wide, and 1 mm deep with a cross-sectional area of 0.8 mm^2) was isolated with petroleum jelly. To simulate a clinical application, the primer was first inserted into a metal matrix and gently air-dried in accordance with the manufacturer's directions (Table 1). Then, the bonding resin was put into the metal matrix and slightly mixed with the primer using a microbrush. The mixture was again air-dried in accordance with the manufacturer's recommendations (Table 1), and all visible air bubbles trapped in the bonding resin were carefully removed. The bonding resin and the primer were added in a ratio of 1:1. A thin strip of polyester sheeting was placed over the bonding resin, and the surface was light-polymerized for 40 seconds. Eight specimens were prepared for each group, and were tested under tensile forces as described for the μTBS test.

Degree of Conversion of the Adhesive Pellicle

The bonding resin was mixed with the primer in a ratio of 1:1 and placed on a thin strip of polyester sheeting in the same way as reported for the UTS. Then, the adhesive was covered with another thin strip of polyester sheeting and light-polymerized for 40 seconds. Each thin adhesive layer was carefully removed with a blade and stored in a dark and dry environment for 24 hours, after which the DC was determined by Fourier transformed infrared analysis (Spectrum 100, Perkin Elmer, Waltham, MA, USA).

The spectra of the polymerized and unpolymerized bonding resins were obtained with 32 scans at a resolution of 4 cm^{-1} in transmission mode. The percentage of unreacted carbon-carbon double bonds (% C=C) was determined from the ratio of absorbance intensities of aliphatic C=C (peak height at 1640 cm^{-1}) against an internal standard before and after specimen polymerization. The aromatic C=C bond (peak height at 1610 cm^{-1}) absorbance was used as an internal standard. The DC was determined by subtracting the % C=C from 100%. Five specimens were tested for each group.

Water Sorption and Solubility

The bonding resin was mixed with the primer in a ratio of 1:1 and placed on a thin strip of polyester sheeting in the same way as reported for the UTS. Ten resin disks of each adhesive were produced in a circular metal mold (5.8 mm diameter, 0.8 mm thick) after isolation with petroleum jelly. The primer and bonding resin were applied in a ratio of 1:1 as previously reported for the UTS test. All visible air bubbles trapped in the adhesives were carefully removed. Next, the solvent evaporation was performed with an air syringe at a distance of 10 cm for 40 seconds. A thin strip of polyester sheeting was placed on top of the adhesive, which was light-polymerized for 40 seconds to allow the specimen to be removed without permanent deformation.

WS and SL were determined according to the International Organization for Standardization (ISO) specification 4049.[15] Immediately after polymerization, the specimens were placed in a desiccator, transferred to a preconditioning oven at 37°C, and left undisturbed for 10 days. After this period, specimens were repeatedly weighed at 24-hour intervals until a constant mass (m_1) was obtained (ie, variation was less than 0.2 mg in any 24-hour period). Thickness and diameter of the specimens were measured using a digital caliper, rounded to the nearest 0.01 mm, and these measurements were used to calculate the volume of each specimen (in mm^3).

After this, specimens were then individually placed in sealed vials containing 10 mL of distilled water (pH 7.2) at 37°C. After fixed time intervals of 1, 2, 3, 4, 5, 6, 7, 14, and 28 days of storage, the vials (15 mL; Eppendorf of Brazil, São Paulo, Brazil) were removed from the oven and left at room temperature for 30 minutes. The specimens were washed in running water, gently wiped with a soft absorbent paper, weighed on an analytical balance (m_2), and returned to the vials containing 10 mL of fresh distilled water. After 28 days of storage, the specimens were dried in a desiccator containing fresh silica gel in an oven at 37°C and left undisturbed for 10 days. They were weighed daily until a constant mass (m_3) was obtained (as previously described). The initial mass determined after the first desiccation process (m_1) was used to calculate the change in mass after each fixed time interval during the 28 days of storage in water. Changes in mass were plotted against the storage time in order to obtain the kinetics of WS during the entire period of water storage.

WS and SL over the 28 days of water storage were calculated using the following formulas: $WS = (m_2 - m_3) / V$ and $SL = (m_1 - m_3) / V$.

Table 2: *Number and Percentage of Specimens (%) According to Fracture Mode on Microtensile Bond-Strength Test; Means and Standard Deviations of Microtensile Bond Strength (μTBS), Nanoleakage (NL), and* In Situ *Degree of Conversion (ISDC)*[a]

Adhesive Systems	Fracture Pattern				μTBS (MPa)	NL (%)	*In Situ* DC (%)
	A	C	A/M	PF			
ALB3	39 (78)	7 (14)	1 (2)	3 (6)	41.5 ± 3.2 B	8.2 ± 2.1 b,c	73.6 ± 4.5 [c]
FSDL	33 (77)	4 (9)	2 (5)	4 (9)	43.5 ± 6.6 B	10.1 ± 2.0 c	66.4 ± 4.5 [d]
OBFL	25 (57)	4 (9)	9 (20)	6 (14)	59.6 ± 5.6 A	2.81 ± 0.8 a	92.7 ± 4.6 [a]
SBMP	37 (76)	8 (16)	1 (2)	3 (6)	43.9 ± 2.5 B	6.9 ± 1.8 b	83.6 ± 4.5 [b]

Abbreviations: A, adhesive fracture mode; A/M, adhesive/mixed fracture mode; C, cohesive fracture mode; PF, premature failures.
[a] *Similar capital (μTBS), lowercase (NL), and superscript letters (DC) are not statistically significant (Tukey test; p<0.05).*

Statistical Analysis

The values originating from the same specimen for μTBS, NL, and ISDC were averaged for statistical purposes. The μTBS, NL and ISDC means for every group were expressed as the average of the seven teeth used per group. Data from μTBS, NL, ISDC, UTS, and DC of the adhesive pellicle were individually analyzed using one-way analysis of variance (ANOVA) and the Tukey *post hoc* test (α=0.05). Data from WS and SL were calculated for each experimental condition, and the data after 28 days were analyzed by one-way ANOVA (adhesive) and the Tukey post hoc test at $\alpha = 0.05$.

RESULTS

Microtensile Bond Strengths

The failure modes of all experimental groups are shown in Table 2. The majority of the specimens (84.4%) presented adhesive/mixed failures. Dentin and resin cohesive failures were observed in 7.0% of the specimens. A number of premature failures (8.6%) were observed.

One-way ANOVA detected significant differences between the μTBS values (Table 2; $p<0.001$). OBFL resulted in the highest mean μTBS (Table 2). All other adhesives had statistically similar mean μTBS ($p>0.05$).

Nanoleakage

Significant differences were detected in the NL values (Table 2; $p<0.0001$). OBFL showed the lowest mean NL (Table 2). SBMP showed intermediate values, but only statistically different from FSDL ($p<0.01$). The mean NL associated with ALB3 was similar to those of SBMP and FSDL ($p>0.05$). Examples of the NL pattern can be seen in Figure 1.

In Situ *Degree of Conversion*

One-way ANOVA detected significant differences in the ISDC means (Table 2; $p<0.00001$). OBFL showed the highest mean ISDC and FSDL showed the lowest (Table 2). FSDL and SBMP showed intermediate values, with the former showing the lower mean ISDC (Table 2).

Ultimate Tensile Strength

Statistically significant differences were detected in the UTS means (Table 3; $p<0.001$). OBFL showed the highest mean UTS (Table 3). All other materials had statistically similar mean UTS ($p>0.05$).

Degree of Conversion

One-way ANOVA detected statistically significant differences (Table 3; $p<0.00001$): OBFL showed the highest mean DC, whereas ALB3 resulted in the lowest (Table 3). FSDL and SBMP showed interme-

Table 3: *Means and Standard Deviation of Ultimate Tensile Strength (UTS), Degree of Conversion of Adhesive Pellicle (DC), Water Sorption (WS), and Solubility (SL) of Each Adhesive System Tested*[a]

Adhesive Systems	UTS (MPa)	DC (%)	WS (μg/mm^3)	SL (μg/mm^3)
ALB3	34.0 ± 5.1 B	65.4 ± 1.9 d	0.14 ± 0.04 [B]	0.05 ± 0.08 [a]
FSDL	34.8 ± 5.3 B	70.7 ± 6.8 c	0.07 ± 0.02 [A]	0.5 ± 0.07 [b]
OBFL	48.8 ± 5.2 A	93.9 ± 3.9 a	0.12 ± 0.02 [A,B]	0.05 ± 0.02 [a]
SBMP	33.3 ± 6.1 B	83.8 ± 4.6 b	0.80 ± 0.22 [C]	0.02 ± 0.03 [a]

[a] *Similar capital (UTS), lowercase (DC), capital superscript (WS), and lowercase superscript letters (SL) are statistically similar (Tukey test; p<0.05).*

diate values, with the latter showing the lowest mean DC (Table 3).

Water Sorption and Solubility

Significant differences were also detected in terms of WS and SL values (Table 3; $p<0.001$ and $p<0.01$, respectively). FSDL and OBFL showed the lowest mean WS; SBMP resulted in the highest mean WS (Table 3; $p<0.001$). FSDL showed an intermediate mean WS, which was similar to that of OBFL (Table 3; $p>0.05$). FSDL resulted in the highest mean SL in comparison with all the other materials (Table 3; $p<0.001$). All other adhesives had statistically similar mean SL ($p>0.05$).

DISCUSSION

The results of this study confirmed that there were several differences between the three-step ER adhesive systems tested. Among the adhesive evaluated, only OBFL and SBMP have been extensively evaluated. When the immediate bond strengths of OBFL and SBMP were compared with each other, controversial results were obtained.[16-23] This lack of consensus among studies can be attributed to methodological differences. A systematic review of bond-strength tests[24] reported that SBMP and OBFL are usually reported as being similar when assessed by shear or microshear bond-strength tests. However, OBFL usually presents significantly higher bond strength values when tested in tensile and microtensile bond-strength tests such as the type observed in the present study.

An earlier study[25] reported that OBFL exhibited a very uniform hybrid layer, whereas the hybrid layer formed with SBMP showed a varying electron density with a less sharply demarcated transition to the unaffected dentin. This observation suggested a less-than-optimal resin infiltration, as has been reported by other authors,[26-29] and this is corroborated by our NL results.

SBMP contains a specific polyalkenoic acid copolymer (PAC) that was primarily added to the composition of the resin-modified glass ionomer Vitrebond (3M ESPE). PAC bonds chemically and spontaneously to hydroxyapatite in glass ionomer materials,[30] which may explain why an ER with PAC showed more enhanced bond strength than a PAC-free adhesive with the same composition.[31] On the other hand, it has been demonstrated that because PAC is a compound with a high molecular weight, it does not dissolve in the adhesive solution, which may lead to phase separation and formation of resin globules within the polymer.[28] In addition, the collagen network can filter it out and the PAC can be deposited as a distinct gel on the exposed collagen network surface.[28,32]

In a more extreme case, the gel can hinder adequate monomer infiltration, and the hybrid layer produced would be constituted of collagen and linear polymer chains, with entrapped residual water and insufficiently removed solvent. The presence of water may reduce the degree of conversion of the material, which is shown by the presence of silver nitrate grains, thus explaining why a higher NL and a lower DC (*in situ* and from the adhesive pellicle) was observed for SBMP when compared with OBFL.

The primer of SBMP contains approximately 50 volume percent (vol%) of water, whereas the OBFL primer contains 23 vol% of water and 29 vol% of ethanol.[25] The high boiling temperature and low vapor pressure of water, when compared with that of ethanol, makes this solvent more difficult to remove in comparison with water-ethanol solutions.[11] The excess water in the adhesive film reduces the degree of conversion of SBMP and diminishes the UTS of the material by preventing molecule approximation during polymerization. A poorly polymerized adhesive is more prone to WS, as could be detected in this study.[33,34]

The adhesives ALB3 and FSDL also had a lower DC than SBMP did, and they did not present such a high WS. Thus, other factors apart from lower DC may be responsible for this finding. SBMP is the only material that contains the hydrophilic monomer HEMA in the bonding resin. HEMA can reduce the vapor pressure of water,[35] leading to solvent retention. Additionally, poly (HEMA) formed after polymerization behaves as a hydrogel, which swells and rapidly takes up water. Water droplets were found to be located adjacent to the adhesive resin-composite interface formed by HEMA-rich adhesives[36,37] The literature has confirmed that SBMP usually showed poor long-term *in vitro* and *in vivo* results when compared with OBFL[22,38] and the clinical results of this material are usually controversial.[3,38]

The bond strength of ALB3 and FSDL was similar to that of SBMP; however, these materials showed the lowest DC (*in situ* or from the adhesive pellicle) and the highest NL values when compared with OBFL and SBMP. This was expected, given that it was recently demonstrated that the DC is negatively correlated with the NL.[39] Conversion of monomer into polymer (percentage of C=C incorporated into polymer chains as C=C) plays an important role in

successful dentin bonding.[40] To some extent it is true that increased permeability of the adhesive layer is observed when there are unreacted monomers within the hybrid layer.[41,42] This results in the formation of a porous hybridoid structure with reduced sealing ability, which is prone to silver nitrate uptake,[43,44] as could be seen in the results of the present study. Unfortunately, the reason why ALB3 and FSDL showed higher amount of NL and lower DC is not clear, and future studies need to be conducted to test these hypothesis.

Earlier studies reported negative solubility values for three-step ER,[45-47] which does not agree with the present findings. Contrary to this study, in which a mixture of primer and bonding resin was used for specimen fabrication, the previous studies only evaluated the bonding resin. The mixture of the primer with the bonding resin affects the mechanical properties of the adhesive interface when compared only with the bonding resin.[48,49]

Materials with a high SL usually present a high level of WS. WS causes polymer swelling, thereby facilitating the elution of unreacted monomers and/or solvents trapped in the polymer network.[40] However, the adhesive system FSDL showed the highest SL in this study without presenting a high WS. This fact along with the low DC of this material suggests that many unbound oligomers and residual monomers were readily leached out without the need for polymer plasticization by water. The high amount of silver nitrate uptake in the adhesive layers produced by FSDL reinforces this hypothesis. Because the manufacturer does not specify the material composition, it is quite difficult to interpret the study findings based on its chemistry. Moreover, as far as we know, no published article with FSDL was found, and future studies need to be conducted to confirm the present results.

The good performance of OBFL in this study is in agreement with the literature findings. OBFL usually showed the best performance in all tests, confirming that it is the gold standard in terms of ER adhesives[2-7] in both laboratory[18,22] and clinical studies.[38,50,51] At least two hypotheses can be used to explain these good results. Sezinando and others[52] speculated that the higher bond-strength durability of this material is due to the presence of glycerol phosphate dimethacrylate, which can interact chemically with hydroxyapatite.[53,54] Additionally, OBFL has a bonding layer with very high filler loading (48 vol%). This filled adhesive, along with the hybrid layer, may also create an artificially elastic cavity wall[55] and act as a potential elastic shock absorber[56] during polymerization of the composite resin. ALB3 also has filler in its composition (>40%), but the benefit of its presence was not confirmed in terms of immediate bond strength and NL into the dentin. Of course, further long-term *in vitro* and clinical studies need to be conducted to prove this hypothesis, mainly because the only clinical study[57] that was found with ALB3 showed very promising results, as was pointed out in the recent systematic review of clinical studies published by Chee and others.[7]

CONCLUSIONS

The evaluation of several bonding (μTBS, NL, and ISDC on dentin) and mechanical (UTS, DC, WS, and SL in water) properties in the short term allowed the adhesives available on the market to be more effectively screened, without requiring long-term studies. OBFL showed the best results in the all properties evaluated, and it can be considered the gold standard in the three-step ER adhesive systems.

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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.

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Departments

Letter to the Editor:

Dear editor,

A colleague of mine sent me a copy of the recent paper in your journal by da Silva et al.[1] In the introduction of the paper it was stated that ". . . during the day ultraviolet radiation makes teeth appear whiter and shinier.". In support of that statement my work was quoted.[2] I understand that presently there is a discussion in the literature on the influence of sunlight UV on tooth appearance. Therefore I wish to state explicitly that in my earlier work[2] neither a reference was made to sunlight, nor to tooth appearance and that therefore the quotation is wrong.

Sincerely yours,

Jaap J. ten Bosch,
Em. Professor in Dental physics,
Univ. of Groningen, the Netherlands
Address; Zuidenveld 30,
NL-9301 JB Roden, the Netherlands

1. da Silva TM, de Oliviera HPM, Severino D, Balducci I, Huhtala MFRL, & Gonçalves SEP (2014) Direct spectrometry: a new alternative for measuring the fluorescence of composite resins and dental tissues *Operative Dentistry* **39(4)** 407-415.

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Author Response:

Dear Editor,

Thank you for notifying and giving us the opportunity to clarify the situation.

After reading what was stated in the paper we wrote related to Dr. Bosch's work, we realized that he is right as to the reference made to sunlight and tooth appearance. The quotation does not belong to his work.

His quotation is related to the first affirmation of the fourth paragraph: "*Dental fluorescence intensity is attributed to the organic components that* . . ." So the reference number of his paper should be "8" and applied to the end of this affirmation above.

We are truly sorry for this and would like to send our apologies to Dr. Bosch.

Best regards,

Tânia Mara da Silva (corresponding author)
Institute of Science and Technology, UNESP- Univ Estadual Paulista
Department of Restorative Dentistry
Avenida Engenheiro Francisco José Longo, 777, Jardim São Dimas, São José dos Campos, SP, ZipCode 12245-000, Brazil.

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Operative Dentistry, Inc. would like to thank our conscientious team of Reviewers for their hard work, tenacity, and dedication in the furthering of operative dentistry around the word. These individuals dedicate innumerable hours in reading, re-reading, and critiquing manuscripts. Submitted articles, good and otherwise, all benefit from these reviewers who help authors present their hard work, as well as verifying that the work we publish is scientifically accurate, clinically relevant, and professionally uplifting. We cannot thank these individuals enough for their contributions, but we do want to publically recognize them.

