# OPERATIVE DENTISTRY

July/Aug 2015
Volume 40
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# OPERATIVE DENTISTRY

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



## Clinical Technique / Case Report



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**Editorial**

# Future Excellence — Everyone's Responsibility

Jeffrey A. Platt, DDS, MS, Editor

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How many times have you been told, "Change is hard"? Nowadays, it has become cliché and using that phrase risks turning off an audience. But, consider the following, "Change is hard, and triple change is exponentially harder." I'm taking the risk that this phrase might keep you reading for at least a few lines. There are major areas in dentistry that are undergoing vast changes and the seasoned members of the profession must continue to look for ways to proactively engage them.

Last fall, I was sitting in the American Dental Association's House of Delegates amid a sea of gray and balding male heads. Oh yes, there was a sprinkling of others throughout, but the clear majority fit the description of aging white male. During the Executive Director's comments to the House, Dr. Kathleen O'Loughlin encouraged us all to look for ways to effectively engage the other growing demographics of our profession. To me, that means we need to figure out how to mentor young dentists so that the profession can benefit from their participation in the years ahead. Perhaps there was no other time in the history of organized dentistry or dental academies when this was of more impact than it is now. The people, the very face of dentistry is changing. Yes, there are still the dental families where third and fourth generations enter the profession, but there are growing numbers of women, African-American, Arabic, Asian, Hispanic and others among new graduates who call themselves dentists. This is significant change. We need to learn how to reach out and mentor this diverse group.

Furthermore, this changing demographic is embedded in a time of dramatic educational change. Increasing understanding of the progression of decay and changing patient treatment needs and desires are changing what is taught in dental schools. With the loss of some of the technically demanding restorative options once taught is a threat that should concern us all. Dr. Robert Keene shared the following quote with me. It is from Dr. Fred Eichmiller and is used with permission.

"Darwin taught us that the species that adapts best to its environment survives. Dentistry is no different. Dental education has changed and teaching of foil, castings, and to a large part clinical excellence has been lost in the process, but we can't reverse that. We need to adapt by promoting excellence outside of dental education. We all learned about 10% of the dentistry we eventually mastered in dental school, and the more valuable 90% was learned after. What has worked for promoting gold and clinical excellence has not been the influence of dental education, but rather organized study clubs. We need to promote our art through continuing education and the refinement of skills within study clubs. While I agree that it would be nice to acknowledge educators that promote gold, they are few in number and dwindling in influence. I would much rather we acknowledge those in our study clubs that are having a more profound influence on younger and newer members. What will shape the future of our profession is not better teaching institutions, but better mentors."

Finally, consider the practice environment where these new members of our profession are landing. Like it or not, many of our new practitioners are ending up in practices that look very different from those that were the mainstay of the dental profession in the twentieth century. When my father mentored me in my early years of private practice, he was mentoring me into essentially the same financial approach to running the business. But, if I would have had the educational debt commonly seen today, it would have been very difficult to do what I did; not impossible, just very difficult. Changing priorities and expectations within our graduates is forcing us to find ways to stay in touch with the new members of our profession. This is particularly difficult when corporate employers are providing continuing edu-

cation and mentoring that may be heavily influenced by their different business model.

So, what are we to do? At the core, we must continue to focus on nurturing the desire to treat our patients and our colleagues with a spirit of excellence – whether providing care or working within our professional organizations. There is no better way to nurture such a desire than to reach out and invest in a new practitioner, to encourage learning beyond what is acquired in school. Bring them to an Academy meeting, a local dental society meeting, or invite them to your study club. Many young dentists are hungry for this. Many, even if they practice in a different environment, desire to learn and become involved on a greater scale. Obviously, mentoring in this way is not a new concept, but there are new demands being put on "the old guard" that make it more challenging. Be willing to move out of your comfort zone and be a supportive friend for those who are the changing face of dentistry. Engage them. They need you.

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**Clinical Technique/Case Report**

# Reestablishing Biology, Function, and Esthetics for Fractured, Immature Incisors

NIP Pini • JY Nagata • D Sundfeld-Neto
L Correr-Sobrinho • AdJ Soares • FHB Aguiar
DANL Lima

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### Clinical Relevance

Dental trauma is a challenge in clinical practice and often needs a multidisciplinary approach for a correct diagnosis and restorative therapy. Pulp revascularization and direct resin composite restorations successfully restored anterior teeth fractures with incompletely formed root apices.

### SUMMARY

**A seven-year-old boy with enamel-dentin fractures on both maxillary central incisors presented to the Piracicaba Dental School–UNICAMP seven days after the trauma. At the clinical evaluation, there were no clinical signs of pulp exposure, neither tooth was mobile, and both affected teeth presented a positive response to sensitivity tests and a negative response for percussion and palpation. The radiographic examination showed an undeveloped root and opened apex for both teeth. Indirect pulp capping was performed on the left maxillary central incisor, followed by a direct restoration. After one month, the patient complained of pain in the left central incisor, which responded negatively to sensitivity testing. Pulp revascularization was performed only on this tooth and was followed for 18 months. During this period, the left maxillary central incisor did not recover sensitivity, although radiographic examination showed apical closure, a slight increase in root length, and the formation of a mineralized barrier between the root canal and sealing material. The technique achieved its goal of restoring biological aspects,**

Nubia Inocencya Pavesi Pini, DDS, MSD, PhD student, Piracicaba Dental School, University of Campinas–FOP/Unicamp, Department of Restorative Dentistry, São Paulo, Brazil

Juliana Yuri Nagata, DDS, MSD, PhD, professor, Federal University of Sergipe, Department of Dentistry, Endodontics Area, Lagarto, Brazil

Daniel Sundfeld-Neto, DDS, MSD, PhD student, Piracicaba Dental School, University of Campinas, Department of Restorative Dentistry, São Paulo, Brazil

Lourenço Correr-Sobrinho, DDS, MSD, PhD, professor, Piracicaba Dental School, University of Campinas, Department of Restorative Dentistry, Dental Materials Area, São Paulo, Brazil

Adriana de Jesus Soares, DDS, MSD, PhD, professor, Piracicaba Dental School, University of Campinas, Department of Restorative Dentistry, Endondontics Area, São Paulo, Brazil

Flávio HB Aguiar, DDS, MSD, PhD, Piracicaba Dental School, University of Campinas, Department of Restorative Dentistry, São Paulo, Brazil

*Débora ANL Lima, DDS, MSD, PhD, assistant professor, Piracicaba Dental School, University of Campinas–UNICAMP, Department of Restorative Dentistry, Piracicaba Dental School, São Paulo, Brazil

*Corresponding author: P.O. Box 52, Piracicaba, São Paulo 13414-903, Brazil; e-mail: deboralima@fop.unicamp.br

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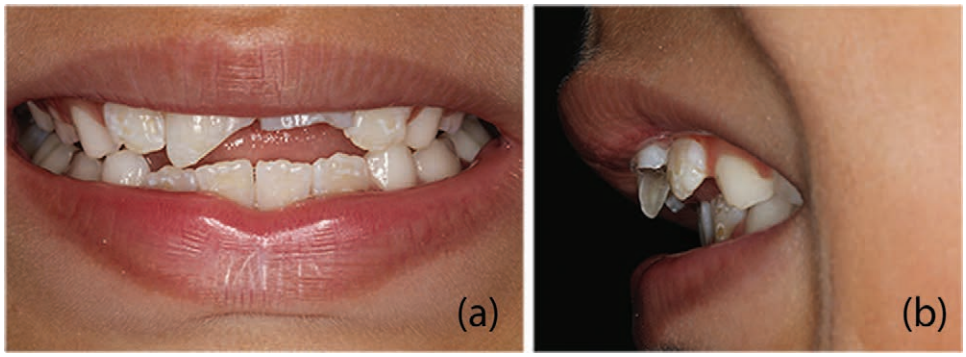


Figure 1. *Initial condition of patient with fracture of both central incisors. (a): Facial view. (b): Lateral view.*

**function, and esthetics of traumatized teeth when using this multidisciplinary approach.**

## INTRODUCTION

Anterior tooth fracture, mainly involving enamel and dentin tissues, is the most frequent dental traumatic injury.[1] Male patients 7 to 15 years old are the most affected,[2-4] and the common etiological factors are falls, sports activities, and traffic accidents.[3] The maxillary incisors are the most commonly involved teeth,[2] especially in patients presenting malocclusion with marked overjet and anterior open bite.[1] In addition, crown fracture of permanent teeth is the most common type of traumatic dental injury.[5] Dental trauma often requires urgent treatment to relieve pain, decrease the exposure of the teeth involved, and restore function, thereby improving prognosis.[6,7]

Dental trauma is of interest to health professionals not only because of its high prevalence but also because of its negative effects on a patient's quality of life.[8] This situation can compromise the emotional state, provide difficulty with eating, negatively influence the appearance of the affected patient, and cause difficulty in speaking clearly.[9,10] Therefore, treating traumatized anterior teeth is integral to restoring normal day-to-day life.[7,11]

The correct diagnosis and evaluation of the case, which frequently involves a multidisciplinary approach, are important to the successful outcome of therapy.[6,7,9] After a traumatic episode, pulp tissue generally loses sensitivity, and a definitive diagnosis must be postponed to avoid unnecessary endodontic treatment. In addition, the diagnosis frequently is more difficult in young patients when considering the incomplete development of the Rachkow plexus, while the patient response may not be reliable because of an immature age. In these situations, a careful diagnosis and delay of endodontic treatment immediately after the trauma is recommended, until the pulp either recovers or presents a definitive sign of deterioration.[12]

The restorative treatment options for dental trauma include direct resin composite restorations, indirect restorations, or reattachment of the dental fragment.[6,13] The choice for treatment is determined by a diagnosis that assesses the extent of periodontal damage, the quality of the remaining tooth structure, and, when applicable, the conservation of the dental fragment.[6,14] The restoration of fractured teeth should reestablish functional and esthetic characteristics, including color, shape, and occlusal contacts.[15] Tooth fragment reattachment is the best option, since it maintains the original characteristics of the teeth.[6,14] In cases in which this treatment is not possible, esthetic materials, such as composite resins, provide excellent results in restoring damaged teeth with minimal sacrifice of additional tooth structure.[1]

Depending on the extension of crown fracture and pulpal damage, endodontic treatment may be necessary. Recently, immature teeth have been treated with pulp revascularization, which has emerged as a promising alternative with more advantages than the traditional treatment of apexification.[16,17] Studies have demonstrated that revascularization promotes root end development and radicular reinforcement, preventing root fractures.[18-20] In addition, this treatment may be performed in one or two sessions without the use of mechanical instrumentation, which could further weaken an immature tooth. This article reports a clinical case in which a multidisciplinary approach, involving endodontics and restorative dentistry, was used to establish a correct diagnosis and to restore maxillary fractured incisors, thereby reestablishing biological, functional, and esthetic factors in a young patient.

## CASE REPORT

A seven-year-old male patient presented to the clinic at the Piracicaba Dental School, State University of Campinas–São Paulo, with a visible fracture (Figure

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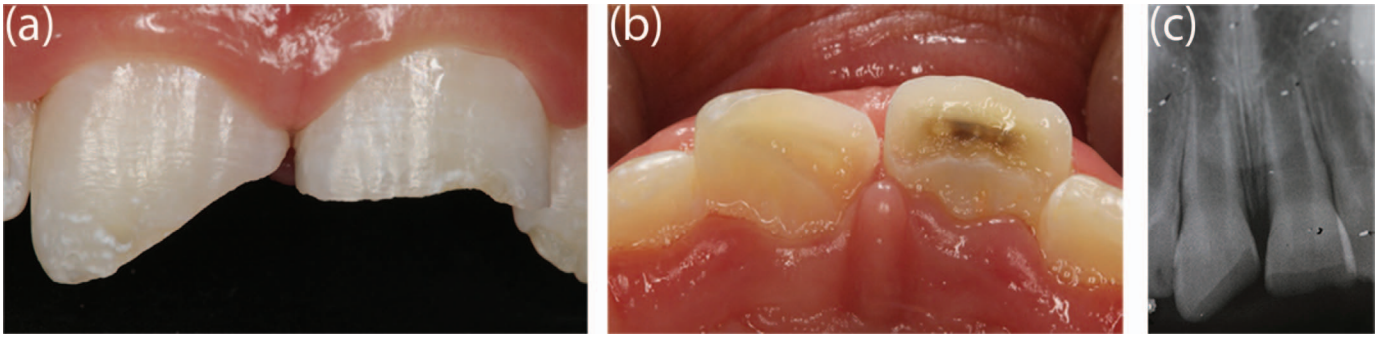


Figure 2. *Clinical (a) and radiographic (b) evaluation of the fractured teeth.*

1) in both maxillary central incisors due to a bicycle accident that happened approximately seven days prior. The patient did not initially complain of pain. After clinical and radiographic examination (Figure 2), the clinician diagnosed a crown fracture including enamel and dentin in both maxillary central incisors without clinical mobility. The patient and his guardian did not save the dental fragments. Clinical examination of the affected teeth determined both to be vital, according to a sensitivity test, and negative for percussion and palpation tests. The radiographic examination showed extensive fracture of the maxillary central incisors, with immature root formation and an open apex, but with normal periapical and periodontal tissues. These results indicated vitality of the damaged teeth, eliminating the need for endodontic treatment at the initial examination. Thus, since there was no pulp exposure, indirect pulp capping of the pulp-dentin complex was suggested for the left central incisor, as this was the least invasive approach.

In the absence of the dental fragments and with partial coronary fracture of the upper central incisors, the restorative treatment was performed using composite resin immediately after the diagnosis. One of the first steps of the direct restoration technique is color selection. This was performed by determining the colors and characteristics of the teeth to be reproduced by the composite resins. In this case, the patient presented with amelogenesis imperfecta, revealing enamel pits and white spots in all of the maxillary anterior teeth, mainly in the incisal third. It was likely that the central incisors were affected too, since the remaining distal incisal angle demonstrated the presence of white stains. These characteristics should be replicated in the restorations to make them as natural as possible and to mimic the restorative material as dental structure. The color was properly selected by inserting a small increment of composite resin on the buccal surface of the tooth, near the region that was to be replaced (Figure 3). The colors selected were A2 and A1 for the medium and incisal thirds of dentin, respectively; Opaque White to reproduce the amelogenisis imperfecta; Translucent White for the incisal third; and Enamel Neutral to reproduce enamel (Amelogen, Ultradent Products Inc, South Jordan, UT, USA).

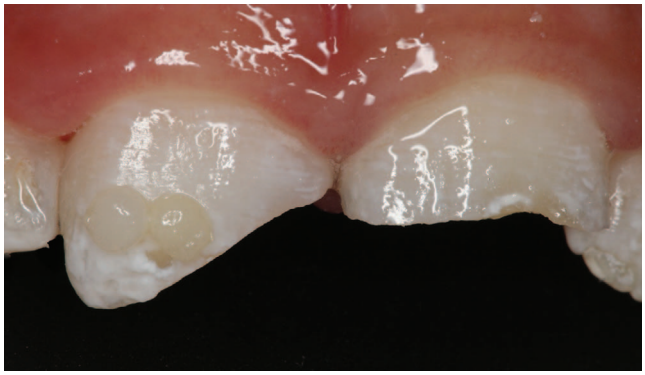

Figure 3. *Shade testing using uncured composite.*

The operatory field was isolated using a rubber dam and stabilized with clamps on the deciduous molars and dental floss in the anterior teeth. The region of fracture was smoothed using a round diamond (1190F, KG Sorensen, Cotia, SP, Brazil) to remove unsupported enamel rods and to enable better adhesion between the restorative material and the tooth structure (Figure 4). As the fracture in the left maxillary central incisor was considered deep, near the dentin-pulp complex, it was protected using a liner. For this procedure, a light-cured calcium hydroxide cement (Ultra-blend, Dentin Shade, Ultradent Products Inc) was applied on the exposed dentin (Figure 5) and light cured for 10 seconds using an LED device (Valo, Ultradent Products Inc). Prior to the restorative procedures, the adjacent lateral incisors were protected using Teflon to limit surface treatments to the central

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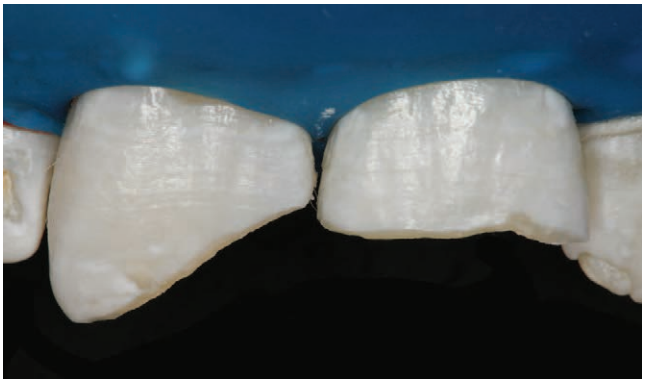

Figure 4. *Isolated operatory field and smoothed enamel margins with a ½ mm bevel.*

incisors. The teeth were etched using 35% phosphoric acid (Ultra-Etch, Ultradent Products Inc) for 15 seconds on dentin and 30 seconds on enamel (Figure 6). Next, the teeth were washed with air/water spray for 30 seconds. After this, the enamel was completely dried with air spray and dentin was covered with sterile cotton to leave a moist substrate to ensure adequate bond to both tissues. Two layers of an etch-and-rinse adhesive system (Single Bond 2, 3M ESPE, St Paul, MN, USA) were applied (Figure 7) and air-dried for 10 seconds, followed by 20 seconds of light curing.

The chosen technique was the stratification technique, which enabled the creation of light and dark variation effects similar to that of the natural dentition. The clinician began the technique on the palatal surface using a polyester matrix (TDV Dental, Pomerode, SC, Brazil) that was stabilized behind the tooth using the clinican's index finger.

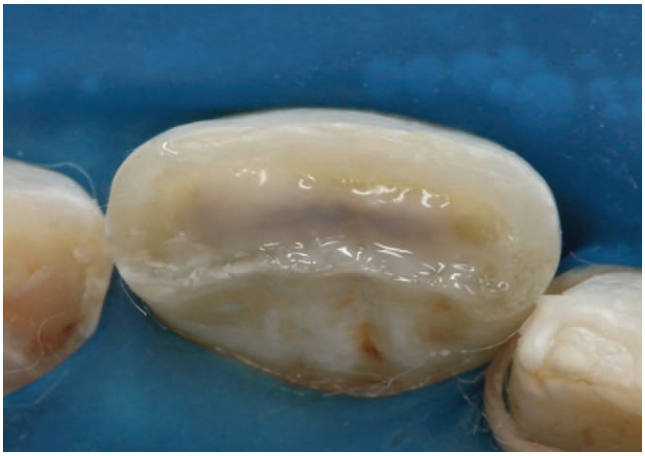

Figure 5. *Left central incisor after pulp-dentin protection covering only the specific pulp horn areas, maintaining enamel margins free of this material to offer less interference with the color matching and potential bond strength.*

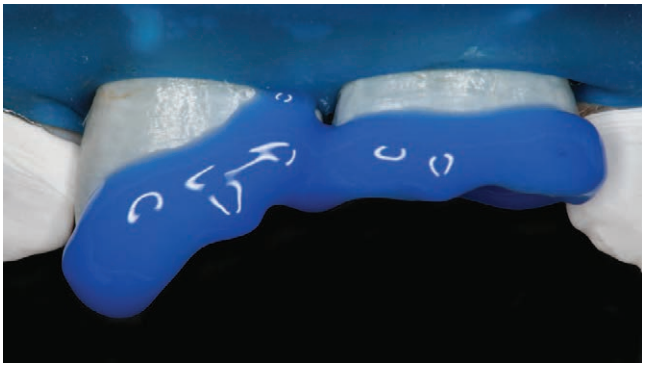

Figure 6. *Conditioning the teeth with 35% phosphoric acid extending beyond the anticipated margin to prevent unattached composite resin.*

With this matrix in position, the first enamel composite resin increment (Enamel Neutral) was inserted on the palatal aspect of the fracture and on the polyester matrix. Light curing was performed with the polyester matrix, clinician's finger, and composite resin in position. The palatal surface of the restoration was then finished (Figure 8). This surface, in both maxillary incisors, was used as the support for the following resin increments. Sequentially, the composite resin selected to reproduce dentin was inserted as indicated earlier, A2 in the middle and beginning of the incisal third (Figure 9a), followed by A1 in the rest of the incisal third (Figure 9b) and into the incisal halo (Figure 9c). Between the dentin and the incisal halo, a more translucent resin was inserted (Translucent White) to reproduce the transparency characteristic of the mamelons (Figure 9c). Small increments of a white opaque resin composite were inserted (Opaque White) to reproduce the amelogenesis imperfecta stains (Figure 10a). Finally, the restoration was finished using a thin layer of enamel composite resin (Enamel Neutral; Figure 10b). All composite resin increments

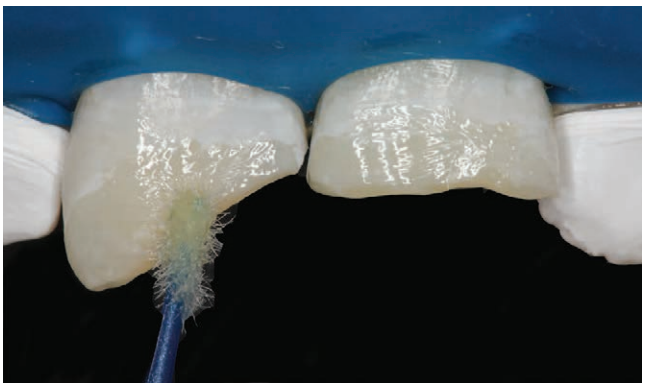

Figure 7. *Applying the adhesive using a microbrush extending beyond the anticipated enamel margins.*

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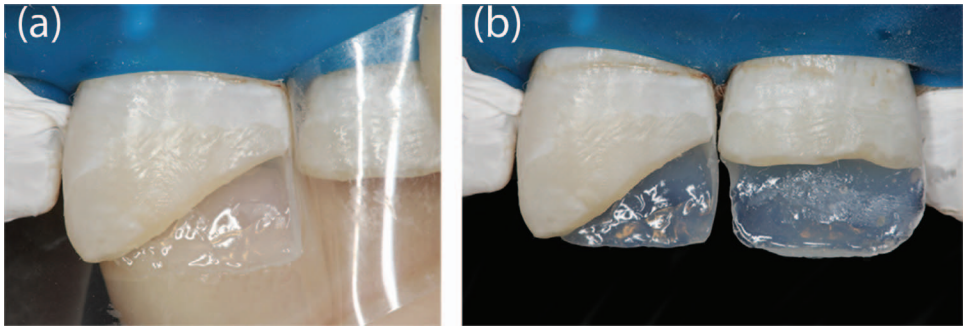


Figure 8. *(a): Construction of palatal surface using the freehand technique. (b): Palatal surfaces of right and left central incisors.*

were light cured with the same LED device for 20 seconds.

After the final light curing, the retainers and rubber dam were removed and the occlusal contacts were checked using carbon paper. As the patient presented overjet, no premature contacts were identified. The patient returned at a later date to have the restorations finished and polished using silicone rubber discs of decreasing grit size (green → yellow → white; Jiffy Polishers, Ultradent Products Inc) followed by a silicon carbide brush (Jiffy Brush, Ultradent Products Inc; Figure 11). After these procedures, the final appearance of the restorations demonstrated the ability of the resin composite materials to reestablish the function and esthetics of the lost dental tissues (Figure 12). In Figure 12a, a slight difference in shade can be noted between the composite resin and the adjacent enamel. Maybe, with the execution of a longer bevel at the enamel margin, this discrepancy could be avoided. However, because of the patient's age and possible future intervention, a more conservative approach was performed with a small bevel of ½ mm.

One month after placing the restorations, the patient presented with pain in the traumatized teeth. A negative result on the sensitivity pulp test associated with pain on percussion revealed an irreversible pulpitis in the left central incisor. Based on the diagnosis, pulp revascularization using triple antibiotic paste (250 mg ciprofloxacin, 400 mg metronidazole, and 50 mg minocycline) was performed.[21] After complete explanation of the procedure, risks, and benefits, informed consent was obtained from the patient's legal guardians to perform pulp revascularization. The cavity access was prepared using a diamond bur (KG Sorensen, Cotia, SP, Brazil), and the root canal was carefully decontaminated with irrigant solutions (6% NaOCl, physiological solution, and 2% chlorhexidine gel). Subsequently, triple antibiotic paste was inserted and left for 21 days. At the second visit, the medication was removed and a manual K-file (Dentsply-Maillefer, Ballaigues, Swiss) was introduced 2 mm beyond the working length to induce bleeding into the canal. A barrier of white MTA (Angelus, Londrina, PR, Brazil) was placed on the blood clot, and the access opening was double sealed with Coltosol (Coltene-Whaledent, Langenau, Germany) followed by composite resin (Amelogen, Ultradent Products Inc). The final restoration with composite resin was performed using the same restorative materials and procedures described earlier.

The pulp revascularization therapy was followed up for 18 months with clinical and radiographic records. The treated tooth did not recover pulp sensitivity, although the patient did not complain of pain on percussion or palpation. Radiographic examination showed apical closure, a slight increase in root length, and the formation of a mineralized

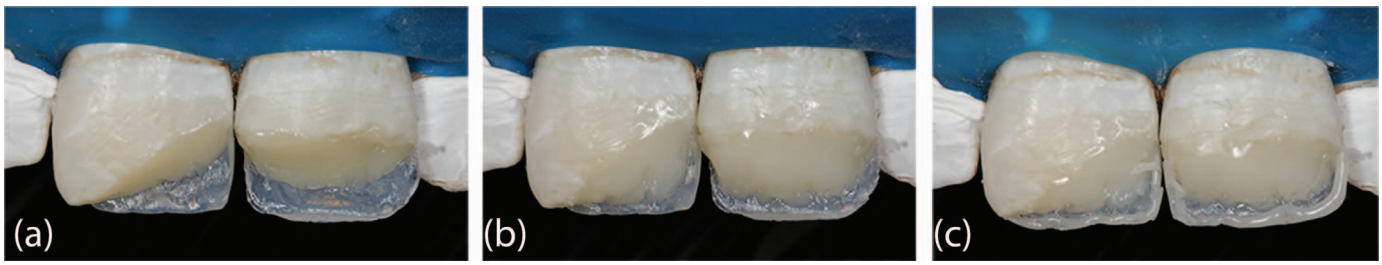


Figure 9. *Dentin stratification of the restoration using composite resin (a): middle third, (b): incisal third, and (c): opaque halo.*

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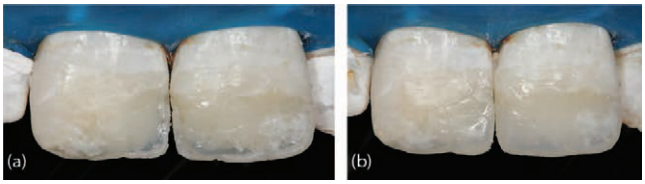


Figure 10. *Enamel stratification of the restoration using composite resin. (a): Translucent and opaque resin in the incisal third and (b): enamel layer.*

barrier between the root canal and the sealing material (Figure 13).

## DISCUSSION

In traumatic injuries, treatment options depend on various factors, including injury type, affected teeth, and time elapsed after trauma.[9] Also, children and adolescents with dental injuries experience a greater impact on daily living than those without injuries.[8] When considering these aspects, it is important to begin treatment of injured teeth as soon as possible.

Crown fractures that involve enamel and dentin are the most common type of dental trauma.[22] Depending on the size of the pulp exposure, the time of contact with saliva, and the macroscopic appearance of pulp tissue, a conservative therapy may be indicated. In these situations, the correct diagnosis is a challenge because of the temporary loss of pulpal sensitivity after trauma.[23,24] The lack of certainty generally leads to conservative treatment and careful follow-up because, although an immediate negative response to pulp testing indicates pulpal damage, this response might not predict pulp necrosis because sensitivity tests assess nerve activity rather than vascular supply, which is ultimately responsible for pulp survival.[25]

Without pulp exposure, indirect pulp capping in cases of dental trauma in younger patients should be encouraged, since it may allow for a more conservative treatment. It is likely that dentin protection aids the pulp-healing process by excluding the exogenous chemophysical factors that may otherwise induce pulp necrosis.[5] In a retrospective study, Wang and others[5] reported that the use of a dentin protector before placing a composite restoration led to a pulp survival of 72% after 36 months of evaluation. In addition, these authors indicate that a delay in treatment up to 10 days, as in this case report, was not a significant factor related to pulp necrosis.

In the present case, considering that the pulp was not directly exposed for both incisors, an indirect pulp cap was performed only for the left maxillary central incisor because its fracture was closer to the pulp chamber based on clinical and radiographic evaluations. Despite this conservative attempt, after one month of follow-up, the pulp tissue was unable to heal itself, possibly because of the intensity of the trauma, and endodontic treatment was necessary. According to Elbay and others,[9] pulp necrosis is the most common posttraumatic complication. Another possible cause for pulpal necrosis is injury to the periodontal supporting tissues,[9] which may have compromised the vascular bundle. In addition, because of the exposure of the dentinal tubules to the oral environment, pathways to the pulp are available for a variety of noxious agents from the oral environment, including bacteria and toxins, which can contribute to pulpal contamination.[5] Moreover, the patient in the current case presented with immature permanent teeth that had thin root walls and open apices. Ideally, these situations require treatment that allows for complete root development.

In this case report, where the tooth fragments were not retrieved, the fractured teeth were restored with composite resin instead of cementing the tooth fragments. The development of adhesive dentistry has introduced a more conservative technique when restoring dental trauma, using the patient's own fragment or using suitable materials, such as composite resins, to restore the fractured tooth. A composite resin restoration should replace the lost dental structures so that they blend with the surrounding structures.[26] In this case, an Amelogen microhybrid resin composite was chosen, since it has adequate properties for esthetic restorations. Da

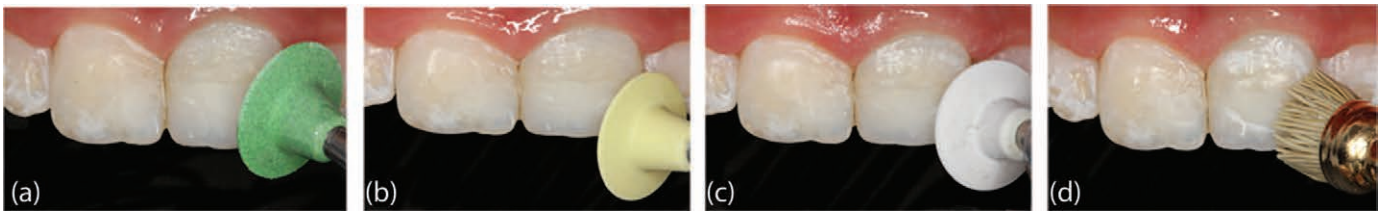


Figure 11. *Finishing and polishing of the restorations using silicone rubber discs (a-c) and a silicon carbide brush (d).*

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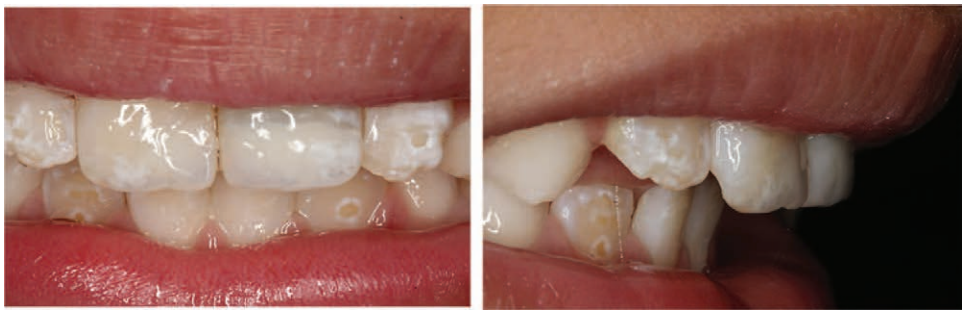

Figure 12. *Final result of restorations. (a): Front view. (b): Lateral view.*

Silva and others[26] reported that this resin composite presents fluorescence values closer to those provided by dentin/enamel, making this material reliable for esthetic restorations. This resin composite also presents with an adequate degree of conversion (around 60%).[27] This parameter is important when restoring teeth, because a low degree of conversion during polymerization may result in a material with unsuitable mechanical and physical properties, such as greater discoloration, degradation,[28] poor wear resistance, and poor color permanence.[29]

The clinical and radiographic follow-ups demonstrated a successful treatment for reestablishing the health of the patient in relation to the biological, functional, and esthetic aspects. Considering pulp revascularization, positive results were demonstrated, since the patient did not complain of pain, and radiographic examination showed apical closure and a slight increase in root length after 18 months (Figure 13). The regenerative treatment has been widely studied and has demonstrated promising outcomes for root end development and radicular reinforcement, which can help prevent root fractures.[30,31] In addition, this treatment provides more advantages when compared with apexification, including shorter treatment time,[32] esthetic rehabilitation, and survival of traumatized teeth. In the present report, no great increase in the thickness of the root walls nor a remarkable increase in root length were observed. Thus, it may be suggested that revascularization is still in progress, and longer periods of follow-up could demonstrate more advanced root development for the current case.

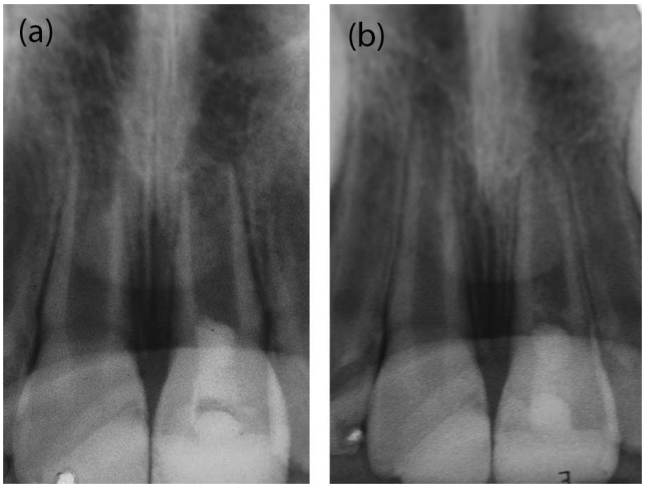


Figure 13. *Radiographs after pulp revascularization (a) and after 18 months (b).*

## CONCLUSION

In the reported case, the patient was completely satisfied with the final results. The treatment achieved its objective of restoring function and esthetics of the fractured anterior teeth in a younger patient.

### Human Subjects Statement

This study was conducted at the Piracicaba Dental School, University of Campinas, Brazil.

### 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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# Maxillary and Mandibular Rehabilitation in the Esthetic Zone Using a Digital Impression Technique and CAD/CAM-fabricated Prostheses: A Multidisciplinary Clinical Report

T Abdel-Azim • A Zandinejad • M Metz
D Morton

### Clinical Relevance

Multidisciplinary treatment planning is an integral part of contemporary dental practice. Collaboration among dental team members is vital to be able to meet the growing esthetic demands of today's dental patients. This involves the use of innovative techniques to accomplish treatment objectives.

*Tamer Abdel-Azim, DDS, University of Louisville School of Dentistry, Oral Health & Rehabilitation, Louisville, KY, USA

Amirali Zandinejad, DDS, MSc, University of Louisville School of Dentistry, Oral Health & Rehabilitation, Louisville, KY, USA

Michael Metz, DMD, MSD, MS, MBA, University of Louisville School of Dentistry, General Dentistry & Oral Medicine, Louisville, KY USA

Dean Morton, BDS, MS, University of Louisville School of Dentistry, Oral Health & Rehabilitation, Louisville, KY, USA

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

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## SUMMARY

**Interdisciplinary treatment planning is necessary in certain clinical situations to optimize esthetic treatment outcomes. Patients presenting with severe wear of their anterior teeth from iatrogenic influences pose a particularly difficult problem in terms of esthetic treatment planning. Collaboration of practitioners from the disciplines of orthodontics, periodontics, and restorative dentistry is essential for the treatment of patients with complex esthetic dental needs. Careful assessment of clinical situations and corresponding specialty consultations are of utmost importance to achieve**

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**more predictable and esthetic treatment outcomes. The purpose of this clinical case is to report to the readership a novel digital fabrication of computer-aided design/computer-aided manufacturing milled acrylic provisional restorations and final lithium disilicate definitive restorations after orthodontic and periodontal therapy with virtual master impressions, casts, and articulation.**

## INTRODUCTION

Dental wear, or the loss of noncarious tooth structure, results from erosive, attritional, or abrasive processes but could result from any combination of these.[1] Erosion is the progressive loss of tooth structure by chemical processes; it is typically defined by wedge-shaped depressions, often found in facial and cervical areas. Attrition is the mechanical wear caused by opposing dental hard tissues rubbing against one another in close proximity; it typically results from mastication or parafunctional tooth alignment. Abrasion is an abnormal wearing away of the tooth structure by causes other than mastication (eg, toothbrush abrasion).[2]

The number of adults with severe tooth wear increases with age.[3] The prevalence of tooth wear increases from 3% at the age of 20 years to 17% at the age of 70 years, and there is a significant association between tooth wear and age.[3] Erosion is associated with the most tooth wear in conjunction with other reasons, such as attrition. Adolescent males are among the highest-risk groups for erosion.[1] Structural tooth loss on the palatal surfaces of maxillary teeth is evidence of erosion caused by gastric reflux in gastroesophageal reflux disease, and this is a common cause of erosion in middle-aged men.[1,4]

Worn dentition is a multifactorial disease process that has varying etiologies and is caused by factors in addition to erosion. For example, bruxism, clenching, and parafunction combined with tooth erosion may significantly increase tooth wear.[5]

Different treatments indicated for patients with tooth wear depend on the severity of the worn dentition and possible loss of a stable and reproducible vertical dimension. Regardless, the etiology of the wear needs to be identified and eliminated before extensive treatment planning of indirect prostheses. Once the cause is determined, treatment could range from a conservative direct composite restoration to full mouth reconstruction with indirect fixed prostheses.[6]

Today's all-ceramic restorations have the advantage of being esthetic, strong, and biocompatible with hard and soft tissue. Recently, new developments in glass ceramics have made all-ceramic restorations stronger and more durable in functional occlusion, which has resulted in more predictable clinical outcomes.[7] The demand for all-ceramic restorations has increased because of market awareness and patient awareness of toothlike crowns with real-tooth optical properties. For example, IPS e.max, a pressed lithium disilicate glass ceramic that has optimized translucency, durability, and strength for full anatomic restorations,[7] has been used for restoring worn dentition and has been associated with positive clinical outcomes and higher patient satisfaction.[7]

Clinical situations that require optimized esthetic results need to be evaluated thoroughly, and typically a multidisciplinary treatment planning approach is necessary. Of particular concern are smile line, facial geometry, emergence profile, tissue geometry, tooth axis (zenith points), tooth shape (golden rule), and proportionality.[8] Merging orthodontic alignment and required spacing for restorative dentistry are necessary in many cases for a more predictable esthetic outcome.[9,10] Additionally, orthodontic treatment can provide protective occlusal movements to enhance long-term prognosis of all-ceramic indirect prostheses.[9,10]

Soft tissue geometry and height play a very important role in the esthetic outcome of any clinical situation.[8] Therefore, proper soft tissue evaluation and corresponding treatment are indicated for any patient seeking optimal esthetic outcomes. A smile that has excessive keratinized gingival tissue in combination with short teeth compromises dental esthetics.[11] Appropriate diagnosis and lengthening of the soft or hard tissue crown are indicated for an optimal esthetic outcome.[11]

Emerging digital technology has introduced many new aspects to contemporary dental practices.[12] Recently, digital impressions have emerged as an alternative to conventional polyvinyl siloxane (PVS) impression techniques and materials.[12] Clinical evaluations of intraoral digital impressions have shown very promising results.[12] All-ceramic crowns fabricated using chair-side scanners have superior marginal fit and improved proximal contact points than those fabricated using conventional impressions.[13]

This clinical case report will present the step-by-step treatment of maxillary and mandibular teeth in a multidisciplinary fashion using fixed appliance orthodontic treatment, crown lengthening, and all-

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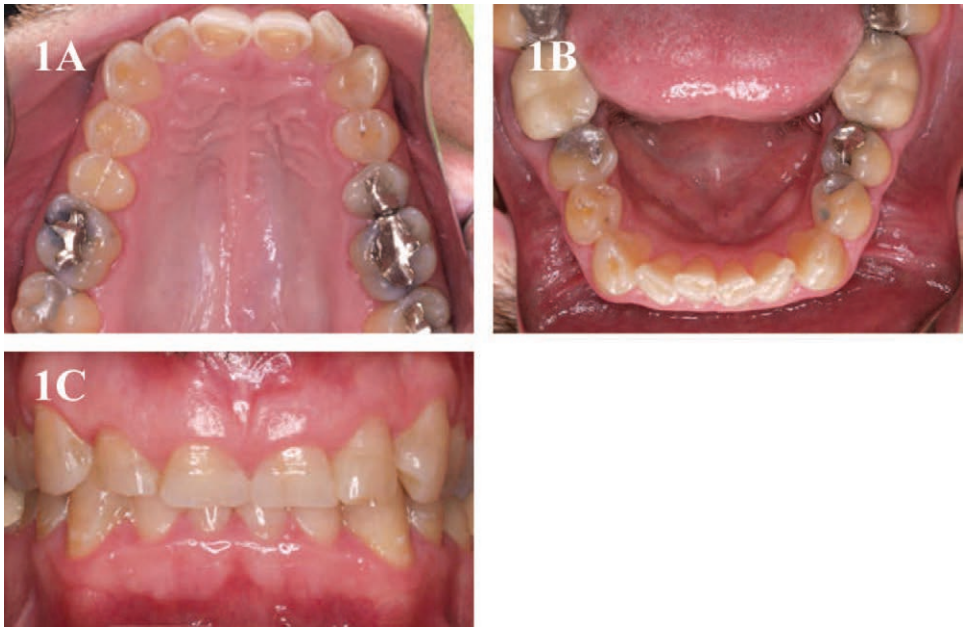


Figure 1. *Preorthodontic treatment. (A): Maxillary occlusal view. (B): Mandibular occlusal view. (C): Frontal view.*

ceramic crowns along with long-term provisional restorations fabricated using a digital impression technique and computer-aided design/computer-aided manufacturing (CAD/CAM) technology. This novel approach bypassed conventional PVS master impression techniques and the need for solid casts and semiadjustable articulation. The creation of a digital impression, occlusal record, and prosthetic fabrication of all-ceramic crowns were performed through the acquisition of digital CAD/CAM files.

## CLINICAL TECHNIQUE REPORT

A healthy 45-year-old man presented to the department of orthodontics for consultation (Figure 1A through C). Initial examination revealed mild maxillary and mandibular anterior crowding as well as excessive anterior overbite/overjet. The patient had moderate to severe lingual wear on his maxillary anterior teeth and moderate wear on the incisal edges of his mandibular anterior teeth due to nocturnal bruxism and daytime clenching. He was wearing a self-prescribed, over-the-counter occlusal splint to slow the wear rate of his teeth. His treatment plan was for comprehensive orthodontic therapy and full-banded fixed orthodontic appliance. He was treated for one year to correct his malocclusion and to achieve sufficient restorative space for indirect prostheses. Of particular importance was reestablishing bilateral canine rise to protect his dentition from excursive interferences during functional movements.

After the conclusion of orthodontic treatment (Figure 2A through C), the patient was referred to the department of oral health and rehabilitation for restorative evaluation and treatment planning. His maxillary and mandibular anterior dentition exhibited moderate to severe wear due to parafunction. His posterior maxillary and mandibular teeth showed no signs of wear, although he had multiple amalgam and direct composite restorations. Although interocclusal space became available after orthodontic treatment, crown-lengthening surgery was indicated to increase abutment height and improve the esthetics of the final restorations.

The treatment plan, which was discussed with and accepted by the patient, was to restore the maxillary and mandibular teeth from the first premolar to the contralateral first premolar with single-unit all-ceramic full coverage restorations preceded by a crown-lengthening procedure of the aforementioned teeth. He understood the benefits and risks associated with the proposed treatment options, and treatment consent was obtained.

Diagnostic impressions were made with irreversible hydrocolloid impression material (Jeltrate Fast Set, Dentsply Caulk, Milford, DE, USA), and solid casts were made from type III dental stone (Orthodontic Stone, Whip Mix Corp, Louisville, KY, USA). Face-bow transfer and maxillomandibular relationship were obtained and used to articulate the diagnostic casts on a semiadjustable articulator (Hanau 2240, Whip Mix Corp). A diagnostic wax-up was completed by a dental laboratory with white-

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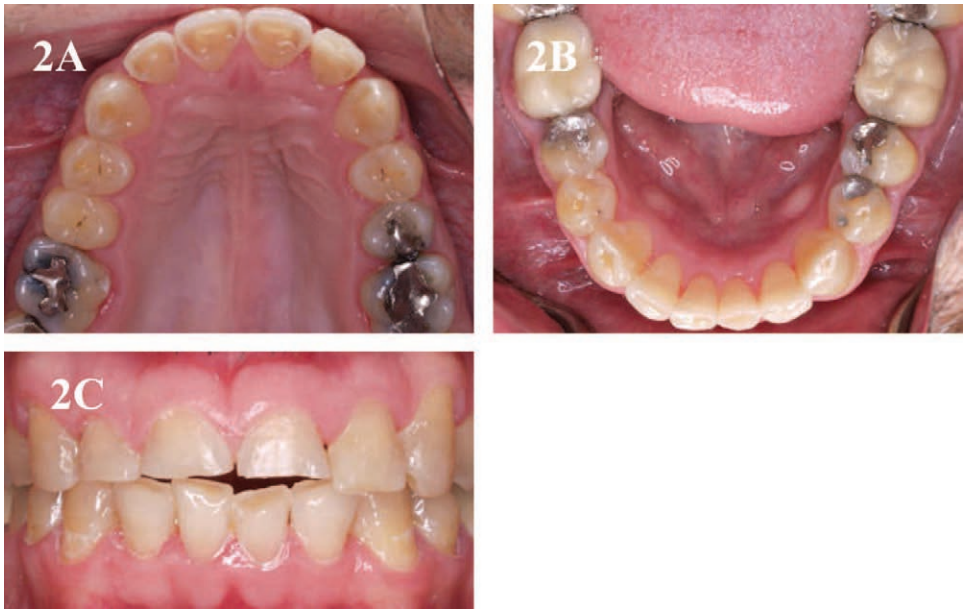


Figure 2. *Postorthodontic treatment. (A): Maxillary occlusal view. (B): Mandibular occlusal view. (C): Frontal view.*

colored wax (Life-like presentation wax, Whip Mix Corp) (Figure 3) and then duplicated. Vacuum-formed material (0.030" Coping Material, Keystone Industries, Cherry Hill, NJ, USA) was used to fabricate a surgical template for crown lengthening, a preparation guide, and a provisional stent.

The crown-lengthening procedure was performed on the facial and palatal/lingual aspects of the abutment teeth using a disposable surgical scalpel blade No. 15 (MDS15215, Medline Industries, Inc, Mundelein, IL, USA) according to the previously mentioned surgical template (Figure 4A,B). Osseous recontouring was accomplished using a surgical handpiece (WS-56E, W & H Dentalwerk, Bürmoos, Austria) and a surgical round bur (Brasseler USA, Savannah, GA, USA). The gingival tissues were sutured using 4(0) interrupted black silk suture (Ethicon, Sommerville, NJ, USA).

After a healing period of eight weeks, maxillary and mandibular abutment teeth preparations were completed according to the vacuum-formed preparation guide under local anesthesia (Septocaine [Articaine HCl 4%], Septodont, Lancaster, PA, USA) with a diamond rotary cutting instrument (Fine Diamonds, Round End; Brasseler USA, Savannah, GA) (Fig. 5). The provisional restorations were fabricated using the vacuum-formed provisional stent and autopolymerizing bis-acryl composite resin Vita shade A1 provisional material (Integrity, Dentsply Caulk, Milford, DE, USA) and cemented with a resin-based temporary cement (TempBond Clear,

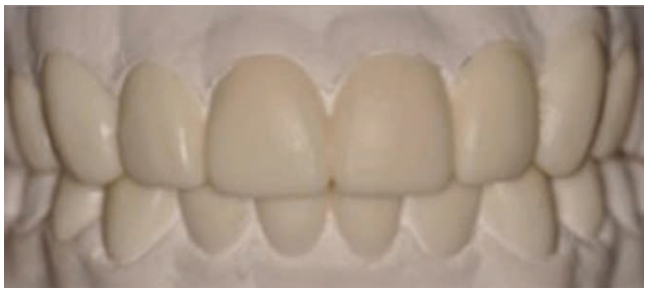

Figure 3. *Pretreatment wax-up (front view).*

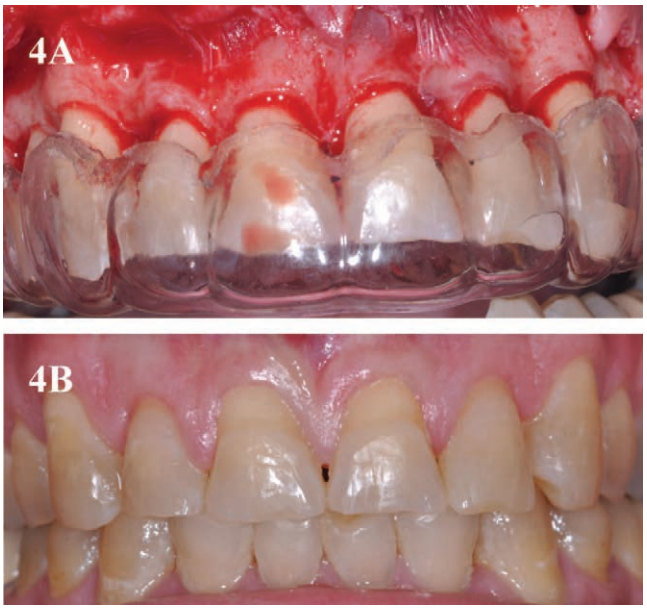


Figure 4. *Periodontal crown lengthening. (A): Crown-lengthening surgery with surgical template. (B): Gingival tissue six weeks after surgery.*

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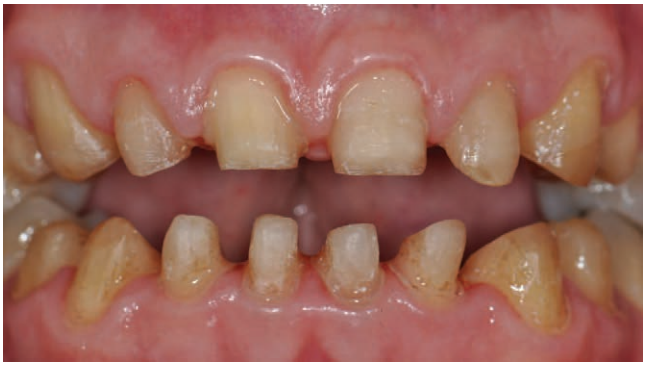

Figure 5. *Abutment teeth preparations (front view).*

Kerr Corp, Orange, CA, USA). The temporary restorations were placed in function for four weeks to allow the patient to evaluate the esthetics, form, and function.

Soft tissue management during the definitive impression was accomplished using a retraction paste (3M ESPE retraction capsule, 3M ESPE, St Paul, MN, USA). The definitive impressions and occlusal record registrations were made using a chairside intraoral digital scanner (iTero, Cadent Ltd, Carlstadt, NJ, USA). The clinician was prompted by the scanner software to capture the prepared abutment teeth and the remaining dentition in a series of occlusal, facial, lingual, and interproximal scans. A virtual interocclusal record was captured with the posterior teeth in maximum intercuspation. The completed scan data were then transmitted to the manufacturer (Cadent, Carlstadt, NJ, USA) for refinement. The data files were subsequently transmitted electronically to a commercial dental laboratory to mark the crown margins and to perform virtual trimming of the margins on the digital image. The digital images of maxillary and mandibular casts were virtually articulated according to the interocclusal record captured during the definitive impression.

The digital file with identified margins and virtual trimming (Figure 6) was approved by the clinician and transmitted to the dental laboratory, which then downloaded the file to the laboratory-based CAD/CAM system (Straumann CARES, Straumann USA, Andover, MA, USA) for design of the definitive restoration.

Individually milled provisional crowns were fabricated to confirm the impression accuracy, the maxillomandibular relationship, and patient satisfaction with the proposed esthetics. The provisional restoration design (Figure 7A) was transmitted to a remote milling center for fabrication (Straumann

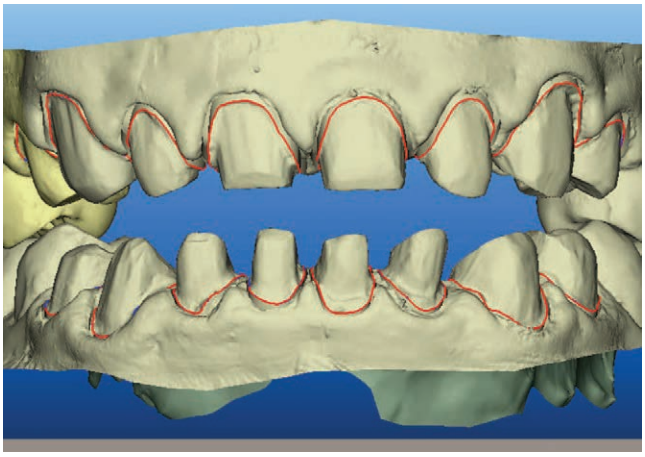

Figure 6. *iTero scans with margins identified (frontal view).*

USA, Arlington, TX, USA). The individually milled provisional crowns were fabricated with polymethyl methacrylate–-based acrylate resin using Vita shade A1 (Polycon ae; Straumann USA). Canine-guided occlusion was designed for the provisional restorations. The proximal and occlusal surfaces were evaluated using articulating paper (AccuFilm, Parkell Inc, Edgewood, NY, USA), all necessary adjustments were made with a low-speed dental handpiece, and the restorations were cemented with resin-based temporary cement (TempBond Clear; Kerr Corp) (Figure 7B,C). No marginal discrepancies were noted with the provisional restorations, which verified the accuracy of the proposed marginal fit. The patient followed the prescribed oral hygiene regimen for home care and was scheduled for a follow-up appointment three weeks after insertion. At the follow-up appointment, the patient was satisfied with the proposed esthetic color and contours of the provisional restorations and reported no functional interferences.

Screenshots of the completed design of the definitive all-ceramic restorations were sent electronically from the dental laboratory to the patient and clinician for review and subsequent approval. The approved design was then sent to the remote milling center for fabrication (Straumann USA). Individual full-contour lithium disilicate restorations (IPS e.max CAD, Ivoclar-Vivadent, Amherst, NY) were milled using Vita shade A1, high-translucency ceramic blocks. High-translucency blocks were chosen because of their "normal" dentin stump shade to be used for optical properties and natural shading. After assessing occlusion and the contours of the crowns, minimal adjustments were made with rotary cutting instruments using a low-speed dental handpiece. Upon completion of all adjustments, the

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Figure 7. *iTero scanned and milled acrylic provisional appliance. (A): iTero CAD of provisional restorations. (B): Frontal view of provisional restorations. (C): Patient's smile after treatment.*

adjusted areas were polished and made ready for final cementation. Canine-guided occlusion was achieved for the definitive crowns.

A high-translucency try-in paste was used to evaluate the dentin stump shade's optical properties and select the final resin luting color. The restorations were verified and luted with a high-translucency, self-adhesive, autopolymerizing resin cement (SpeedCEM, Ivoclar-Vivadent) (Figure 8). The residual cement was removed and postinsertion instructions were given to the patient. The patient was then enrolled in a hygiene program with a six-month recall interval.

## DISCUSSION

Tooth wear is difficult to treat. It often presents at an advanced stage and with multiple underlying factors, which complicates its diagnosis and necessitates a multidisciplinary treatment approach. The workflow in this current case report presented a novel method for digital impression capture using a chairside intraoral scanner and electronic data transfer to the dental laboratory. This serves to facilitate the digital design of the restoration between the clinician, dental technician, and restoration manufacturer at a centralized production facility.

The digital design of the provisional and definitive restorations can be advantageous in allowing the CAD software to replace the wax-up procedures for individual teeth that were traditionally done by the dental technician. To remove the waxing and casting/pressing techniques cuts down on human error and the lengthy time required to make simple contour changes. Electronic mail was used to facilitate communication with the patient, which gave him the opportunity to play an active role throughout the critical phases of the CAD design. The process of controlling the soft tissue during the scanning phase is extremely technique sensitive. Therefore, the operator must provide an uncontaminated scanning surface for the iTero scanner to properly identify the preparation margins. Many times the technique sensitivity for the iTero scanner is less forgiving than traditional PVS impression materials. Obtaining clear and visible margins through proper tissue retraction and adequate isolation are paramount for accurate data acquisition by the scanner as no impression material is introduced into the sulcus, unlike the case with conventional impression techniques. Also, operator experience in orienting the scanning module (wand) and the need to capture sufficient data points are essential.

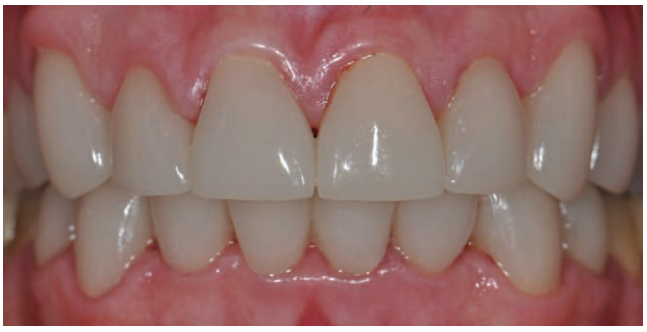

Figure 8. *Frontal view of iTero Milled IPS e.max definitive restorations.*

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## SUMMARY

This clinical report presented a multidisciplinary treatment protocol for treatment of excessive anterior teeth wear associated with parafunctional habits. It emphasized the collaboration between practitioners in the fields of orthodontics, periodontics, prosthodontics, and dental technology to achieve an optimal esthetic outcome for the patient. Fabrication of the milled provisional and definitive all-ceramic restorations was accomplished through a completely digital approach. Long-term milled provisional restorations were used to confirm esthetics and digital articulation. The use of CAD/CAM definitive restorations that were identical to the approved designs of the provisional restorations ensured a predictable clinical outcome and patient satisfaction, while eliminating dental laboratory errors and additional steps and, thus, maximizing efficiency. This case report presents a novel treatment protocol for CAD/CAM milling of long-term provisional and definitive restorations that did not require the production of physical casts or mounted articulation.

### Human Subjects Statement

This work was conducted at the University of Louisville School of Dentistry.

### 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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# Predictable Restorative Work Flow for Computer-Aided Design/ Computer-Aided Manufacture–Fabricated Ceramic Veneers Utilizing a Virtual Smile Design Principle

WS Lin • A Zandinejad • MJ Metz
BT Harris • D Morton

### Clinical Relevance

This clinical report demonstrates the use of a digital restorative work flow to achieve predictable esthetic clinical outcomes using computer-aided design/computer-aided manufacture–fabricated lithium disilicate ceramic veneers.

## SUMMARY

**The purpose of this case report was to present the use of a contemporary digital photograph–assisted virtual smile design principle, an intraoral digital impression, and computer-aided design/computer-aided manufacture–fabricated lithium disilicate ceramic veneers to treat a patient with esthetic needs in the maxillary anterior region. By using the proposed digital restorative work flow, this case report demonstrated an effective communication pathway between the patient, clinician, and dental laboratory technician. Effective communication can help to achieve a more predictable and satisfactory esthetic outcome.**

*Wei-Shao Lin, DDS, School of Dentistry, University of Louisville, Louisville, KY, USA

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

Michael J Metz, DMD, MSD, MS, MBA, School of Dentistry, General Dentistry and Oral Medicine, University of Louisville, Louisville, KY, USA

Bryan T Harris, DMD, School of Dentistry, Oral Health and Rehabilitation, University of Louisville, Louisville, KY, USA

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

*Corresponding author: 501 S. Preston St., Room 310, Louisville, KY 40292, USA; e-mail: WeiShao.Lin@Louisville.edu

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## INTRODUCTION

Digital dentistry provides dental clinicians and technicians with a new perspective in their daily practice. However, the work flow involved in digital dentistry is significantly different when compared to the conventional treatment protocol in terms of prosthetic design and fabrication. Digital photograph–assisted virtual smile design (digital treatment planning/presentation process),[1-3] digital impression (digital data acquisition),[3,4] and computer-aided design/computer-aided manufacture (CAD/CAM) restorations[5-7] are all gaining popularity in modern clinical dentistry.