Be it known here, and throughout the world, that the following named individuals have contributed real and invaluable service to the profession of Dentistry by volunteering their time and talents to the cause of peer-review for Operative Dentistry, Inc. The time of service listed below is for the 2014 publication year.

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Clinical Decision Making on Extensive Molar Restorations

T Laegreid • NR Gjerdet • A Johansson
A-K Johansson

Clinical Relevance

Extensive loss of posterior tooth substance, which traditionally was restored with amalgam or indirect restorations, is more commonly being restored with resin-based composite restorations. The choice between prescribing direct or indirect techniques when restoring extensive posterior defects has challenged clinical decision making.

SUMMARY

Extensive loss of posterior tooth substance, which traditionally was restored with amalgam or indirect restorations, is more commonly being restored with resin-based composite restorations. Using a questionnaire, we aimed to survey dentists' clinical decision making when restoring extensive defects in posterior molar teeth. The questionnaire, which included questions on background information from the dentists, clinical cases with treatment options, and general questions about restoring extensive posterior defects, was sent to 476 dentists. The response rate was 59%. Multiple logistic regressions were used to investigate the different associations. Most of the respondents preferred a direct composite restoration when one cusp was missing, while indirect restorations were most preferred when replacing three or four cusps. Younger dentists and dentists working in the private sector had a greater tendency to choose an indirect technique compared with older colleagues. Generally, the most important influencing factor in clinical decision making was the amount of remaining tooth substance. Factors that appeared to be less important were dental advertisements, use of fluoride, and dietary habits. Female dentists perceived factors such as oral hygiene, patient requests, and economy to be more important than did their male colleagues.

*Torgils Laegreid, DDS, PhD, University of Bergen, Department of Clinical Dentistry–Cariology, Bergen, Norway

Nils Roar Gjerdet, DDS, PhD, University of Bergen, Department of Clinical Dentistry–Biomaterials, Bergen, Norway

Anders Johansson, DDS, PhD, University of Bergen, Department of Clinical Dentistry–Prosthodontics, Bergen, Norway

Ann-Katrin Johansson, DDS, PhD, University of Bergen, Department of Clinical Dentistry–Cariology, Bergen, Norway

*Corresponding author: Postboks 7804, Bergen, N-5020, Norway; e-mail: torgils.lagreid@iko.uib.no

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INTRODUCTION

Clinical decision making is an important component of everyday dentistry, and its outcome depends on a large number of different crucial factors. Improved biological and mechanical properties of composite restorative materials have broadened their indications during the past few decades.[1] Extensive loss of posterior tooth substance, which traditionally was restored with amalgam or indirect restorations, is more commonly being restored with resin-based

composite restorations. In some countries, the use of dental amalgam has been restricted,[2] and direct composite restorations are often considered to be the most viable treatment alternative. These changes have challenged the clinical decision making of dentists and, specifically, the choice between prescribing direct or indirect techniques when restoring extensive posterior defects has been rendered difficult. Suggested treatment options must primarily be based on an individual clinical assessment, but other factors such as patient requests and economy may also contribute to the decision making.[3,4]

While there is little scientific information available concerning the choice of restorative treatment for extensive loss of tooth substance in posterior teeth, more research has been carried out on treatment options for intracoronal Class I and II cavities in posterior teeth.[5-12] These studies focus on the differences and changes in the use of amalgam and composite but generally do not distinguish between the different outline forms and designs of the restorations.

From a clinical decision-making point of view, the choice between direct and indirect restorative techniques has several dimensions. First, the use of direct restoratives is increasingly based on adhesion and the principle of minimally invasive dentistry, and this procedure minimizes the risk of iatrogenic damage to the tooth and surrounding tissues.[13] On the other side, the mechanical strength of direct restoratives can be inferior to that of indirect restoratives in many situations,[14] and this may call into question the use of such materials in extensive posterior defects. Finally, the immediate costs of doing an indirect restoration are higher compared with those of a direct restoration,[15] and in addition, indirect work is more time-consuming.

The available literature is sparse concerning the choice between direct and indirect restorative treatment of extensive defects in posterior teeth. While most studies concentrate on the choice between composite and amalgam, a few studies discuss the direct/indirect approach.[4,8,16-19] A recent report concluded that "there is no high quality evidence that supports or rejects the practice of placing an indirect restoration on a heavily restored vital molar rather than a direct restoration to ensure longer tooth survival."[20] While some authors suggest that the restorative treatment of posterior teeth is moving toward a direct technique,[14] others predict an enhanced future use of indirect restorations in posterior teeth.[8]

The primary aim of this study was to survey Norwegian dentists' clinical decision making when restoring extensive defects in posterior molar teeth. A secondary aim was to evaluate the importance of influencing factors when it comes to the choice of treatment. Finally, the third aim was to study the associations between treatment choice, operator, and patient-related factors.

METHODS AND MATERIALS

Questionnaire

A questionnaire was designed to obtain information from dentists regarding their clinical decision making when restoring extensive posterior defects. The first part included background factors such as sex, age, dental education, workplace, and type of patients attending their clinic.

The second part comprised descriptions of four different clinical cases with details of medical history and clinical examination, and the dentists were asked to rank the three most appropriate treatment options, based on their own clinical judgment. The cases were illustrated by photographs of a lower first molar with one to four missing cusps (Figure 1).

Finally, the third part included questions about different possible factors influencing the treatment planning, frequency of use of the different restorative options, and clinical problems deemed associated with the treatment.

A pilot of the questionnaire was sent to 10 dentists, and final corrections were made after feedback from respondents.

Subjects

The names and addresses of all 768 members of the Bergen branch of the Norwegian Dental Association (Bergen Dental Association) were received from the local membership administrator. Retired dentists, dental students, orthodontists, and oral surgeons were excluded from the study, and the final selection comprised 476 dentists who were included in the survey.

The questionnaires, together with a cover letter and a stamped return envelope, were sent to the recipients by mail. Three weeks after the initial mailing, a reminder announcement was published in the local dental journal. Six weeks after the initial mailing, a reminder letter with a questionnaire was sent to the nonresponders.

To achieve anonymity, each subject in the mailing list was assigned a code. The coded mailing list was

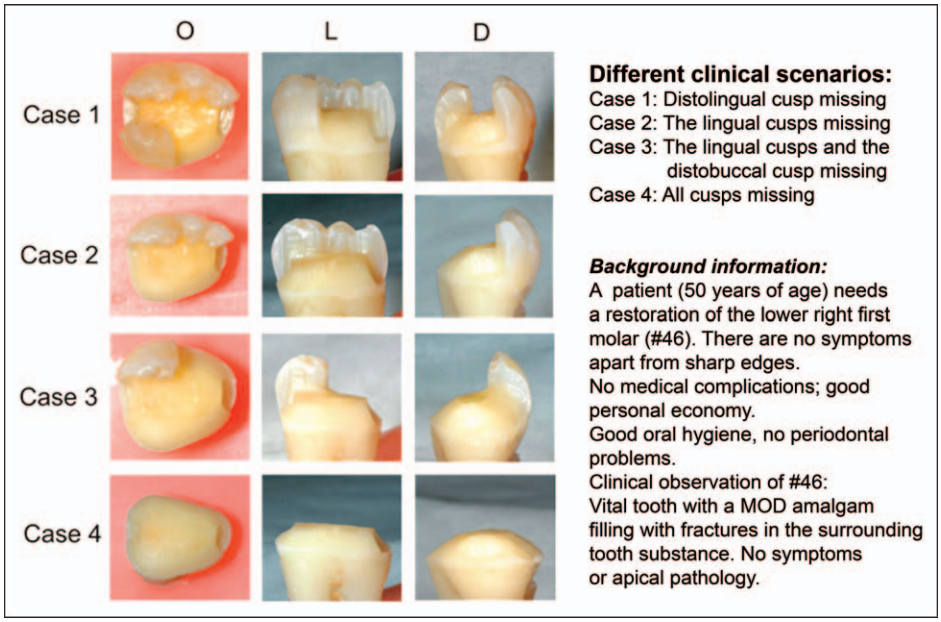

Figure 1. *Four clinical situations for the lower right first molar as presented in the questionnaire, seen from an occlusal (O), lingual (L), and distal (D) view. In the presented situations, the amalgam restoration is removed. An extracted molar was prepared to illustrate the cases. Patient-related information (right) was also given in the questionnaire.*

used to send out the reminders to nonrespondents. It was not possible to identify the respondents from the returned questionnaires without the coded mailing list. The respondents were free to withdraw from the survey at any time.

Privacy Protection

The study was reported to the commission of privacy protection at the Norwegian Social Science Data Services.

Statistical Methods

The chi-square goodness-of-fit test was used to evaluate the representativity of the sample of responding dentists. The treatment choices were dichotomized into direct and indirect treatment alternatives to simplify the statistical calculations. Multiple logistic regressions were used to investigate the different associations presented in this article. p values less than 0.05 were considered statistically significant. All statistical analyses were performed using the PASW Statistics 18.0 software (SPSS, Inc, Somers, NY).

RESULTS

Response and Background Data

Nineteen of the questionnaires were returned because of unknown addresses. Consequently, 457 dentists remained available for the survey. The final response constituted 270 dentists (59%).

The respondents (142 women and 128 men) had a mean age of 46.4 years (range, 25-71 years). Eight respondents reported that they did not work clinically and were therefore excluded from the statistical analyses. The distribution of the respondents according to age, sex, and employment sector is shown in Table 1. Sixty-two percent of the respondents reported that their practice was located in the City of Bergen, which has about 250,000 inhabitants.

Table 1: *Distribution of the Responders according to Sex, Age, and Employment Sector*

Age Group, y	Public Dental Sector[a]		Private Dental Sector		Total
	Women	Men	Women	Men	
<30	7	0	10	6	23
30-39	25	0	25	16	66
40-49	18	2	18	18	56
50-59	11	13	12	38	74
≥60	4	12	5	18	39
Total	65	27	70	96	258[b]

[a] *Included in the category "Public dental sector" are dentists working full-time or part-time in the Public Dental Service, staff at the Dental University Clinic in Bergen, at hospitals, or at other public services in Hordaland County, Norway.*
[b] *Of the 262 dentists available for statistical analysis, three did not report their age and one did not report employment status.*

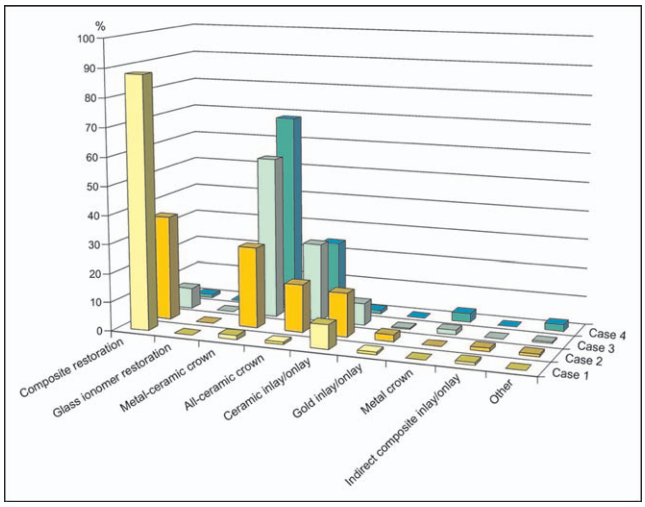

Figure 2. *The proportionate distribution of the respondents' first treatment choice in case 1 (n=266), case 2 (n=267), case 3 (n=266), and case 4 (n=264). The "nonclinical" respondents are included.*

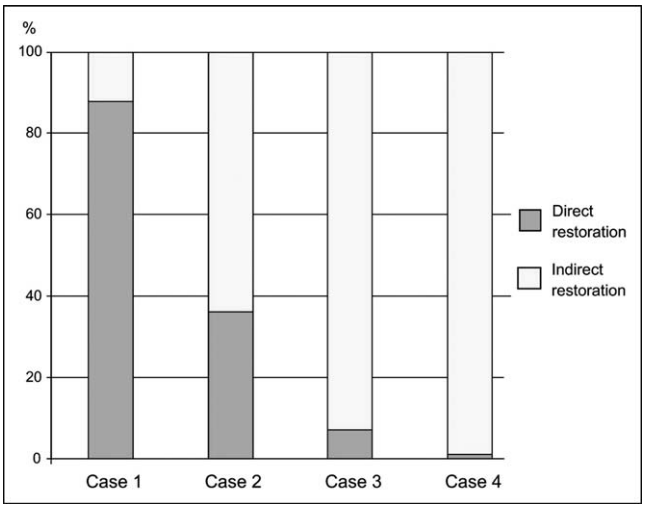

Figure 3. *The proportionate distribution of the respondents' first treatment choice (dichotomized into direct and indirect restorations) in case 1 (n=266), case 2 (n=267), case 3 (n=266), and case 4 (n=264). The "nonclinical" respondents are included.*

The remaining 38% had their practices in surrounding smaller towns or rural areas.

The chi-square goodness-of-fit test indicated that there were no significant differences in the distribution of responding dentists in this survey compared with the distribution of dentists in Norway according to age, sex, and employment sector.

Clinical Cases

Figure 2 shows the distribution of the first treatment choice of the respondents in each of the four cases. In the following statistics, these treatment choices were dichotomized into direct and indirect treatment alternatives (Figure 3). Composite was the only direct material preferred by the respondents when restoring the four cases.

Most of the respondents chose a direct composite restoration for case 1. This is in contrast to cases 3 and 4, in which indirect restorations were the preferred treatment option. A more even distribution between direct and indirect treatment was present in case 2, and this was chosen as the indicator case for further statistical analyses.

Age, Sex, Workplace, and Location of Practice

When including age, sex, workplace, and location of the practice as independent variables in a logistic regression analysis with the treatment choice in case 2 (direct or indirect treatment as a dependent variable), only age and employment status were found to have a significant influence on the restorative decision making (Table 2). A significantly greater proportion of the younger dentists (≤50 years) chose to restore the tooth with an indirect restoration compared with their older colleagues (odds ratio [OR]=1.9, 1.0-3.7). A significantly greater proportion of the dentists working in the private sector chose an indirect restoration compared with those from the public sector (OR=5.7, 3.1-10.6).

Use of Restorative Materials in General

The frequency of use of different restorative materials in extensive posterior defects in general, reported by the respondents, is shown in Table 3. Direct composite restorations and metal-ceramic crowns are the most reported treatment alternatives. Most of the respondents reported that they, to a slight extent or seldom, used restorative materials

Table 2: *Multiple Logistic Regression Analysis with the Treatment Choices Made by the Respondents in Case 2 as a Dependent Variable (Direct Technique=0; Indirect Technique=1) and the Dentist-Related Factors Sex, Age, Employment Status, and Practice Area as Independent Variables*[a]

Independent Variable	β	*p* Value	OR	95% CI for OR
Sex (female)	0.2	0.519	1.3	0.6-2.5
Age (≥50 years)	0.7	0.041	1.9	1.0-3.7
Employment status (private sector)	1.7	<0.001	5.7	3.1-10.6
Practice area (urban)	0.5	0.071	1.7	1.0-3.1

Abbreviations: CI, confidence interval; OR, odds ratio.
[a] *Nagelkerke* R^2=0.23.

Table 3: *Distribution of Answers to the Question, "How Often Do You Use the Listed Treatment Options in Your Clinical Work when Restoring Molars with Extensive Defects?"*

	n	Always (%)	Often (%)	Occasionally (%)	Little (%)	Seldom or Never (%)
Glass ionomer	256	0.4	4.7	22.3	25.0	47.7
Composite	260	1.9	83.5	10.0	3.8	0.8
Composite inlay/onlay	255	0.0	1.6	10.2	23.1	65.1
Gold inlay/onlay	256	0.0	1.6	9.8	28.5	60.2
Ceramic inlay/onlay	256	0.0	6.3	20.3	32.8	40.6
All-ceramic crown	260	0.4	32.3	36.9	22.3	8.1
Metal-ceramic crown	260	1.2	77.7	17.7	3.1	0.4
Metal crown	259	0.0	4.2	22.8	29.0	44.0

such as glass ionomer cement, gold/composite/ceramic inlays or onlays, and full metal crowns.

Multiple logistic regression (Table 4) showed that dentists working in the public sector used significantly more glass ionomer cement than those in private practice (OR=14.8, 2.3-93.1), while private dentists used significantly more ceramic (OR=3.3, 1.6-6.5) and metal-ceramic crowns (OR=6.7, 3.2-14.1). Male dentists used significantly more ceramic inlays or onlays compared with female dentists (OR=3.9, 1.0-14.7). Dentists working in urban areas used significantly more ceramic crowns than did dentists in rural areas (OR=2.6, 1.4-5.0).

Influencing Factors

The factors reported to influence the choice of restorative material in extensive posterior defects are shown in Table 5.

Generally, the most important factor reported was the amount of remaining tooth substance. Other important factors were patient request, presence of parafunctional oral habits, caries activity, and lectures. Factors that were less important for the respondents were advertisements, use of fluoride, and dietary habits.

Multiple logistic regression (Table 6) after dichotomizing the categories into "very much influence" and "much, neither much or little, little and no influence," showed that female dentists perceived factors such as oral hygiene (OR=3.5, 1.3-9.3), patient requests (OR=2.0, 1.0-3.8), and patient's economy (OR=2.2, 1.1-4.4) to be significantly more important than their male colleagues did.

A significantly greater proportion of dentists aged 50 years or younger reported that factors such as remaining tooth substance (OR=1.9, 1.1-3.3) and moisture control (OR=2.6, 1.3-5.1) were more important, compared with their older colleagues, while scientific literature was more important (OR=2.3, 1.0-5.3) for the older dentists (Table 6).

A significantly greater proportion of dentists working in urban areas reported a very important influence of factors such as secretion of saliva (OR=2.6, 1.1-6.3) and moisture control (OR=2.0, 1.1-3.7) compared with their colleagues from more rural areas.

DISCUSSION

The dental health care system in Norway is organized in two different sectors: a public dental health care sector and a private sector. The public

Table 4: *Significant Findings in the Multiple Logistic Regression Analyses with the Frequency of Use of Different Materials as a Dichotomized Dependent Variable* (Treatment Choice: Always or Often=1; Sometimes, Little, Seldom, or Never=0) and the Dentist-Related Factors Sex, Age, Employment Status, and Practice Area as Independent Variables[a]*

Independent Variable	Treatment Choice*	β	*p* Value	OR	95% CI for OR
Employment status (public sector)	Glass ionomer cement	2.7	0.004	14.8	2.3-93.1
Employment status (private sector)	Ceramic crown	1.2	0.001	3.3	1.6-6.5
	Metal-ceramic crown	1.9	<0.001	6.7	3.2-14.1
Sex (male)	Ceramic inlay/onlay	1.4	0.044	3.9	1.0-14.7
Practice area (urban)	Ceramic crown	1.0	0.002	2.6	1.4-5.0

Abbreviations: CI, confidence interval; OR, odds ratio.
[a] *The nonsignificant findings are not shown.*

Table 5: *Distribution of Answers when the Respondents Were Asked, "How Do You Feel the Listed Factors Influence Your Treatment Choice when Restoring Molars with Extensive Defects?"*

	n	Very Much (%)	Much (%)	Neither Much nor Little (%)	Little (%)	No Influence (%)
Acquirement factors						
Advertisements	259	0	3.1	24.3	51.7	20.8
Scientific literature	259	13.1	49.4	29.7	7.7	0
Courses/lectures	258	20.9	58.9	17.8	2.3	0
Patient-related factors						
Age of the patient	259	7.3	49.0	30.5	12.4	0.8
Oral hygiene	259	13.1	61.4	22.0	3.5	0
Caries activity	260	21.9	62.3	15.0	0.8	0
Secretion of saliva	259	13.9	47.5	31.7	6.2	0.8
Diet	258	3.5	22.1	56.6	14.3	3.5
Use of fluoride	259	2.7	22.4	52.9	17.4	4.6
Patient requests	260	28.8	56.5	13.5	1.2	0
Patient economy	259	25.5	51.7	19.7	2.7	0.4
Tooth-related factors						
Occlusion	260	16.2	54.2	23.8	5.4	0.4
Remaining tooth substance	259	46.3	51.7	1.9	0	0
Available cervical enamel	259	15.8	46.7	29.0	6.9	1.5
Parafunctional habits	259	23.9	59.5	14.7	1.9	0
Moisture control	260	28.1	49.2	18.5	3.8	0.4

dental health system provides free care for all children aged 0-20 years, all mentally and physically handicapped people, and institutionalized and elderly people. All other people have to pay the costs for dental care themselves. There are almost 5000 dentists in Norway, and that represents about 1000 inhabitants per dentist.

The target group for this questionnaire survey was dentists providing restorative treatment as a main activity in their clinical practice. Therefore, retired dentists, dental students, orthodontists, oral surgeons, and dentists reporting that they did not work clinically were excluded from the study. The study population was considered representative for Norwegian dentists in general, based on statistical comparison with available official statistics.[21] Very limited information could be retrieved about the nonresponders, so a meaningful nonresponse analysis could not be performed.

The questionnaire used in the present study is based on the Paper Patient Cases method, which has been found to be useful in studies dealing with

Table 6: *Statistically Significant Findings of Multiple Logistic Regression Analyses with the Influence of Different Factors to the Treatment Choice as a Dichotomized Dependent Variable* (Influencing Factor: Much, Neither Much nor Little, Little or No=0; Very Much Influence=1) and the Dentist-Related Factors Sex, Age, and Practice Area as Independent Variables*[a]

Independent Variable	Influencing Factor*	β	*p* Value	OR	95% CI for OR
Sex (female)	Oral hygiene	1.3	0.013	3.5	1.3-9.3
	Patient request	0.7	0.040	2.0	1.0-3.8
	Patient economy	0.8	0.029	2.2	1.1-4.4
Age (>50 years)	Scientific literature	0.8	0.045	2.3	1.0-5.3
Age (≤50 years)	Remaining tooth substance	0.6	0.031	1.9	1.1-3.3
	Moisture control	0.9	0.007	2.6	1.3-5.1
Practice area (urban)	Secretion of saliva	1.0	0.034	2.6	1.1-6.3
	Moisture control	0.7	0.032	2.0	1.1-3.7

Abbreviations: CI, confidence interval; OR, odds ratio.
[a] *No significant differences were found according to employment status.*

clinical decision making in restorative dentistry.[22] Realistic and illustrative clinical cases with background information were presented to a large number of dentists, who were then asked questions about their educational and professional background and their clinical decision making.

The treatment options available for the respondents in this study could all be tentatively viable options as restorative materials and techniques for posterior teeth.[14] To simplify statistical analyses, the options were dichotomized into direct and indirect restoratives. Dental amalgam was left out because its use is forbidden by law in Norway.

The results from the present study indicate, in general, good agreement in clinical decision making among the respondents when restoring a deficient molar lacking one, three, or four cusps. The borderline between direct and indirect technique selection seems to be concentrated on restorations involving two cusps. For that reason, case 2 was considered to be the most interesting clinical situation for studying the decision making among dentists and can be considered as an indicator case in the analysis.

As mentioned before, there is a lack of scientific information concerning the choice of restorative treatment for extensive loss of tooth substance in posterior teeth. In a Kuwaiti questionnaire survey from 2010,[8] the authors found that fewer than 20% of the respondents would have preferred a direct restoration for the retreatment of a failed molar restoration involving two cusps. Although the respondents could have chosen between both amalgam and composite as direct alternatives, this proportion is much lower than the results of the present study, which shows that 36% of the respondents would have chosen a direct composite restoration in a comparable clinical situation. Another finding from the Kuwaiti study mentioned above was that male dentists, older dentists, and dentists working in the public sector each had a greater tendency to place indirect restorations when the molar needed a restoration that involved two cusps. These results contrast with our results, showing that a greater proportion of younger dentists and dentists working in the private sector would prefer indirect restorations compared with their older colleagues. The fact that older dentists use direct composite more frequently in extensive molar defects has also been found in another study.[6]

The tendency among dentists in the public sector to use a direct technique in our study may be explained by the age group of patients. The dental care of patients aged up to 20 years is a public responsibility in Norway, and this is a major patient group in the Norwegian Public Dental Service. The use of indirect restorations in children and adolescent is not very common and is often complicated and involves higher direct costs. The same differences between private and public dentists' treatment choices, as found in this study, were also reported in a Finnish study from 2002.[18] In Kuwait, dental treatment is free of charge in public clinics,[8] and this lack of financial constraint can be an explanatory factor for the increased use of indirect alternatives among public sector dentists. In addition, public clinics in Kuwait do not only treat children but also have a substantially greater proportion of adult patients compared with Norway, which, in turn, may explain the increased use of indirect restorations (Professor R. Omar, Faculty of Dentistry, Kuwait University, Kuwait, personal communication).

Patient-related factors such as preference and costs were also considered important by the respondents in this study. This indicates that the patient request has an influence on the treatment, which is in accordance with some but not with other studies.[16,17,19]

Another finding in the present study is that female dentists consider factors such as patient requests and economy to be more important than do their male colleagues in clinical decision making. This is comparable with the results of a 1996 study[3] in which it was reported that female dentists sought their patient's opinion more frequently than male dentists did. It was suggested that female dentists had a greater interest in esthetics than did their male counterparts.

In the present study, the amount of remaining tooth substance was clearly considered the most important factor influencing the treatment choice. The perceived importance of such a technical tooth-related factor has been reported in other studies[16,19] and is confirmed by the present study.

Resin-based composite has replaced amalgam as the primary direct restorative material in posterior teeth in countries such as Sweden and Norway, since amalgam is no longer an available option. An issue is to what extent the governmental restrictions on the use of dental amalgam have influenced the clinical decision making among dentists. In Norway, the use of amalgam was banned in 2008. Nevertheless, the decrease in use of amalgam was noticeable begin-

ning in the 1990s,[11] and amalgam constituted fewer than 10% of the restorations in 2002.[23] However, in a study carried out between 2001 and 2004, most of the participating dentists still preferred amalgam in more challenging restorations with respect to caries activity, lesion depth, and tooth type.[24] This indicates that many Norwegian dentists were forced to change their treatment strategies when restoring extensive posterior defects after 2008.

As the present study indicates a shift toward the use of direct resin-based composite when restoring extensive posterior defects in Norway, an important question concerning longevity and socioeconomic effects arises. Comparative clinical studies of extensive posterior restorations are few, but interesting. In a prospective longitudinal evaluation of extensive restorations in permanent teeth,[25] it was found that the median survival times were 12.8 years for amalgam, 7.8 years for resin-based restorations, and 14.6 years for crowns. The authors concluded that "extensive amalgam restorations but not composite resin restorations can be used as appropriate alternatives to crowns." Other studies have reported the same conclusion as the above-mentioned study.[26,27] On the other side, a recently published retrospective study[28] showed better survival of composite restorations compared with amalgam after 12 years.

In a review from 2004,[14] the mean annual failure rate (AFR) for different indirect posterior restorations was calculated: indirect composite inlays and onlays (2.9%), ceramic inlays and onlays (1.9%), computerized designed and manufactured (CAD/CAM) ceramic inlays and onlays (1.7%), and cast gold inlays and onlays (1.4%). Another review from 2007 estimated the mean AFR for crowns, which varied from 1.0% to 3.4%, depending on what type of material was used.[29] The mean AFR calculated from these long-term studies involving indirect restorations is lower than the failure rate calculated for extensive direct composite restorations (4.2%).[30]

The choice between direct techniques, in which the restoration is inserted and set directly into the tooth, and indirect techniques, in which the restoration is produced outside the mouth, has a great impact on the initial cost of the treatment. An indirect technique may enhance the price of the restoration multifold.[31,32] For the patient and eventually a third party such as insurance companies and governmental institutions, it is also important to have information about the cost-effectiveness in the long term. Such long-term costs of dental restorations are dependent on both the initial price and the expected longevity of the restoration.

Historically, amalgam has been regarded as the most cost-effective treatment in posterior teeth compared with other direct and indirect restoratives.[31-37] Some studies have also concluded that it is not cost beneficial to replace failed extensive amalgam restorations with indirect alternatives.[32,37,38] Internal differences in long-term costs between the direct alternatives amalgam, resin-based composite, and glass ionomers are also reported, with amalgam as the least expensive.[36]

None of these studies include extensive cusp-covering resin-based restorations. For that reason, the information about the cost-effectiveness of extensive direct posterior composite restorations in relation to indirect prosthetic alternatives is sparse.

The reparability of resin-based composite restorations is documented,[39,40] and such repair procedures are less expensive than total replacements, and they thus reduce the long-term costs. This may contribute to an opinion that extensive direct posterior composite restorations are more cost-effective than their indirect counterparts, although the latter have better survival rates.

CONCLUSION

The results of this study indicate that the choice of restorative technique in posterior teeth (direct vs indirect) is influenced by a range of factors related both to the patient and the operator. In addition, they reveal a variation in clinical decision making among dentists.

Since clinical decision making has an influence on oral health care, both economically and biologically, further research on this topic is needed.

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.

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Film Thickness of Dentin Desensitizing Agents on Full Crown Preparations: Influence of Product and Gravity

RD Bannister • RV Roudsari • JD Satterthwaite

Clinical Relevance

Dentin sensitivity is considered a side effect of crown preparation. It has been suggested that dentin desensitizing agents can help to reduce post-cementation sensitivity; however, it is not known how these agents affect the morphology of crown preparation.

SUMMARY

Objective: To determine the thickness of resin layer formed when dentin desensitizing agents are applied to teeth prepared for full crown restorations.

Design: *In vitro* measurements of resin layer thickness.

Methods and Materials: Forty caries-free human premolar teeth were prepared as for a full metal-ceramic crown restoration with a retention groove placed mesiobuccally. Stratified allocation created five groups of eight teeth, which were treated with various desensitizing agents. Four teeth within each group were treated upright, and four were treated while inverted, resulting in a total of 10 experimental groups. Teeth were sectioned and resin layer thickness measured under an environmental scanning electron microscope at certain sites across the section.

Results: Analysis was carried out using three-way analysis of variance. On flat tooth surfaces, light-cured resins (Prime & Bond and Seal & Protect) formed layers of 16.2 ± 8.9 μm and 23.4 ± 10.6 μm, respectively. More concave sites had significantly thicker layers ($p<0.05$) than flat or convex sites. At the internal shoulder angle, mean thicknesses were 84.1 ± 27.8 μm and 104.3 ± 56.6 μm, respectively. At the retention groove, figures were 86.6 ± 3.13 μm and 136.2 ± 72.0 μm. Differences between these two resins were not significant ($p>0.05$). Light-cured resins formed significantly thicker layers on inverted samples at the occlusal indentation only ($p=0.004$), with a mean of 66.9 ± 21.6 μm; upright samples had a mean of 36.6 ±

Richard D Bannister, BDS, MSc, Swan Dental Practice, Harrogate, United Kingdom

*Reza Vahid Roudsari, DDS, MSc, PGDip, MFDS, The University of Manchester, School of Dentistry, Manchester, United Kingdom

Julian D Satterthwaite, BDS, MSc, PhD, FDS, MFDSRCS, FDS(Rest Dent), FHEA, The University of Manchester, School of Dentistry, Manchester, United Kingdom

*Corresponding author: Higher Cambridge Street, Manchester, M15 6FH, UK; e-mail: reza@manchester.ac.uk

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12.4 μm. Self-activating resins (Pain-Free Desensitizer, Viva Sens, and Gluma Desensitizer) formed no consistent layers.

Conclusion: Within the limitations of this *in vitro* study, light-cured resins consistently pooled in convex areas of crown preparations. A great portion of retention grooves can potentially become occluded by resin. The self-activating products tested did not form significant layers.

INTRODUCTION

Sensitivity after cementation of crowns occurs in approximately 10% of patients.[1] Therefore, exposing vital dentin, particularly with crown preparations, represents a challenge to the pulp, which may over time result in loss of vitality and endodontic pathology. Brännström and others[2] noted that some patients are, for unknown reasons, less able or unable to form secondary dentin. Such patients may well be at a greater risk of sensitivity or bacterial contamination after restorative procedures. Postcementation studies suggest that about 10% of patients receiving crowns or bridges will report sensitivity in the following weeks and months.[1,3,4] Furthermore, Valderhaug and others[5] found that approximately 10% of crowned teeth become nonvital after 10 years. Thus, reducing the insult to the pulp is fundamental to restorative dentistry. To reduce the risk of postoperative sensitivity and irritation to the pulp, several methods have been advocated including 1) utilizing the sealing/soothing properties of temporary or permanent luting cements, 2) using antiseptic agents, 3) coating the preparation with fluoride or other varnishes, 4) sealing the preparation with a dentin bonding agent (DBA), 5) applying one of the two groups of chemicals specifically marketed as dentin desensitizing agents (DDAs) or desensitizers, 6) performing laser or ozone therapy, and 7) performing iontophoresis.

Scanning electron microscope (SEM) studies[6–9] have related sensitivity in teeth to patency of dentinal tubules, that is, the fluid is able to flow outward. The greater the number and diameter of exposed tubules, the greater the permeability of the dentin and the likelihood of sensitivity. Dentin permeability and sensitivity are both reduced when the dentin tubules are occluded.[10]

The primary use of DDAs is to control hypersensitivity associated with exposed root dentin and cervical wear lesions. DDAs can be divided into two groups: the first group includes resins that normally require etching or conditioning with or without light-curing, forming a seal over the dentinal surface; and the second group requires the rubbing action of a chemical against the tooth with a brush or cotton pellet, precipitating various proteins or crystals into and around the dentinal tubules. The latter are not light-cured.

Studies on film thickness of DDAs are few. It is not known whether different types of modern DDAs form a layer across crown preparations, whether this is affected by gravity, or whether the layer is of sufficient thickness to cause a clinical or technical challenge. Hypothetically, this film thickness can reduce the available restorative space and result in either esthetically compromised or overcontoured restorations. It may also have a negative effect on the auxiliary retentive features by occluding them.

The aim of this study was to measure the film thickness of the DDAs over features of full crown preparations, such as chamfer and shoulder margins and retention grooves. The objectives were to 1) assess the effect of the gravity over this film thickness and 2) determine if specific crown preparation features are more prone to be affected by the DDAs. The null hypothesis was that the film thickness of the DDAs makes no difference to the morphology of full crown preparations.

METHODS AND MATERIALS

Tooth Selection

Relevant ethical approval was obtained. Caries-free, human premolar teeth, extracted for orthodontic reasons, were collected. Teeth with unusually shaped crowns were rejected. Forty-five teeth were selected. After storage in distilled water and thymol, the teeth were individually mounted in dental plaster so they could be handled without touching the coronal surfaces and located securely during preparation. When mounting, care was taken to ensure that the long axis of the teeth was perpendicular to the base of the plaster mount and the crowns of the teeth were exposed 1 mm coronal to the cementoenamel junction.

Material Selection

Before tooth preparation, five DDAs were obtained to represent a range of chemistries and modes of action. These are presented in Table 1.