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The original intent of standardized film-based clinical photographs was to provide the practitioner with a documentation tool to compare photographic records before and after treatment.[8] Modern digital photography eliminates the delay between image capturing and the development process of previous film-based photography.[9] Additionally, it has been proposed as an effective tool for diagnosis, treatment planning, and communication.[9-11] Presentation and computer design software can be used to evaluate a patient's esthetic needs and utilized to create a virtually designed esthetic treatment plan.[2,3] The smile analysis principles[12-14] are applied in the evaluation process, and the proposed virtually designed esthetic plan can be presented to the patient for immediate feedback and approval.[2,3] The approved virtually designed esthetic plan can serve as an effective communication tool between the patient, clinician, and dental technician throughout the course of treatment. Ideal communication of the entire restorative team can allow for a more predictable clinical outcome.[2,3,11]

The basic work flow for digital impression and CAD/CAM restoration includes the following: 1) capture the intraoral teeth geometry with a scanning device, 2) creation of a virtual definitive cast from captured data and/or optional physical definitive cast for subsequent computer-aided restorations design, and 3) computer-aided restoration manufactured chairside, in a dental laboratory, or at a centralized production center.[15-17] Machineable lithium disilicate ceramic block (IPS e.max CAD, Ivoclar Vivadent, Amherst, NY) became commercially available in 2006 with a partially crystallized blue-violet color. The partially crystallized state allows the block to be milled easier and more rapidly during the CAD/CAM process without excessive diamond bur wear or damage to the ceramic crystal.[18] Although lithium disilicate ceramic was initially available only as a substructure material, it had been suggested for fabrication of full-contour monolithic restorations and/or ceramic veneers. Extrinsic staining and glazing characterization is ideal because of its translucent properties, resin luting characteristics, and shade variabilty.[19-21] This report describes the work flow of a virtually designed esthetic plan, a digital impression, and CAD/CAM-fabricated lithium disilicate veneers to achieve a more predictable clinical esthetic outcome.

## TECHNIQUE DESCRIPTION

A 45-year-old Caucasian female presented to the dental clinic (Dental Associates, School of Dentistry, University of Louisville, Louisville, KY) with concerns of her smile, including existing composite resin restorations on her maxillary anterior teeth. The patient reported that her maxillary anterior teeth had been restored with direct composite resin multiple times throughout the years. The patient consented to a comprehensive treatment plan to include lithium disilicate ceramic veneers for her maxillary anterior teeth (canine to canine) for a better esthetic outcome. A periodontal soft tissue recontouring surgery was proposed to the patient for the improvement of the soft tissue architecture; however, the patient declined the surgical proposal after full disclosure of the proposed esthetic benefits.

Diagnostic casts were made with irreversible hydrocolloid impression material (Jeltrate Alginate, Dentsply Caulk, Milford, DE) and poured with type III dental stone (Buff Stone, Whip Mix, Louisville, KY). A face-bow transfer and maxillomandibular relationship were obtained and used to articulate the diagnostic casts in a semiadjustable articulator (Hanau Modular Articulator System, Whip Mix). The length and width of the right maxillary central incisor were measured on the diagnostic cast. The measurements were recorded as 10.5 mm and 8 mm, respectively. The desired midline and incisal edge positions for central incisors were determined during the intraoral examination. After a thorough examination, the desired midline was determined to be shifted to the left by 0.5 mm, and the length of central incisors was determined to be increased by 1 mm. Digital photographs were taken showing the patient's frontal smile view (Figure 1A) and retracted intraoral view. The retracted intraoral digital photograph was taken with contraster (PhotoMed International, Van Nuys, CA) showing the maxillary anterior teeth (Figure 1B). The clinical digital photograph with intraoral frontal view was then imported into the presentation software (Keynote iWork, Apple, Cupertino, CA). A ruler was digitalized as an image file in JPEG format with a scanner (All-in-One Printer, Hewlett-Packard, Palo Alto, CA). The digital ruler file was then imported into the presentation software (Keynote iWork) on the same slide as the clinical digital photograph. The digital ruler was then resized, while maintaining the height-to-width ratio, until it matched the measurements obtained from the diagnostic cast. The calibrated digital ruler was then copied and positioned at the bottom and left side of the slide (Figure 2). The calibrated digital ruler served as an accurate representation of actual teeth dimensions and was used to transfer the virtually designed esthetic plan

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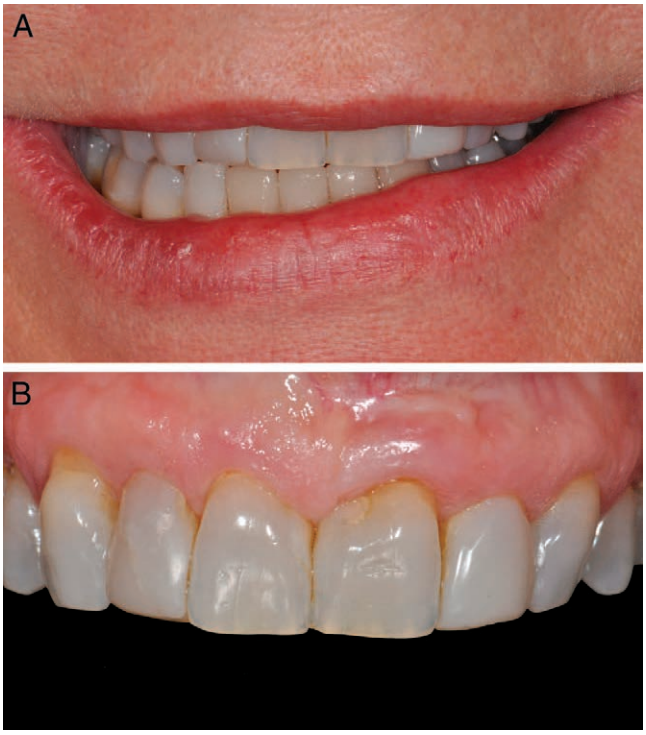


Figure 1. *Pretreatment condition. (A): Smile, frontal view. (B): Intraoral facial view.*

to the diagnostic casts during the wax-up procedure. The desired midline and incisal edge positions for central incisors were also transferred and marked in the presentation software (Keynote iWork; Figure 2).

The "Draw With Pen" tool under the "Shapes" menu toolbar in the presentation software (Keynote iWork) allows users to create custom-shaped objects.

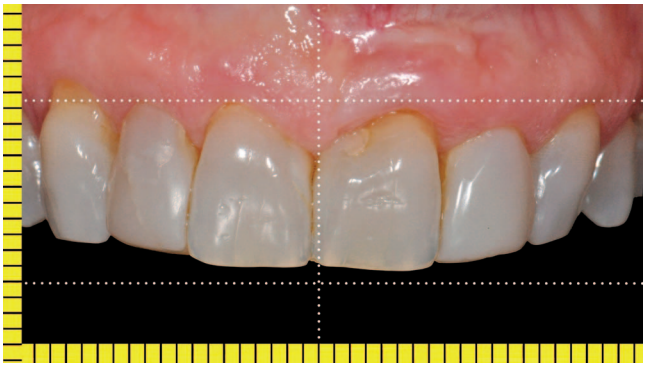

Figure 2. *The screen shot demonstrated the calibrated digital ruler (mm) placed at the bottom and the left-hand side of the screen. The apical horizontal dotted line represented the soft tissue zenith at the right maxillary central incisor. The incisal horizontal dotted line represented the desired positions of maxillary central incisors as determined during the clinical examination. The vertical dotted line represented the desired midline as determined during the clinical examination.*

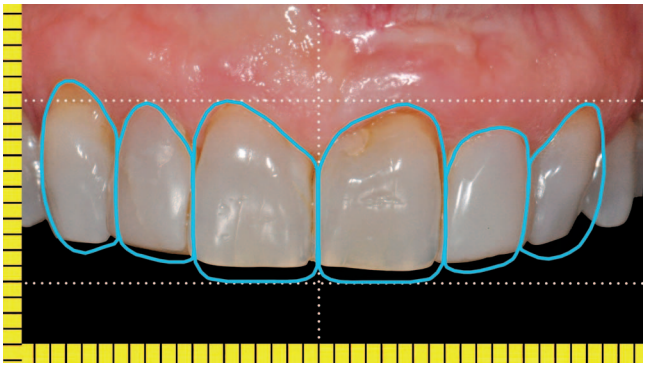

Figure 3. *The customized virtually designed esthetic plan was created in the presentation software.*

This tool was used in this clinical report to draw the desired tooth-shaped objects on the slide. Each tooth-shaped object was drawn and adjusted on the corresponding tooth to mimic the desired definitive restoration outline. The digital photograph–assisted virtually designed esthetic plan was completed with all tooth-shaped objects representing the desired posttreatment tooth contours and alignment (Figure 3). During the development of the esthetic plan, the desired incisal edge positions, midline position, and tooth proportions should be carefully considered. This proposal of a virtually designed esthetic plan was demonstrated to the patient and was modified according to direct feedback from the patient. The "Graphic Inspector" tool under the "Inspector" menu toolbar in the presentation software (Keynote iWork) allowed users to change the color, opacity, and style of the drawing line for the shape objects. The color, opacity, and drawing line of the superimposed tooth-shaped objects in digital photograph–assisted virtually designed esthetic plans can be altered using the "Graphic Inspector" tool to provide patients with different visual perceptions. Additionally, a virtually designed esthetic plan can facilitate communication between clinician and patient. In this clinical report, white color fills with 50% opacity and transparent drawing lines on all the tooth-shaped objects were used (Figure 4). The approved virtually designed esthetic plan was then used to communicate between the clinician and dental technician a virtual esthetic plan–guided diagnostic wax-up. Maxillary diagnostic wax-up was completed using tooth color wax (Diagnostic Wax, Blue Dolphin, Morgan Hill, GA; Figure 5) and then duplicated to fabricate a vacuum-formed preparation guide.

For additional confirmation of the clinical outcome of the virtual esthetic plan–guided diagnostic wax-up, the preparation guide can be fitted onto the abutment teeth prior to the abutment preparation

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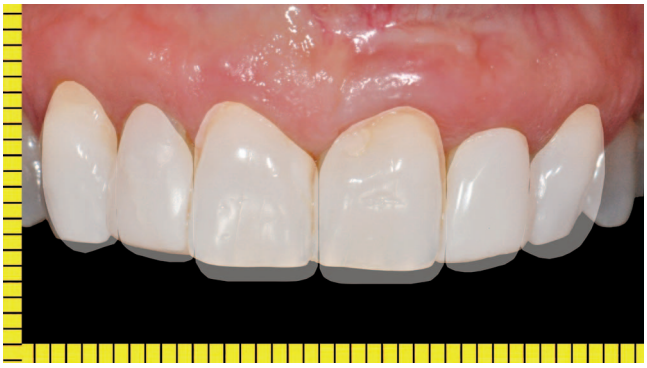

Figure 4. *The opacity of the drawings of the virtually designed esthetic plan can be increased to give the patient different visual perceptions during the decision-making process.*

(Figure 6). Optional trial insertion (mock-up) can be achieved by using autopolymerizing composite resin material (Integrity, Dentsply Caulk) injected into the preparation guide and inserted into the patient's mouth. Maxillary abutment tooth preparations were completed with the use of the vacuum-formed preparation guide under local anesthesia (Xylocaine Dental, Dentsply Pharmaceutical, York, PA) with a diamond-cutting instrument (Fine Diamonds, Round End Parallel, Brasseler USA, Savannah, GA). The double-cord technique was used for soft tissue management during intraoral scanning procedures (Cadent iTero, Cadent Ltd, San Jose, CA). To facilitate the subsequent intraoral digital impression procedures, both cords were left in the soft tissue to retract the soft tissue away from the abutment tooth finish lines (Figure 7). The scanner software (Cadent iTero) prompted a guided scanning sequence with a series of scans at each abutment, followed by additional scans for recording the opposing dentition, and the interocclusal record registration (Figure 8). The interim veneers were fabricated with autopolymerizing composite resin material (Integrity A1, Dentsply Caulk) using the acrylic preparation

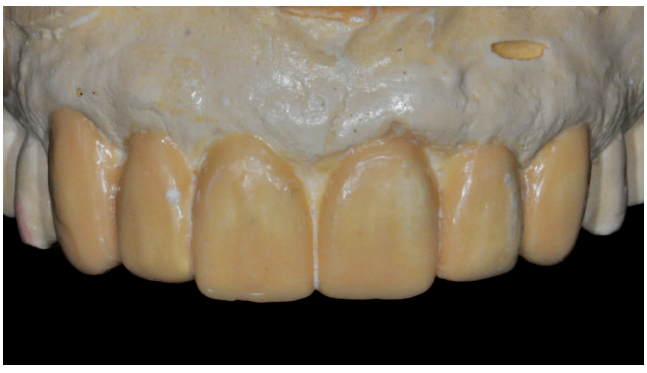

Figure 5. *Completed virtual esthetic plan–guided diagnostic wax-up.*

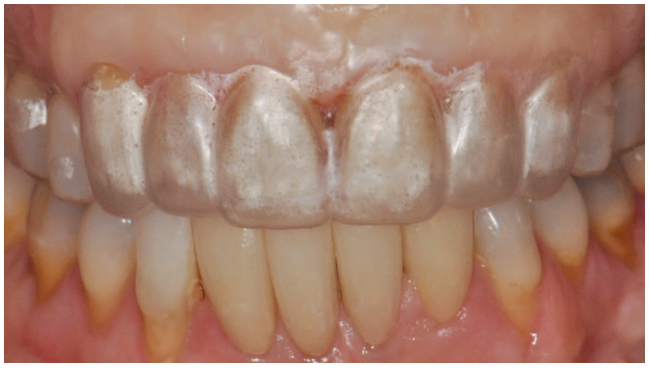

Figure 6. *A preparation guide was fitted on the abutment teeth prior to the abutment preparation for additional visual confirmation of the clinical outcome of the virtual esthetic plan.*

guide. The interim veneers were luted with interim cement (TempBond Clear, Kerr Corp, Orange, CA).

The completed scan data were then transmitted to the manufacturing company (Cadent iTero), undergoing a modeling process. Milled polyurethane definitive casts were fabricated and articulated on a specifically designed hinge articulator (iTero Articulator, Cadent; Figure 9). The milled polyurethane definitive casts and diagnostic wax-up were digitalized with a laboratory-based scanner (Straumann CARES, CS2 scanner, Institut Straumann AG, Basel, Switzerland), and all the scanned data were then imported into the CAD/CAM software (Straumann CARES). The scanned diagnostic wax-up was used as a design template for the definitive veneer restorations (Figure 10). The digital ruler in the CAD/CAM software was used to confirm the dimensions of the designed CAD/CAM restorations to ensure that the definitive restorations shared the same dimensional specifications as the virtual esthetic plan–guided diagnostic wax-up (Figure 11). The approved design was sent to a centralized

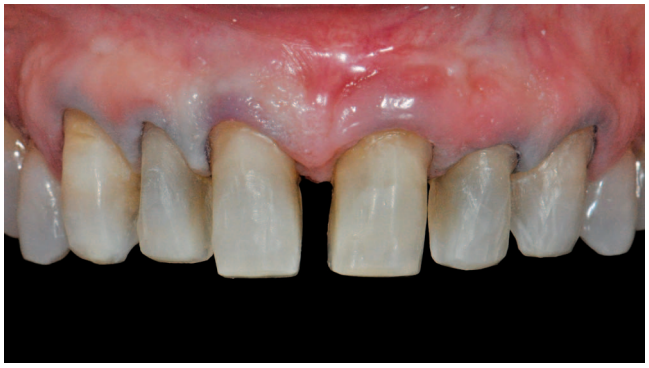

Figure 7. *Completed maxillary abutment tooth preparations. Both cords were left in the periodontal sulcus to complete the soft tissue management for the intraoral scanning procedures.*

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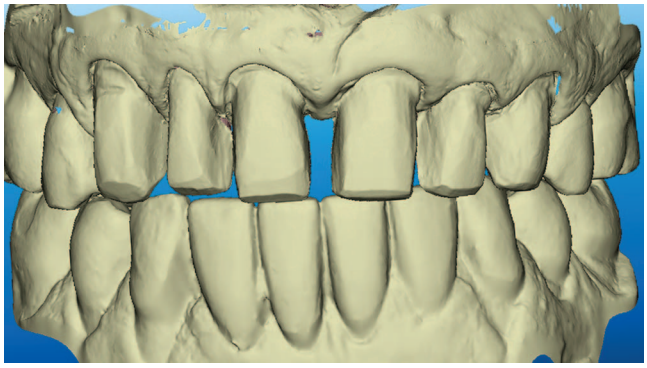

Figure 8. *Completed maxillary definitive impression, mandibular impression for the opposing dentition, and the interocclusal record.*

production center, and anatomic full-contour veneers were milled from machineable lithium disilicate ceramic blocks (IPS e.max CAD, LT, Ivoclar Vivadent). Cutback of the milled veneers was performed, and low-fusing nanofluorapatite glass-ceramic veneering porcelain (IPS e.max Ceram, Ivoclar Vivadent) was used to complete the layering process. Additional characterization and a glazing process for the layered definitive lithium disilicate ceramic veneers was completed with low-fusing nanofluorapatite glass ceramic (IPS e.max Ceram Shades and Essences, Ivoclar Vivadent; Figure 12).

During the insertion appointment, the intaglio surfaces of veneers were etched with 5% hydrofluoric acid gel (IPS Ceramic Etching Ge, Ivoclar Vivadent) for 20 seconds. The etched surfaces were rinsed and dried with oil-free air. Ceramic primer (Monobond Plus, Ivoclar Vivadent) was applied on all treated lithium disilicate surfaces for 60 seconds and then dried with oil-free air. The abutment teeth were etched with 37% phosphoric acid (Scotchbond Etchant; 3M ESPE, St Paul, MN) for 30 seconds and rinsed with water. The teeth were dried with oil-free

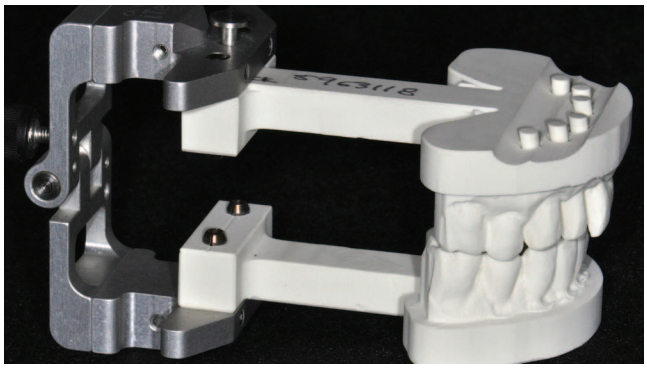

Figure 9. *Milled polyurethane definitive casts articulated on a specifically designed hinge articulator.*

Figure 10. *The scanned data of diagnostic wax-up (blue color) were used as a design template for the designs of CAD definitive restorations (light gray color).*

air, and a single-component bonding agent (Adper Single Bond Plus Adhesive, 3M ESPE) was applied on the etched surface. The CAD/CAM-fabricated, layered lithium disilicate veneers were adhesively luted with dual-polymerizing resin cement (Transparent Variolink II, Ivoclar Vivadent). The excess luting agent was removed, and the patient was given home care instructions (Figure 13A,B).

## DISCUSSION

The use of feldspathic porcelain veneers was introduced into dentistry in the early 1980s and has steadily increased in popularity for the conservative restoration of unesthetic anterior teeth.[22] When adhesively bonded to enamel, the evidence suggests that the estimated cumulative survival for feldspathic porcelain veneers is 95.7% at five years and ranges from 64% to 95% at 10 years across three studies.[23] Other nonfeldspathic ceramics were also used to fabricate veneers; however, the clinical evidence of

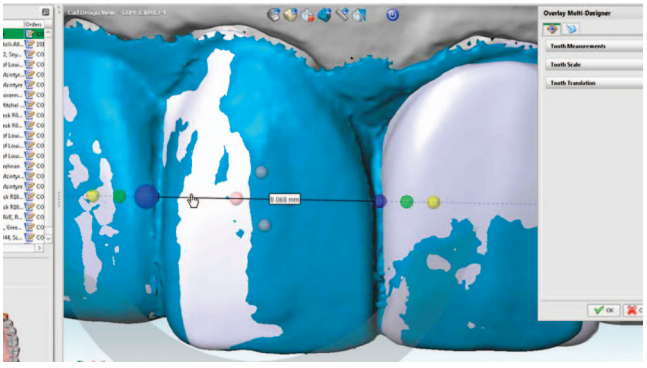

Figure 11. *The digital ruler was used to ensure the dimensions of the designs for the future CAD/CAM-fabricated lithium disilicate ceramic veneers. The three-dimensional measurements on the CAD should have a close resemblance to the virtual esthetic plan–guided diagnostic wax-up.*

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Figure 12. *CAD/CAM-fabricated lithium disilicate ceramic veneers after layering and characterization.*

these veneers is very limited. A systematic review and meta-analysis suggested that the long-term outcome (more than five years) of nonfeldspathic porcelain veneers is only sparsely reported in the literature, and most studies have focused on pressed leucite-reinforced glass ceramic (IPS Empress, Ivoclar Vivadent) with a five-year pooled cumulative estimated survival at 92.4%.[24] Recently, CAD/CAM-fabricated lithium disilicate ceramic veneers have been proposed in various clinical case reports[21,25] and is the choice of treatment in this report.

Although the clinical outcome can be satisfactory, clinicians should utilize this treatment modality with caution. The digital design of definitive restorations was used in the report, where the CAD software was chosen over the conventional wax-up procedure produced by a dental technician. Dental laboratory technicians will require additional training and production experience in digital restorative dentistry to provide consistent definitive restorations. There are also some limitations facing the clinician with this digital work flow as well. The success in utilizing this new treatment protocol depends on the clinician's capacity to operate and maintain the presentation software and intraoral scanning machinery.

## SUMMARY

In this case report, the clinician used presentation software (Keynote iWork) to design a virtual esthetic plan during the treatment planning and presentation process. A digital photograph–assisted virtual esthetic plan can serve as an effective communication tool. The patient can provide direct feedback, and the virtual esthetic treatment plan can be immediately modified to satisfy the patient's esthetic expectations. The approved virtual esthetic plan can be transferred to a calibrated dental laboratory along

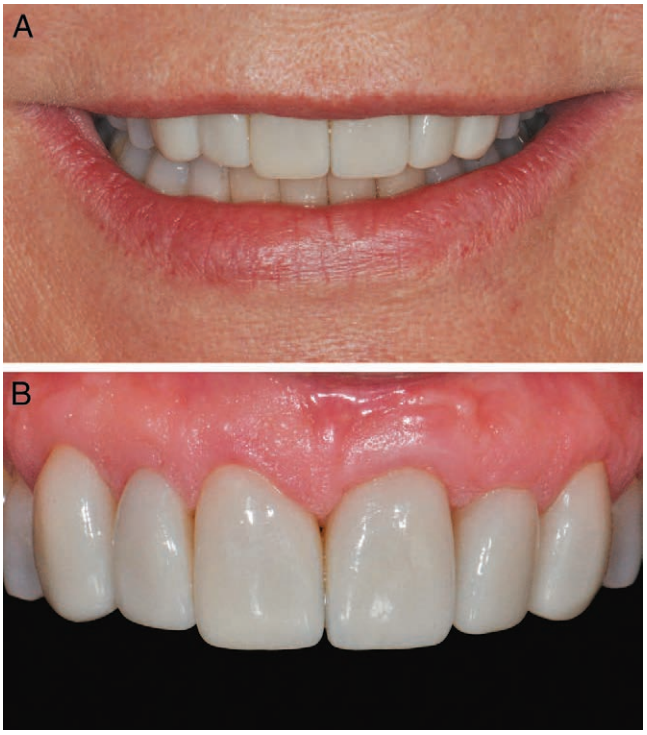


Figure 13. *Posttreatment condition. (A): Smile, frontal view. (B): Intraoral facial view.*

with the diagnostic casts. The esthetic plan then becomes a tool to relay the patient's personal preferences to the dental laboratory technician while performing the diagnostic wax-up. The resulting diagnostic wax-up can then be scanned with the laboratory-based scanner and serve as a design template for the definitive restorations. This helps ensure that the CAD/CAM-fabricated definitive restorations will follow the initial esthetic plan to achieve a predictable clinical result.

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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

# Failure Strengths of Composite Additions and Repairs

D Tantbirojn • C Fernando • A Versluis

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### Clinical Relevance

Repair of defective composite restorations is a common practice, but few studies have quantified the strength of various types of repair materials and techniques. This study provides evidence that may help clinicians choose the most promising techniques and materials.


### SUMMARY

**Purpose: When adding composite to a cured composite restoration, the intent is to achieve the same failure strength as the original restorative material. This study evaluated the failure strengths of added or repaired composite using various chemical and/or mechanical surface treatments.**

**Methods: Failure strengths were determined using a four-point bending test. Beam-shaped specimens were fabricated by adding new composite to cured composite (Filtek Supreme Ultra). The cured composites were either fresh or aged seven days (N=10-14). The composite surfaces were left unground or were ground before treatment with various combinations of roughening, acid etching, silane, and dental adhesives (conventional Adper SingleBond Plus or new multimode Scotchbond Universal) and/or tribochemistry (CoJet system). Monolithic composite specimens were the control. Failure strengths were statistically analyzed using one-way analysis of variance and the Fisher protected least significant difference ($\alpha$=0.05).**

**Results: Failure strengths (mean ± standard deviation) when composite was added to unground freshly cured composites (111±25 MPa) and aged composites using a new multimode adhesive with (102±22 MPa) or without (98±22 MPa) tribochemical treatment were not significantly lower than the monolithic specimens (122±23 MPa). Grinding the surfaces of freshly cured composite significantly reduced failure strength, either with (81±30 MPa) or without (86±31 MPa) use of conventional adhesive. Failure strengths of aged composites were also significantly lower (51±21 MPa with SingleBond Plus), even after tribochemical treatment (71±29 MPa with SingleBond Plus; 73±35 MPa with Silane-Visiobond).**

**Conclusions: Using a new multimode adhesive when adding composite to freshly cured or aged composite substrates recovered the fail-**



Daranee Tantbirojn, DDS, MS, PhD, Department of Restorative Dentistry, College of Dentistry, University of Tennessee Health Science Center, Memphis, TN, USA

Christini Fernando, BS, Biology Department, School of Sciences, Christian Brothers University, Memphis, TN, USA

*Antheunis Versluis, PhD, Department of Bioscience Research, College of Dentistry, University of Tennessee Health Science Center, Memphis, TN, USA

*Corresponding author: 875 Union Ave., Memphis, TN 38163; e-mail: antheun@uthsc.edu


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**ure strength to that of the original monolithic composite.**

## INTRODUCTION

Composite repair for defective restorations is departing from the traditional approach of removing the whole existing restoration. Repair, rather than total replacement, is one of the main concepts of minimally invasive dentistry.[1] The main benefits of composite repair are that it preserves tooth substance, reduces the risk of pulpal complications, reduces costs for patients, and reduces treatment time.[2,3] Eighty-eight percent of dental schools in the United States and Canada and 95% of dental schools in Japan reported teaching repair of defective composite restorations.[2,3] Dental schools that did not include composite repair in their curriculum indicated poor experiences with restoration repairs and viewed the repair procedure as subjective and potentially increasing clinical risks.[2]

From a material point of view, adding fresh composite to a cured composite in a repair procedure raises questions about the adhesion and strength of the restoration.[4-7] To achieve chemical adhesion, unreacted double bonds in cured resin-based composites are essential when adding a new layer of material.[8] Freshly cured composites usually have a relatively high percentage of unreacted double bonds. Methacrylate groups in restorative composites remain 25%-55% unreacted after polymerization.[9] For aged composites, low bond strengths of added layers have been attributed to a reduced number of unreacted double bonds.[4]

Various surface treatment techniques have been used to improve repair bond strength by increasing surface roughness and/or chemically treating the substrate surfaces. Examples are bonding agent application, acid etching, surface abrasion, silica coating, and silanization.[10-14] As more than half of the components of composite restorative materials are ceramic-based filler particles, surface treatment and/or application of an adhesive that promotes affinity to both the methacrylate-based resin and the ceramic-based fillers should be advantageous. For instance, a tribochemical system (CoJet, 3M ESPE, St Paul, MN, USA) that combines sandblasting, a proprietary silica coated sand, silanization, and adhesive resin has been reported to increase repair bond strengths.[12,13] Moreover, a recently introduced multimode adhesive (Scotchbond Universal, 3M ESPE) combines methacryloxydecyl phosphate monomer and silane to enable bonding to various substrates, including metal, nonglass ceramic, and glass ceramic.[15] The indication for use of this new universal adhesive includes intraoral repair of existing composites.

The objectives of this study were to investigate failure strength of added or repaired composite using tribochemical pretreatment and/or the new multimode adhesive and to compare the results with traditional techniques of surface roughening, acid etching, and bonding agent. Two types of substrates were investigated: (1) freshly cured composite, representing a clinical situation where composite is added in the same visit as the restoration placement, and (2) aged composite, representing a clinical situation where composite is added (repaired) at a later visit. Monolithic composite served as the unrepaired control. The hypothesis was that with an appropriate surface treatment, the failure strength of added or repaired composite could be the same as the cohesive strength of monolithic composite.

## METHODS AND MATERIALS

Beam-shaped composite specimens were fabricated using Filtek Supreme Ultra (3M ESPE). The composition and application information for all materials used is summarized in Table 1.