Tooth Preparation

Tooth preparation was standardized using a laboratory clamp and a straight handpiece, aligned

Table 1: *Products Used*

Trade Name	Key Specifications	Directions to Use
Seal & Protect	Dentsply DeTrey, Konstanz, Germany This product is the only light-cured dentin desensitizing agent available that is claimed to infiltrate and coat the dentin in the same manner as a dentin bonding agent.	Clean dentin with rubber cup and pumice Wash and dry; do not desiccate Apply liquid liberally Leave undisturbed for 20 s Remove excess with air Light-cure for 10 s Apply second layer Remove excess with air Light-cure for 10 s Remove oxygen-inhibited layer with a cotton pellet
Prime & Bond NT	Dentsply DeTrey, Konstanz, Germany This product is a single-bottle, fifth-generation dentin bonding agent. Though not a desensitizer as such, it can theoretically function as one by blocking dentinal tubules.	Clean dentin with rubber cup and pumice Wash and dry Total etch of enamel and dentin, starting with enamel; no more than 15 s for dentin Wash and dry; do not desiccate Apply liquid liberally Leave undisturbed for 20 s Remove excess with air Light-cure for 10 s Remove oxygen-inhibited layer with a cotton pellet
Pain-Free	Parkell, Edgewood, NY, USA With this product, monomers are claimed to flow into dentinal tubules; calcium within the collagen reacts with the oxalic acid to form calcium oxalate crystals, cross-linked to the tooth structure by a resin network.	Clean tooth surface with moist cotton wool Dry surface with a fresh cotton roll Mix one drop of each of the two components Apply with a cotton pellet for 20-30 s, using a rubbing/pumping motion to encourage the liquid to penetrate the surface
Viva Sens	Ivoclar Vivadent, Schaan, Liechtenstein This material claims to act via infiltration of the monomer and formation of calcium fluoride ions and proteins in the dentinal tubules. Potassium fluoride may have a nerve-inhibiting effect.	Use precoated brush to mix liquid well Clean tooth surface thoroughly Dry with gentle air Gently rub liquid onto the tooth for 10 s Disperse and dry gently with air for 10 s Do not rinse, eat, drink, or brush for 30 min afterward
Gluma	Gluma, Heraeus Kulzer, Hanau, Germany Gluma is claimed to cause the precipitation and coagulation of plasma proteins, reducing dentin permeability and occluding peripheral tubules. It also has an antibacterial effect.	Clean tooth surface and rinse Apply a small amount of liquid with pellets or brushes Leave for 30-60 s Dry carefully until fluid film has dispersed Rinse thoroughly

vertically using a spirit-level (Figure 1). A flat, immobile stage was created such that the tooth could be moved around the cutting bur. Height was adjusted on the upright of the clamp for each tooth such that the finish line would be level around its circumference. Each tooth was prepared for a full-coverage metal-ceramic crown. A 1.5-mm shoulder was cut on the buccal surface and a 0.5-mm chamfer on the lingual surface. A retention groove was placed in the mesiobuccal aspect of each tooth, measuring 1.0-mm deep at its gingival floor. The occlusal reduction was aided by guide grooves to represent 1.5-mm of clearance.

Bulk reduction was carried out using the straight handpiece set to a cutting speed of 40,000 revolutions/min and a flat-ended, tapered diamond bur (#KTSM0043, Skillbond, High Wycombe, UK) that was cooled with a constant stream of water from a handheld syringe. Once the preparation form had been established, fine finishing was carried out by hand with an air-turbine handpiece (SUPERtorque 660, KaVo, Charlotte, NC, USA) and the aid of binocular loupes at 2.5× magnification (Micro 250N, SurgiTel, Ann Arbor, MI, USA). Round (Hi-Di #637, ISO 198-020, Dentsply, Surrey, UK) and flat-ended (Hi-Di #557, ISO 173-013, Dentsply) tapered diamond burs were used of coarse and fine grain. All procedures were undertaken by the same operator (RDB).

Forty-five teeth were selected and prepared as described previously. One tooth was set aside as the control. Four teeth were set aside to run two pilot studies to calibrate the ESEM and choose the best dye material. The remaining teeth were divided into two groups according to whether they appeared larger or smaller than average. Five groups of eight teeth were then created, ensuring an equal balance of large and small teeth across each test group. Each

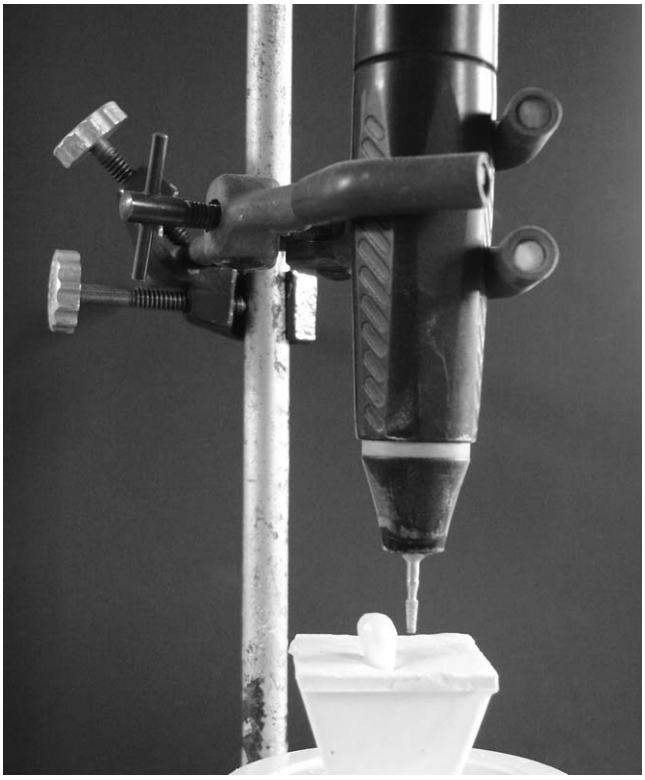

Figure 1. *Straight handpiece mounted vertically in laboratory clamp.*

group would be treated with a particular DDA; groups were named accordingly: PB (Prime & Bond), SP (Seal & Protect), PF (Pain-Free), VS (Viva Sens) and G (Gluma).

To test the effect of the gravity on the flow of each solution, half of the samples in each group (with an equal balance of larger and smaller teeth) were mounted upside-down, resulting in four teeth in each test group. This was to mimic the variation of tooth orientation that might be encountered clinically. Manufacturers' instructions were followed to apply the DDAs (Table 1). One layer of each material was applied in each group except for the SP (for which two layers were needed according to the instructions). Samples from the PB and SP groups had the oxygen inhibition layer gently removed with a damp cotton pellet. Samples from PF, VS, and G groups were left for 30 minutes (per the instructions for VS) before sectioning.

The teeth were sectioned buccolingually in half using a diamond disc (#SCSMO016 Intensiv, Skillbond), which was positioned in the clamped straight handpiece and water-cooled. Holding the tooth by the root so as to not contaminate the coronal prepared area, the cut surface of each section was then polished to a fine grit with abrasive discs (Soflex, 3M ESPE, St Paul, MN) used in sequence with water. Each section was mounted by the root on a small ball of adhesive putty (Blu-Tack, Bostik-Findley, Paris-La Défense, France) such that it could be transported without fear of damage to the prepared surfaces. In addition, midcoronal sections were prepared from selected slices to allow examination of the retention groove. Specimens were then mounted on a specimen plate for examination under a Field-Emission Gun Environmental Scanning Electron Microscope (FEG-ESEM XL30, Philips, Eindhoven, Netherlands) and a voltage of 10 kV. To avoid operator bias, the specimens were coded using a random number generator (SPSS software version 13.0, IBM Corporation, Armonk, NY) prior to being examined under the microscope.

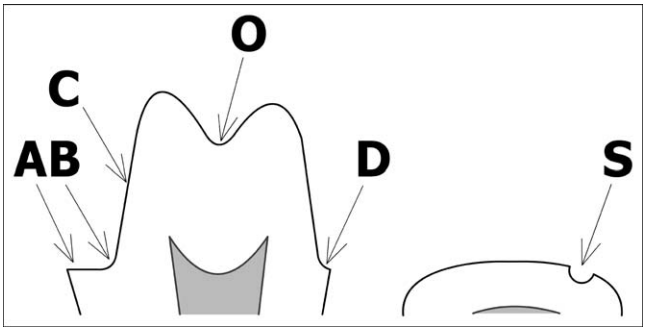

Figure 2. *Measuring positions along tooth section. (A): Shoulder edge. (B): Internal shoulder angle. (C): Midpoint of buccal wall. (D): Internal chamfer angle. Abbreviations: O, occlusal indent; S, deepest part of retention groove.*

Measurements in micrometers (μm) were taken at five points along the section of the tooth (Figure 2) and for slices containing the retention groove. The maximum thickness found at each site was recorded.

RESULTS

Group PB: Prime & Bond

A measurable layer formed at most sites with this material (Table 2). As suggested by the ESEM images, PB had a greater tendency for pooling in concave areas of the preparation, regularly reaching thicknesses of 50+ μm in these areas (Figure 3). The internal shoulder angle (point B) and retention groove (point S) showed the most pooling. Flatter areas seldom exceeded 20 μm.

There was a trend for thicker resin layers when the sample was prepared upside-down. One-way analysis of variance (ANOVA) revealed a significant difference at three sites: the buccal wall (p=0.038), the internal shoulder angle (p=0.05), and the occlusal indent (p=0.003).

Table 2: *Results for Prime & Bond (μm)*

Measuring Site	N	Minimum	Maximum	Mean	SD
Shoulder edge	8	0.0	16.3	8.26	4.69
Internal shoulder angle	8	50.8	121	84.13	27.75
Buccal wall	8	1.9	30.1	16.24	8.85
Internal chamfer angle	8	11.8	96.5	64.36	29.08
Occlusal indent	8	25.3	101	57.79	29.85
Retention groove	3	83.2	89.4	86.57	3.13

Table 3: *Results for Seal & Protect (μm)*

Measuring Site	N	Minimum	Maximum	Mean	SD
Shoulder edge	8	0.0	21.0	4.95	8.35
Internal shoulder angle	8	26.8	199	104.28	56.57
Buccal wall	8	10.3	39.5	23.36	10.55
Internal chamfer angle	8	39.3	339	92.06	100.59
Occlusal indent	8	21.3	57.5	45.73	13.09
Retention groove	3	88.6	219.0	136.20	71.97

Group SP: Seal & Protect

A layer of resin was seen at most sites (Table 3); at times this was visible to the naked eye. SP was also seen to accumulate at concave areas of the preparation while forming thinner layers on flat surfaces, approximately ≤25 μm (Figure 4).

Extreme values were obtained from one of the upright samples (#302), which reached ≥200 μm at three sites (B, D, and S). This may be why values generally dropped for the inverted group. Nonetheless, one-way ANOVA revealed no significant differences between the sites. If sample #302 is left out of the calculations, a significant difference is found only at site D (p=0.009). In this case the data skewed in the opposite direction because the four inverted samples all had higher values than the three upright samples.

Group PF: Pain-Free

Minimal surface coverage was seen; however, a degree of infiltration into dentin was apparent (Figure 5). In most cases, no surface layer was observed (Table 4). The greatest thickness seen with this product was 22.1 μm, and all mean values by site were <3 μm. Significance testing is not appropriate for such low numbers; calculating the median was more relevant, and this was 0 μm for all sites.

Group VS: Viva Sens

No consistent surface layer was observed (Table 5) for the VS group. The highest recorded value was 32.6 μm. Although a value of 27 μm was recorded in a retention groove, the other two readings were zero. Despite this, all mean values for this product were <10 μm, and the median value was 0 μm. No statistical analyses were appropriate.

Group G: GLUMA

Minimal to no coverage was seen with GLUMA (Table 6). The highest recorded value was 57.1 μm. No mean was greater than 8 μm, and the median was 0 μm at all sites. No statistical analyses were used.

Univariate Analysis of the Data Between the Groups

The measurements were also subjected to three-way ANOVA analysis followed by Tukey *post hoc* test based on the type of DDA used, orientation of the mounting, and position where the measurement was taken on the preparation. ANOVA confirmed that

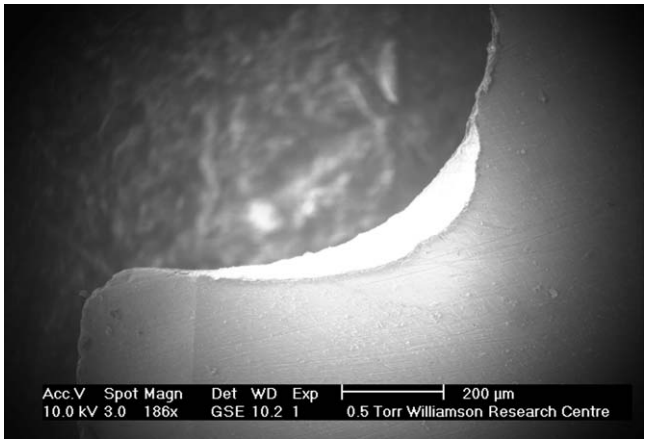

Figure 3. *Point B: a clear pooling effect is seen in the Prime & Bond group. Thickness = 82 μm.*

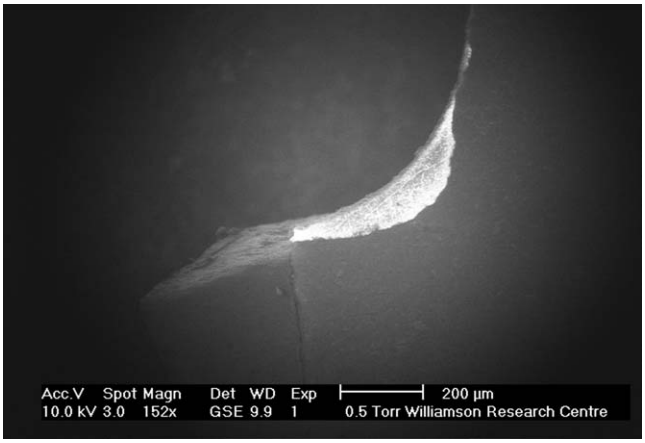

Figure 4. *Point B: 108 μm in the Seal & Protect group.*

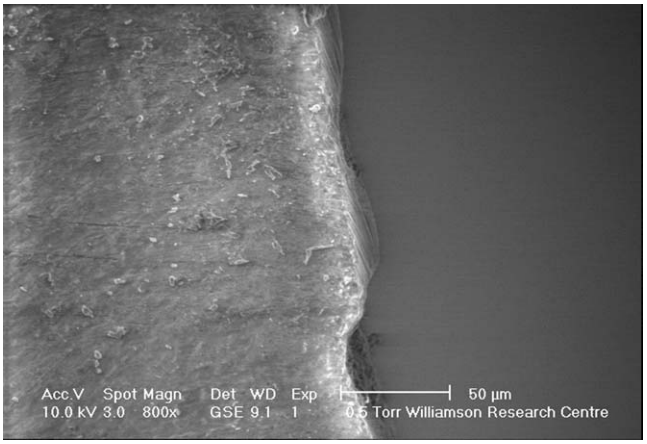

Figure 5. *Point C: 0 µm in the Pain-Free group. Note that the outer tooth layer appears to be infiltrated with resin.*

there were significant differences based on the type of DDA used ($p<0.001$) and the position on the preparation ($p<0.001$); however, orientation of the tooth had no significant influence ($p=0.538$). It was also confirmed that the combination of type of DDA and preparation position had significantly different effects on the results ($p<0.001$).

The *post hoc* Tukey test confirmed that the DDAs could be subdivided into two subsets: subset 1, consisting of PF, G, and VS, and subset 2, consisting of PB and SP. The two subsets were significantly different ($p<0.001$). Subset 2 showed significantly higher readings; however, there was no significant difference within each subset ($p=1.000$ and $p=0.322$, respectively).

The Tukey test based on the preparation position subdivided the data set into three subsets: subset 1, consisting of positions A, C, and O; subset 2, consisting of positions O, D, and B; and subset 3, consisting of positions D, B, and S. There was no significant difference within the subsets ($p=0.064$, $p=0.086$, and $p=0.205$, respectively), but the three subsets were significantly different ($p<0.001$) compared with each other (Table 7).

Table 4: *Results for Pain-Free (µm)*

Measuring Site	N	Minimum	Maximum	Mean	SD
Shoulder edge	8	0.0	12.4	1.55	4.38
Internal shoulder angle	8	0.0	18.0	2.25	6.36
Buccal wall	8	0.0	22.1	2.76	7.81
Internal chamfer angle	8	0.0	0.0	0.0	0.0
Occlusal indent	8	0.0	5.1	0.64	1.80
Retention groove	3	0.0	8.1	2.70	4.68

Table 5: *Results for Viva Sens (µm)*

Measuring Site	N	Minimum	Maximum	Mean	SD
Shoulder edge	8	0.0	0.0	0.0	0.0
Internal shoulder angle	8	0.0	8.2	1.02	2.90
Buccal wall	8	0.0	14.0	1.75	4.95
Internal chamfer angle	8	0.0	32.6	5.09	11.47
Occlusal indent	8	0.0	15.7	3.26	6.20
Retention groove	3	0.0	27.6	9.20	15.93

DISCUSSION

Results were comparable with those of other studies. On a flat, nonconcave surface, the mean thickness of various adhesive resins ranged from 13 to 115 µm[11] and from 3 to 48 µm.[12] At point C (the buccal wall) the adhesive resins had mean thicknesses within both of these ranges: 16.2 ± 8.85 µm (PB), and 23.4 ± 10.55 µm (SP).

At concave areas of the preparations (the buccal chamfer or shoulder), means of 20-355 µm[11] and 14-183 µm[12] have been recorded. At point B (the internal shoulder angles), values of 84.1 ± 27.8 µm (PB), and 104.3 ± 56.6 µm (SP) were found in this study, which again lie within these ranges.

Our ESEM images and measurements show minimal or no coverage with the precipitating-type DDAs, VS and G, and with the self-polymerizing DDA, PF. Previous SEM studies have shown minimal or no surface layer on dentin after treatment with various precipitation-type DDAs and a thin layer with PF.[13–16] G formed a layer up to 327 µm thick[11] but this was after sealing with light-cured adhesive and therefore can be discounted. PF forms a layer of clinically negligible thickness (N. Gendusa, personal communication, 2006) as does G.[11]

The methods used are similar to those in the literature. Pashley[11] and Peter and others[12] added resins to crown preparations before sectioning, polishing, and examining them microscopically. Previous studies have used a fluid cell system to simulate pulpal pressure, first described by Pashley[11] and Paul

Table 6: *Results for GLUMA (µm)*

Measuring Site	N	Minimum	Maximum	Mean	SD
Shoulder edge	8	0.0	11.4	1.43	4.03
Internal shoulder angle	8	0.0	57.1	7.14	20.19
Buccal wall	8	0.0	0.0	0.0	0.0
Internal chamfer angle	8	0.0	0.0	0.0	0.0
Occlusal indent	8	0.0	16.2	2.03	5.73
Retention groove	3	0.0	10.0	3.33	5.77

Table 7: *Tukey* Post Hoc *Testing of the Measurements (μm) By the Position on the Preparation*

Position	Subset		
	1	2	3
Shoulder edge	3.24		
Buccal wall	8.82		
Occlusal indent	21.89	21.89	
Internal chamfer angle		32.30	32.30
Internal shoulder angle		39.76	39.76
Retention groove			47.60
Significance (*p*)	0.064	0.086	0.205

and Scharer.[17] This was unnecessary in the current study as permeability was not being measured.

Although this project succeeded in demonstrating the pooling of DDAs at preparation angles, consistent surface layers were not achieved within DDA types, and a highly variable degree of cover was observed. It was difficult at times to distinguish a genuine resin layer rather than depth of the sample or other artifact. In addition, a gap beneath the layer or a peeling effect was sometimes seen (Figure 6). This peeling may be due to damage from sectioning and polishing or from dehydration of the sample. For this reason, ESEM examination was carried out within three hours of preparation.

Precipitation-type DDAs, where present, appear in much thinner sections than the light-cured DDAs. It is likely that these thin sections are more friable than their counterparts and hence more prone to being dislodged or lost in the process of sectioning and polishing. The manufacturer of VS advises that the patient should wait half an hour after application before eating or brushing teeth, presumably to give the material sufficient time to harden within the dentinal tubules; therefore, a half-hour delay was used in this study for all of the non-light-cured materials. Nonetheless, materials that are susceptible to being removed by tooth brushing are presumably also susceptible to the forces of sectioning and polishing. It is therefore impossible to say whether sites that recorded a zero in fact had a resin layer present before sectioning.

Only PB formed a consistent layer close to the shoulder angle (point A). Three of the eight SP samples had a layer present whereas the other products showed scarcely a sign of any covering (an occasional crystal-like structure was seen). This may be because PB was the only material used that had a dedicated enamel etching step. In addition, resin may be thinned further by the proximity of the

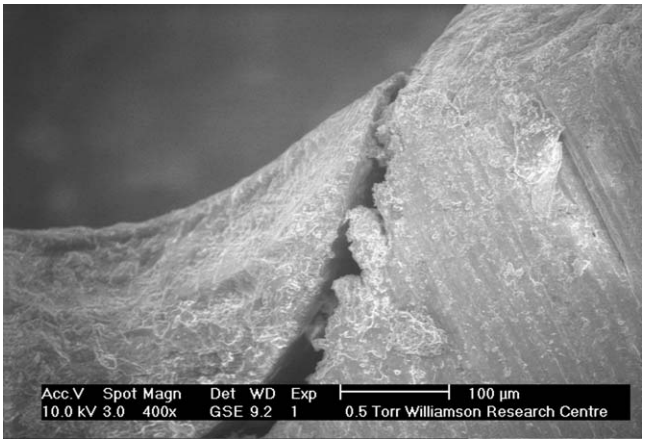

Figure 6. *Gap formation between the dentin desensitizing agent and the tooth.*

convex margin.[18] Peter and others[12] also found their lowest thickness values in this region (3 ± 1 μm for the material Syntac). It should also be noted that pooling of the material in certain areas (for example, point A at the shoulder edge) can have a negative effect on the marginal adaptation of the final restoration.

When studying the ESEM images, it was found that a number of sites had not been prepared into dentin, and resin was thus applied to cut enamel. In particular, this was seen at the occlusal indent and buccal wall. In the case of PB and SP, this did not appear to matter, as both products are capable of enamel bonding. The remaining three DDAs, however, did not appear to bond to enamel, perhaps because they are designed to enter dentinal tubules; on enamel surfaces they have no suitable bonding site. Most crown preparations carried out *in vivo* do not involve the removal of all enamel;[19] hence, this was maintained in this study.

The question raised earlier was whether the presence of a resin layer at the key sites of a crown preparation might influence other aspects of crown design or its success. Most of the surface areas of a tooth prepared for a conventional crown consist of flat or convex surfaces. Adding an adhesive desensitizing resin such as PB or SP forms a layer 16.2-23.4 μm thick on flat surfaces *in vitro*, resulting in a combined value of 19.8 ± 10.1 μm for both products. A tooth with exactly 1 mm of clearance from its opposing tooth will, after the addition of resin, still have 0.98 mm of clearance, which is a difference of 2%. This is unlikely to make any practical difference.

A standard metal-ceramic crown requires 1.5 mm of reduction on the buccal surface, including space for the luting cement. PB and SP have been shown to

pool at the buccal shoulder angle *in vitro*, forming a layer with a mean thickness of 94.2 ± 44.3 μm but reaching values as high as 199 μm. A space of 1.5 mm available for crown fabrication would thus be reduced to approximately 1.4 mm, a difference of 7%, or as little as 1.3 mm, a difference of 13%. If the tooth was underprepared initially, the effect of the resin layer will be further magnified. As mentioned earlier, Begazo and others[20] found widely varying shoulder widths and shoulder angles between 3446 tooth preparations for the same type of crown; however, in another survey, Poon and Smales[21] noted that metal-ceramic crowns in particular had underprepared cusps and buccal shoulders. Despite the use of binocular loupes in our study, once sectioned, many of the teeth were also found to have underprepared buccal shoulders, with a minimum width of 1.25 mm.

When looking at retention grooves, this study found that a layer 89.9 ± 6.67 μm thick was formed with PB and SP. This appeared sufficient under the ESEM to block out a significant portion of the groove; in one extreme case (219 μm) the groove appeared almost totally occluded. The necessary dimensions of a groove in order to have a positive effect on crown retention are subject to debate;[22–24] however, results suggest that the degree of pooling that occurs with light-cured DDAs in retention grooves *in vitro* may be sufficient to render the groove ineffective.

Gravity made a significant difference at one site only: the occlusal indent. Resin layers on upside-down samples were some 30 μm thicker than those on upright samples, suggesting that gravity has made a difference: excess resin has flowed down the mesial and distal walls and across into the occlusal reduction. With a thickness of 66.9 ± 21.62 μm, the layer is unlikely to cause technical problems, unless the occlusal surface has been underprepared. It is also noteworthy that in this study the teeth were mounted upside-down to confirm if gravity has any effect on the results. In real clinical scenarios the operative angles will differ according to the jaw of the operation and position of the tooth; therefore, the results of this study cannot be extrapolated to clinical scenarios with confidence.

There is no clear evidence for any one DDA being more effective on crown preparations than any other, though many different types have their advocates. Available products are either designed for dentin bonding (DBAs) or the treatment of root hypersensitivity (DDAs); no products are specifically designed for preparation sealing. An ideal product would be universally effective at eliminating sensitivity, compatible with all luting or bonding cements, and thin enough in application so as not to risk encroaching on crown space.

DDAs have been successfully used in this way since at least 1992,[25] and with 1-2 million tubules exposed by the average preparation,[19] the theory of blocking or sealing the tubules is plausible. A greater evidence base from further well-designed clinical trials would go some way to solving a problem that has hitherto been dealt with by anecdote as much as by fact. In the meantime, a carefully planned and executed crown preparation, with a view to reducing the risks caused by such factors as oral bacteria, under- and over-preparation, and poor impressions or provisional restorations is unlikely to cause significant sensitivity, and a DDA may well be unnecessary.

CONCLUSIONS

Within the limitations of this study, the following conclusions could be suggested:

1. Light-cured desensitizing resins form a surface layer across crown preparations, pooling in concave areas such as internal chamfer angles and retention grooves. In certain instances, this may affect the space requirements of the subsequent crown.
2. When using light-cured desensitizing resins, the effect of gravity should be taken into account as these materials have a tendency to pool in accordance to tooth position and gravity.
3. Self-curing or precipitating-type desensitizing resins do not form a surface layer.
4. Evidence for the use of desensitizers in sealing crown preparations is scarce. Although their use is unlikely to cause problems, adherence to sound principles in the various stages of crown preparation, provisional restoration, and cementation is at present a more reliable solution for preventing postoperative sensitivity.

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.

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Evaluation of Flexural, Diametral Tensile, and Shear Bond Strength of Composite Repairs

TA Imbery • T Gray • F DeLatour
C Boxx • AM Best • PC Moon

Clinical Relevance

The use of a dentin bonding agent produced more reliable composite repairs than chemical primers.

SUMMARY

Objective: Repairing composite restorations may be a more conservative treatment than replacing the entire restoration. The objective of this *in vitro* study was to determine the best repair method by measuring flexural, diametral tensile, and shear bond strength of repaired composites in which the surfaces were treated with chemical primers (Add & Bond or Silane Bond Enhancer), a bonding agent (Optibond Solo Plus [OBSP]), or mechanical retention with a bonding agent.

Methods: Filtek Supreme Ultra shade B1B was placed in special molds to fabricate specimens that served to test the flexural, diametral tensile, or shear strength of the inherent resin substrate. The same molds were modified to make specimens for testing repair strength of the resin. Repairs were made immediately or after aging in deionized water at 37°C for seven days. All repair sites were finished with coarse Sof-Lex discs to simulate finishing new restorations or partially removing aged restorations. Repair surfaces were treated with one of the following: 1) phosphoric-acid etching and OBSP; 2) Add & Bond; 3) phosphoric-acid etching, Silane Bond Enhancer, and OBSP; or 4) quarter round bur, phosphoric-acid etching, and OBSP. Specimens were placed back in the original molds to fabricate specimens for diametral tensile or flexural testing or in an Ultradent jig to make specimens for shear bond testing. Composite resin in shade B5B was polymerized against the treated surfaces to make repairs. Two negative control groups for the three testing methods consisted of

*Terence A Imbery, DDS, assistant professor, Virginia Commonwealth University (VCU) School of Dentistry, General Practice, Richmond, VA, USA

Trever Gray, dental student, VCU School of Dentistry, Richmond, VA, USA

Frank DeLatour, dental student, VCU School of Dentistry, Richmond, VA, USA

Charles Boxx, dental student, VCU School of Dentistry, Richmond, VA, USA

Al M Best, PhD, VCU School of Dentistry, Periodontics, Richmond, VA, USA

Peter C Moon, PhD, VCU School of Dentistry, General Practice, Richmond, VA, USA

*Corresponding author: 520 North 12th S, Lyons Bldg, Richmond, VA 23298, USA; e-mail: taimbery@vcu.edu

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specimens in which repairs were made immediately or after aging without any surface treatments. Controls and experimental repairs were aged (water 37°C, 24 hours) before flexural, diametral tensile, or shear testing in an Instron Universal testing machine at a crosshead speed of 0.5 mm/min.

Results: Experimental flexural repair strengths ranged from 26.4% to 88.6% of the inherent substrate strength. Diametral tensile repair strengths ranged from 40% to 80% of the inherent substrate strength, and shear bond strength repairs ranged from 56% to 102%. Geometric means were statistically analyzed with two-way analysis of variance on their log-transformed values. Significant differences were determined using Tukey honestly significant difference ($p<0.05$).

Conclusions: Depending on the mechanical property being tested, surface treatments produced different results. OBSP produced more consistent results than chemical primers.

INTRODUCTION

Despite the esthetic advantages of composite resins, restoration failure continues to be a problem. Studies have shown a significant increase in the size of cavity preparations when restorations are replaced.[1,2] Additionally, the esthetics of resins pose a problem of identifying margins at time of replacement and distinguishing resin from tooth structure. Complete removal of a defective composite restoration may result in unnecessary removal of additional tooth structure. Repairing an existing composite restoration may be a more conservative approach, especially if the defective restoration is very extensive or approximates the pulp. An alternative to replacing an entire restoration is to remove only the defective portion so that a repair can be accomplished. Minimally invasive repair of composites may extend the longevity of the restoration without further damaging teeth.[3]

The vast majority of composite restorations are placed in incremental layers and rely on the presence of the oxygen-inhibited layer of unpolymerized resin to bond the next increment.[4,5] The amount of unreacted methacrylate groups available for bonding on the surface of a composite decreases significantly after 24 hours.[1] Additionally, composites undergo hydrolytic degradation, which leads to swelling and crack propagation within the matrix and breakdown of the coupling agent, resulting in loss of filler particles.[2] As a result, bonding to aged or contaminated composite is unpredictable. Successful repairs depend on the development of an adequate interfacial bond between the old and new composite resin. Methods have been proposed to enhance the bonding through micromechanical and chemical means with repair strengths varying from 25% to 82% of the inherent strength of the composite substrate.[6-11]

When performing composite repairs most North American dental schools teach the use of diamond finishing burs to create micromechanical retention followed by phosphoric acid etching and placement of a bonding agent before composite placement.[3] Add & Bond (Parkell Inc, Edgewood, NY, USA) is a one-step primer marketed for composite repairs. The manufacturer claims repair strengths that surpass the inherent cohesive strength of the composite. Another chemical method is to silanate the resin before placing a bonding agent. This means placing Si-O groups on the exposed filler particles and matrix that can bond with the matrix of the repair resin.

The purpose of this *in vitro* study was to determine the optimal repair protocol by evaluating the flexural, diametral tensile, and shear bond strengths of composite repairs. The surface treatments were 1) phosphoric-acid etching and Optibond Solo Plus (OBSP; Kerr, Romulus, MI, USA); 2) Add & Bond, Parkell Inc, Edgewood, NY, USA ; 3) phosphoric-acid etching and Silane Bond Enhancer (Pulpdent Corp, Watertown, MA, USA) followed by OBSP; and 4) mechanical retention, phosphoric-acid etching and OBSP. Our null hypothesis was that there would not be any differences among the four repair protocols or any difference between the inherent strength of the resin substrate and the repair.

METHODS AND MATERIALS

The dental materials utilized and their compositions are listed in Table 1. The nano-composite Filtek Supreme Ultra (3M ESPE, St Paul, MN, USA) was used to make all specimens in this study by photocuring the resin for 40 seconds with SmartLite IQ2 (Dentsply/Caulk, Milford, DE, USA) at an intensity of 500 mW/cm^2. When fabricating all specimens, a glass plate was placed against Mylar strips to compress the resin into the molds using finger pressure. Excess resin was removed before photocuring. To delineate the repair joint during testing, two different shades of composite were used; B1B to make the original specimens and B5B as the repair composite. External surfaces of the specimens were visually examined, and a sharp knife was used to

Table 1: *Materials Used in the Study*

Product	Manufacturer
Filtek Supreme Ultra	3M ESPE, St. Paul, MN, USA
• BisGMA, UDMA,	
• TEGDMA, PEGDMA	
• Filler: 20 nm silica and 4-11 μm zirconium	
Optibond Solo Plus	Kerr, Orange, CA, USA
• Alkyl dimethacrylate	
• Barium aluminoborosilicate glass	
• Fumed silica (silicon dioxide)	
• Sodium hexafluorosilicate	
• Camphorquinone	
Add & Bond	Parkell, Edgewood, NY, USA
• Uncured methacrylate resin	
• Ester monomers	
• Camphorquinone	
Silane Bond Enhancer	Pulpdent, Watertown, MA, USA
• Silane	
• Alcohol	
• Acetone	

Abbreviations: BisGMA, bisphenol glycidyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

remove any burrs or irregularities that would prevent exact placement of the specimen into custom-made molds or testing jigs. Specimens were either repaired immediately or after being aged by placing them in 37°C deionized water for seven days. Twelve specimens were made for each control and experimental group.

Flexural Strength

Composite resin (shade B1B) was placed in bulk in a stainless steel mold measuring 2-mm by 2-mm by 24-mm. The curing light tip measured 7 mm in diameter and was placed four times along the length of the beam, overlapping the previous photo-cured composite. These beams served as the positive control (n=12) for flexural strength testing. An additional 120 composite resin beams measuring 2-mm by 2-mm by 12-mm were made in a similar fashion. These were divided into 10 groups (eight experimental and two negative control groups) of 12 specimens. Five groups of 12 beams were repaired immediately and the other five groups were used for repairing aged specimens. One end of each beam was roughened with a coarse Sof-Lex (3M ESPE) disc for five seconds to simulate clinical finishing or partial removal of a composite resin restoration and then thoroughly rinsed for 10 seconds with air/water aerosol. The roughened end of the beam was treated with one of the experimental surface treatment protocols as explained in Table 2, placed back in the original mold, and composite resin in shade B5B was photo-cured against the treated end to make beams measuring 2-mm by 2-mm by 24-mm. Two negative controls consisted of immediate and delayed repairs without any surface modifications or treatments.

Diametral Tensile Strength

A stainless steel mold was used to fabricate cylindrical composite specimens in shade B1B measuring 6 mm in diameter and 3 mm thick. The resin was placed in bulk and photo-cured against Mylar strips through both open ends of the mold. These served as the positive control (n=12). An additional 120 specimens were made in the same manner using a split mold to make half cylindrical shaped specimens. Half of these were repaired immediately and the other half repaired after aging. The flat diametral end of each cylindrical specimen was roughened with a coarse Sof-Lex disc for five seconds then thoroughly rinsed for 10 seconds with air/water aerosol. Surface treatments were applied as described in Table 2, and specimens were placed back in the original mold; repairs were made by photo-curing shade B5B composite resin against the treated surface through both open ends of the mold. Negative control groups consisted of immediate and aged repairs without any surface modifications or treatments.