### Monolithic Composite

Monolithic beam specimens ($2 \times 2.5 \times 25$ mm) were fabricated by filling a stainless steel mold with a restorative composite placed between two glass slides. The composite beams were light-cured from the top and the bottom (VIP junior light curing unit, BISCO Inc, Schaumburg, IL, USA) through the glass slides. The light tip was moved along the top ($4 \times 20$ seconds) and bottom ($4 \times 20$ seconds) to cover the entire length of the beams. The intensity of the light source was 600 mW/cm$^2$, as indicated by a radiometer (Model 100, Demetron Research Corp, Danbury, CT, USA). After curing, the beams were taken out of the mold, flash was removed, and the specimens were finished by wet grinding with 600-grit silicon carbide paper. The sample size of the monolithic group was 6.

### Adding to Freshly Cured Unground Composite

Half-length beam specimens ($2 \times 2.5 \times 12.5$ mm) were made by filling half the stainless steel mold (Figure 1A) with composite against a metal insert. The half-beams were light-cured following the same procedure as the full-length monolithic beams ($2 \times 20$ seconds from the top and $2 \times 20$ seconds from the

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Table 1: *Materials Used in the Study*

| Material | Composition | Application |
|---|---|---|
| Filtek Supreme Ultra Restorative Composite[a] (A2 body shade), lot N323761 and N367652 | Silane treated ceramic 60%-80%; silane treated silica 1%-10%; UDMA 1%-10%; bisphenol A polyethylene glycol diether dimethacrylate 1%-10%; Bis-GMA 1%-10%; silane treated zirconia 1%-10%; polyethylene glycol dimethacrylate <5%; TEGDMA <5%; 2,6-di-tert-butyl-p-cresol <0.5% | Light-cure 20 s |
| Scotchbond Universal Etchant,[a] lot 476313 | Water 50%-65%; phosphoric acid 30%-40%; synthetic amorphous silica fumed, crystalline free 5%-10%; polyethylene glycol 1%-5%; aluminum oxide <2% | Etch 15 s, rinse with water 10 s, blot dry |
| Adper Single Bond Plus Adhesive,[a] lot 364852 | Ethyl alcohol 25%-35%; Bis-GMA 10%-20%; silane treated silica (nanofiller) 10%-20%; HEMA 5%-15%; copolymer of acrylic and itaconic acid 5%-10%; glycerol 1,3-dimethacrylate 5%-10%, water <5%, UDMA 1%-5%; diphenyliodonium hexafluorophosphate <0.5%; EDMAB <0.5% | Apply 15 s, air dry 5 s, light-cure 10 s |
| CoJet Sand Blast-Coating Agent 30 mm,[b] Lot 471342 | Aluminum oxide >97%; amorphous silica 3% | Sand blast 3 s |
| ESPE Sil Silane Coupling Agent,[b] lot 470876 | Ethyl alcohol >97%; 3-methacryloxypropyl trimethoxysilane <3%; methyl ethyl ketone <2% | Apply, allow to dry 30 s |
| Visio Bond Light-curing Bonding Agent,[b] lot 464854 | 2-propenoic acid, 2-methyl-, [(3-methoxypropyl)imino]di-2,1-ethanediyl ester 1%-5%; 2,2-dimethoxy-2-phenylacetophenone <0.3%; dicyclopentyldimethylene diacrylate >95%; 2-propenoic acid, 2-methyl-, 2-[(2-hydroxyethyl)(3-methoxyproply)amino]ethyl ester <0.8% | Apply 10 s, air thin 5 s, light-cure 20 s |
| Scotchbond Universal Adhesive,[a] lot 475230 | Bis-GMA 15%-25%; 2-hydroxyethyl methacrylate 15%-25%; decamethylene dimethacrylate 5%-15%; ethanol 10%-15%; water 10%-15%; silane treated silica 5%-15%; 2-propenoic acid, 2-methyl-, reaction products with 1,10-decanediol and phosphorous oxide 1%-10%; copolymer of acrylic and itaconic acid 1%-5%; dimethylaminobenzoat <2%; camphorquinone <2%; (dimethylamino)ethyl methacrylate <2%; methyl ethyl ketone <0.5% | Apply 20 s, air dry 5 s, light-cure 10 s |
| MicroEtcher II Intraoral Sandblaster,[c] Lot 23131-9 | Equipment | Sand blast 3 s |

*Abbreviations: Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; EDMAB, ethyl 4-dimethyl aminobenzoate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate*
[a] *3M ESPE, St Paul, MN, USA.*
[b] *3M ESPE AG, Seefeld, Germany.*
[c] *Danville Materials, San Ramon, CA, USA*

bottom). After the half-beams were cured, the metal insert was removed and composite was inserted into the other half of the mold (Figure 1B), which was then light-cured ($2 \times 20$ seconds from top and $2 \times 20$ seconds from bottom). The final full-length beams were removed from the molds and finished using the same procedures as described for the monolithic beams. The sample size of the freshly cured composite group was 10.

### Fabrication of Half-Beams

Half-length beam specimens ($2 \times 2.5 \times 12.5$ mm) were made by filling half the stainless steel mold with composite against the metal insert and light-cured following the same procedure as described previously. After curing, the half-length beams were removed from the molds and flash was removed. All surfaces, including the surface where composite would be added, were finished by wet grinding with 600-grit silicon carbide paper. Composite was added to the half-beam specimens either immediately (freshly cured) or after being stored dry for 7 days at room temperature (aged).

### Adding to Freshly Cured Composite

The freshly cured half-beam specimens were (1) ground wet using 600-grit silicon carbide paper; or (2) ground wet using 600-grit silicon carbide paper etched with phosphoric acid gel (Scotchbond Universal Etchant, 3M ESPE) for 15 seconds, rinsed with water, and blotted dry. After adhesive was applied for 15 seconds (Adper SingleBond Plus, 3M ESPE),

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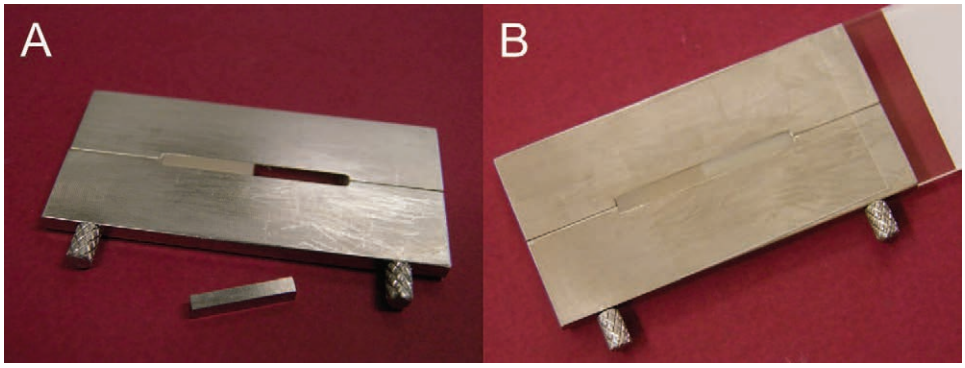


Figure 1. *Specimen fabrication. (A): Half-beam composite specimens (2 × 2.5 × 12.5 mm) were made in a stainless-steel mold. The metal insert was removed after curing. (B): Composite was added to repair the half-beam to full-length (2 × 2.5 × 25 mm).*

the specimens were air dried for 5 seconds and light-cured for 10 seconds. The surface-treated half-beam specimens were placed back into the mold, and the new composite was added to fabricate a full-length beam. The added composite was light-cured (2 × 20 seconds from top and bottom). The full-length beams were removed from the molds and finished using the same procedures as described for the monolithic beams. Sample size of the freshly cured composite groups was 10.

### Repair of Aged Composite

After wet grinding with 600-grit silicon carbide paper, the interfaces of the aged composite half-beam specimens were treated using one of the following protocols:

- Etch/SingleBond Plus: Etched with Scotchbond Universal Etchant for 15 seconds, rinsed with water, blotted dry, applied Adper SingleBond Plus adhesive for 15 seconds, air dried 5 seconds, and light-cured 20 seconds.
- Cojet/SingleBond Plus: Tribochemical treatment (CoJet sand-blast) for 3 seconds, applied Adper SingleBond Plus adhesive for 15 seconds, air dried 5 seconds, and light-cured 20 seconds.
- Cojet/Silane/Visiobond: Tribochemical treatment (CoJet sand-blast) for 3 seconds, applied ESPE Sil Silane Coupling Agent and allowed to dry 30 seconds, applied Visio Bond for 10 seconds, air-thinned 5 seconds, and light-cured 20 seconds.
- Etch/Universal: etched with Scotchbond Universal Etchant for 15 seconds, rinsed with water, blotted dry, applied Scotchbond Universal Adhesive for 20 seconds, air dried 5 seconds, and light-cured 20 seconds.
- Cojet/Universal: Tribochemical treatment (CoJet sand-blast) for 3 seconds, applied Scotchbond Universal Adhesive for 20 seconds, air dried 5 seconds, and light-cured 20 seconds.

The treated half-beam specimens were placed back into the mold, and new composite was inserted into the other half of the mold to fabricate a full-length beam. The added composite was light-cured (2 × 20 seconds from top and bottom). The repaired full-length composite beams were removed from the molds and finished using the same procedures as described for the monolithic beams. Sample sizes of the repaired aged composite groups were 10-14.

### Flexural Failure Strength Test

All specimens were stored dry at room temperature for 24 hours before testing. Before they were tested, the dimensions (length, height, and width) of each beam specimen were measured with a digital caliper. Width and height were determined as the average of three locations along each beam length. These dimensions were used in the failure strength calculations of each tested beam. Failure strengths were determined using a four-point bending test (Figure 2). Specimens were loaded until failure in a universal testing machine (Instron Electromechanical Testing System, Series 5567, Instron, Norwood, MA, USA) at a rate of 0.5 mm/min. Load at failure (N) was recorded. Failure strength (MPa) was calculated from the relationship: $3\,FL/(4BH^2)$, where F was the load at failure, L was the distance between the lower supports (20 mm), B was the beam width, and H was the beam height.[16] Failure strength comparisons between the tested groups were statistically analyzed with one-way analysis of variance followed by the Fisher protected least significant difference ($\alpha$=0.05).

## RESULTS

Failure strengths and sample sizes are shown in Table 2. It was observed that specimens with lower failure loads usually fractured along the interface of the repair, whereas specimens with higher failure

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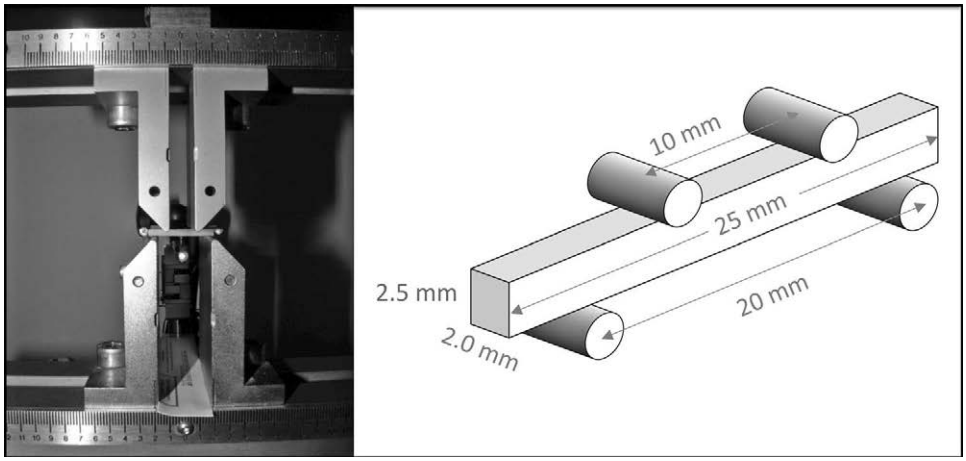


Figure 2. *Four-point bending test setup and diagram of the specimen dimensions and loading points.*

loads often failed cohesively. Monolithic beams, representing the strength of the original unrepaired composite, had the highest failure strength values of 122 ± 23 MPa (mean ± standard deviation). When new composite was added to unground freshly cured composite, the failure strength (111±25 MPa) was not significantly different from the values found for the monolithic specimens.

When the freshly cured composite surface had been ground and new composite material was added, the failure strength significantly decreased by about 25%, regardless of whether adhesive was used (86±31 MPa without adhesive; 81±30 MPa with Adper SingleBond Plus).

When new composite was added to aged specimens, failure strength decreased more than 50% when a conventional etch-and-bond technique had been used (51±21 MPa). CoJet sandblasting slightly increased the failure strength (71±29 MPa), but it was still about 35% lower than the original strength, even after application of silane coupling agent (73±35 MPa). Repairs using the universal adhesive, however, recovered about 80% of the original strength, yielding failure strengths that were not significantly different from those of the monolithic specimens regardless of whether CoJet sand-blast treatment was used (98±22 MPa without CoJet sand-blast; 102±22 MPa with CoJet sand-blast).

Table 2: *Sample Size and Failure Strengths (Mean ± Standard Deviation)*[a]

| Group | Sample size | Failure strength (MPa) |
|---|---|---|
| Aged: Grind, Etch/SingleBond Plus | 12 | 51 ± 21 a |
| Aged: Grind, CoJet/SingleBond Plus | 11 | 71 ± 29 a,b |
| Aged: Grind, CoJet/silane/Visiobond | 14 | 73 ± 35 b |
| Fresh: Grind, Etch/SingleBond Plus | 10 | 81 ± 30 b,c |
| Fresh: Grind | 10 | 86 ± 31 b,c |
| Aged: Grind, Etch/Universal | 11 | 98 ± 22 c,d |
| Aged: Grind, CoJet/Universal | 10 | 102 ± 22 c,d |
| Fresh: No grinding | 10 | 111 ± 25 d |
| Monolithic | 6 | 122 ± 23 d |

[a] *Same letters denote values not significantly different (one-way analysis of variance followed by the Fisher protected least significant different test, $\alpha$=0.05).*

## DISCUSSION

This study examined different techniques and adhesive materials for adding or repairing composite restorations. The effectiveness of the addition or repair techniques was evaluated 24 hours after a repair was made by measuring the maximum flexural strength in composite beams fabricated by adding new composite to a cured composite. Flexure was chosen to test the strength of the composite beams because bending involves both tensile and compressive stress conditions and thus can be argued to be more inclusive than a uniaxial tensile test.[12] Shear tests have also been used to evaluate repair strength,[13] but stress conditions in shear tests are not well defined.[17] Furthermore, the choice of a four-point instead of a three-point loading condition provided a wider failure area with the same stress conditions (because the longitudinal stress is constant between the two upper supports) and thus accommodated both cohesive and interfacial failures. The significance of this choice was evident during the testing, where specimens that failed at lower load values were more likely to fail along the interface, while specimens with relatively high failure loads tended to fail cohesively (ie, away from the repair interface).

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The sample sizes among the groups varied between 6 and 14. The International Standard ISO 4049 for dental resin-based filling materials specifies at least five specimens for the flexural strength test.[18] The monolithic composite group had a sample size of six. Sample-size number was increased for specimens in groups with added or repaired composite because they had higher variation in failure strengths than the monolithic beams. Because a sample size of 9 has 95% confidence to detect a mean difference of 2/3 of the standard deviation between groups, sample size was increased to 10.[19] Some specimens in the aged composite groups had failure loads substantially lower than the group means. As these low values may represent actual repair complications, extra samples were added for each substantially lower value instead of excluding those samples. The final sample sizes are shown in Table 2.

Two clinical conditions were simulated in this *in vitro* study: (1) adding composite to a freshly cured composite to represent adding composite to a restoration within the same visit, and (2) adding composite to an aged composite to represent adding composite in a subsequent visit after the composite restoration reached its maximum possible conversion.

Freshly cured composite can be expected to have the highest number of unreacted double bonds for chemical adhesion of added composite. Failure strength did not decrease significantly compared with the monolithic specimens when composite was added directly to freshly cured composite. When the attachment surface was ground, which simulates finishing of a restoration within the same visit, the failure strength reduced significantly even when the surface was re-etched and a conventional bonding agent was applied. Other *in vitro* studies reported mixed results for bonding agent application on repair strength.[4,11,13] Under clinical conditions, re-etching and application of a bonding agent cleans the surface of contamination and provides better adaptation of the added composite. Nevertheless, clinicians should be aware that adding a new layer of composite within the same visit to correct inadequate contact, contour, surface voids, or shade mismatch may reduce the strength of a restoration.

When composite restorations chip, wear down, develop defective margins or have confined areas of recurrent decay, while large portions of the restorations remain intact and radiographically sound, adding new composite to the old (aged) restorations is highly desirable within the concepts of minimal intervention and preservation of tooth structure. Although partial removal of composite restoration is conservative and avoids potential pulp injuries, the bonding to tooth structure when the whole restoration is removed can be better ensured than bonding to old composite. Therefore, composite repair techniques are sometimes viewed as unreliable and potentially increasing clinical risks.[2] Indeed, the current study found that adding new composite to aged composite could result in a substantial (37% to 54%) reduction in failure strength compared with repair of fresh composite. This strength reduction may be attributed to the more complete polymerization of aged composites, which should have fewer remaining unreacted double bonds for chemical adhesion of the new layer.[4,8]

To improve bond strength of repairs made to aged composites, mechanical and/or chemical surface treatments are often recommended.[10-14] It has been shown that surface roughening by aluminum oxide sandblasting increased composite repair strength more than roughening with a diamond bur.[14] A tribochemical system, like the CoJet Sand Blast, is a step further than just sandblasting. The CoJet system blasts proprietary silica-coated sand with high energy to leave a ceramic coating (silicatized) on the surface to enhance chemical bonding by subsequent silanization.[20] Previous studies achieved good repair strengths with this tribochemical system.[12,13] The same tribochemical system was used in the present study and significantly increased the failure strengths of the repairs to aged composite by 40% compared with surface roughening with silicon carbide paper. Interestingly, no additional improvement in failure strength was found when a silane coupling agent was subsequently used to silanize the interface after Cojet Sand Blast.

Besides the conventional bonding agents, a new generation of multimode adhesive was tested for repairing aged composites, which combines methacryloxydecyl phosphate monomers for adhesion to nonglass ceramic substrates and silane for adhesion to glass-ceramic surfaces.[15] Surfaces that are ground in preparation of composite repair are likely to have a high fraction of exposed ceramic fillers. This may explain why the new multimode adhesive yielded the highest failure strengths for repair of the aged composites, regardless of tribochemical treatment. The failure strength values that were found with this new generation of adhesive were not significantly lower than the monolithic strength. This

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confirmed the hypothesis that it is possible to repair composite without a significant reduction in failure strength, depending on the treatment of the attachment surface, in this case treatment with a multimode adhesive.

Ultimately, the durability of repaired composite restorations must be proven under clinical conditions. However, clinical studies cannot quantify the strength of repairs, which requires *in vitro* studies. Despite its limitations, an *in vitro* study can thus provide important insight about repair procedures and indicate the most promising techniques. The results of the current study indicate promising strength values with a new multimode adhesive. The multimode adhesive, which is already part of the enamel/dentin bonding procedure, is simple to use and does not require extra armamentarium, such as a tribochemical system. The ability to make additions or repairs to composite restorations without lowering the failure strength will contribute to the longevity of the dentition and thus oral health.

## CONCLUSION

- Adding new composite to aged or ground freshly cured composite significantly reduced composite failure strength when using a conventional bonding agent, regardless of surface treatment.
- Repair of aged composite using a new multimode adhesive, with or without tribochemical treatment, achieved failure strengths that were not significantly lower than those of additions to freshly cured unground composite or monolithic unrepaired composite.
- Tribochemical treatment alone increased repair strength but was not as effective as the multimode adhesive.

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### Conflict of Interest

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

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# Influence of Etching Protocol and Silane Treatment with a Universal Adhesive on Lithium Disilicate Bond Strength

VK Kalavacharla • NC Lawson • LC Ramp
JO Burgess

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### Clinical Relevance

Optimal bond strength to lithium disilicate is achieved by exposure to at least 20 seconds of 5% hydrofluoric acid (HF) followed by a coat of silane and then a universal adhesive. If an additional silane step is not taken prior to applying a universal adhesive, the use of 9.5% HF for 60 seconds can increase bond strength.


## SUMMARY

**Objectives: To measure the effects of hydrofluoric acid (HF) etching and silane prior to the application of a universal adhesive on the bond strength between lithium disilicate and a resin.**

**Methods and Materials: Sixty blocks of lithium disilicate (e.max CAD, Ivoclar Vivadent) were sectioned into coupons and polished. Specimens were divided into six groups (n=10) based on surface pretreatments, as follows: 1) no treatment (control); 2) 5% HF etch for 20 seconds (5HF); 3) 9.5% HF etch for 60 seconds (9.5HF); 4) silane with no HF (S); 5) 5% HF for 20 seconds + silane (5HFS); and 6) 9.5% HF for 60 seconds + silane (9.5HFS). All etching was followed by rinsing, and all silane was applied in one coat for 20 seconds and then dried. The universal adhesive (Scotchbond Universal, 3M ESPE) was applied onto the pretreated ceramic surface, air thinned, and light cured for 10 seconds. A 1.5-mm-diameter plastic tube filled with Z100 composite (3M ESPE) was applied over the bonded ceramic surface and light cured for 20 seconds on all four sides. The specimens were thermocycled for 10,000 cycles (5°C-50°C/15 s dwell time). Specimens were loaded until failure using a universal testing machine at a crosshead speed of 1 mm/min. The peak failure load was used to calculate the shear bond strength. Scanning electron microscopy images were taken of representative e.max specimens from each group.**


Vamsi K Kalavacharla, BDS, MS, UAB, Birmingham, AL, USA

*Nathaniel C Lawson, DMD, MS, University of Alabama at Birmingham, Clinical and Community Sciences, Birmingham, AL, USA

Lance C Ramp, DMD, PC, UAB School of Dentistry, Comprehensive Dentistry, Birmingham, AL, USA

John O Burgess, DDS, University of Alabama at Birmingham, Prosthodontics and Biomaterials, Birmingham, AL, USA

*Corresponding author: SDB 603, 1720 2nd Avenue S, Birmingham, AL 35294-0007, USA; e-mail: nlawson@uab.edu

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**Results: A two-way analysis of variance (ANOVA) determined that there were significant differences between HF etching, silane treatment, and the interaction between HF and silane treatment ($p<0.01$). Silane treatment provided higher shear bond strength regardless of the use or concentration of the HF etchant. Individual one-way ANOVA and Tukey post hoc analyses were performed for each silane group. Shear bond strength values for each etch time were significantly different ($p<0.01$) and could be divided into significantly different groups based on silane treatment: no silane treatment: 0 HF < 5% HF < 9.5% HF; and RelyX silane treatment: 0 HF < 5% HF and 9.5% HF.**

**Conclusions: Both HF and silane treatment significantly improved the bond strength between resin and lithium disilicate when used with a universal adhesive.**

## INTRODUCTION

Lithium disilicate is a dental ceramic that mimics the esthetics and strength of natural tooth structure. The 70% crystal phase of this unique glass-ceramic material refracts light naturally and provides superior structural reinforcement, imparting a greater flexural strength than is associated with traditional feldspathic porcelain or leucite-reinforced glass ceramics.[1,2] Lithium disilicate crowns may be placed by either traditional cementation techniques or adhesive bonding. One clinical trial has shown similar survival of lithium disilicate crowns cemented with resin-modified glass ionomer cements and bonded with resin cements.[3,4] In clinical situations involving short clinical crowns or overtapering of the crown preparation, adhesive bonding is recommended.[5] *In vitro* studies[6,7] have shown superior bond strength when lithium disilicate is bonded to tooth as compared to traditional cementation. Additionally, bonding with a resin composite cement may also improve the fracture strength of lithium disilicate crowns.[8]

Prior to bonding lithium disilicate crowns, etching with hydrofluoric acid (HF) is recommended.[9] The action of HF etching on the microstructure of these ceramics is by dissolution of the glassy phases of porcelain.[5,6] This phase is partially dissolved to create an appropriate microstructure that increases surface area for bonding.[7-10] Studies[11-15] have shown that HF etching with lithium disilicate improved bond strength. These studies have etched with 9.5%-10% HF for 60 seconds[13,15] or 20 seconds[11,12] and with 5% HF for 20 seconds.[14] Since there has not been an evaluation on the effect of HF concentration on bond strength, this study will compare two reported etching protocols: 9.5% HF for 60 seconds[13,15] and 5% HF for 20 seconds.[14] Silanes are a class of organic molecules that contain one or more silicon atoms. The specific silane used in dentistry is 3-methacryloxypropyltrimethoxysilane. It is used as a chemical coupler, linking organics (resin-based materials) to inorganics (eg, porcelain, some oxidized metals, and glass fillers in resin-based composites).[16] Infrared spectroscopy has shown that silane has the potential to react with hydroxyl (-OH) groups present on the surface of silica in ceramics and the methacrylate group of a bonding agent or resin cement.[17,18] A study by Panah and others[15] demonstrated that the bond strength to lithium disilicate was significantly improved with silane treatment and further improved with HF etching and then use of silane. Nagai and others[14] concluded that silane improved the bond strength to lithium disilicate. Additionally, they discovered that HF etching improved bond strength more in unsilanated specimens than in those that were silanated.[14]

The introduction of universal adhesives presents a new simplified approach for bonding ceramic to resin cements. Universal adhesives contain silane and a monomer called 10-methacryloxydecyl dihydrogen phosphate (MDP) that help bond ceramic to the resin in a cement. A study by Amaral and others[19] has shown the ability of a universal adhesive to bond zirconia to a resin; however, the effectiveness of the adhesive has not been thoroughly investigated with lithium disilicate.

This study examined how surface treatment (HF and silane) affects shear bond strength between a resin composite and lithium disilicate treated with a universal adhesive (Scotchbond Universal, 3M ESPE, St Paul, MN, USA). The purpose of this study was to determine if it is necessary to apply silane prior to a universal adhesive. Additionally, we wished to determine if HF etching improves bond strength to lithium disilicate and to compare the effectiveness of 5% HF (20 seconds) and 9.5% HF (60 seconds). The null hypotheses of this study were that there is no difference in the bond strength when silane is applied prior to a universal adhesive and that there is no difference in bond strength with two different etching protocols prior to a universal adhesive application.

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Table 1: *Materials Used in this Study*

| Material | Brand Name | Manufacturer | Lot No. |
|---|---|---|---|
| Lithium disilicate | e.max | Ivoclar Vivadent | — |
| 5% Hydrofluoric acid etchant | Ceramic etching gel | Ivoclar Vivadent | R05638 |
| 9.5% Hydrofluoric acid etchant | Porcelain etchant | Bisco | 1200006564 |
| Silane | RelyX Ceramic Primer | 3M ESPE | N371615 |
| Universal adhesive | Scotchbond Universal | 3M ESPE | 472585 |
| Resin composite | Z100 | 3M ESPE | N352896 |

## METHODS AND MATERIALS

All materials used for specimen preparation are described in Table 1. Blocks of lithium disilicate (e.max CAD, Ivoclar Vivadent, Amherst, NY, USA) in bisque (blue, metasilicate) form were sectioned into rectangular coupons using a low-speed cutting device (Isomet, Buehler Ltd, Lake Bluff, IL, USA) and sintered according to the recommended protocol. To establish a uniform surface, each specimen was polished with a rotational polishing device using 180- and 320-grit silica carbide abrasive paper under a steady stream of water. The polishing was done by rotating the specimen 90° every one minute with each grit, for a total of four minutes. The specimens were finished with 0.5-μm $Al_2O_3$ slurry, rotating the specimens 90° every 30 seconds for a total of two minutes. All specimens were subjected to ultrasonic cleaning in distilled water for 15 seconds.

The specimens were divided into groups (n=10) based on surface pretreatments, as follows (Table 2): 1) no treatment (control); 2) 5% HF etch for 20 seconds (5HF); 3) 9.5% HF etch for 60 seconds (9.5HF); 4) silane (S); 5) 5% HF for 20 seconds + silane (5HFS); and 6) 9.5% HF for 60 seconds + silane (9.5HFS).

Groups 5HF, 9.5HF, 5HFS, and 9.5HFS were etched with HF. For the etching procedure using 5% HF (Ceramic etching gel, Ivoclar), a drop of etchant was evenly spread for 20 seconds over the bonding surface of the ceramic using a microbrush. The surface was cleaned with water for 10 seconds. The 9.5% HF (Porcelain etchant, Bisco, Schaumburg, IL, USA) was applied for 60 seconds and cleaned with water for 10 seconds. Groups S, 5HFS, and 9.5HFS then received one coat of silane (RelyX Ceramic Primer, 3M ESPE), which was applied for 20 seconds using a microbrush and then air dried for 10 seconds with room-temperature air. Scotchbond Universal adhesive (3M ESPE) was applied onto all pretreated ceramic surfaces for 20 seconds using a microbrush, followed by air thinning for 10 seconds. The adhesive was cured for 10 seconds with an LED curing light (1200 mW/cm$^2$; Elipar S10, 3M ESPE). Curing light output was tested after finishing every 10 specimens to check for uniformity of light output using a radiometer (Power Max, Molectron Detector Inc, Portland, OR, USA).

A 5-mm-long, transparent, plastic tube with an internal diameter of 1.5 mm was filled with Z100 composite (shade A2, 3M ESPE). Z100 composite was chosen to represent an adhesive resin cement, such as Rely X Unicem (3M ESPE), as both materials are composed of a methacrylate-based monomer reinforced with silanated inorganic fillers (70 wt% for RelyX Unicem and 85 wt% for Z100). Z100 resin composite was used instead of a cement in order to prevent cohesive fractures within the bonded post. The composite filled tube was affixed to the surface of each ceramic specimen and light cured (Elipar

Table 2: *Etching, Silane, and Adhesive Procedure*

| Group | Etch Concentration, % | Etching Time, s | Silane Treatment Duration, s | Universal Adhesive Treatment Duration, s |
|---|---|---|---|---|
| Control | None | — | — | 20 |
| 5HF | 5 | 20 | — | 20 |
| 9.5HF | 9.5 | 60 | — | 20 |
| S | None | — | 20 | 20 |
| 5HFS | 5 | 20 | 20 | 20 |
| 9.5HFS | 9.5 | 60 | 20 | 20 |

*Abbreviations: 5HF, 5% hydrofluoric acid (HF) etch for 20 s; 9.5HF, 9.5% HF etch for 60 s; S silane with no HF; 5HFS, 5% HF for 20 s + silane; 9.5HFS, 9.5% HF for 60 s + silane.*

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Figure 1. *Composite tube affixed to the coupon of lithium disilicate.*

S10, 3M ESPE) on four sides for 20 seconds per side (Figure 1). After storage in deionized water for 24 hours at 37°C, the specimens were thermocycled for 10,000 cycles (six days) between 5°C and 50°C water baths (15-second dwell time). Prior to shear bond strength testing, the plastic tube was removed to reveal a cylinder of composite. The specimens were mounted into a steel fixture in a universal testing machine (Instron 5565, Canton, MA, USA). A sharpened stylus applied a shear load to the side of the composite cylinder until failure at a crosshead speed of 1 mm/min (Figure 2). The peak failure load and surface area of the composite cylinder were used to calculate the shear bond strength (MPa). An additional specimen from both etching conditions was sputter-coated and imaged under a scanning electron microscope (SEM) for observation of the etching pattern.