Shear Bond Strength

Specimens were made using the same mold as used to make the positive control diametral specimens. A positive control group consisted of photo-curing the resin (shade B1B) without a Mylar strip on one of the open ends of the mold. This left an air-inhibited layer on the surface to which a column of resin (shade B5B) 2.0 mm in height and 2.38 mm in diameter was bonded perpendicular to the surface. An additional 120 specimens were made in a similar fashion but photo-cured against Mylar strips on both open ends of the mold. Half were used for immediate repairs and the other half were used to make repairs of aged specimens. Experimental groups consisted of treating the repair site with a coarse Sof-Lex disc for five seconds, thoroughly rinsing for 10 seconds, and completing surface treatments as described in Table 2. The specimens were placed in a bonding jig (Ultradent, South Jordan, UT, USA), and a column

Table 2: *Surface Treatment Protocols*

Product	Treatment Protocol
Optibond Solo Plus	1. Apply H_3PO_4 etchant for 10 seconds and rinse with air/water aerosol for 10 seconds.
	2. Apply Optibond Solo Plus with a scrubbing motion. Evaporate the solvent for 10 seconds using air from an air/water syringe.
	3. Repeat step 2 and photo-cure for 20 seconds with SmartLite IQ2[a] with an intensity of 500 mW/cm^2
Add & Bond	1. Apply a thin layer of Add & Bond and agitate for 10 seconds. Do not rinse, dry, or light-cure this layer. Place the resin over the surface and photo-cure both the resin and underlying Add & Bond for 40 seconds with a SmartLite IQ2 with an intensity of 500 mW/cm^2.
Silane Bond Enhancer Optibond Solo Plus	1. Apply H_3PO_4 etchant for 10 seconds and rinse with air/water aerosol for 10 seconds.
	2. Apply Silane Bond Enhancer and allow it to evaporate for 60 seconds. If not completely evaporated, then lightly dry with air.
	3. Apply Optibond Solo Plus with a scrubbing motion as previously described.
Mechanical Retention and Optibond Solo Plus	1. Using a quarter round bur in a high-speed handpiece, place a retentive point in the resin to the depth of 1 mm.
	2. Etch with H_3PO_4 and apply Optibond Solo as previously described.

[a] *Dentsply/Caulk, Milford, DE, USA.*

of composite-resin (shade B5B) 2.0 mm in height and 2.38 mm in diameter was photo-cured perpendicular to the treated surfaces.

Testing

All specimens were stored in deionized water (37°C) for 24 hours before testing. Specimens were placed in custom-made testing jigs and repair sites were loaded until failure in an Instron Universal Testing Machine (Model #TTC, Instron Corporation, Canton, MA, USA) at a crosshead speed of 0.5 mm/min. For diametral tensile testing, a compressive load was applied diametrically without the use of soft pads between the specimen and platen. A three-point bending test was used for flexural strength testing with a distance of 20 mm between the two supporting struts. For shear testing, a chiseled bar 1.0-mm thick was used to apply the force at the repair site. Forces required for failure were recorded in pounds and flexural strength calculated using the formula $(3 \times \text{Load} \times \text{Length})/(2 \times \text{Width} \times \text{Thickness}^2)$. Diametral strength was calculated using the formula $(2 \times \text{Load})/(3.14 \times \text{Diameter} \times \text{Thickness})$. Shear bond strength was calculated by dividing the load by the surface area. All bond strengths were converted to megapascals (MPa) and geometric means statistically analyzed with two-way analysis of variance (ANOVA) using SAS version 9.3, JMP Pro version 11.0 software (SAS Institute Inc, Cary, NC, USA) at a significance level of 5%. Independent variables were surface treatment and repair time (immediate or aged). Significant differences within groups were determined using Tukey honestly significant difference (HSD) multiple comparison at a confidence level of $p<0.05$. All specimens were visually examined using loupes with 4× magnification, and failures classified as cohesive within the resin, adhesive with failures occurring between the two resin interfaces, or mixed.

RESULTS

The results for the three testing methods were not normally distributed. Therefore, geometric means, which are less affected by skewed data, were calculated and analyses (ANOVA and Tukey HSD multiple comparison) performed on the log scale of the geometric means.

Flexural Strength

The minimum, maximum, geometric means, and standard deviations for flexural strength are listed in Table 3 and failure modes in Table 4. The mean flexural strength of the intact resin beam was 102.9 MPa. The mean (geometric) repair strengths ranged from a low of 27.2 MPa (aged repairs using Add & Bond) to a high of 91.2 MPa (immediate repair using OBSP). All immediate and aged repairs were statistically similar to the intact resin beam except aged repairs using OBSP or Add & Bond. At best, 88% of the inherent flexural strength was achieved when the specimen was repaired immediately using OBSP. Aged specimens repaired using Silane Bond Enhancer with OBSP (68.6MPa) had more than twice the repair strength achieved using only OBSP (29.0 MPa). The immediate negative control (no surface treatment, primer, or dentin bonding agent) obtained 43% of the inherent flexural strength, and the aged negative control obtained 17% of the

Table 3: *Flexural Strength (MPa)*[a]

Treatment Groups	Minimum	Maximum	SD	Mean
Control				
Positive	67.1	151.0	27.8	102.9[A]
Immediate/no treatment	25.2	75.5	15.7	44.1[ABC]
Aged/no treatment	0.0	75.5	20.8	17.3[C]
Immediate				
OBSP	30.2	174.5	44.3	91.2[A]
Add & Bond	26.9	120.8	28.6	80.6[A]
Silane Bond Enhancer/OBSP	26.8	130.9	33.6	67.7[AB]
Mechanical retention/OBSP	18.5	141.0	33.7	54.1[AB]
Aged				
OBSP	5.0	122.5	35.4	29.0[BC]
Add & Bond	6.7	107.4	32.8	27.2[BC]
Silane Bond Enhancer/OBSP	12.6	122.5	32.2	68.6[AB]
Mechanical retention/OBSP	8.4	104.0	31.8	44.9[ABC]

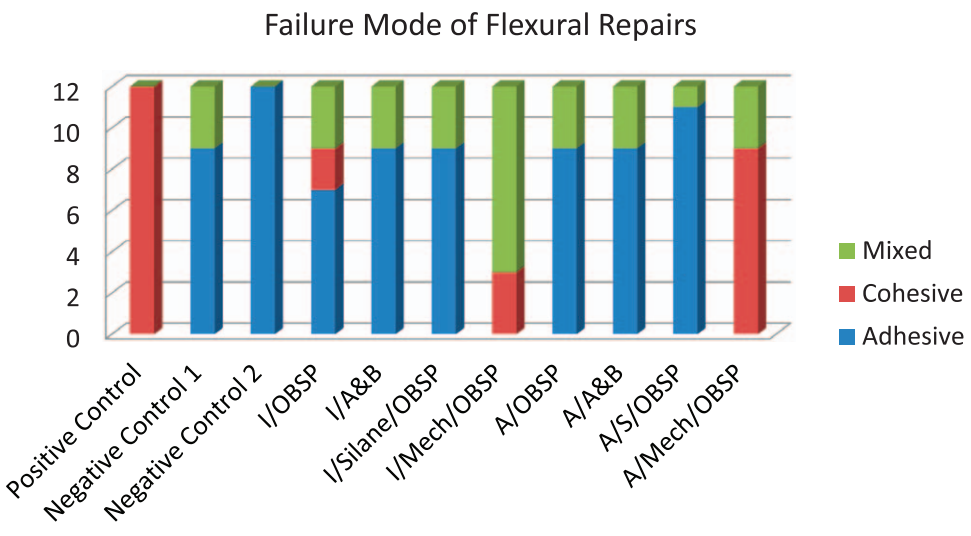
[a] *Geometric means were compared using two-way analysis of variance on the log-transformed values. Means not sharing the same superscript letter are significantly different (at α=0.05).*

inherent substrate's strength. Most of the failures were classified as mixed or adhesive in nature.

Diametral Tensile Strength

The minimum, maximum, geometric means, and standard deviations are reported in Table 5 and failure modes in Table 6. The mean (geometric) diametral tensile strength of the inherent resin substrate was 59.0 MPa. Mean experimental diametral tensile repair bond strength ranged from a low of 18.0 MPa (immediate repairs using Add & Bond) to a high of 47.2 MPa (immediate repairs using OBSP). The diametral tensile bond strengths of all experimental groups were statistically less than the positive control except for immediate repairs using OBSP (47.2 MPa) or Silane Bond Enhancer with OBSP (44.6 MPa). Eighty percent of the inherent diametral tensile strength of the composite was obtained by an immediate repair using OBSP. The immediate negative control obtained 56% of the inherent diametral tensile strength and the aged negative control 19%. The failure mechanism was predominately mixed and adhesive.

Table 4: *Failure Mode of Flexural Strength Repairs*



Abbreviations: I, immediate; A, aged; OBSP, Optibond Solo Plus; A&B, Add and Bond; Mech, mechanical retention with a quarter round bur.

Table 5: *Diametral Tensile Strengths (MPa)*[a]

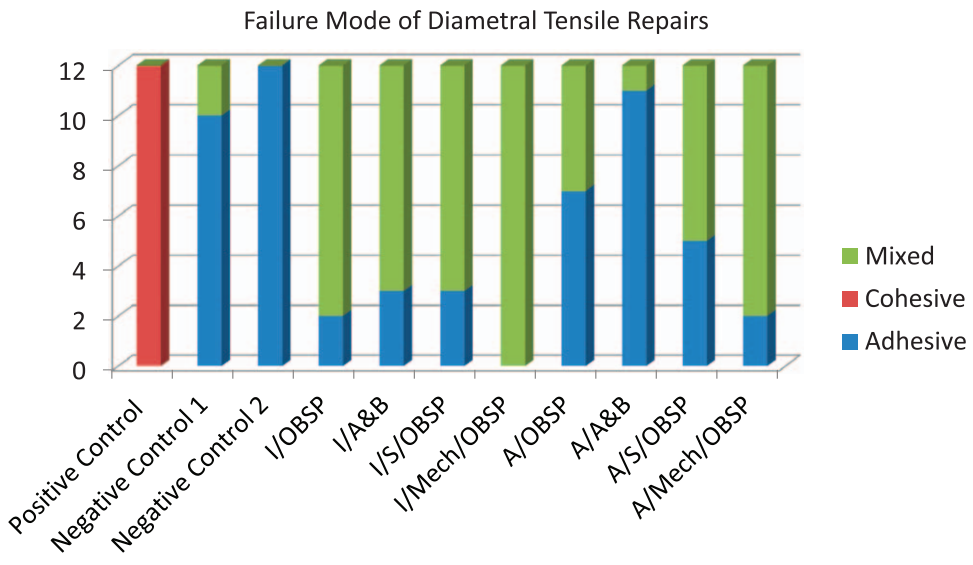
Treatment Groups	Minimum	Maximum	SD	Mean
Controls				
Positive	48.2	71.8	8.2	59.0[A]
Immediate/no treatment	21.3	52.8	10.1	32.9[BDC]
Aged/no treatment	7.1	22.8	4.8	11.3[F]
Immediate				
OBSP	26.5	72.3	15.1	47.2[AB]
Add & Bond	12.0	32.8	7.3	18.0[E]
Silane Bond Enhancer/OBSP	27.2	64.9	12.5	44.6[ABC]
Mechanical retention/OBSP	14.8	44.7	10.2	29.1[DC]
Aged				
OBSP	18.9	41.4	5.9	28.7[D]
Add & Bond	15.8	36.2	6.7	23.6[ED]
Silane Bond Enhancer/OBSP	16.9	40.2	7.0	26.9[D]
Mechanical retention/OBSP	17.5	34.0	5.4	26.9[D]

[a] *Geometric means were compared using two-way analysis of variance on the log-transformed values. Means not sharing the same superscript letter are significantly different (at α=0.05).*

Shear Bond Strength

The minimum, maximum, geometric means, and standard deviations are illustrated in Table 7 and failure modes in Table 8. Mean (geometric) shear bond repair strengths ranged from a low of 9.4 MPa (aged/no treatment) to a high of 19.2 MPa (immediate/mechanical retention and OBSP). All experimental groups were statistically similar to the positive control (18.8 MPa) except for the group that was aged and treated with Silane Bond Enhancer and OBSP (10.6 MPa). Two experimental groups were numerically greater, although not statistically stronger, than the positive control. Both of these groups included repairs made using mechanical retention and OBSP repaired immediately (19.2 MPa) and repaired after aging (18.9 MPa). The immediate negative control achieved 60% of the inherent substrate shear bond strength; the negative aged control obtained 50%. Failure mechanisms in experimental groups demonstrated predominantly mixed or adhesive failures.

Table 6: *Failure Mode of Diametral Strength Repairs*



Abbreviations: I, immediate; a, aged; OBSP, Optibond Solo Plus; A&B, Add and Bond; mech, Mechanical retention with quarter round bur.

Table 7: *Shear Bond Strengths (MPa)* [a]

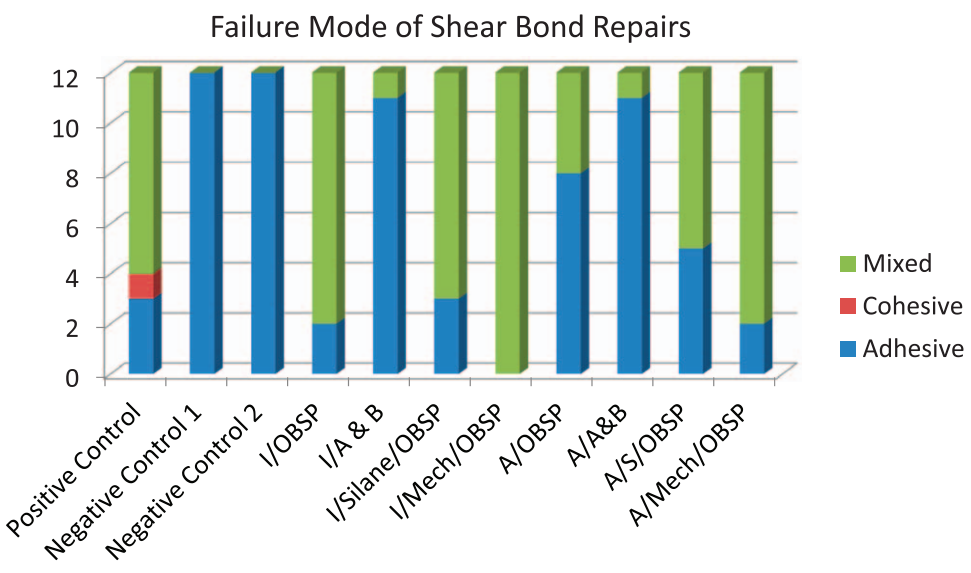
Treatment groups	Minimum	Maximum	SD	Mean
Controls				
Air-inhibited layer present	11.5	27.5	4.9	18.8^{A}
Immediate/air-inhibited layer removed	7.5	17.5	3.3	11.3^{BC}
Aged/no treatment	4.0	16.5	4.4	9.4^{C}
Immediate				
OBSP	8.0	22.5	4.7	13.6^{ABC}
Add & Bond	7.5	20.0	4.3	13.3^{ABC}
Silane Bond Enhancer/OBSP	9.5	33.5	6.6	18.3^{A}
Mechanical retention/OBSP	10.5	24.0	4.0	19.2^{A}
Aged				
OBSP	10.6	22.0	3.5	17.4^{AB}
Add & Bond	10.0	20.0	2.5	16.6^{AB}
Silane Bond Enhancer/OBSP	3.5	30.5	8.4	10.6^{BC}
Mechanical retention/OBSP	11.5	26.5	5.1	18.9^{A}

[a] *Geometric means were compared using two-way analysis of variance on the log-transformed values. Means not sharing the same superscript letter are significantly different (at α=0.05).*

DISCUSSION

Repair bond strength differences exist depending on the surface treatments. Additionally, differences exist between the original substrate and repairs; therefore, both null hypotheses are rejected. Repairs occur by three mechanisms: 1) micromechanical bonding due to surface irregularities, 2) chemical bonding between the two resin matrices, and 3) chemical bonding with the filler.[12] The purpose of using coarse Sof-lex discs with approximately 100 μm particle size is to simulate the immediate clinical finishing of the restoration. For aged specimens the Sof-lex disc served to remove the contaminated superficial layer of resin that would have been exposed to saliva. Additionally, their use creates micromechanical irregularities, resulting in increased surface area and free energy. North American dental schools that teach composite repairs recommend roughening the composite surface with a diamond bur.[3] However, the degree of coarseness of

Table 8: *Failure Mode of Shear Bond Strength Repairs*



Abbreviation: I, immediate; A, aged; OBSP, Optibond Solo Plus; A&B, Add and Bond; Mech, mechanical retention with quarter round bur.

the diamond bur was not identified. Depending on the manufacturer, a course diamond bur may have particle size ranging from 25 μm to 100 μm. We chose to use coarse Sof-lex discs because their particle size was similar to the range of commonly used coarse diamond burs. Additionally, many diamonds burs have a 4° to 7° tapper, which would result in a slanted repair junction.

Filler particles in composite resin are coated with silane, which allows chemical bonding to the resin matrix. We anticipated that surface treatment with Silane Bond Enhancer followed by application of the dentin bonding agent would result in an additive effect compared with using only OBSP. This was the result in immediate repairs under shear testing (18.3 MPa compared with 13.6 MPa) and aged repairs under flexural testing (68.6 MPa compared with 29.0 MPa). When tested under flexural forces, Silane Bond Enhancer used with OBSP (immediate and aged specimens) provided statistically similar bond strengths to each other and to the intact substrate (Table 3). Silane Bond Enhancer had very little effect in diametral testing. The silane coupling agent on the filler particles must be replaced for adequate repairs to occur because surface preparation with Sof-lex discs removes the filler's silane layer.[13] Improved repair strength tested under shear forces has been reported when the resin is first silicoated followed by silanization and application of a bonding agent.[14] Our study produced comparable shear bond strengths without prior silicoating.

The manufacturer claims that Add & Bond produces repair strengths as strong as the composite's inherent strength but this study never produced values as numerically strong. However, the values were statistically similar for immediate repairs tested under flexural forces (102.9 MPa compared with 80.6 MPa) and in shear testing immediate repairs (13.3 MPa compared with18.8 MPa) and aged repairs (16.6 MPa compared with 18.8 MPa). In diametral tensile testing, Add & Bond was significantly weaker than the intact substrate for both the immediate repairs (18. 0MPa) and the aged repairs (23.6 MPa) compared with the intact substrate (59.0 MPa).

We anticipated an additive effect combining mechanical retention with OBSP but obtained mixed results. It was beneficial for aged repairs under flexural forces (44.9 MPa) compared with only OBSP (29.0 MPa) but not for immediate repairs, which had 40% reduction in repair strength. Mechanical retention combined with OBSP produced decreased diametral tensile repair strength of immediate repairs compared with only OBSP (29.1 MPa compared with 47.2 MPa). Mechanical retention with OBSP was most beneficial under shear forces, obtaining the highest shear bond strengths for immediate repairs (19.2 MPa) and aged repairs (18.9 MPa). The mixed results may be related to the size of the repair site area. Flexural specimens had a 4 mm^2 surface area, shear specimens 4.44 mm^2, and diametral specimens 18 mm^2. The diametral tensile specimens may benefit from additional retentive points instead of a single point. A quarter round bur is 0.5 mm in diameter and results in a retentive feature with a 90° shoulder and a rounded bottom. This is recommended by Vivas and others,[15] who demonstrated improved flexural and shear bond strength compared with acid etching alone. Shen and others[16] suggested that a quarter round bur may cause an area of stress concentration and should be eliminated by rounding the shoulder or widening the entrance. As an alternative, Yesilyurt and colleagues[17] recommended the use of micromechanical retention produced by air abrasion or a diamond bur.

The use of bonding agents has been shown to significantly enhance the shear bond strength of composite repairs that were aged from one to 12 weeks before repair.[18] The bonding agent is able to enter the microscopic mechanical features of the resin. Etching with phosphoric acid was used in our protocol because repairing a composite resin usually involves not only removing a portion of the restoration but also adjacent enamel and dentin. Surface imaging performed by Fawzy and others[19] demonstrated no significant change in the composite's surface morphology after acid etching. The acid's action on resin may be limited to superficial cleaning.[20] Several studies have not shown an increase in bond strength when composite surfaces were etched with phosphoric acid.[6,7,10]

In a study in which composite specimens 4 mm thick were aged, Rodrigues and others[21] calculated 50% water saturation occurred within nine days. Additionally, Biradar and others[22] determined that maximum water absorption occurred in the first week. Therefore, we chose seven days of water storage for our aging protocol. Water diffuses through the polymer chains and interface boundaries between the resin and filler particles. Hydrolytic deterioration of the resin results in elution of components and plasticization of the resin. Properties such as hardness, wear resistance, strength, and fracture toughness are affected by water absorption. Additionally, water absorption reduces the available unreacted methacrylate carbon double bonds neces-

Figure 1. *Diametral tensile failure. The fracture occurred along the repair site and through adjacent composite resin. This is referred to as a cleft fracture and was classified as a mixed fracture.*

sary to chemically react with the repairing composite. The amount of water absorption is affected by the resin and the filler content, coupling agent, and curing time and distance.[23] Increasing the amount of filler content and improving its bond to the resin matrix decreases water absorption. As a result, hybrid composites have been shown to provide better repair strength than microfilled resins.[24] Although composites are hydrophobic, water absorption may increase their hydrophilicity allowing better penetration of the dentin bonding agent during the repair process. Resins containing bisphenol glycidyl methacrylate have lower conversion rates compared with other matrices.[25.] Therefore, bis-GMA containing resins have more unreacted carbon double bonds to chemically react with the repairing composite resulting in higher repair strengths than resins containing different matrices.[8] Ultimately, repair strengths are dependent on the microstructure and composition of the parent and repair materials as well as penetration of the adhesives into the retentive features of the aged resin.

The application of a bonding agent for immediate repairs may not be necessary as Rinastiti and others[26] have demonstrated. The survival rate of free radicals in photo-cured resin is 14 days.[27,28] There probably exists sufficient unreacted carbon double bonds to react with the repair composite. Because the negative control groups were able to form a repair suggests that unreacted vinyl groups (C=C) in the original substrate remain available, but to a lesser degree. Negative control groups ranged from a low of 17% of the inherent substrate strength (aged flexural repairs without surface treatment) to a high of 60% (immediate shear bond strengths).

Shear testing had the largest percentage of mixed and cohesive failures. (Table 8). Della Bona and van Noort[29] suggested that shear testing measures the cohesive strength of the underlying composite rather the adhesive strength of the repair. The underlying composite will fracture only if the repair is stronger. However, it may also be an indication that the underlying composite was weakened because of water absorption and damage from using a Sof-lex disc. Flexural repairs had mainly adhesive and mixed failures at the repair site (Table 4). In shear testing there were 10 cohesive failures in the aged specimens using mechanical retention and OBSP. This may be the result of weakening of the parent substrate by the presence of mechanical retention in an already weakened matrix and stress concentration induced by the preparation design.

Repaired composites are subjected to complex intraoral forces during mastication and parafunctional habits. Of the three tests used in this study to replicate intraoral forces; diametral tensile is the most difficult to interpret. Failure must occur in the center of the specimen due to tensile forces if the diametral test is to yield useful results.[30] However, there is debate as to whether failure is actually caused by tensile forces acting at the center of the specimen.[30,31] When the specimen is loaded at a point, the fracture is due to shear and compressive forces at the loading point.[31] If a specimen is loaded over a small flattened area (an arc less than 0.2× the diameter) or beneath soft pads, there are significant effects on the stress distribution patterns and diametral tensile values.[31] All diametral tensile specimens in our study always failed along the diametral plane, and many exhibited cleft fractures, which we classified as mixed failures (see Figure 1). We believe that because point loading at the repair site continued, the specimens may have deformed resulting in a change from point loading to distributed loading. When this occurred stresses deviate from the recommended ideal tensile stresses at the center to more complex combined stresses.[31] This may be the reason we observed mixed failures in specimens with lower diametral tensile strengths. Although our study did not exactly follow the testing methodology outlined in American National Standards Institute/American Dental Association Specification 27 for light-cured resins, the results we obtained were not drastically different from those obtained in studies that did.[32,33]

The minimally required composite repair strength that is necessary to survive clinically is not known. However, if the repair strength approaches the

inherent strength of the nonrepaired composite or if a repair fails cohesively, this is an indication that the approach selected for repair may be clinically successful. In this study, several experimental groups were statistically equal to the flexural, diametral tensile, and shear strength of the inherent composite. *In vitro* studies have generally shown decreased repair strength of the composite repair. However, other studies have shown benefits of repairs on the longevity of the restoration.[34] Although the repair strength may be much lower than the inherent physical property being tested, there may not be a direct correlation to the clinical performance and longevity of the restoration.[35-38] Some clinical situations may involve immediate repair of a composite restoration. If a clinician determined that a suboptimal restoration was placed (open margin, inadequate proximal contact) and enough time remained in the appointment, then the clinician may decide to perform an immediate repair. If sufficient time is not available, it may be seven days before the patient could be reappointed to accomplish the repair. However, if the patient is new to the practice and the composite's composition is not known, there may not be a universally acceptable repair technique as composites of different compositions react differently when repaired.[39]

CONCLUSIONS

1. Repairing composite is unpredictable, and surface treatments produce different effects depending on the mechanical property being tested.
2. Composite repair strengths are generally lower than the inherent strength of the composite.
3. The combination of three mechanical tests was not able to determine an optimal protocol for composite repair.

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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.

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Influence of pH on the Effectiveness of Hydrogen Peroxide Whitening

CRG Torres • E Crastechini • FA Feitosa
CR Pucci • AB Borges

Clinical Relevance

Verification that pH influences the bleaching efficacy will contribute to the development of more efficient bleaching products.

SUMMARY

Objective: To evaluate the influence of pH on the bleaching effect of hydrogen peroxide on chromogen agents.

Method: Hydrogen peroxide 50% was mixed with red wine or with an alcoholic solution of tobacco in glass cuvettes, resulting in final peroxide concentrations of 16.97% and 21.12%, respectively. The pH of this mixture was measured and adjusted with 3.3 M HCl solution or 2.5 M NaOH solution to obtain the final pH values of 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, and 9.0. After mixing, the color of these solutions was evaluated in a reflectance spectrophotometer; readings were repeated after 10 minutes for the wine solution and 20 minutes for the tobacco solution. Ten samples were prepared for each solution at each pH. Color changes (Delta E) were calculated. The data were statistically analyzed using analysis of variance one-way and Tukey tests, with a significance level of 5%.

Results: There were significant differences among the different pH values for the wine and tobacco solutions (p=0.0001). The Tukey test showed that for both solutions, pH 9.0 resulted in a significantly greater bleaching effect than the other values tested.

Conclusion: The efficacy of hydrogen peroxide bleaching is directly proportional to the increase in its pH.

*Carlos R G Torres, DDS, PhD, UNESP – Univ. Estadual Paulista, Institute of Science and Technology, Department of Restorative Dentistry, São Paulo, Brazil

Erica Crastechini, DDS, MS, UNESP – Univ. Estadual Paulista, Institute of Science and Technology, Department of Restorative Dentistry, São Paulo, Brazil

Fernanda Alves Feitosa, DDS, MS, UNESP – Univ. Estadual Paulista, Institute of Science and Technology, Department of Restorative Dentistry, São Paulo, Brazil

Cesar Rogério Pucci, DDS, MS, PhD, UNESP – Univ. Estadual Paulista, Institute of Science and Technology, Department of Restorative Dentistry, São Paulo, Brazil

Alessandra Bühler Borges, DDS, MS, PhD, UNESP – Univ. Estadual Paulista, Institute of Science and Technology, Department of Restorative Dentistry, São Paulo, Brazil

*Corresponding author: Av Eng Francisco Jose Longo, 777, Jd São Dimas, São Jose dos Campos, São Paulo, 12245-000, Brazil; e-mail: carlosrgt@ict.unesp.br or carlosrgt@gmail.com

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INTRODUCTION

Tooth bleaching is a treatment widely used in the dental clinic to improve the esthetics of discolored teeth. Changes in tooth color may be of intrinsic or extrinsic causes. The intrinsic causes might be from endogenous origin, such as hemorrhage or disorders during odontogenesis caused by metabolic or infectious diseases, or even with the intake of certain medications. Extrinsic changes are from external

sources, such as pigments from tea; beverages containing chromogenic agents, such as coffee and wine; medications such as chlorhexidine or iron compounds; or even habits such as using tobacco. These substances are deposited on the enamel's surface and can be removed by a simple prophylaxis. However, with time, they can penetrate through the pores of the enamel and become intrinsic.[1-3]

Pigments, also called chromogens, have in common a structure shaped like a complex carbon chain, with many double bonds, which absorbs most ambient light affecting the tooth structure. It is known that the color observed in any structure actually corresponds to the light wavelength being reflected by it. For the bleaching to occur, it is necessary that the chromogen agents' carbon chains are broken, turning them into simpler molecules, reducing light absorption and increasing reflection.[3]

Hydrogen peroxide is the most widely used dental bleaching agent, manufactured at low concentration for at-home techniques or high concentration for in-office techniques.[4-6] It is a highly unstable molecule and decomposes according to a sequence of reactions, which can be influenced by incident light, pH, temperature, interactions with transition metals, and other factors.[7-9] Initially, it decomposes into hydrogen cations (H^+) and the perhydroxyl anion (HO_2^-; equation 1). The H^+ release explains their behavior as a weak acid. The perhydroxyl anion interacts with another peroxide molecule and results in the formation of the free radicals hydroxyl $(HO)^\bullet$ and perhydroxyl $(HO_2)^\bullet$, also called active species (equation 2). The hydroxyl radicals react with more peroxide and result in the formation of more perhydroxyl radicals and water (equation 3). With the completion of the reaction, all of the peroxide is converted to water. Obviously, the description above is simplified due to the possible formation of intermediates and other active species.

$$H_2O_2 \rightarrow H^+ + HO_2^- \quad (1)$$

$$HO_2^- + H_2O_2 \rightarrow HO_2^\bullet + HO^\bullet + OH^- \quad (2)$$

$$HO^\bullet + H_2O_2 \rightarrow HO_2^\bullet + H_2O \quad (3)$$

Free radicals are unstable oxidants, which have in their structure an unpaired electron. To become stable, free radicals bind with electrons from other organic molecules that contact with the pigments.[3] The pigment molecules are broken down into simpler chains in a redox reaction, which changes its behavior and decreases the optical absorption of light.[4,5]

The decomposition of hydrogen peroxide can be initiated with or without the presence of a catalyst. The radicals are formed slowly, without the presence of a catalyst in a reaction called self oxidation-reduction.[9] In the presence of metal ions or enzymes, that reaction can be accelerated.[10] The same is observed with increasing temperature.[11]

It is very important to develop and produce dental bleaching gels that are the most efficient and safe as possible, making the technique simpler for the dentist and more comfortable for the patient. In relation to the efficacy of the bleaching procedure, the oxidative activity of the hydrogen peroxide molecule is strongly dependent on several factors. When we think about oxidation of chromogenic molecules, the action of hydrogen peroxide is essentially the same, whether we are bleaching wood pulp, cotton, or cloth in industrial processes; dirty clothes or dishes in the home; or chromogenic molecules inside the intercrystalline spaces of enamel. Therefore, to develop a more efficient dental bleaching gel, we have to improve the activity of the hydrogen peroxide molecule itself, boosting the chemical reaction that promotes the bleaching. For that, we have to understand and control all variables that exert a direct effect on free radical generation and availability.

The direct effect of pH on bleaching effectiveness was previously demonstrated in the industrial bleaching of cotton fibers or wood pulp. Hydrogen peroxide solutions with a higher pH are used to increase the efficacy of the process.[12] In relation to dental bleaching gels, a large number of products present an acidic pH in order to increase the product's shelf life, since hydrogen peroxide is more stable in an acidic environment. However, this low pH can promote enamel demineralization. Several studies have shown that bleaching in acidic pH can produce changes in chemical composition and surface morphology, calcium loss, and reduction in hardness and fracture resistance.[13-17] On the other hand, studies of dental bleaching agents with alkaline pH have shown an increased bleaching efficacy,[18] reducing its deleterious effects on enamel surface properties.[19] However, there is a lack of literature in relation to the influence of pH on bleaching of chromogens commonly found in the oral cavity that are responsible for tooth darkening, such as wine and tobacco. Therefore, determining a pH that is safe and allows for bleaching of chromogenic substances available in the oral environment with maximum efficacy is very important for the development of new formulations and products.

For a preliminary study on the influence of bleaching agent formulation on their efficacy, the use of teeth is laborious. Thus, an *in vitro* method for testing the action of peroxide on chromogens without the use of tooth substrate is simple, and a larger number of combinations can be assessed, avoiding the complexities resulting from chemical diffusion and optical transmission within the tooth.[18] When we use tooth structure, other variables are present, such as tooth age, mineralization status, initial color, diameter and number of dentin tubules, and differences in the numbers of organic and inorganic compounds.

Given the importance of objective parameters for the development of new formulations, the aim of this study was to determine the optimal pH to maximize the efficacy of the bleaching effect of hydrogen peroxide on chromogens. The null hypothesis tested was that the pH does not influence the bleaching efficacy.

METHODS AND MATERIALS

To evaluate the bleaching effect of hydrogen peroxide *in vitro,* its action on the colored dye solutions containing known chromogens was measured by a reflectance spectrophotometer (CM 2600d, Konica Minolta, Osaka, Japan), as proposed by Maiolo and others[20] and Young and others.[18] Wine (Santome, Itupeva, São Paulo, Brazil) and an alcoholic solution of tobacco *(Nicotianatabacum L.)* were used as chromogen agents.[21,22] To prepare the solution, a portion of 80 g of dry tobacco was mixed and chopped into slices of 7 mm (maximum) in 100 mL of alcohol 54°Gay-Lussac. The solution was allowed to rest for 24 hours in a closed container. After this time, the solution was filtered to remove the solid portion of tobacco and then stored in a capped bottle under refrigeration at 5°C until use.

To evaluate the effect of hydrogen peroxide bleaching on the wine chromogens, 1.27 mL of wine, 1.27 mL of hydrogen peroxide to 50% (Cosmochemistry, Barueri, São Paulo, Brazil), and 1.20 mL of ultra-pure water (type 1) were mixed in a glass cuvette with an optical path of 10 mm and a total capacity of 4.0 mL (G4, Biocell Buckets, Taboao da Serra, São Paulo, Brazil), resulting in 16.97% final peroxide concentration. For the tobacco, 1.0 mL of the tobacco solution, 1.61 mL of hydrogen peroxide 50%, and 1.20 mL of ultra-pure water were mixed, resulting in 21.12% final peroxide concentration. The pH of this mixture was measured with a pH meter (SevenMulti, Mettler Toledo, Schwerzenbach, Switzerland), equipped with an electrode (Inlab Viscous, Mettler Toledo), which was calibrated using solutions with pH 4.01 and 6.86.

For the final adjustment on different pH values, a portion of the ultra-pure water in the mixture was replaced by a 3.3 M HCl or 2.5 M NaOH solution. The final pH of the mixture was set at 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, and 9.0. The final concentration of hydrogen peroxide solution into the wine mixture was 20% (w/v) and of the tobacco mixture was 25% (w/v).

A custom-made device for adjustment in the spectrophotometer was used at the time of color reading, so that three sides of the cuvette were covered with a standard white background, which had the chromaticity coordinates $L^* = 93.10$, $a^* = -1.27$, and $b^* = 4.95$ (Leneta, The Leneta Co, Mahwah, NJ, USA), as shown in Figure 1.

The reading frame of the spectrophotometer was adjusted to contact the front part of the cuvette. The entire set was covered with a dark chamber adapted to the shape of the spectrophotometer so that the reading of the solution's color was held in the dark (Figure 1). The apparatus was adjusted to the D65 standard illuminant, small area view mode with the specular component included. The observer angle was set to 2° and ultraviolet emission at 100%. Three consecutive measurements were carried out, obtaining an average of L* a* b* chromatic coordinates. The L* value is a measure of lightness from 0 (perfect black) to 100 (full white), the a* axis represents red (positive a*) to green (negative a*), ranging from +120 to −120, and the b* axis represents yellow (positive b*) to blue (negative b*), also ranging from +120 to −120.[23] The data were analyzed by the software Spectramagic NX (Konica Minolta Inc, Tokyo, Japan).