A two-way analysis of variance (ANOVA) and Tukey post hoc analysis were used to analyze the differences in the bond strength values based on HF etching and silane treatment ($\alpha$=0.05).

Table 3: *Bond Strength of Lithium Disilicate to Composite Resin with Different Surface Treatments (Mean ± Standard Deviation [Group Abbreviation])*[a]

| | Bond Strength, MPa | |
|---|---|---|
| | **No Silane Application** | **Silane Application** |
| No HF | 1.82 ± 2.0 A (control) | 12.55 ± 5.0 A (S) |
| 5% HF | 19.08 ± 3.0 B (5HF) | 40.47 ± 4.2 B (5HFS) |
| 9.5% HF | 24.93 ± 2.6 C (9.5HF) | 37.50 ± 5.1 B (9.5HFS) |

*Abbreviations: 5HF, 5% hydrofluoric acid (HF) etch for 20 s; 9.5HF, 9.5% HF etch for 60 s; S silane with no HF; 5HFS, 5% HF for 20 s + silane; 9.5HFS, 9.5% HF for 60 s + silane.*

[a] *Groups in each column with similar capital letters are not statistically different.*

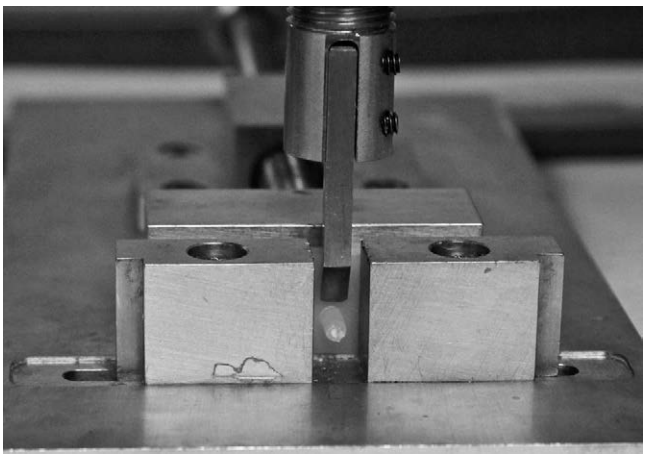

Figure 2. *Lithium disilicate specimen mounted in the universal testing device.*

## RESULTS

The means and standard deviations of the bond strength values for each group are presented in Table 3. The two-way ANOVA determined that there were significant differences between HF etching, silane treatment, and the interaction between the HF and silane groups ($p<0.01$). As the factor "silane treatment" was found to be significant, silane treatment was shown to provide higher shear bond strength without HF etching and at both concentrations of HF etching. Individual one-way ANOVA and Tukey post hoc analyses were performed for the silane and no silane groups. Shear bond strength values for each etch concentration were significantly different ($p<0.01$) and could be divided into significantly different groups based on silane treatment: no silane treatment: no HF < 5% HF < 9.5% HF; and RelyX silane treatment: no HF < 5% HF and 9.5% HF. Therefore, the significant interaction could be explained by the fact that without silane treatment, 9.5% HF etching produced higher bond strength than did 5% HF etching, whereas this was not the case if the material was treated with silane. The SEM images of the 5% HF etched lithium disilicate showed the presence of elongated crystals after partial disintegration of the silica matrix (Figures 3A and 3B). The 9.5% HF etched surface showed a more distinct etching pattern with more dissolved matrix (Figures 3C and 3D).

## DISCUSSION

The use of HF etching improved resin-lithium disilicate bond strength regardless of whether or not silane was applied. Therefore, we can reject the null hypothesis that HF etching will not affect the

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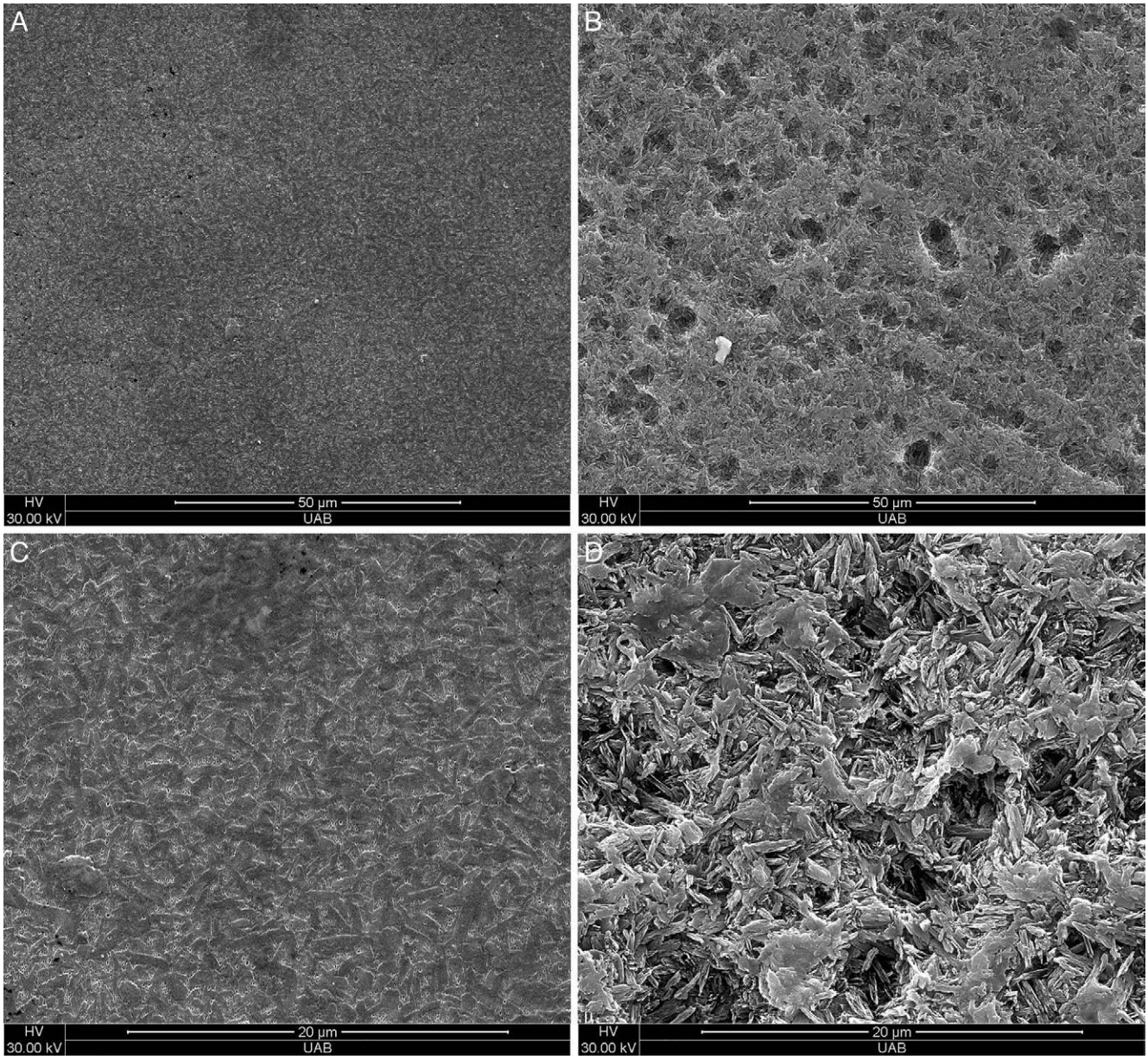


Figure 3. *Etching pattern of lithium disilicate etched for 20 seconds with 5% HF acid at 3000× (A) and 10,000× (B) and for 60 seconds with 9.5% HF acid at 3000× (C) and 10,000× (D).*

bond strength of resin bonded to lithium disilicate pretreated with a universal composite. The concentration/application time of HF etchant did not have a significant effect on bond strength for the specimens that were given a coat of silane. The specimens that were not silanated, however, showed significantly greater bond strength with 60 seconds of 9.5% HF than with 20 seconds of 5% HF. Although the study clearly demonstrates that optimal bonds are achieved by HF etching and silanating, if an additional silane step is not taken prior to applying a universal adhesive, the use of 9.5% HF for 60 seconds can increase bond strength. Perhaps the explanation for this difference is that the specimens that did not receive silane were more dependent on the micromechanical retention provided by the etch pattern in the ceramic. The SEM images show that there is considerably deeper etching pattern present on the specimen etched for 60 seconds at 9.5% HF (Figure 3A-D). Therefore, the nonsilanated specimens could achieve higher bond strengths with a deep etching pattern in the 9.5% HF. The specimens that were silanated, on the other hand, could rely on the chemical bond facilitated by the silane molecules.

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The choice to etch for 20 seconds with 5% HF and for 60 seconds with 9.5% HF was based on the manufacturer's recommendations. Previous studies have suggested that etching with 4.9% HF for over 90 seconds[20] or with 9% HF for 120 seconds[21] decreases the strength of the lithium disilicate. As the bond strength in this study did not improve when increasing the etching concentration from 5% HF (20 seconds) to 9.5% HF (60 seconds), it is beneficial to adopt a regimen of 5% HF at 20 seconds. This etching protocol will save the clinician time and preserve the strength of the lithium disilicate.

The results of this study also reveal that silane treatment prior to application of a universal adhesive significantly improved the bond strength regardless of the method of etching. We can therefore reject the null hypothesis that there is no difference in bond strength between resin and lithium disilicate when a silane treatment is applied. This result suggests that the constituent silane in the universal adhesive was not effective in optimizing the ceramic-resin bond. Clinicians should therefore pretreat lithium disilicate with a coat of silane prior to applying the universal adhesive. A study by Panah and others[15] showed that microshear bond strength between lithium disilicate and composite resin improved from 4.10 MPa to 14.58 MPa when silane was applied. Additionally, the microshear bond strength improved from 14.04 MPa to 24.70 MPa when silane was applied to lithium disilicate that had been HF etched.[15] This study further confirms that lithium disilicate should undergo both sufficient etching and silanization prior to bonding.

One limitation of this study is that only one brand of universal adhesive was tested. There are several commercially available universal adhesives that have unique compositions. The specific chemicals and concentrations used in each brand of adhesive may have different interactions with the method of surface pretreatment.

## CONCLUSIONS

Optimal bonds are achieved by HF etching and application of silane to lithium disilicate prior to application of a universal adhesive. When using the universal adhesive in conjunction with silane, it is beneficial to adopt a regimen of 5% HF at 20 seconds in order to minimize surface damage to the ceramic while preserving the bond strength. If an additional silane step is not taken prior to applying a universal adhesive, the use of 9.5% HF for 60 seconds can increase bond strength. As the constituent silane and MDP in the universal adhesive were not effective in optimizing the ceramic-resin bond, silane should always be applied to lithium disilicate prior to bonding.

### 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 except for the following: Dr Burgess has, through the University of Alabama at Birmingham, contracts and research grants from the following: GC, 3M ESPE, Ivoclar, Dentsply, Septodont, Glidewell, Discus, VOCO, Shofu, Ultradent, Noritake, and DMG. However, Dr Burgess received no honoraria, nor did he collect any fee, for the submission of this manuscript.

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# Effect of Phosphoric Acid Pre-etching on Fatigue Limits of Self-etching Adhesives

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

### Clinical Relevance

The effect of phosphoric acid pre-etching of enamel and dentin prior to the use of self-etching adhesives is dependent on the mineralized substrate and the adhesive material.

## SUMMARY

**The purpose of this study was to use shear bond strength (SBS) and shear fatigue limit (SFL) testing to determine the effect of phosphoric acid pre-etching of enamel and dentin prior to application of self-etch adhesives for bonding resin composite to these substrates. Three self-etch adhesives—1) G- ænial Bond (GC Corporation, Tokyo, Japan); 2) OptiBond XTR (Kerr Corp, Orange, CA, USA); and 3) Scotchbond Universal (3M ESPE Dental Products, St Paul, MN, USA)—were used to bond Z100 Restorative resin composite to enamel and dentin surfaces. A stainless-steel metal ring with an inner diameter of 2.4 mm was used to bond the resin composite to flat-ground (4000 grit) tooth surfaces for determination of both SBS and SFL. Fifteen specimens each were used to determine initial SBS to human enamel/dentin, with and without pre-etching with a 35% phosphoric acid (Ultra-Etch, Ultradent Products Inc, South Jordan, UT, USA) for 15 seconds prior to the application of the adhesives. A staircase method of fatigue testing (25 specimens for each test) was then used to determine the SFL of resin composite bonded to enamel/dentin using a frequency of 10 Hz for 50,000 cycles or until failure occurred. A two-way analysis of variance and Tukey post hoc test were used for analysis of SBS data, and a modified *t*-test with Bonferroni correction was used for the SFL**

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

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

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

Donal D Scheidel, DDS, associate professor, Diagnostic Sciences, Creighton University School of Dentistry, Omaha, NE, USA

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

Mark A Latta, DMD, MS, professor, General Dentistry, dean, Creighton University School of Dentistry, Omaha, NE USA

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

*Corresponding author: Wayne W Barkmeier, DDS, MS, 2500 California Plaza, Omaha, NE 68178, USA; e-mail: wbark@creighton.edu

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**data. Scanning electron microscopy was used to examine the area of the bonded restorative/tooth interface. For all three adhesive systems, phosphoric acid pre-etching of enamel demonstrated significantly higher ($p<0.05$) SBS and SFL with pre-etching than it did without pre-etching. The SBS and SFL of dentin bonds decreased with phosphoric acid pre-etching. The SBS and SFL of bonds using phosphoric acid prior to application of self-etching adhesives clearly demonstrated different tendencies between enamel and dentin. The effect of using phosphoric acid, prior to the application of the self-etching adhesives, on SBS and SFL was dependent on the adhesive material and tooth substrate and should be carefully considered in clinical situations.**

## INTRODUCTION

With the increased popularity of adhesive restorative dentistry, many dental manufacturers have introduced self-etching adhesive systems to the profession. Self-etch adhesive systems are promoted as being more efficient for bonding procedures in that they require fewer treatment steps to condition tooth surfaces for bonding resin-based materials. However, self-etching adhesive systems are not able to etch enamel as effectively as the phosphoric acid used in etch-and-rinse adhesive systems, and most published work[1-7] indicates that self-etching adhesive systems provide lower resin composite to enamel bond strengths than do etch-and-rinse adhesive systems, which may be related to their lower etching capability. In order to achieve a durable bond to enamel with self-etching adhesive systems, selective etching of enamel with phosphoric acid before the application of self-etching adhesives has been proposed.[8-13] However, it may be difficult to precisely etch only the enamel region, and there is certainly the possibility of affecting exposed dentin.

A major concern in adhesive dentistry is whether the resin monomer of a self-etching adhesive infiltrates into the entire depth of demineralized dentin. Incomplete penetration of a resin monomer into the demineralized dentin might lead to bond degradation from oral fluids and bacterial enzymes.[14-16]

The ability of adhesive agents to bond resin-based materials to tooth structures has been measured extensively in the laboratory using various methods to determine bond strengths. Common laboratory methods employed to determine shear bond strength (SBS) or microtensile bond strength (μ-TBS) use a monotonically increasing load until bond failure occurs. These standardized tests provide valuable information regarding the ability of adhesive agents to bond resin-based materials to demineralized tooth structures. However, this type of force is not the likely mode of failure for bonds in the oral cavity, where failure is considered to result from repeated loading over many months or years, and at lower force levels. Adhesive bonds to both enamel and dentin substrates in the mouth are subjected to repeated stress over long periods of time by the process of loading on tooth structure or restorations that apply compressive, flexural, or tensile stresses to the bonds.

Cyclic loading of specimens to elicit failure is often referred to as fatigue testing.[17-21] A common method of fatigue testing is the staircase method,[22] in which the load on a specimen is increased or decreased by a fixed amount depending on whether the preceding specimen survived or failed, respectively. With this type of test, a parameter called the fatigue limit, which represents the load (stress) at which half the specimens fail during the cycling period, can be calculated.

Limited information is available regarding the ability of enamel bonds produced by self-etching adhesives to resist the forces of fatigue cycling.[19-21] Further research is also needed regarding dentin bonding with self-etching adhesives and the relationship of shear fatigue limit (SFL) and SBS. The purpose of this study was to use fatigue testing to examine the effect of phosphoric acid etching of enamel and dentin, prior to the application of self-etching adhesives, for bonding resin composite, the latter group being included to test the effect of inadvertent exposure of dentin to phosphoric acid during selective enamel etching. The null hypothesis proposed was that pre-etching with phosphoric acid does not affect the SBS and SFL regardless of tooth substrate (enamel/dentin) and self-etching adhesive system (material).

## METHODS AND MATERIALS

### Study Materials

The materials used in this study are shown in Table 1. The self-etching adhesives used were 1) G- ænial Bond [GB]; (GC Corporation, Tokyo, Japan); 2) OptiBond XTR [OX]; (Kerr Corp, Orange, CA, USA); and 3) Scotchbond Universal [SU]; (3M ESPE Dental Products, St Paul, MN, USA). The phosphoric acid pre-etching agent was Ultra-Etch (Ultradent Products Inc, South Jordan, UT, USA). The resin composite used for the bonding procedure was Z100 Restorative (Shade A2; 3M ESPE).

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Table 1: *Study Materials*

| | Manufacturer | Main Components | Code |
|---|---|---|---|
| **Adhesive** | | | |
| G-ænial Bond, Lot No. 1102221 | GC Corporation<br>Tokyo, Japan | 4-META, UDMA, TEGDMA, phosphoric acid monomer, acetone, water, silanated colloidal silica, initiator | GB |
| OptiBond XTR<br>Primer: Lot No. 4483016<br>Adhesive: Lot No. 4544058 | Kerr Corp<br>Orange, CA, USA | Primer: GPDM phosphate monomer, HEMA, dimethacrylate monomers, acetone, ethyl alcohol, water, initiator<br>Adhesive: ethyl alcohol, dimethacrylate monomers, barium aluminoborosilicate glass, fumed silica, sodium hexafluorosilicate | OX |
| Scotchbond Universal,<br>Lot No. 451192 | 3M ESPE Dental Products<br>St Paul, MN, USA | MDP phosphate monomer, HEMA , dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane | SU |
| **Pre-etching agent** | | | |
| Ultra-Etch,<br>Lot No. G017 | Ultradent Products Inc<br>South Jordan, UT, USA | 35% Phosphoric acid | |
| **Resin composite** | | | |
| Z100 Restorative,<br>Lot No. N416713 (Shade A2) | 3M ESPE Dental Products<br>St Paul, MN, USA | Zirconia/silica, 0.01-3.5 μm<br>Filler load: 84.5% weight, 66% volume | |

*Abbreviations: GPDM – glycero-phosphate-dimethacrylate; HEMA – hydroxyethylmethacrylate; 4-META – 4-methacryloxyethyl trimellitate anhydride; MDP – methacryloyloxydecyl dihydrogen phosphate; TEGDMA – trietyleneglycoldimethacrylate; UDMA – urethanedimethacrylate*

## Specimen Preparation

The enamel/dentin bonding sites were prepared by sectioning extracted human molar teeth mesiodistally and then removing approximately two-thirds of the apical root structure. The buccal and lingual tooth sections were mounted with Triad DuaLine (DENTSPLY International, York, PA, USA) in 25-mm-diameter brass rings. The enamel/dentin bonding surfaces were ground flat to 4000 grit using a water coolant and a sequence of carbide polishing papers (Struers Inc, Cleveland, OH, USA).

Metal rings machined from 304 stainless steel with an inner diameter of 2.4 mm, an outer diameter of 4.8 mm, and a length of 2.6 mm were used to bond resin composite (Z100 Restorative) to enamel/dentin surfaces for SBS and SFL tests. The bonding procedure resulted in a resin composite cylinder inside the ring that was 2.36 mm in diameter and approximately 2.5 mm in length. The ring was left in place for the tests.

## SBS Tests

Fifteen specimens each were used to determine initial SBS to enamel/dentin with and without phosphoric acid (15 seconds) pre-etching prior to the application of the adhesive. The adhesive agents were applied according to manufacturers' directions, as shown in Table 2.

Following the treatment of the enamel or dentin flat ground tooth surface with the adhesive agent, the metal ring was positioned over the bonding site and secured in place by clamping in a custom fixture. The resin composite material was placed into the ring using a condenser and polymerized for 40 seconds with a Spectrum 800 Curing Unit (DENTSPLY Caulk, Milford, DE, USA) set at 600 mW/cm$^2$. The bonded specimens were stored for 24 hours in distilled water at 37°C before testing. The specimens were loaded to failure at 1 mm/min using a MTS Insight machine and TestWorks 4 software (MTS Systems Corporation, Eden Prairie, MN, USA). A metal rod with a chisel-shaped end was used to apply the load on the metal ring immediately adjacent to the flat ground tooth surface. The SBS values (MPa) were calculated from the peak load at failure divided by the bonded surface area. After testing, the bonding-site tooth surfaces and resin composite cylinders were observed under an optical microscope (MZ16; Leica Microsystems Ltd, Heerbrugg, Switzerland) at a magnification of 20× to examine the bond failure site. The type of bond failure was based on the percentage of substrate area (adhesive - resin composite - enamel/dentin) observed on the debonded cylinders and tooth bonding sites and was recorded as 1) adhesive failure, 2) cohesive failure in composite, 3) cohesive failure in enamel or dentin, or 3) mixed failure—partial adhesive and partial cohesive.

## SFL Testing

A staircase method of fatigue testing described by Draughn[22] was used for SFL testing. Test specimens were made as described above for SBS testing. The lower load limit was set near zero (0.4 N), and the initial maximum load applied was 50%-60% of the SBS

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Table 2: *Application Protocol for Pre-etching and Self-etching Adhesives*

| Method code | Pre-etching Protocol | Adhesive Application Protocol |
|---|---|---|
| With | 1. Enamel/dentin surface was phosphoric acid conditioned for 15 s.<br>2. Conditioned surface was rinsed with water for 15 s (three-way dental syringe) and air-dried. | |
| Without | Phosphoric acid pre-etching was not performed. | |
| Adhesive | | |
| GB | | 1. Adhesive applied to air-dried enamel/dentin surface for 10 s.<br>2. Adhesive light-cured for 10 s. |
| OX | | 1. Primer applied to air-dried enamel/dentin surface with rubbing action for 20 s. Medium air pressure applied to surface for 5 s.<br>2. Adhesive applied to primed surface with rubbing action for 15 s and then air-thinned for 5 s.<br>3. Primer/adhesive light-cured for 10 s. |
| SU | | 1. Adhesive applied to air-dried enamel/dentin surface with rubbing action for 20 s.<br>2. Gentle stream of air applied over the liquid adhesive for 5 s or until adhesive no longer moved and the solvent has completely evaporated.<br>3. Adhesive light-cured for 10 s. |

*Abbreviations: GB, G- ænial Bond; OX, OptiBond XTR; SU, Scotchbond Universal.*

determined for each of the adhesive systems tested. The load was applied at a frequency rate of 10 Hz with an ElectroPuls E1000 machine (Instron Worldwide Headquarters, Norwood, MA, USA) using a sine wave for 50,000 cycles or until failure occurred (Figure 1a,b). The load was incrementally adjusted upward or downward (depending on survival or failure) approximately 10% of the initial load. For each test condition 25 specimens were used to determine the SFL. After testing, the specimens were examined to define the location of the bond failure in the same manner as described above for SBS.

### Scanning Electron Microscopy (SEM) Observations

The restorative/tooth interfaces were observed by SEM. For the ultrastructure observation of the restorative/tooth interface, bonded specimens (stored in 37°C distilled water) were embedded in epoxy resin and then longitudinally sectioned with a diamond saw (Isomet Low Speed Saw, Buehler, Lake Bluff, IL, USA).

The sectioned surfaces were polished to a high gloss with abrasive discs (Fuji Star Type DDC, Sankyo Rikagaku Co Ltd, Saitama, Japan) followed by diamond pastes down to 0.25-μm particle size (DP-Paste, Struers, Ballerup, Denmark). The specimens were dehydrated in ascending grades of *tert*-butyl alcohol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for two hours) and then transferred from the final 100% bath to a critical-point dryer (Model ID-3, Elionix, Tokyo, Japan) for 30 minutes. The polished surfaces were then subjected to argon-ion beam etching (EIS-200ER, Elionix) for 45 seconds with the ion beam (accelerating voltage 1.0 kV, ion current density 0.4 $mA/cm^2$) directed perpendicular to the polished surfaces. The surfaces were coated in a vacuum evaporator with a thin film of gold. Observation was done under a SEM (FE-8000, Elionix) at an operating voltage of 10 kV.

### Statistical Analysis

A two-way analysis of variance (ANOVA) and Tukey post hoc test were used for analysis of SBS data, and a modified *t*-test with Bonferroni correction was used for SFL data.

## RESULTS

### SBS and SFL—Enamel

The study results for SBS and SFL for resin composite bonded to enamel with the three adhesive systems with and without phosphoric acid pre-etching are shown in Table 3.

The two-way ANOVA revealed that the factors (pre-etching vs no pre-etching and adhesive system) significantly influenced the SBS values ($p<0.001$). Interaction between the two factors was not statistically significant ($p=0.154$).

The SBS of resin composite to enamel produced by the three self-etching adhesives, after phosphoric

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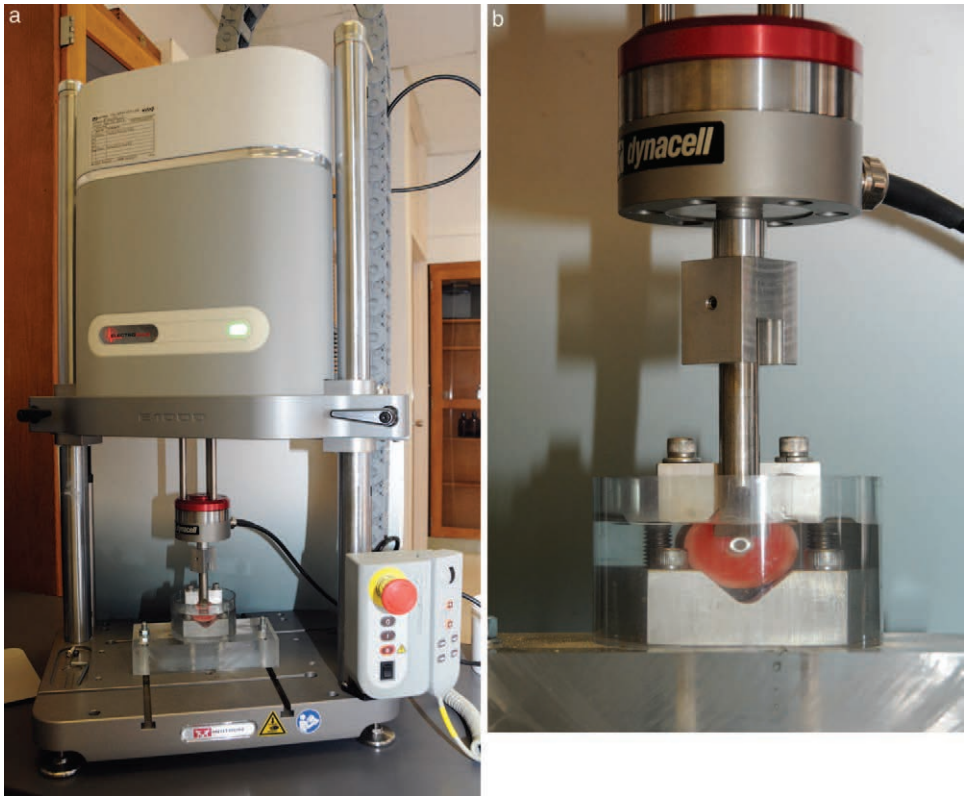


Figure 1. *(a) ElectroPuls E1000 testing machine. (b) Test specimen mounted in custom fixture on ElectroPuls E1000 testing machine.*

acid pre-etching, ranged from 42.0 ± 3.5 to 48.1 ± 6.1 MPa. The SBS for the same adhesives for enamel surfaces that were not pre-etched with phosphoric acid ranged from 27.7 ± 3.8 to 34.2 ± 3.8 MPa. For all three of the self-etching adhesives, the SBS of resin composite to enamel was significantly higher ($p<0.05$) in the phosphoric acid pre-etching group compared to the group without pre-etching.

There appeared to be a trend toward differences in the failure mode between enamel pre-etching groups and the groups without pre-etching. All three self-etching adhesive systems exhibited more cohesive failures in enamel with pre-etching when compared to surfaces that were not pre-etched. The predominant failure mode without phosphoric acid pre-etching of enamel was adhesive failure for all the self-etching adhesives.

The SFL of self-etching adhesives was significantly higher in specimens with phosphoric acid etching of enamel (19.6-25.1 MPa) than in specimens without phosphoric acid pre-etching (12.9-17.8 MPa). The predominant failure mode for all the self-etching adhesives without phosphoric acid pre-etching was adhesive failure. However, for the groups with phosphoric acid pre-etching, mixed failures and cohesive failures in enamel were increased for all of the self-etching adhesives. OX demonstrated a higher ratio of SFL/SBS than did SU and GB, regardless of whether the enamel was pre-etched with phosphoric acid or not pre-etched.

### SBS and SFL—Dentin

The results for SBS and SFL of resin composite bonded to dentin are shown in Table 4. The two-way ANOVA revealed that the factors (pre-etching vs no pre-etching and adhesive system) significantly influenced the SBS values ($p<0.001$) to dentin. Interaction between the two factors was not statistically significant ($p>0.05$).