An initial reading of the L*a*b* chromatic coordinates was performed immediately after mixing and then after 10 minutes for wine and 20 minutes for the tobacco solution. The waiting time between the first and second readings of the spectrophotometer was determined based on previous tests, performed in order to determine a significant color variation, that is, $\Delta E \geq 3.0$, which is visually perceptible.[24,25] The variation in the color of solutions containing the chromogen agents after bleaching can be seen in Figure 2. During the pilot study, it was observed that it takes more time for a significant lightening of the tobacco solution compared with wine, and because of this, a longer time was set for it.

All procedures were performed at 24°C (±1°C) and a relative humidity between 30% and 36%. After the

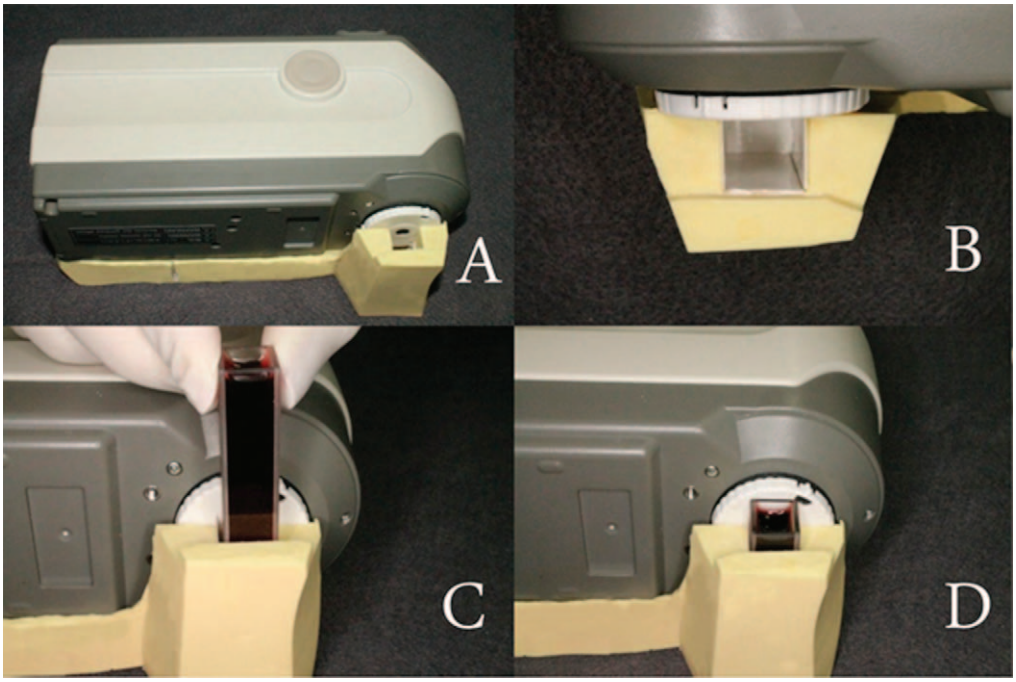

Figure 1. *(A): Support for the spectrophotometer with the device positioned. (B): View of the approximate area where the cuvette holder was placed. The walls are lined with white standardized background (Leneta). (C): Cuvette with stained solution being positioned in support. (D): Cuvette in position and in contact with the spectrophotometer's frame reading.*

respective periods, the solutions became clearer, indicating that hydrogen peroxide promoted the oxidation of chromogens (Figure 2). Then, a final color reading was performed, and the software calculated the values of the general color variation (ΔE) using the formula $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$, according to the instructions of the Commission Internationale de l'Éclairage.[23]

The data were analyzed using statistical parametric tests of one-way analysis of variance (ANOVA) and Tukey, with a significance level of 5%.

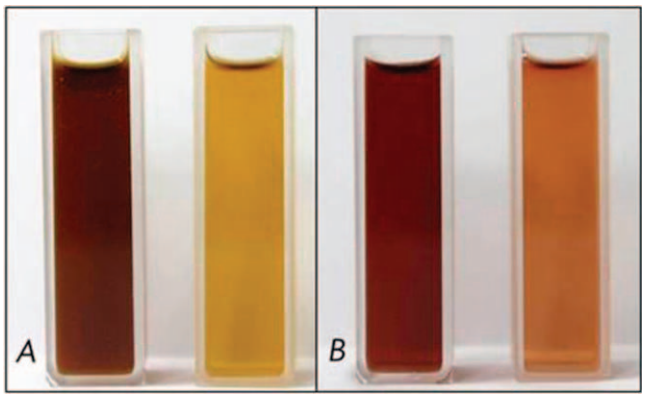

Figure 2. *(A): Tobacco solution at baseline and after 20 minutes of bleaching. (B): Wine solution at baseline and after 10 minutes of bleaching.*

RESULTS

The ANOVA test showed significant differences for color variation considering the different pH of wine (p=0.0001, F=37.61, df=6) and tobacco solutions (p=0.0001, F=281.22, df=6).

Table 1 presents the results of the Tukey test for the wine and tobacco solutions. There were no significant differences in the bleaching effect, comparing the wine solutions with pH between 3 and 5. For solutions with pH between 6 and 8, the whitening effect was significantly higher, and the solution with pH 9 presented the greatest color change. The mean ΔE values for the wine solutions are shown in Figure 3. Among the tobacco solutions with pH values between 3 and 5, no significant variation was observed in the bleaching effect. From pH 6 onward, the alkalinity significantly increased the bleaching effect of hydrogen peroxide on the colored solution. Figure 4 shows the mean values of ΔE for the different pH values of tobacco solution.

DISCUSSION

Wine is produced from grapes and contains anthocyanin, which is responsible for the reddish and purple pigmentation in fruits and vegetables.[26,27] The carbon chain of this chromophore is shown in Figure 5A.[28] The polyphenols contained in tobacco

Table 1: *Mean Values and Standard Deviation (SD) of the Parameter ΔE for Wine and Tobacco Solutions*

Group	Wine[a]	Tobacco[b]
pH 3.0	9.09 (2.40)a	1.25 (0.91)A
pH 4.0	9.07 (0.75)a	1.07 (0.12)A
pH 5.0	8.95 (0.55)a	1.84 (0.27)A
pH 6.0	12.20 (1.17)b	3.33 (0.24)B
pH 7.0	4.12 (0.54)b	6.22 (0.90)C
pH 8.0	12.82 (1.22)b	8.57 (0.84)D
pH 9.0	18.50 (3.41)c	11.23 (1.18)E

[a] *Different lowercase letters mean significant differences among the groups for wine solutions ($p<0.05$).*
[b] *Different capital letters mean significant differences among the groups for tobacco solutions ($p<0.05$).*

plants play an important role in physiological processes of the plant metabolism. They are considered vital components of tobacco, especially because of their contribution to the sensory properties of taste, color, bitterness, and antioxidant properties. One of the main polyphenol components of tobacco is rutin,[29-31] whose structure is shown in Figure 5B.[31] According to the measured values of ΔE, it is evident that the chromogen solution present in tobacco is more resistant to oxidation by hydrogen peroxide than the one present in wine. Such a difference may be due to the chemical structure of these molecules.

The factors that change the decomposition of hydrogen peroxide include impurities, temperature, pH, and metal ions from solutions.[8,10,32] As it concerns pH, the results reported in our study, directly measuring the color of the solution containing the chromogen, clearly show that the efficacy of

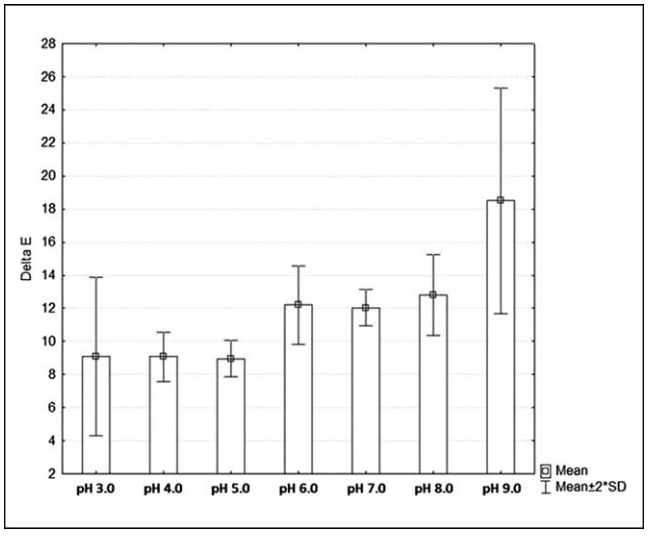

Figure 3. *Mean ΔE values in relation to the increase in the pH of wine.*

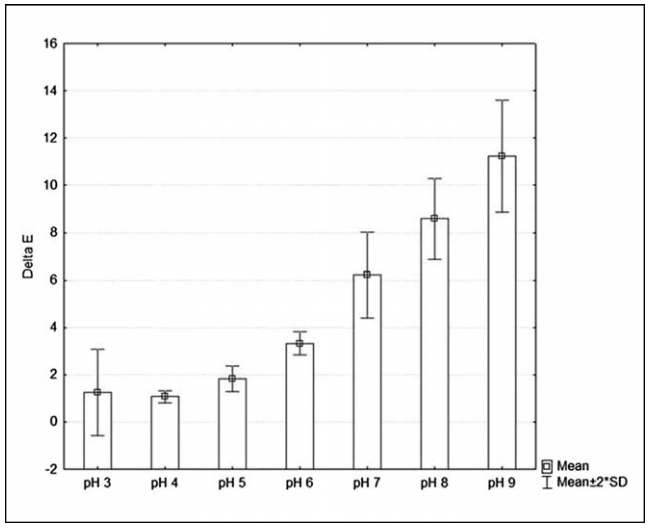

Figure 4. *Mean ΔE values in relation to the increase in the pH of the tobacco.*

hydrogen peroxide bleaching is directly proportional to the pH of the solution. The significant increase in bleaching outcomes occurs from pH 6.0, with maximum effectiveness achieved at pH 9.0, both for wine and tobacco solutions; thus, the null hypothesis was rejected.

Similar results were obtained in a previous study investigating the chemical activity of hydrogen peroxide on chromogens of a tea solution, by measuring the absorbance of the solution as a

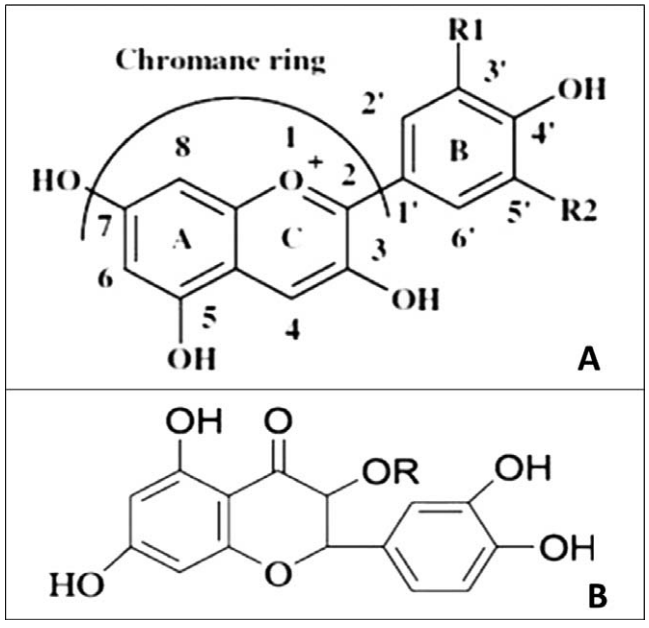

Figure 5. *(A) Chemical structure of the basic anthocyanin, wine chromogen. (B): Chemical formula of rutin, tobacco chromogen.*

function of time. The authors found an increase in the speed of the reaction between pH 8 and 9.[18] In studies with human teeth, an increase in the whitening efficacy of bleaching gels with alkaline pH was also observed, when compared with acid gels.[19,33] In addition, alterations in enamel surface properties were investigated, and there was no evidence of erosion when gels with neutral or alkaline pH were used,[19] different from what occurred in studies on dental tissues in acidic environments.[8,34]

Chen and others[8] investigated hydrogen peroxide decomposition using a system in which two bottles were connected and sealed. They showed that the decomposition was faster and more violent when the peroxide was mixed with 20% sodium hydroxide than when mixed with hydrochloric acid and ether. Adbel-Halim and Al-Deyab[11] evaluated the bleaching in cotton fabrics using hydrogen peroxide and concluded that the rate of H_2O_2 decomposition elevates significantly with the increase of the pH from 5 to 11 and that the pH exhibits a large effect on the time required for the complete decomposition of the hydrogen peroxide and for the bleaching effect.

These positive results obtained with the increase in pH values can be explained based on the chemical reactions involved in the process. According to Brooks and Moore,[35] hydrogen peroxide decomposes into H^+ and perhydroxyl (equation 1). The latter leads to the formation of free radicals (equation 2), which are the active species of oxidizing chromogens. Stoichiometric experiments showed that the formation of perhydroxyl ion is influenced by pH; thus, the higher the pH, the more ions are formed, leading to more free radical production. The perhydroxyl anion is the primary key species responsible for the bleaching outcomes. In one experiment with cotton fabric, the degree of bleaching obtained was directly related to the increase in pH and the concentration of perhydroxyl anion in the whitening solution.[35] An increase of oxygen release as a result of the increased pH has also been observed.[36-38]

Another theory is that the traces of heavy metals such as iron, in contact with the hydrogen peroxide, form unstable peroxides or complex per-ions, which result in its decomposition. However, when the pH is high, there is the formation of insoluble iron hydroxide (colloidal hydroxide), which exerts higher catalytic activity for the peroxide decomposition than the complex peroxide or per-ions. With a further increase of pH and excess of alkalinity, these hydroxides are redissolved and release the metal ions, which increase the catalytic activity even more, therefore increasing the bleaching effect.[39]

On the other hand, other studies have shown similar efficacy comparing whitening gels with acid and neutral pH.[6,32] These results might be due to the use of manufactured bleaching gels with different formulations, which may contain other ingredients in the formula, such as thickeners, fluoride, potassium nitrate, and others (depending on the manufacturer), which could interfere with the final result of bleaching in a more expressive way than just the pH.

Many bleaching solutions and gels manufactured in a single bottle, ready for use, are acidic or neutral, in order to make the chemical stabilization of hydrogen peroxide easier, preventing their decomposition. This aims to prevent the formation of oxygen and water inside the bottle, thus enhancing the validity of the product, although its whitening efficacy is suppressed.[8,9,40,41] The high level of hydrogen peroxide stability and its reduced catalytic activity in acid systems were attributed to the lack of an initiator necessary to decompose hydrogen peroxide into free radicals.[41] The perhydroxyl ion, which is supposed to start the decomposition of hydrogen peroxide in alkaline solutions, is present in insignificant amounts in acidic solutions since the ionization of hydrogen peroxide is not favored.

There are also gels on the market in which an alkalizing agent should be mixed with hydrogen peroxide during clinical use, so that it is used with a higher pH,[30] thereby improving the bleaching efficacy and preventing dental surface demineralization.[18] The effects of alkaline pH in soft tissues range from mild irritation to severe ulcers. Thus, during the bleaching treatment, the direct contact between gel and soft tissues must be avoided. The possibility of burns is another reason for increasing attention to the total isolation from soft tissues to the teeth using a gingival barrier or a rubber dam.

These results show the importance of the formulation of bleaching products with a pH higher than 6, to achieve better bleaching outcomes and reduce damage to dental tissue, which would make the treatment more effective and safe. Products that are stored in separate bottles and mixed at time of use seem to be the best option, keeping the peroxide at an acidic pH for stability and immediate alkalinization during clinical use. This can be achieved by a self-mixing syringe, in which the gel is prepared only at the time of use.

The hydrogen peroxide molecule has strong oxidation activity. When in contact with oral tissues, the molecule is decomposed, producing highly reactive free radicals. For dental bleaching procedures, two main techniques are available. The at-home bleaching technique uses hydrogen peroxide gel in low concentrations, up to 10%, in trays or strips applied over the patient's teeth with no special isolation, without major irritative effects on the soft tissues. However, for the in-office technique, highly concentrated hydrogen peroxide gels, up to 38%, are applied over the enamel but with some kind of previous gingival isolation, preventing the gel from contacting the soft tissues. If this contact occurs, a chemical burning is always expected by the oxidative effect of hydrogen peroxide, even in a neutral pH. According to the OECD Guidelines for Testing of Chemicals, related to acute dermal irritation/corrosion, substances exhibiting pH extremes such as <2.0 and >11.5 may have strong local effects, identifying those substances as corrosives to skin or mucosa.[42] If the pH is >2.0 and <11.5, it is assumed as not corrosive or irritative in relation to pH. In our study, the pH range tested was 3-9. Therefore, no irritative effects are expected for a bleaching compound in this range, with the possible aggressive effects related to the oxidative action of hydrogen peroxide by itself.

Clinical application of whitening products with an alkaline pH needs further study since other variables inherent to *in vivo* treatment may represent changes in the bleaching efficacy. This *in vitro* study aimed to investigate the influence of pH as an isolate factor, but it is known that other factors as mentioned above also influence the efficacy of the bleaching solutions. The next step after confirmation of the importance of alkalinity to whitening solutions is to perform tests in extracted teeth and subsequently in clinical applications. Factors such as the influence of high pH on the stability of other components of the formulation, the issue of potential pulp irritation caused by alkalinity, and product stability require further analysis.

CONCLUSIONS

The efficacy of hydrogen peroxide bleaching is directly proportional to the increase of its pH. The significant increase in bleaching outcomes occurs from pH 6.0, with maximum effectiveness achieved with pH 9.0.

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.

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