The SBS of resin composite to dentin using GB was significantly lower in specimens with phosphoric acid pre-etching than in specimens without phosphoric acid etching. The SBS of SU and OX to dentin with phosphoric acid pre-etching tended to decrease compared to values associated with no pre-etching, but there was no significant difference ($p>0.05$) in SBS.

The failure site locations for OX and SU with phosphoric acid pre-etching tended to exhibit more mixed failures when compared to those with no pre-

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Table 3: *Influence of Phosphoric Acid Pre-etching of Enamel on Shear Bond Strength (SBS) and Shear Failure Limit (SFL), in MPa (Standard Deviation) [Failure-mode Percentages]* [a & b]

| $H_3PO_4$ | With SBS | Without SBS | With SFL |
|---|---|---|---|
| OX | 48.1 (6.1) a,A [73.0/6.7/13.3/6.7] | 34.2 (3.8) a,B [80.0/0.0/6.7/13.3] | 25.1 (1.8) a,A [50.0/33.3/16.7/0.0] |
| SU | 44.7 (6.1) ab,A [60.0/6.7/20.0/13.3] | 27.7 (3.8) b,B [86.7/0.0/0.0/13.3] | 21.7 (2.3) b,A [54.5/9.1/27.3/9.1] |
| GB | 42.0 (3.5) b,A [80.0/0.0/13.3/6.7] | 28.1 (4.0) b,B [93.3/0.0/0.0/6.7] | 19.6 (4.8) b,A [83.3/0.0/8.3/8.3] |

*Abbreviations: GB, G-* ænial *Bond; OX, OptiBond XTR; SBS, shear bond strength; SFL, shear fatigue limit; SU, Scotchbond Universal.*
[a] *Same lowercase letters in vertical columns are not different at the 5% significance level. Same capital letters between columns indicate no difference at the 5% significance level in $H_3PO_4$ pre-etching vs no pre-etching groups of the same adhesive.*
[b] *Failure mode percentages (adhesive failure/cohesive failure in resin composite/cohesive failure in enamel/mixed failure).*

etching. The predominant SBS failure mode for GB was adhesive failures for all the specimens tested regardless of phosphoric acid pre-etching or no pre-etching.

There was not a significant difference ($p>0.05$) between phosphoric acid pre-etching (20.3-25.3MPa) and no phosphoric acid pre-etching (17.6-22.6 MPa) for the SFL of SU and OX; however, the SFL tended to decrease in specimens with phosphoric acid pre-etching for these two self-etching adhesive systems. The SFL of the GB self-etching adhesive was significantly lower ($p<0.05$) with phosphoric acid pre-etching compared to values associated with no phosphoric acid pre-etching.

The predominant SFL testing failure mode with phosphoric acid pre-etching was adhesive failure for all of the self-etching adhesives. However, mixed failure and cohesive failure in dentin were increased for OX and SU without phosphoric acid pre-etching. For all the adhesives without phosphoric acid pre-etching of dentin, a higher ratio of SFL/SBS was found compared to the same adhesives with phosphoric acid pre-etching.

### SEM Observations

SEM observations of the restorative-enamel interface are shown in Figures 2 through 4. The restorative-enamel interface of all adhesives showed excellent adaptation regardless of phosphoric acid pre-etching or no pre-etching. The resin tags into the enamel surfaces were longer for the groups with phosphoric acid pre-etching when compared to those associated with no pre-etching.

SEM observations of the restorative-dentin interface are shown in Figures 5 through 7. The restorative-dentin interface of all adhesives showed excellent adaptation to the dentin surface. For the groups with phosphoric acid conditioning and using SU and OX, a hybrid layer of approximately 3-5 μm was found between resin adhesive and tooth structure (Figures 5a,b and 7a,b), compared to a layer of 2-5 μm for the GB adhesive (Figure 6a,b). For the SU and GB groups without pre-etching with phosphoric acid there was formation of a thin transitional layer between the adhesive resin and tooth structure (Figures 5c,d and 6c,d). A dentin hybrid layer was observed for OX regardless of phosphoric acid pre-etching (Figure 7a,b) or no phosphoric acid pre-etching (Figure 7c,d). The thickness of the hybrid layer using OX with phosphoric acid pre-etching (Figure 7a,b) was greater (3-5 μm) when compared to dentin surfaces without pre-etching with phosphoric acid (Figure 7c,d).

## DISCUSSION

Laboratory bond strength tests are a common approach to evaluating the potential effectiveness of adhesive systems. Over the years, many studies have

Table 4: *Influence of Phosphoric Acid Pre-etching of Dentin on Shear Bond Strength (SBS) and Shear Failure Limit (SFL), in MPa (Standard Deviation) [Failure-mode Percentages]* [a & b]

| $H_3PO_4$ | With SBS | Without SBS | With SFL |
|---|---|---|---|
| OX | 44.8 (8.1) a,A [33.3/13.3/20.0/33.3] | 50.9 (4.9) a,A [0.0/33.3/40.0/26.7] | 22.2 (6.2) a,A [63.6/0.0/9.1/27.3] |
| SU | 39.2 (6.7) b,A [33.3/6.7/40.0/20.0] | 42.6 (4.0) b,A [20.0/20.0/46.7/13.3] | 17.6 (1.4) b,A [81.3/0.0/0.0/18.7] |
| GB | 24.6 (2.8) c,A [100.0/0.0/0.0/0.0] | 31.1 (3.8) c,B [100.0/0.0/0.0/0.0] | 11.5 (2.8) c,A [100.0/0.0/0.0/0.0] |

*Abbreviations: GB, G-* ænial *Bond; OX, OptiBond XTR; SBS, shear bond strength; SFL, shear fatigue limit; SU, Scotchbond Universal.*
[a] *Same lowercase letters in vertical columns are not different at the 5% significance level. Same capital letters between columns indicate no difference at the 5% significance level in $H_3PO_4$ pre-etching vs no pre-etching groups of the same adhesive.*
[b] *Failure mode percentages (adhesive failure/cohesive failure in resin composite/cohesive failure in dentin/mixed failure).*

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Table 3: *Influence of Phosphoric Acid Pre-etching of Enamel on Shear Bond Strength (SBS) and Shear Failure Limit (SFL), in MPa (Standard Deviation) [Failure-mode Percentages] [a & b] (ext.)*

| $H_3PO_4$ | Without SFL | With Ratio SFL/SBS | Without Ratio SFL/SBS |
|---|---|---|---|
| OX | 17.8 (1.9) a,B [90.0/0.0/0.0/10.0] | .522 | .520 |
| SU | 12.9 (1.5) b,B [100.0/0.0/0.0/0.0] | .485 | .466 |
| GB | 13.4 (2.5) b,B [100.0/0.0/0.0/0.0] | .467 | .477 |

been reported in the literature regarding the bonding effectiveness of adhesive systems to mineralized tooth structures using shear bond strength tests and μ-TBS testing. These types of laboratory tests routinely use a monotonically increasing force to the bonded assembly until failure occurs, while in most clinical situations, adhesively bonded restorations are typically subjected to subcritical loading during function.[17-21,23] While repeated loads typically encountered in the oral cavity are insufficient to provoke failure, they induce damage by generating cracks that grow over time and eventually result in deterioration of adhesively bonded restorations through marginal failure or, in extreme cases, bulk fracture.

Fatigue can be defined as the degradation or failure of mechanical properties after repeated applications of stress at a level well below the ultimate fracture strength of the material or interface.[24] Consequently, fatigue tests provide not only information on the ability of a material or interface to resist the development of cracks but also information related to the endurance characteristics of a bonding system (ie, materials and technique).

A popular method of fatigue testing, referred to as the staircase method, involves selecting a starting stress of approximately one-half of the ultimate strength. The load is applied at a set frequency until the specimen survives a specific number of cycles or fails during the cycling. Draughn[22] developed an analytical approach to determining the fatigue strength of materials using the staircase method. Previous studies[19] have demonstrated that fatigue limits are much lower than the initially measured SBS and may be on the order of 40%-60% of that strength.

In the present study, SBS values were determined for two single-step self-etching adhesives (SU and GB) and a two-step self-etching adhesive (OX) to provide a relative comparison of the bonding performance to both enamel and dentin using a resin composite material. The results of these SBS tests were subsequently used as baseline values for SFL testing. The results of this study demonstrated that OX produced higher SBS and SFL to enamel ($p<0.05$), when compared to SU and GB, with the exception of the SBS of SU when the enamel was pre-etched with phosphoric acid. Additionally, regardless of phosphoric acid pre-etching or no pre-etching, OX demonstrated a higher ratio of SFL/SBS than did SU and GB. These results were consistent with those of previous studies[4,25-27] comparing multiple-step self-etching adhesives and single-step self-etching adhesives with SBS and μ-TBS testing of enamel and dentin.

Recently, single-step self-etching adhesives have been advocated to reduce the number of application steps and eliminate technique-sensitive factors that may negatively impact the ability of resin materials to bond to enamel and dentin. Single-step self-etching adhesives combine the functions of a self-etching primer (acid monomer) and a bonding agent. These types of adhesives typically contain both hydrophilic and hydrophobic monomers and require a high concentration of solvent to keep them in solution.[28,29] Additionally, most incorporate water, which is essen-

Table 4: *Influence of Phosphoric Acid Pre-etching of Dentin on Shear Bond Strength (SBS) and Shear Failure Limit (SFL), in MPa (Standard Deviation) [Failure-mode Percentages][a & b] (ext.)*

| $H_3PO_4$ | Without SFL | With Ratio SFL/SBS | Without Ratio SFL/SBS |
|---|---|---|---|
| OX | 25.3 (3.7) a,A [23.0/0.0/38.5/38.5] | .496 | .497 |
| SU | 20.3 (2.7) b,A [75.0/0.0/8.3/16.7/0] | .449 | .477 |
| GB | 15.9 (2.2) c,B [100.0/0.0/0.0/0.0] | .467 | .511 |

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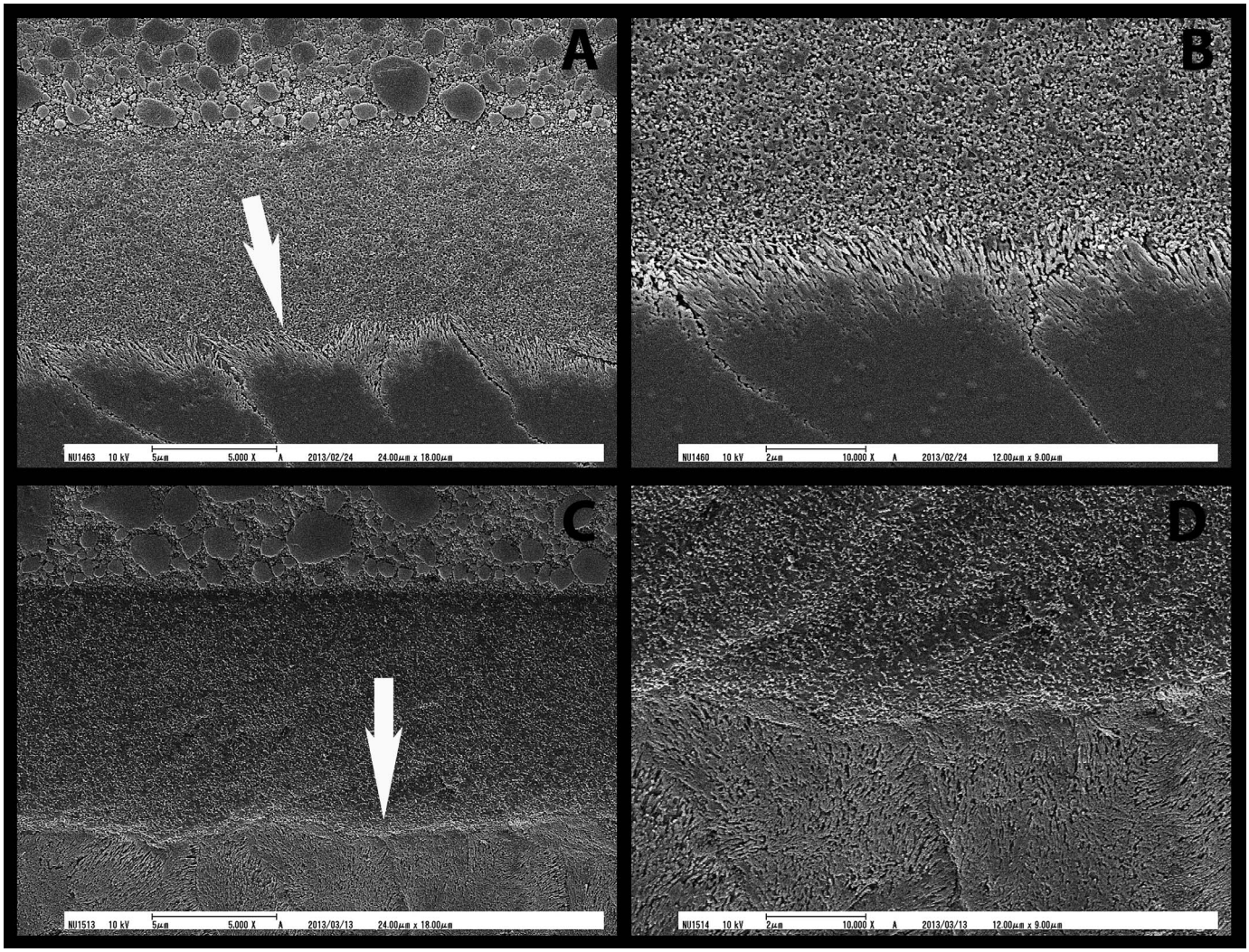


Figure 2. *(a) Scotchbond Universal—restorative/enamel interface with phosphoric acid pre-etching (5000×). (b) Scotchbond Universal—restorative/enamel interface with phosphoric acid pre-etching (10,000×). (c) Scotchbond Universal—restorative/enamel interface without phosphoric acid pre-etching (5,000×). (d) Scotchbond Universal—restorative/enamel interface without phosphoric acid pre-etching (10,000×).*

tial as an ionization medium, to enable self-etching activity to occur.[30]

Previous studies[31,32] have reported that one of the characteristics or vulnerabilities of single-step adhesives is phase separation, which may result in droplets and blisters in the adhesive layer and at the interface of the tooth substrate and the adhesive layer. In addition, single-step self-etching adhesives may contain high concentrations of water, which purportedly lower the degree of conversion.[29] These drawbacks might reduce the mechanical properties of an adhesive layer and also produce a weak point, resulting in crack initiation and propagation. The composition (Table 1) of the two-step adhesive system in this study (OX) may result in improved physical or mechanical properties when compared to the single-step adhesives (SU and GB). Improvement in physical properties may relate closely to the higher SBS and SFL exhibited by OX.

In the present study, phosphoric acid pre-etching of enamel significantly increased SBS and SFL. The range of SBS was 42.0 ± 3.5 to 48.1 ± 6.1 MPa for phosphoric acid pre-etched enamel surfaces, compared to 27.7 ± 3.8 to 34.2 ± 3.8 MPa for surfaces that were not pre-etched. The SFL ranged from 19.6 ± 4.4 to 25.1 ± 1.8 MPa for phosphoric acid pre-etched enamel surfaces, compared to a range of 12.9 ± 1.5 to 17.8 ± 1.9 MPa for surfaces without pre-etching. SEM observations also revealed that resin-tags into the enamel bonding sites were longer and more extensive for the groups with phosphoric acid pre-etching when compared to groups without

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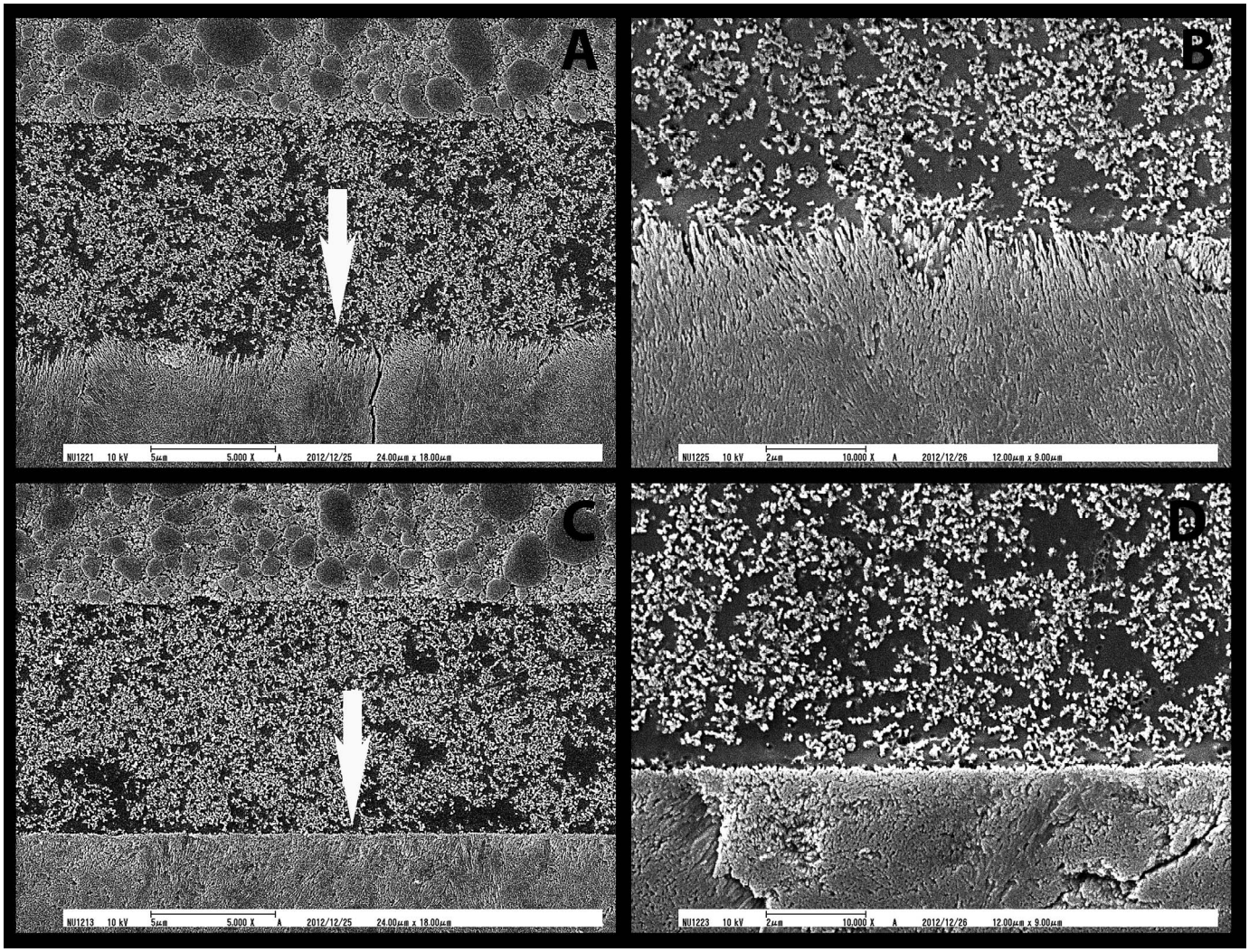


Figure 3. *(a) G-*ænial *Bond—restorative/enamel interface with phosphoric acid pre-etching (5000×). (b) G-*ænial *Bond—restorative/enamel interface with phosphoric acid pre-etching (10,000×). (c) G-*ænial *Bond—restorative/enamel interface without phosphoric acid pre-etching (5000×). (d) G-*ænial *Bond—restorative/enamel interface without phosphoric acid pre-etching (10,000×).*

phosphoric acid pre-etching (Figures 2a through 4d). Therefore, the null hypothesis that phosphoric acid pre-etching does not affect the SBS and SFL of enamel was rejected for all of the adhesive systems.

Some recent studies[1-7] on bonding to enamel have found that self-etching adhesive systems, whether two-step or one-step systems, have inferior bond strengths compared with total-etch systems. In addition, pre-etching of enamel with phosphoric acid was shown[12,33,34] to improve the bond strengths of both two-step and single-step self-etching adhesives when compared with bond strengths achieved without pre-etching. Also, SEM studies[1,7,35] examining the morphology of enamel surfaces revealed that the application of a self-etching primer and single-step adhesives did not create as deep of an enamel etching pattern as did phosphoric acid conditioning.

Over the years phosphoric acid has become the standard procedure for conditioning to modify the enamel structure prior to the application of adhesive bonding agents. The infiltration of an adhesive resin monomer into the porous zone results in the formation of resin-tags, thereby establishing micromechanical retention to the etched enamel. Phosphoric acid treatment of enamel increases the bonding area and wettability of the adherent surface. Treating enamel with phosphoric acid improves the surface free energy by about 30% compared with treating with a silica-carbide paper (grit #180, #600, and #2000) ground enamel surface without phosphoric acid treatment.[36]

With the introduction of self-etching dental adhesives to the profession, phosphoric acid pre-etching

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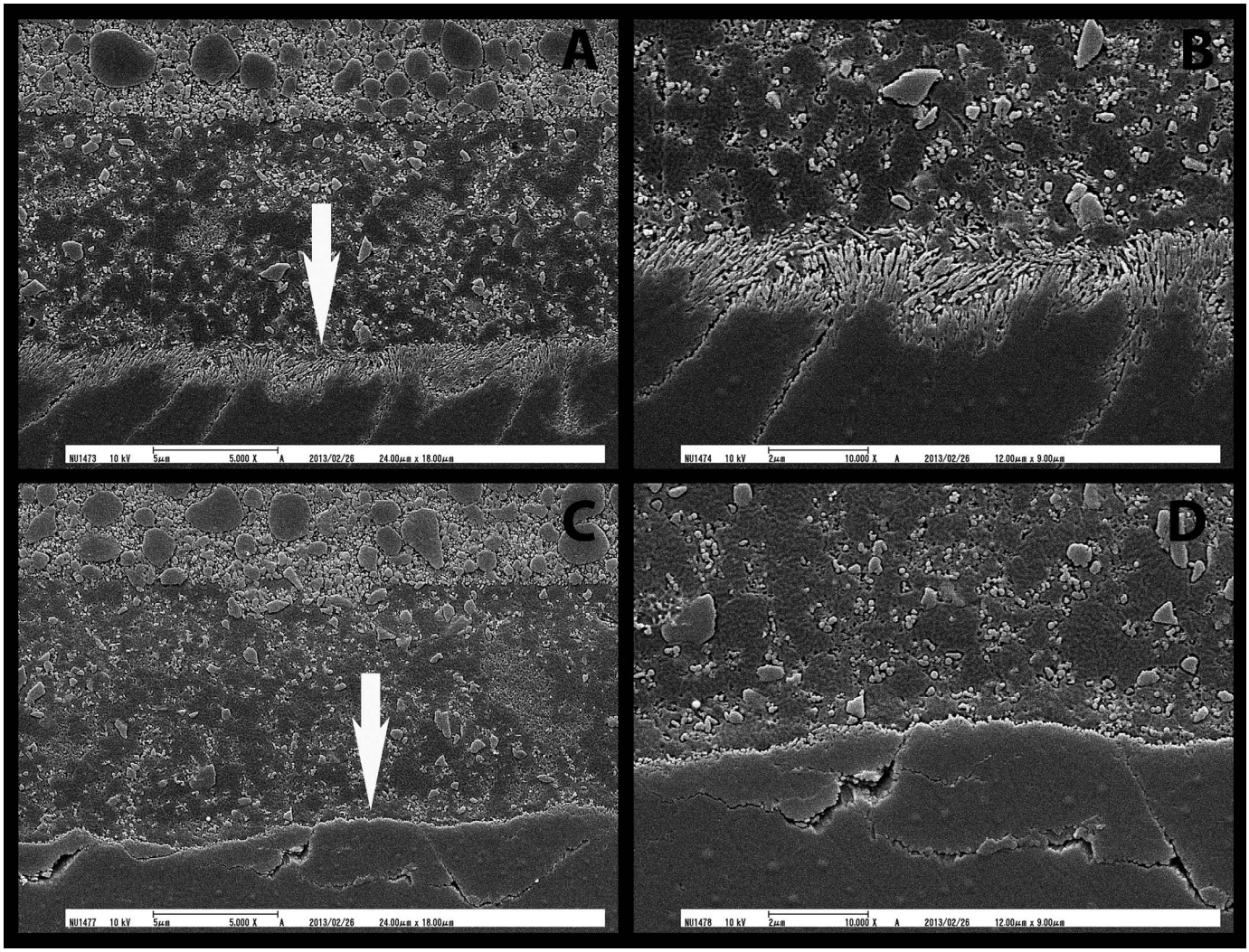


Figure 4. *(a) Optibond XTR—restorative/enamel interface with phosphoric acid pre-etching (5000×). (b) Optibond XTR—restorative/enamel interface with phosphoric acid pre-etching (10,000×). (c) Optibond XTR—restorative/enamel interface without phosphoric acid pre-etching (5000×). (d) Optibond XTR—restorative/enamel interface without phosphoric acid pre-etching (10,000×).*

of enamel is not routinely recommended by manufacturers. The evidence from several studies,[7,12,19] including the present study, comparing phosphoric acid pre-etching of enamel vs no pre-etching clearly shows increased enamel bond strengths following pre-etching.

Bonding resin-based materials to dentin continues to be a challenge. 2-hydroxyethyl methacrylate (HEMA) is a water-soluble methacrylate monomer frequently employed in dental adhesives. HEMA purportedly enhances the wetting properties of the adhesive solution and the penetration efficacy of the adhesive into demineralized dentin as a result of its polar properties and small dimensions.[37,38] HEMA has been reported[39] to have the capability of increasing the bond strength to dentin. Because of its hydrophilic character, HEMA is frequently used as an ingredient to improve miscibility of both the hydrophobic and hydrophilic components of adhesive solutions to prevent phase separation.[31]

While HEMA has known advantages for adhesive bonding to mineralized tooth structures, concerns have been expressed about the use of HEMA in adhesive dental products. One of the reported drawbacks of HEMA is the potential for allergenic issues, especially to the skin.[40,41] Other problems reported[29,42] include deterioration of mechanical properties of the polymerized adhesive caused by its characteristics, which enhance water uptake, swelling, and staining.

In an effort to reduce possible allergenic problems and to achieve long-term durability, some single-step

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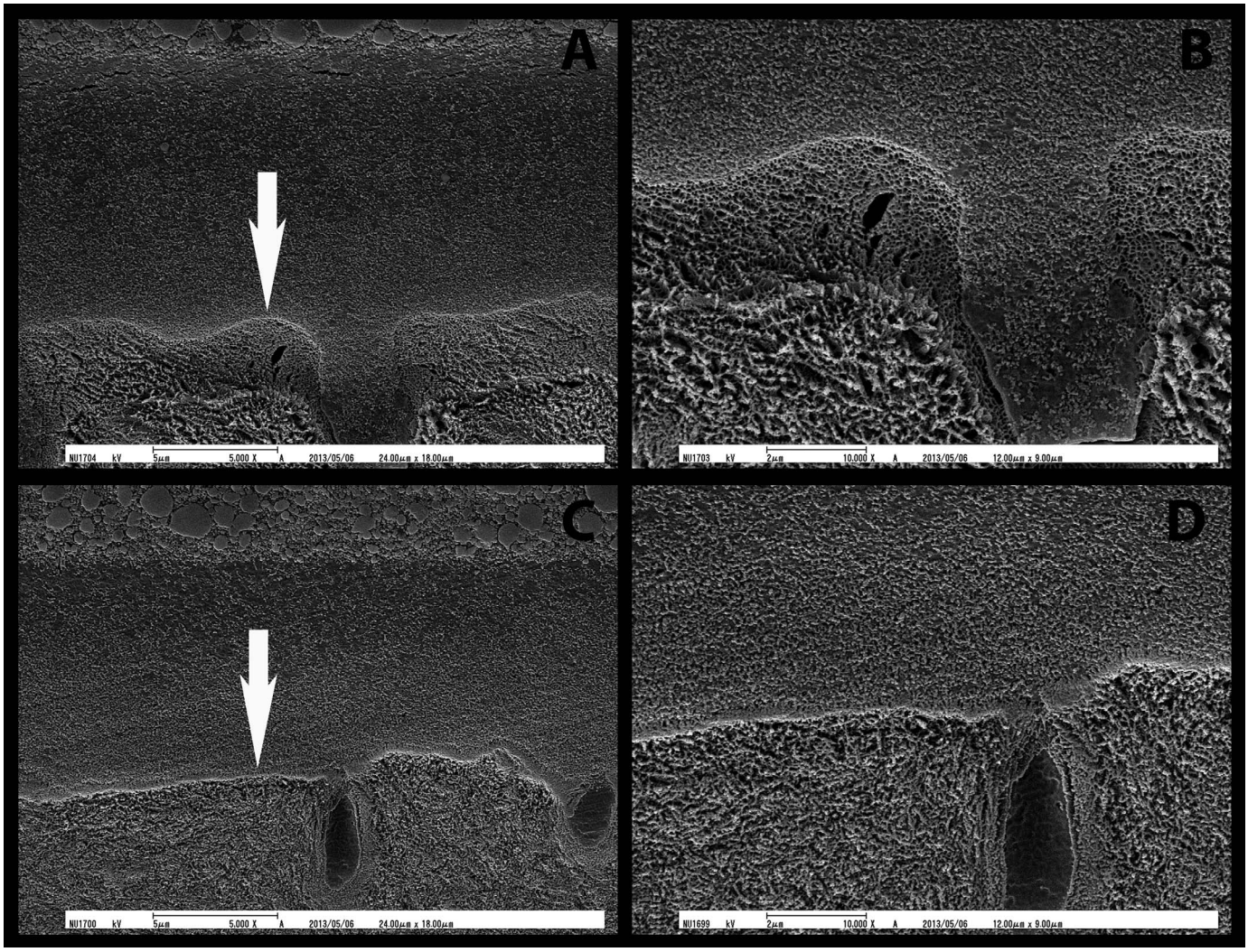


Figure 5. *(a) Scotchbond Universal—restorative/dentin interface with phosphoric acid pre-etching (5000×). (b) Scotchbond Universal—restorative/dentin interface with phosphoric acid pre-etching (10,000×). (c) Scotchbond Universal—restorative/dentin interface without phosphoric acid pre-etching (5000×). (d) Scotchbond Universal—restorative/dentin interface without phosphoric acid pre-etching (10,000×).*

self-etching adhesives do not include HEMA in the composition. In this study, SU and OX contain HEMA; however, GB does not contain HEMA as an ingredient and is classified as a HEMA-free, single-step self-etching adhesive. The results of this study show that the SBS and SFL to dentin for SU and OX tended to decrease with phosphoric acid pre-etching of the dentin surface prior to the application of the adhesives but that this decrease was not significant ($p>0.05$). For the HEMA-free GB adhesive, phosphoric acid pre-etching of the dentin surface significantly decreased ($p<0.05$) SBS and SFL, and the values were significantly less ($p<0.05$) than for OX and SU. Therefore, the null hypothesis that phosphoric acid pre-etching does not affect the SBS and SFL of dentin was not rejected for SU and OX, but it was rejected for GB.

The SEM examinations of the adhesive interface with the mineralized tooth structures revealed differences among the self-etching systems and with phosphoric acid pre-etching vs no pre-etching. With phosphoric acid pre-etching of dentin, a hybrid layer of approximately 2-5 μm was observed with the GB adhesive (Figure 6a,b), compared to a hybrid layer in the range of 3-5 μm for both the SU and OX adhesives (Figures 5a,b and 7a,b). A typical hybrid layer was not found with SU and GB when phosphoric acid pre-etching was not used on dentin (Figures 5c,d and 6c,d). For the OX adhesive, a hybrid layer was found with or without the use of phosphoric acid pre-etching of dentin; however, the width of the hybrid layer with phosphoric acid pre-etching (Figure 7a,b) was approximately double (3-5

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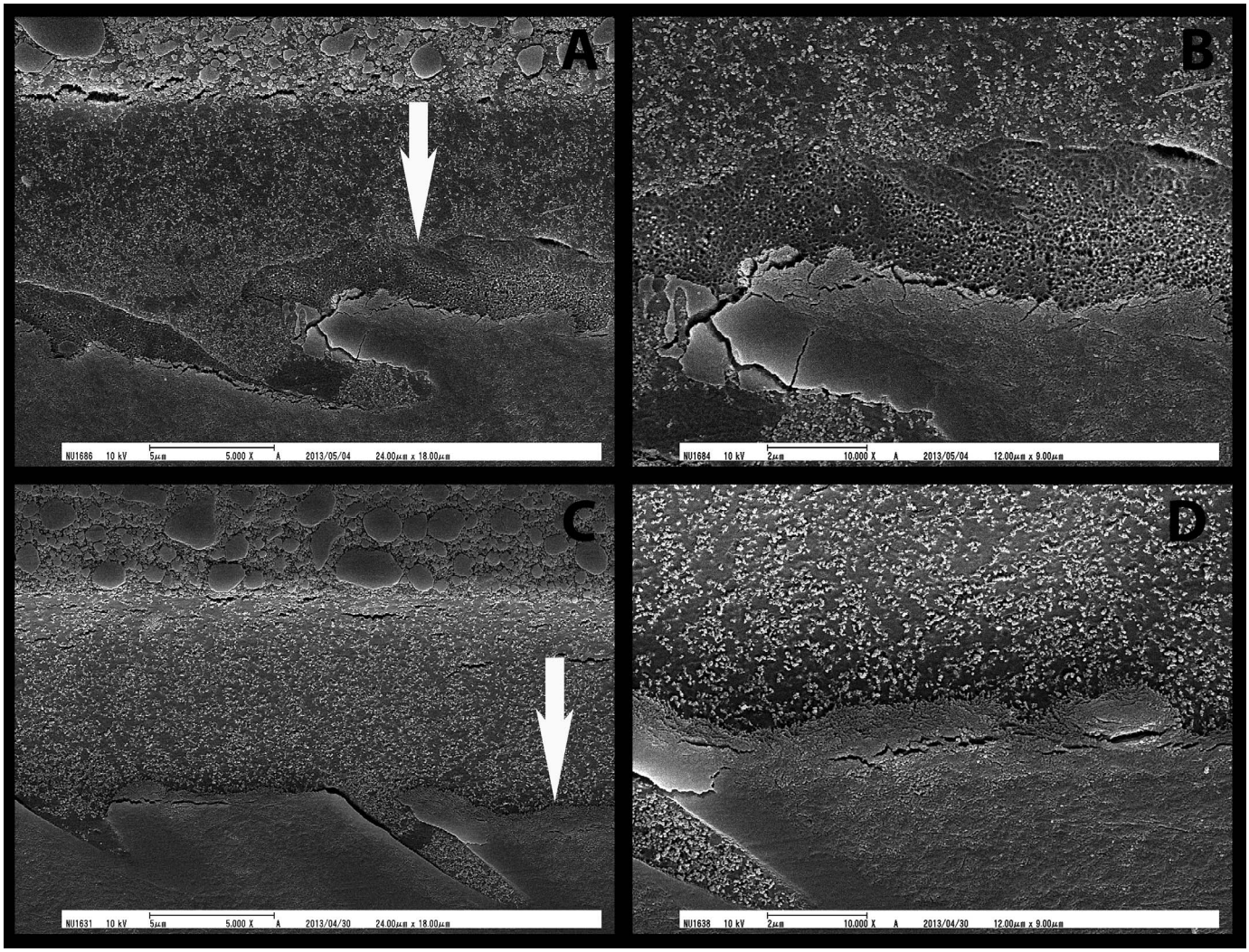


Figure 6. *(a) G-*ænial *Bond—restorative/dentin interface with phosphoric acid pre-etching (5000×). (b) G-*ænial *Bond—restorative/dentin interface with phosphoric acid pre-etching (10,000×). (c) G-*ænial *Bond—restorative/dentin interface without phosphoric acid pre-etching (5000×). (d) G-* ænial *Bond—restorative/dentin interface without phosphoric acid pre-etching (10,000×).*

μm) that found without phosphoric acid pre-etching (Figure 7c,d).

Phosphoric acid conditioning of dentin, followed by air-drying of the treated surface, has been shown[43] to result in collapsed collagen fibrils that inhibit resin monomer penetration into the entire depth of the decalcified dentin. Adhesives that contain hydrophilic HEMA and water, such as the SU single-step self-etching adhesive and the OX two-step self-etching adhesive, may help to re-expand collapsed collagen fibrils and enhance the penetration of adhesive monomers into demineralized dentin. In an effort to further facilitate bonding to mineralized tooth structures, SU also contains 10-methacryloxydecyl dihydrogen phosphate (MDP) and Vitrebond copolymer. The MDP purportedly has the potential to develop chemical bonds to hydroxyapatite, and the Vitrebond copolymer is included to develop ionic bonds to hydroxyapatite and/or collagen.[44,45] However, even if hydrophilic components of an adhesive have penetrated into demineralized surfaces, it is possible that the resin components themselves may have been hampered in penetrating the exposed collagen network, leading to a decrease in bond strength.

The GB adhesive demonstrated statistically significant ($p<0.05$) lower SBS and SFL values when using phosphoric acid pre-etching of the dentin. While a HEMA-free single-step adhesive may have benefits for long-term bonding durability,[46-48] it might induce droplets and blisters in the adhesive layer or on the interface due to phase

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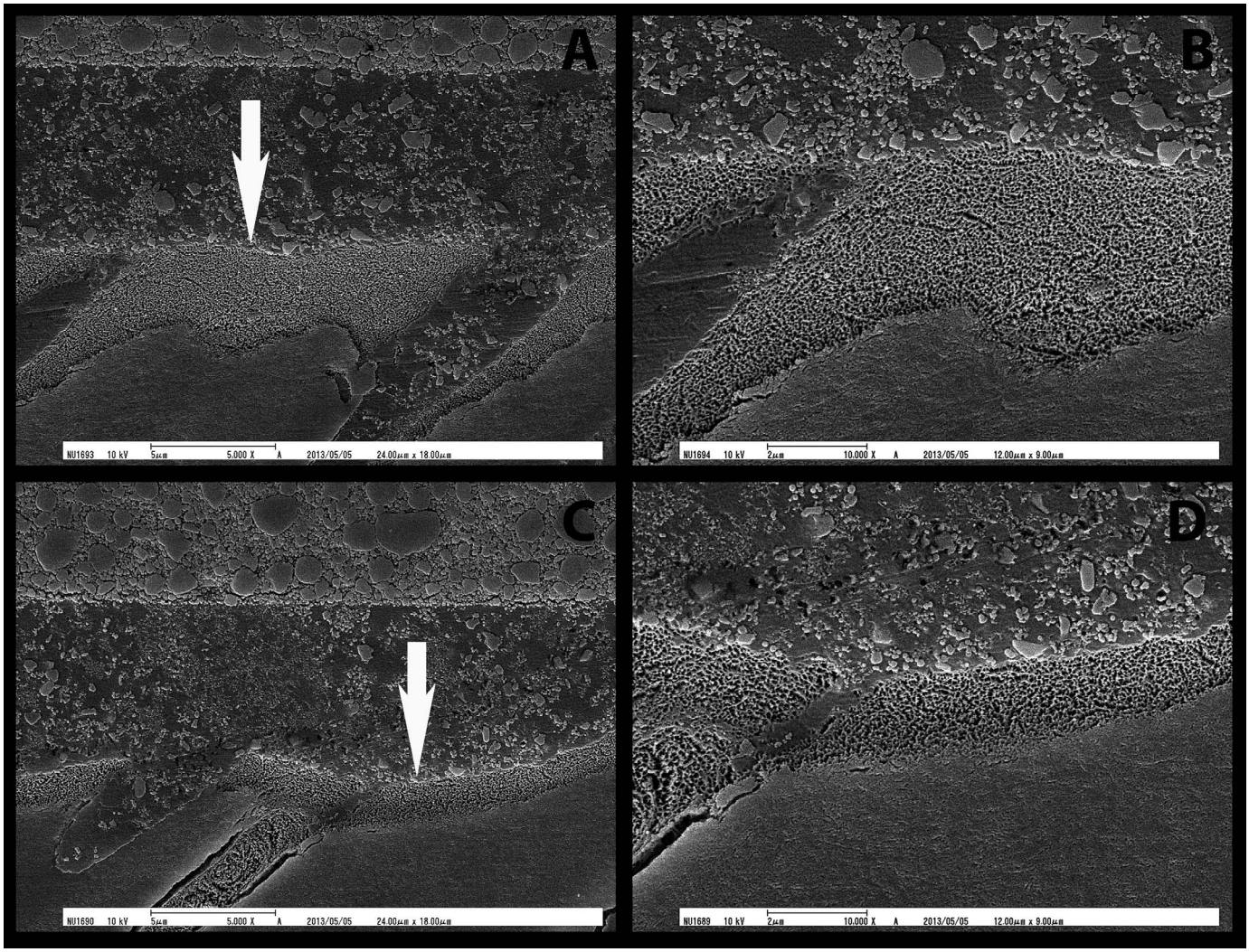


Figure 7. *(a) Optibond XTR—restorative/dentin interface with phosphoric acid pre-etching (5000×). (b) Optibond XTR—restorative/dentin interface with phosphoric acid pre-etching (10,000×). (c) Optibond XTR—restorative/dentin interface without phosphoric acid pre-etching (5000×). (d) Optibond XTR—restorative/dentin interface without phosphoric acid pre-etching (10,000×).*

separation or the more hydrophobic nature of the components.[31] It may be possible that these defects provide vulnerable regions for crack initiation and propagation from load stress resulting in lower bond strength. In addition, a previous study[49] of the GB adhesive demonstrated that resin tag formation, produced under vacuum treatment of dentin, did not contribute to the μ-TBS. Therefore, the principal method of adhesion to the dentin for GB was not dependent on resin tag formation but on chemical bonding to the hydroxyapatite. From this perspective, using phosphoric acid pre-etching on dentin may have extensively removed hydroxyapatite from the surface, which is necessary to achieve chemical bonding, resulting in an adverse effect on the dentin bonding of GB. The results of the present study clearly demonstrated a decrease in SBS and SFL with the GB system when the dentin surface was pre-etched with phosphoric acid and support the theory that the loss of mineralized dentin from the surface resulted in lower adhesion because of reduced chemical bonding.

Fatigue failure is a result of nucleation of microcracks, propagation and eventual coalescence of cracks leading to catastrophic failure.[24] Nucleation is considered to occur at flaws in the materials or at interfaces, such as scratches, voids, and inclusions, which might be weak points where high stress intensities can develop under load applications. In addition, it is important to consider the influence of different adherent substrates, as well as mechanical properties of adhesives, and crack propagation from repeated subcritical loading.

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Manufacturers' directions for self-etching adhesives now often give practitioners a choice of whether to use phosphoric acid pre-etching on enamel. The SBS and SFL data in the present study clearly show that phosphoric acid conditioning of enamel prior to the application of the three adhesive systems tested improved the adhesion of a resin composite material (Table 3). The opposite was true for dentin. The SBS and SFL to dentin for all three systems were decreased with phosphoric acid pre-etching (Table 4).

It is interesting to note that the system (OX) that produced the highest SBS and SFL values also yielded the highest ratio of SFL/SBS for both enamel and dentin. While the SBS values provide a relative ranking of the adhesive characteristics of a system, the SFL value is a better measure of endurance. The data in Table 3 for enamel and in Table 4 for dentin demonstrate clear differences in the resistance of bonds to failure with cyclic loading.

It might be speculated that the reason for differences in SFL between enamel and dentin were caused by not only physical properties of adherent substrates themselves but also by adhesive layer conversion after evaporation of water and solvent. Enamel is homogeneous in nature and is primarily composed of hydroxyapatite. In comparison, dentin is heterogeneous, consisting of hydroxyapatite and collagen. Overall, the water content of dentin is significantly higher than that of enamel, and this may influence the effectiveness of evaporation of solvents, which may result in a reduction of the degree of conversion, leading to lower mechanical properties. From the results of the failure modes observed in dentin bonding, adhesive mode failures tended to increase in SBS and SFL specimens with phosphoric acid pre-etching, except in the case of the GB adhesive (GB demonstrated 100% adhesive failure for all conditions). On the other hand, failure modes of enamel for all three adhesives demonstrated a different trend between SBS and SFL; adhesive mode failures tended to decrease in SBS, when the use of phosphoric acid pre-etching was compared to the same self-etch system without the use of pre-etching, but in SFL adhesive mode failures tended to increase.

The clinical relevance of fatigue testing gains strength from an earlier study[50] showing a larger percentage of gap formation at resin composite/enamel margins for self-etching systems when compared to etch-and-rinse adhesives. Frankenberger and Tay[50] conducted a study comparing marginal gap formation of Class II resin composite restorations bonded with both etch-and-rinse and self-etching adhesives using simultaneous mechanical loading and thermocycling (thermomechanical fatigue loading, TML). In reporting the results for marginal quality in enamel, these authors reported that while all adhesive systems showed a significant loss ($p<0.05$) of gap-free margins after TML compared to results prior to TML, etch-and-rinse systems performed significantly better than did self-etching systems. Dentin margins, like enamel, showed a high percentage of gap-free margins before TML. All the adhesive systems exhibited a significant decline in the percentage of gap-free dentin margins after TML. The results of their study clearly show the effect of TML fatigue loading on both enamel and dentin margins of adhesively bonded Class II resin composite restorations. In addition, clinical studies[13,47,51-54] have found increased marginal breakdown for restorations bonded with self-etching adhesives compared to those with etch-and-rinse adhesives. Since some of the self-etch adhesives may have laboratory bond strengths that are similar to those of the etch-and-rinse adhesives, the reason for more and earlier failures may be attributed to lower fatigue resistance.

Generally, failure in fatigue testing is the result of cumulative damage over time. How different adherent substrates, especially enamel and dentin, are related to crack propagation and bond failures requires further investigation. Additional fatigue testing is needed to investigate the long-term durability of adhesively bonded materials to mineralized tissues.

## CONCLUSIONS

Phosphoric acid pre-etching of enamel produced significantly higher ($p<0.05$) SBS and SFL of a resin composite bonded to enamel using three self-etching adhesive systems when compared to similar values associated with no pre-etching. The use of phosphoric acid pre-etching on dentin prior to the application of the three adhesive systems resulted in a decrease in SBS and SFL when compared to values obtained using the adhesives without pre-etching with phosphoric acid.

SBS and SFL testing of bonds using phosphoric acid etching prior to application of the self-etch adhesives clearly demonstrated different tendencies between enamel and dentin. The **e**ffect of phosphoric acid pre-etching (prior to application of self-etch adhesives) on bond performance was adhesive

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system and tooth substrate dependent. The results of this study have significant implications for the selection and technical use of self-etching adhesive systems.

### 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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# Contamination of Composite Resin by Glove Powder and Saliva Contaminants: Impact on Mechanical Properties and Incremental Layer Debonding

NM Martins • GU Schmitt • HL Oliveira
MM Madruga • RR Moraes • MS Cenci

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### Clinical Relevance

The handling technique is important for the mechanical performance and incremental layer bond strength of composite restorations. Composites can be digitally manipulated, but gloves should be clean to avoid the negative effects of contamination.

## SUMMARY

**This study investigated the influence of digital manipulation of a composite resin (Z250; 3M ESPE, St Paul, MN, USA) with gloves contaminated with powder and/or human stimulated saliva on the mechanical properties and incremental layer debonding of the restorative. The six groups tested were powdered gloves with or without saliva, powder-free gloves with or without saliva, powdered gloves with saliva cleaned with 70% ethanol, and no digital manipulation or contamination (control). Diametral tensile strength, flexural strength, flexural modulus, and incremental layer shear bond strength were evaluated. Each composite increment was digitally manipulated for 10 seconds. Data from each test were separately analyzed using analysis of variance and the Student-Newman-Keuls test (α=0.05). No significant differences for diametral tensile strength were observed. Manipulation of the composite using powder-free gloves with saliva or using gloves cleaned with ethanol generated higher flexural strength and modulus**

Nathalia M Martins, DDS, Federal University of Pelotas, Pelotas, Brazil

Gabriela U Schmitt, Federal University of Pelotas, Pelotas, Brazil

Héllen L Oliveira, DDS, MSc, Graduate Program in Dentistry, Federal University of Pelotas, Pelotas, Brazil

Marina M Madruga, DDS, MSc, Graduate Program in Dentistry, Federal University of Pelotas, Pelotas, Brazil

Rafael R Moraes, DDS, MS, PhD, Federal University of Pelotas, Pelotas, Brazil

*Maximiliano S Cenci, DDS, MS, PhD, Federal University of Pelotas, Pelotas, Brazil

*Corresponding author: R. Gonçalves Chaves. 457 Pelotas, N/A 96015560, Brazil; e-mail: cencims@gmail.com

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**compared to the other groups. The control group and the group manipulated using powdered gloves with saliva generally showed lower mechanical performances. Lower incremental layer bond strength was observed for the group manipulated with powdered gloves without saliva. The control group and the groups manipulated with powdered gloves with saliva or cleaned with ethanol showed higher shear bond strengths. Most of the failures were cohesive. In conclusion, digital manipulation might be important for the composite resin to achieve better mechanical performance and incremental layer bond strength, provided that the gloves are not contaminated. Cleaning the gloves with ethanol might avoid the negative effects of digital manipulation using contaminated gloves.**

## INTRODUCTION

The insertion of composite resin into a cavity preparation is one of the most important factors affecting the clinical performance of adhesive direct restorations. This insertion can be carried out through direct application of the material with special devices, such as capsules or compules, or the composite can be removed from its container (eg, syringes) and placed in the cavity with a hand instrument.[1] When the material is removed from the syringe with a hand instrument, clinicians might perform unintentional digital manipulation of the composite or even intentionally manipulate the material in order to homogenize it and facilitate its insertion and accommodation into the cavity. This direct contact with gloves used for restorative procedures could, however, cause contamination of the restorative material by powder from the gloves, by saliva, or by other sources.

Direct digital manipulation of composite resins could affect their mechanical properties because it might introduce organic and inorganic debris[2-7] and leave porosities[8-10] inside the materials. In this context, a major reason for the failure of composite restorations is fracture of the restorative material,[11-13] which has been hypothesized to potentially occur due to the propagation of pre-existing cracks between increments of the restorative[14] or the incorporation of air bubbles during its insertion into the cavities. Therefore, the variables related to the presence of these defects into restoratives should be further explored.

Although some studies have reported on the contamination of composite resins by organic or inorganic agents,[2-7] these studies have usually focused on the effects of surface contamination of the composite and its consequences on the bond strength to the dental substrata/adhesive systems[2,3,5-7] or to the composite increments.[4,15] However, the literature does not address the effects of this contamination on the cohesive strength of the material, which could be contaminated by intentional or unintentional direct contact with the gloves used during the restorative procedure. In addition, there is no evidence that protocols for cleaning/decontaminating the gloves could reduce the potential harm from contamination to the composites' mechanical properties.

The aim of this study was to evaluate the influence of digital manipulation of a composite resin with powdered and powder-free gloves, contaminated or not with saliva, on the mechanical properties and the incremental layer bond strength of the restorative. The null hypothesis tested was that contamination is not detrimental to the material's properties.

## METHODS AND MATERIALS

### Experimental Design

This *in vitro* study involved a completely randomized and blinded design, considering the effect of different contamination conditions of latex gloves for clinical procedures used to digitally manipulate a composite resin on the mechanical properties of the restorative. The factor under investigation was "glove contamination" at six levels: powdered glove without saliva, powder-free glove without saliva, powdered glove with saliva, powder-free glove with saliva, powdered glove with saliva cleaned with 70% ethanol, and the control group, which was defined by no digital manipulation or contamination. The response variables assessed were diametral tensile strength, flexural strength, flexural modulus, and shear bond strength between composite increments. Sixty specimens were prepared for each test with a photoactivated microhybrid composite resin (Z250, 3M ESPE, St Paul, MN, USA; shade A2), according to the specifications of each test (n=10 per group for each test, with a total of 180 specimens).

### Preparation of the Gloves and Digital Manipulation of the Composite

Powdered and powder-free disposable latex gloves were used (Supermax Glove Manufacturing, Selangor, Malaysia). The classification of the gloves was based on the manufacturer's information, with cornstarch powder in powdered gloves. Each glove

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was removed from its respective pack immediately before use. In the groups contaminated with saliva, in order to simulate the clinical condition of using a contaminated glove, the gloves were prepared by wetting the surface with stimulated human saliva collected using paraffin film (Parafilm M, American National Can, Chicago, IL, USA) from a single healthy donor. The gloves were allowed to dry at room temperature for 24 hours to simulate the conditions where the gloves were contaminated but not soaked in saliva. For the other experimental conditions, the gloves were removed from the box and immediately used. Digital manipulation of each composite increment was carried for 10 seconds, obtaining a final round shape for the increment. In the control group, the composite was removed from the syringe with a titanium-coated spatula and placed into the molds (according to the test) without any digital manipulation.

### Diametral Tensile Strength

Disc-shaped specimens (diameter 4 mm, thickness 2 mm) were prepared by filling a cylindrical metal mold using three increments of the composite resin. Each increment was photoactivated for 20 seconds using a light-emitting-diode curing unit (Radii, SDI, Bayswater, VIC, Australia) with 600-mW/cm$^2$ irradiance. The top surface of the specimens was covered with a polyester strip and a glass slide, and hand pressure was applied before photoactivation. The specimens were wet polished with 600-grit SiC abrasive papers and stored in distilled water at 37°C. After 24 hours, the diametral compressive test was carried out in a mechanical testing machine (DL500, EMIC, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/min until fracture of the specimen. Diametral tensile strength was calculated in MPa.

### Flexural Strength and Modulus

Bar-shaped specimens (25 × 2 × 2 mm) were prepared by filling a split metal mold using four increments of the composite resin. Each increment was photoactivated for 20 seconds. The top surface of the specimens was covered with a polyester strip and a glass slide, and hand pressure was applied before photoactivation. The specimens were wet polished with 600-grit SiC abrasive papers and stored in distilled water at 37°C. After 24 hours, a three-point bending test was carried out in the mechanical testing machine at a crosshead speed of 0.5 mm/min until fracture of the specimen. Flexural strength (MPa) and modulus (GPa) were calculated from the load-displacement curve.

### Incremental Layer Bond Strength

Epoxy resin molds with a square-shaped cavity (5 × 5 mm, thickness 2 mm) were used. The cavities were filled with the composite resin and covered with a polyester strip and a glass slide. Hand pressure was applied in order to create a flat composite surface, and the material was photoactivated for 20 seconds. In order to obtain specimens for evaluating the incremental layer bond strength by shear testing, elastomer molds with a cylindrical-shaped orifice (diameter 1.2 mm, thickness 0.5 mm) were positioned onto the composite surfaces. The orifices were filled with composite resin, which was manipulated according to the different contamination conditions, and photoactivated for 20 seconds. The specimens were stored in distilled water at 37°C. After 24 hours, a thin steel wire (diameter 0.2 mm) was looped around each resin cylinder and aligned with the bonded interface. The shear bond strength test was conducted in the mechanical testing machine at a crosshead speed of 0.5 mm/min until failure of the bonding. Bond strength data were calculated in MPa. The fractured specimens were examined under a stereomicroscope at 40× magnification for classification of the failure modes: interfacial (adhesive) failure or cohesive failure within the composite. Representative fractured surfaces were gold coated and analyzed by scanning electron microscopy (SEM; SSX-550, Shimadzu, Tokyo, Japan).

### Statistical Analysis

Data from each response variable of all tests were separately analyzed using one-way analysis of variance followed by the Student-Newman-Keuls *post hoc* test at a 5% significance level.

## RESULTS

Results for the mechanical properties tested are shown in Table 1. No significant differences for diametral tensile strength were observed between groups; however, the power of the performed statistical test was below 0.8. For flexural strength and modulus, the power of the performed statistical test was 1. Manipulation of the composite using powder-free gloves with saliva or using gloves cleaned with ethanol generated significantly higher flexural strength and modulus compared with the other groups. The control group (no digital manipulation) and the group defined by digital manipulation using powdered gloves with saliva generally showed

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Table 1: *Means (Standard Deviations) for the Mechanical Properties Tested (n=10)*[a]

| Contamination Condition | Diametral Tensile Strength, MPa | Flexural Strength, MPa | Flexural Modulus, GPa |
|---|---|---|---|
| Powdered glove without saliva | 41.8 (7.0) A | 87 (25) B | 9.4 (1.4) B |
| Powder-free glove without saliva | 42.0 (7.3) A | 59 (18) C | 8.4 (6.4) BC |
| Powdered glove with saliva | 44.0 (9.0) A | 51 (20) C | 7.5 (2.9) CD |
| Powder-free glove with saliva | 36.3 (8.5) A | 131 (31) A | 13.6 (1.5) A |
| Powdered glove with saliva cleaned with ethanol | 45.7 (5.7) A | 150 (26) A | 13.8 (1.9) A |
| Control (no digital manipulation) | 45.5 (12.8) A | 49 (29) C | 6.0 (1.1) D |

[a] *Distinct letters in each column indicate significant differences (p<0.05).*

significantly lower flexural strength and modulus than the other groups.

Results for incremental layer bond strength are shown in Figure 1 (the power of the performed statistical test was 0.854). Significantly lower bond strength between composite increments was observed for the group manipulated with powdered gloves without saliva. The control group and the groups manipulated with powdered gloves with saliva or cleaned with ethanol showed higher bond strengths. The groups manipulated with powder-free gloves, either contaminated or not by saliva, showed intermediate results. SEM pictures illustrating the failure modes are shown in Figure 2. The failure analysis showed that most of the failures (~92%) were mainly cohesive, with only one interfacial debonding observed for each group, except for the group defined by manipulation with powdered gloves without saliva, which showed only cohesive failures.

## DISCUSSION

The present study showed that composite resins can be handled with gloves used for procedures, but these gloves should be cleaned to remove powder remains and other contaminants. This is important because in several countries, composite resins are sold mainly in syringes or other packs, and clinicians have to remove the material with hand instruments from their containers. During this procedure, some dentists may digitally manipulate composites in order to facilitate the placement of the material into the cavity preparation, whereas others may unintentionally touch them with the gloves during the placement of restorations. There are no specific studies in the literature reporting the effects of contamination of composites through digital manip-

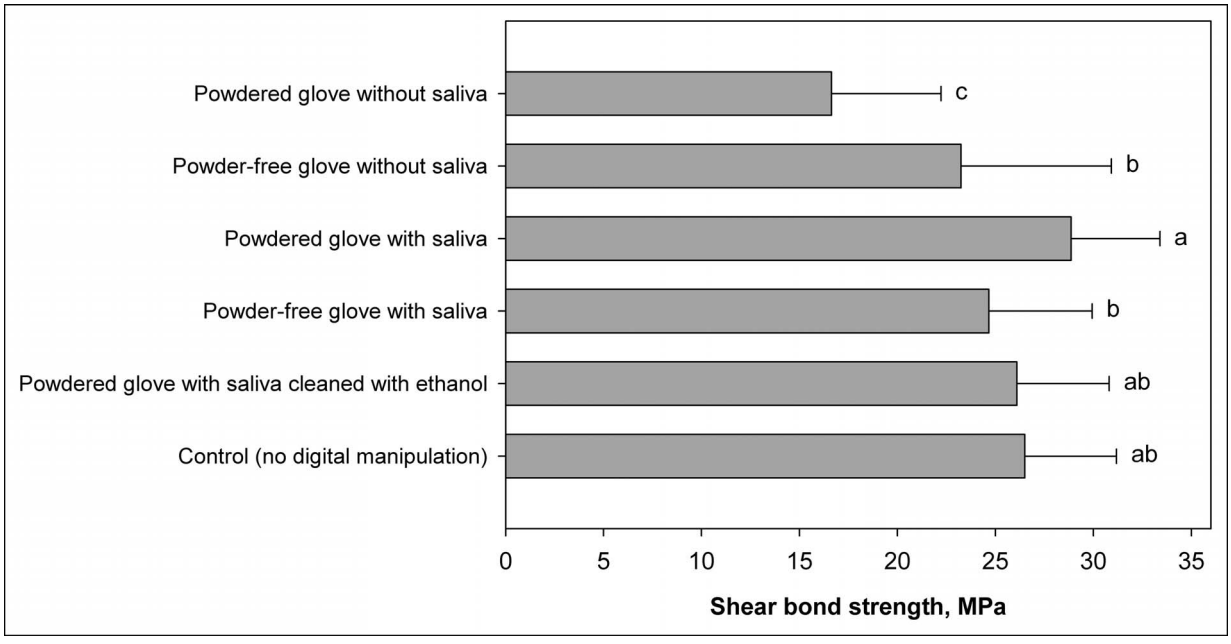


Figure 1. *Results for the bond strength between composite increments (bars are means + standard deviations, n=10). Distinct letters indicate significant differences (p<0.05).*

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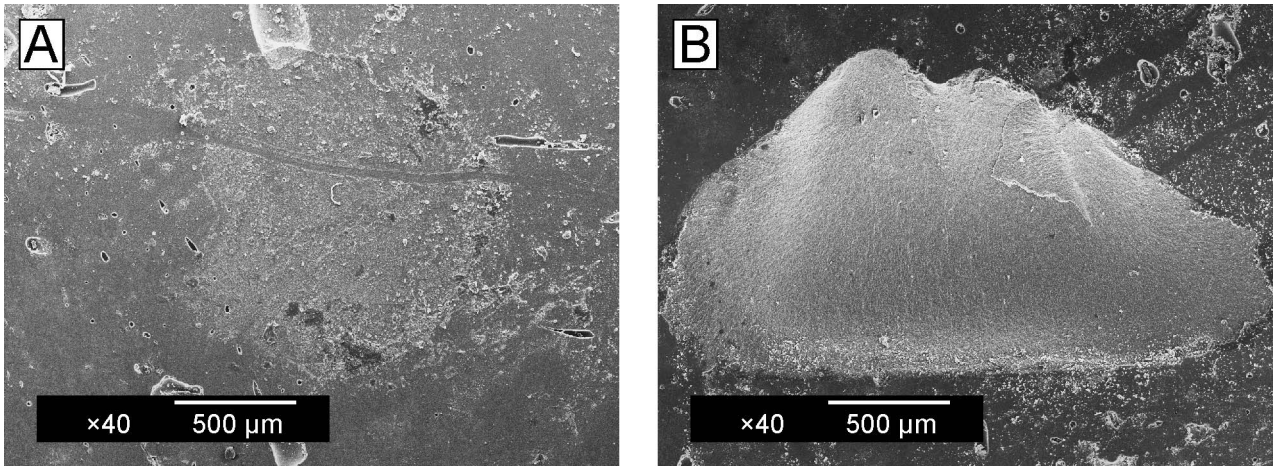


Figure 2. *SEM pictures illustrating the failures modes. (A): Adhesive failure characterized by an interfacial failure between the increments. (B): Cohesive failure within the composite.*

ulation with latex gloves in the mechanical properties of the composite resins. Some studies, however, show the negative influence of contamination by blood,[2] saliva,[3,4,15] latex gloves[5,7] and cleaning agents in the adhesive properties of restoratives to the dental substrata. Moreover, there is some evidence that the technique used for composite placement can directly affect the incorporation of bubbles and porosities in the material.[1,8-10,16-18]

The contamination with saliva and/or blood has been cited in the literature as one of the main problems during adhesive procedures,[19,20] and digital manipulation of composites with powder-containing gloves or contaminated by saliva is a form of contamination between the increments of the composite resin. A direct comparison to our results with data from previous studies was not possible because most studies analyze different variables, such as contamination and decontamination of dental substrate, contamination and decontamination between increments of composites, and the effects of techniques for insertion of restorative material on the formation of voids or porosities, while the present study assessed the effects of contamination sources on the mechanical properties of composite resins.

In the present study, saliva-contaminated gloves were left to dry before digital manipulation of the composite. This procedure was chosen because the study did not focus on the moisture effects of saliva on properties of composite resins. It is unlikely to expect that clinicians would deliberately use a moist, saliva-contaminated glove for touching the composite; however, the glove could be contaminated with saliva and dry before the composite is manipulated. In such a situation, only the solid saliva components, such as proteins, amino acids, and enzymes, would be present on the gloves to act as contaminants,[15] and the dentist would not be aware of that contamination source.

The results from the diametral tensile strength test showed no statistically significant difference among groups, demonstrating that contamination by powder and/or saliva did not directly affect this property. This result supports the null hypothesis of the study. The diametral tensile test examines the ability of the material to support multidirectional forces, and in the present study, all groups had values above 34 MPa, fulfilling the recommendation of the American Dental Association for direct restorative composites to be used in posterior restorations.[21] In addition, the diametral tensile strength values reported here are in line with other results reported in the literature for different commercial composite resins.[22] However, this test might not be sensitive enough to detect the effects of contamination among the layers of the composite placed with an incremental technique, explaining the lack of differences among groups under this test. However, it should be acknowledged that the power of the performed statistical test for diametral tensile strength data was below 0.8 and that different results might be observed if a larger sample size was used.

The overall results found from the flexural strength and flexural modulus evaluations showed that gloves not cleaned before digital manipulation can cause negative effects on these properties of composites. Even the manipulation with powder-free gloves seems to cause adverse effects on the flexural strength and modulus of composites, and this could

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be explained by the presence of contaminants in the gloves other than just the powder. Also, the control group without digital manipulation presented lower flexural strength and modulus, which could be attributed to the presence of voids and porosities among composite increments. This finding was previously reported in the literature for the direct placement of the composites into the cavity.[16] It is interesting to note that the disinfection of contaminated gloves with ethanol seems to have a positive effect on flexural strength and modulus; this finding indicates that digital manipulation procedures could be performed if the gloves are decontaminated. These results also reinforce the negative influence of contamination on the properties of composite resins, rejecting the null hypothesis of the study.

Regarding the incremental layer bond strength test, results supported that the presence of powder in procedure gloves interfered negatively in the union between the increments, which was also showed by Oskoee and others,[7] who compared the effects of contamination of the adhesive surface (bovine enamel) from latex gloves with and without powder used to place composite restorations. The presence of saliva on the gloves may counteract the negative effect produced by the presence of powder, and it could be speculated that the presence of small remains of saliva proteins between the increments is not as damaging as the presence of powder from the gloves. As for the results of flexural strength and flexural modulus, cleaning the gloves with alcohol seems to be a good option to avoid negative effects from contamination by the gloves.

Results of this study provide evidence that the digital manipulation of composite resins with gloves might influence their mechanical properties. Clinicians should bear this in mind during placement of adhesive restorations and avoid touching the adhesive materials and composites with contaminated gloves. It is important to acknowledge the limitations of this *in vitro* study, which represents only an indication of the performance of the specific materials tested here. While there may be a correlation between laboratory tests and the clinical performance of restorations, the former are used primarily to guide the effects of changes in composition or the evolution of their properties. Knowledge of the mechanical properties is essential for the correct use of these materials and to estimate the clinical long-term performance,[23] but the best evidence would be achieved with randomized and controlled clinical trials or long-term prospective and retrospective studies on the longevity of restorations.

## CONCLUSIONS

Within the limitation of this *in vitro* study, the following conclusions can be drawn:

- The presence of powder in the gloves seems to be more damaging for the mechanical properties and incremental layer bond strength than the presence of saliva.
- Cleaning the gloves with ethanol might be an alternative to avoid the negative effects of digital manipulation of the composite using contaminated gloves.
- Digital manipulation might be indicated for composite resin restorations to achieve improved mechanical properties, provided that the gloves are not contaminated.

### 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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# Surface Treatments that Demonstrate a Significant Positive Effect on the Shear Bond Strength of Repaired Resin-modified Glass Ionomer

D Welch • B Seesengood • C Hopp

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**Clinical Relevance**

The appropriate surface treatment used to repair resin-modified glass ionomer is not well defined. A definitive protocol needs to be established as a guideline for the conservative repair of this material.


## SUMMARY

**This study examined surface treatment options used to repair resin-modified glass ionomer (RMGI; GC Fuji II LC, GC America). Two hundred forty specimens were equally divided into four different water/temperature cycling environmental conditions. The conditions were 1) five-minute delay, 2) one-week delay with one thermocycle, 3) 500 thermocycles, and 4) 24-hour delay in a dry environment, followed by 500 thermocycles. Within each of the condition groups, the specimens were equally divided again into three different surface treatment groups with 20 specimens in each. The treatment groups comprised A) sanding, B) sanding and acid etch, and C) sanding, acid etch, and dental bonding agent. Our results suggest that RMGI is extremely susceptible to the simultaneous exposure of temperature cycling and water during the first 24 hours. Our main results reflect that 1) during the first five minutes after the initial placement, the surface treatments made no difference in terms of the shear bond strength (NS); and 2) when we weakened the RMGI by exposing it to water and temperature cycling immediately after initial placement, each of the treatments (A<B<C) had a significant incremental increase in bond strength ($p<0.05$). As such, given that a RMGI is partially a composite resin, the surface treatment with a dental bonding agent did have a significant positive**



*Dan Welch, PhD, Southern Illinois University School of Dental Medicine, Growth Development and Structure, Alton, IL, USA

Brooke Seesengood, BA, Southern Illinois University School of Dental Medicine, Biology, Alton, IL, USA

Christa Hopp, DMD, Southern Illinois University School of Dental Medicine, Restorative, Alton, IL, USA

*Corresponding author: 2800 College Ave, Alton, IL 62002, USA; e-mail: dwelch@siue.edu


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**effect on the micromechanical bond strength of the repair.**

## INTRODUCTION

Resin-modified glass ionomer (RMGI) restorative materials are generally believed to have advantages over regular glass ionomer, such as reduced sensitivity to moisture contamination,[1] and are often used for Class 3 and Class 5 lesions.[2] Clinically, the retention of RMGIs is favorable, with a failure rate under 3% over the course of 13 years.[3] However, the repair of a previous restoration is sometimes required as a result of the presence of voids, marginal chipping, lack of contour,[2] or color changes. The initial color match may be favorable, but it appears that they may not always be color stable.[3] The benefits of a repair of such minor defects include the preservation of the tooth and a reduction in cost.[4] A longitudinal clinical study conducted by Gordan and others[5] in 2006 indicates that restorations with marginal adaptation and stained margins are better off being repaired rather than replaced. Despite the need for a clear protocol on the noninvasive repair of a RMGI, an appropriate evidence-based approach in the literature has been largely neglected.

In 1998, the first attempt to address this issue was made simultaneously by Yap and others[6] and Shaffer and others.[2] Unfortunately, Yap and others[6] found that none of the surface treatments that they tried demonstrated significantly higher repair bond strength than did the control. Shaffer and others[2] simulated the harsh conditions of the oral environment by thermocycling the specimens 500 times between 5°C and 55°C after the specimens had been stored for two days in 37°C water. With these conditions, they found that the time of repair significantly affected the bond strength of two of the three materials tested.[2] However, water by itself does not seem to affect the strength of RMGIs.[7] In 2000, Yap and others[8] followed their initial research question and found that surface conditioning with maleic acid and resin application gave the highest repair bond strengths for those that were in three and six months of storage. However, in 2010, Maneenut and others[9] found that acid treatment shows little effect on the surface of a RMGI under scanning electron microscopy (SEM) evaluation.

Few articles have explored the repair of RMGI with consideration of surface treatment prior to application and variations in moisture and temperature cycling before the repair process. The results obtained to date have been mixed. In this study, we wish to validate and extend these previous results by examining each of the known factors simultaneously in order to help resolve the inconsistencies and gaps in our knowledge. In essence, a definitive protocol needs to be established as a guideline for the conservative repair of this material.

## METHODS AND MATERIALS

Specimens were created using epoxy resin plates with inverted cone recesses (6-mm outer diameter and 5-mm inner diameter). The plates were filled with RMGI (Shade A1), which was purchased in capsule form (GC Fuji II LC, GC Corporation, Tokyo, Japan). The RMGI was mixed for 10 seconds using an amalgamator with the speed set at M3 (Caulk Vari-Mix II-M, Dentsply International, York, PA, USA). For each sample, the RMGI was extruded into the recessed cone, which was supported by a glass slab held underneath. The RMGI was then cured according to the manufacturer's directions with a quartz-tungsten-halogen light for 20 seconds (Optilux 501, Kerr, Danbury, CT, USA) at an intensity of $>750$ W/cm$^2$. The glass was removed and the RMGI was examined for the presence of a uniform surface. If satisfactory, the specimens were then placed into 12 respective groups with 20 samples in each group. There were four environmental conditions with one of three surface treatments given to each individual group, for a grand total of 240 specimens (see Figure 1). The environmental conditions were as follows: 1) five-minute delay, 2) one-week delay with one thermocycle, 3) 500 thermocycles, and 4) 24-hour delay in a dry environment, followed by 500 thermocycles. We programmed an environmental testing chamber (Cincinnati Sub-Zero Products Inc, Cincinnati, OH, USA) to either 1) thermocycle once between 5°C and 55°C and then maintain the temperature at 37°C with 95% humidity for one week or 2) thermocycle 500 times between 5°C and 55°C and then maintain the temperature at 37°C with 95% humidity, which required 10 days to simulate the temperature and moisture variations of the oral environment. Each of the four environmental conditions contained three treatment groups, as follows: A) sanded, B) sanded and acid etched, and C) sanded and acid etched, followed by application of a dental bonding agent. The purpose of these groups was to separately assess the impact that each significant factor has on the strength of the material. The sanded samples were abraded

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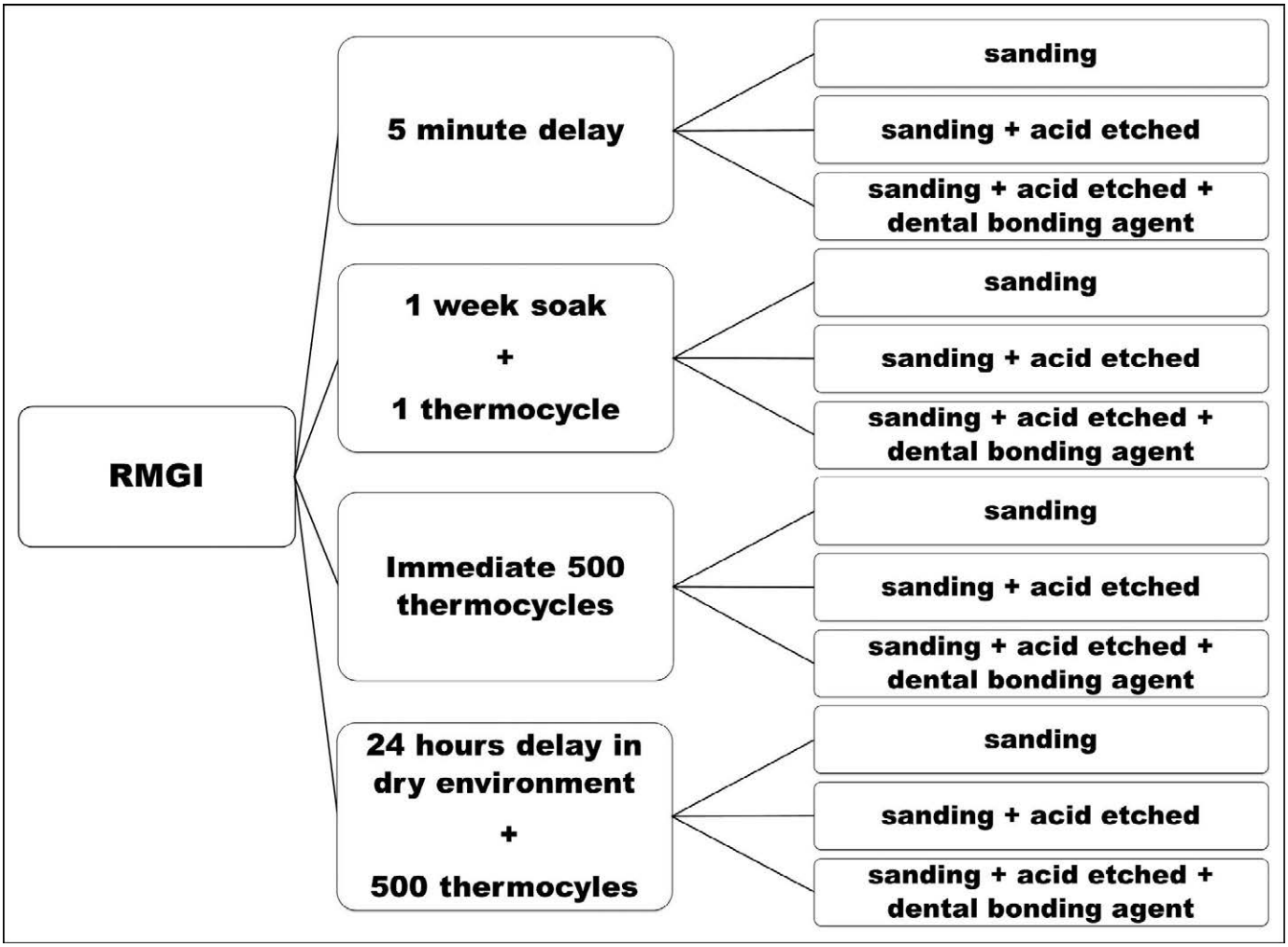


Figure 1. *Two hundred forty specimens were equally divided into four different water/temperature cycling environmental conditions and were equally divided again into three different surface treatment groups (n=20).*

with wet 800-grit silicon carbide paper (Leco, St Joseph, MI, USA) until smooth and then rinsed and dried to insure a uniform matt surface. The coarseness of the 800-grit carbide paper was intended to simulate a typical diamond bur. The acid etchant treatment comprised 37.5% phosphoric acid (Kerr, Orange, CA, USA). The acid etchant was applied for 15 seconds using a disposable applicator brush, and then the samples were rinsed for 15 seconds using water. The dental bonding agent (Optibond Solo Plus, Kerr) was applied for 15 seconds using a disposable applicator brush, air-thinned for three seconds, and then cured with a quartz halogen light for 20 seconds, according to the manufacturer's directions. After the respective treatments, a plastic matrix with a standardized 4-mm-diameter window was placed over the sample, and then a second epoxy resin plate with a window was placed over the plastic to standardize the bonding interface. The two epoxy resin plates were temporarily fixed in place with screws and wing nuts to prevent any movement. The RMGI was then syringed through the window and cured according to the manufacturer's directions. The bonded samples were placed on the universal testing machine (5960 Dual Column Tabletop Instron, Norwood, MA, USA) to test shear bond strength (see Figure 2).

Analysis of variance was used to test the effects of the surface treatments for each environmental condition. Paired *t*-test post hoc comparisons were made, with Bonferroni corrections to control for the probability of false positives, due to multiple com-

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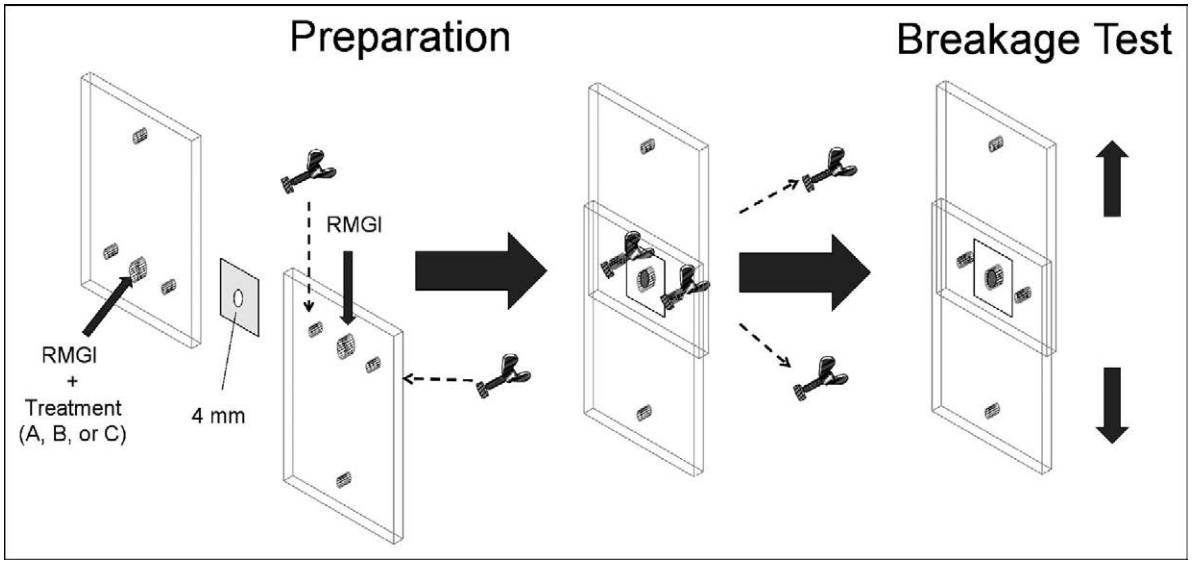


Figure 2. *Flow chart. Diagram demonstrating the sequence from specimen preparation to breakage test.*

parisons. A value of $p < 0.05$ was used to determine statistical significance for this study.

## RESULTS

A series of SEM images (13×, 10 kV) were used in our assessment of the breakage (ABT-32, Topcon, Livermore, CA, USA). The mode of failure was documented as A) adhesive failure, B) mixed failure, or C) cohesive failure (see Figure 3). The failure modes of the groups are presented in Table 1. Initially, we compared the specimens that received a sanding surface treatment only for the following conditions: 1) five-minute delay, 2) one-week delay with one thermocycle, 3) 500 thermocycles, and 4) 24-hour delay in a dry environment, followed by 500 thermocycles. We found that the RMGI is extremely susceptible to temperature cycling and water exposure during the first 24 hours ($p<0.05$) (see Figure 4). This was an experimental negative control condition that would not occur in a clinical setting, in which saliva and moisture are always an issue. We did this to evaluate the properties of the material independently and in order to emphasize how important it is to control moisture as much as possible in a clinical setting. We also compared the surface treatments (A-C) for each environmental condition (1-4) (see Figure 5). We found that 1) during the first five minutes, the surface treatments made no difference (NS); 2) when we temperature cycled the specimens once and had them soak in water for one week, those with sanding, acid etch, and dental

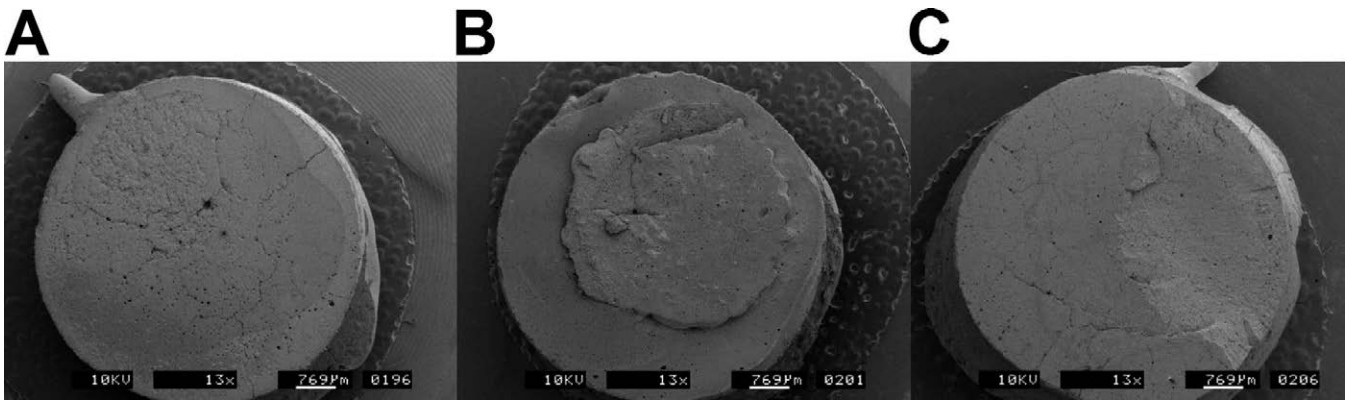


Figure 3. *The mode of failure was documented as (A) adhesive failure, (B) mixed failure, or (C) cohesive failure.*

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Table 1: *The Failure Modes of the Groups Are Presented*

| Environmental Condition | Surface Treatment | Adhesive | Mixed | Cohesive | n |
|---|---|---|---|---|---|
| 5 min | sand | 9 | 11 | 0 | 20 |
| 5 min | sand + acid etch | 10 | 10 | 0 | 20 |
| 5 min | sand + acid etch + bonding agent | 5 | 15 | 0 | 20 |
| 1 wk soak + 1 thermocycle | sand | 5 | 7 | 8 | 20 |
| 1 wk soak + 1 thermocycle | sand + acid etch | 4 | 11 | 5 | 20 |
| 1 wk soak + 1 thermocycle | sand + acid etch + bonding agent | 1 | 13 | 6 | 20 |
| 500 thermocycles | sand | 13 | 6 | 1 | 20 |
| 500 thermocycles | sand + acid etch | 16 | 2 | 2 | 20 |
| 500 thermocycles | sand + acid etch + bonding agent | 7 | 8 | 5 | 20 |
| 24-h delay + 500 thermocycles | sand | 0 | 10 | 10 | 20 |
| 24-h delay + 500 thermocycles | sand + acid etch | 6 | 11 | 3 | 20 |
| 24-h delay + 500 thermocycles | sand + acid etch + bonding agent | 0 | 6 | 14 | 20 |

bonding agent (C) were significantly stronger than those that were sanded and acid etched (B) ($p<0.05$); 3) when we temperature cycled the specimens immediately, each of the treatments (A<B<C) had a significant incremental increase in bond strength ($p<0.05$); and 4) when we let the samples dry for 24 hours prior to the temperature cycling and water, we also found that all treatments had significant differences in bond strength relative to each other, with the sanding and acid etch being the weakest (B) ($p<0.05$).

## DISCUSSION

The main finding of our study was that a dental bonding agent will improve the tensile bond strength between an old and new layer of RMGI but has no significant effect during the first few minutes, while the glass ionomer is still maturing. This verifies and extends previous results in which surface conditioning with maleic acid and resin application gave the highest repair bond strengths for those that were in three and six months of storage.[8] Previous research seems to indicate that the strength of RMGI materials is not affected by early water contact.[7] However, we have demonstrated that temperature cycling and water exposure together during the first 24 hours does in fact weaken the bond (see Figure 4). This is because a RMGI hardens by two separate reactions: 1) a fairly rapid photochemical-initiated polymerization of the resin and 2) a slower acid-base reaction between the glass powder and an organic acid.[10] As such, the physical properties of the glass ionomer depend on the formation of a fairly insoluble polysalt matrix. This matrix takes time to change from being a mostly soluble calcium polyacrylate to a more stable aluminum polyacrylate during the first 24 hours.[11] As an experimental control, we added a fourth condition in which there was a 24-hour delay prior to the water and temperature cycling. These results clearly demonstrate that the vulnerability of the material dissipates after the first day (Figure 3). Clinical application of the results can be interpreted as follows: if repair of a RMGI is necessary on the day of the initial placement, the only surface treatment needed is roughening the surface prior to placement; if repair is necessary more than 24 hours after the initial placement, a bonding agent should be used to strengthen the bond.

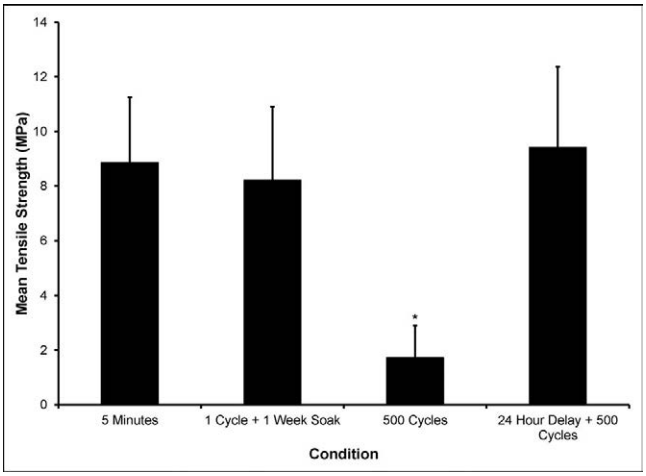


Figure 4. *RMGI is extremely susceptible to temperature cycling and water exposure during the first 24 hours. * = statistically significant difference (p<0.05).*

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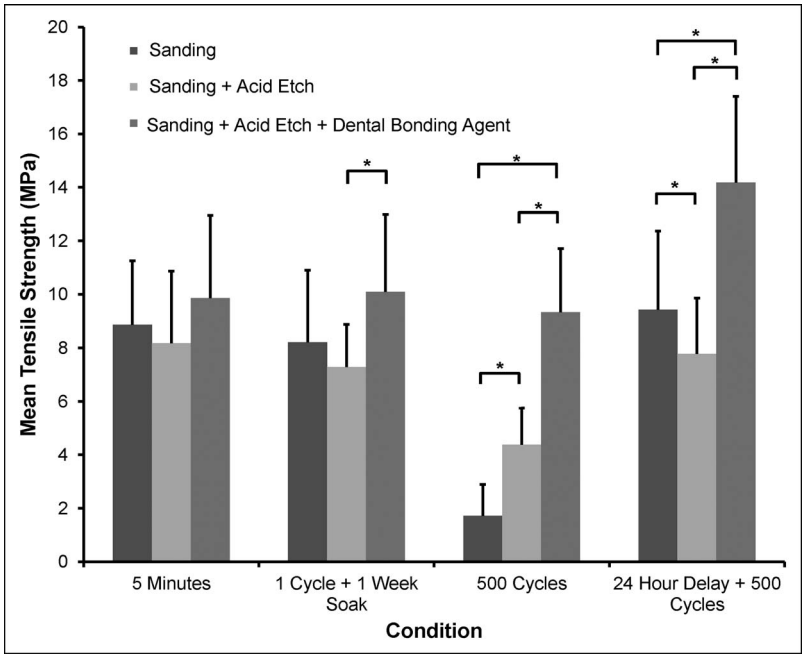


Figure 5. *Comparison of the mean tensile strength (MPa) measurements for the environmental conditions vs surface treatments.*

The focus of this study was to determine the best approach for the clinical situation when RMGI is the chosen material. An example would be a pediatric patient, when the advantages of RMGI (speed/ease of use) overshadow any other undesirable properties of the material. However, composite resin can be used as a viable alternative to repair RMGI in some clinical situations. In fact, other investigators have examined that approach experimentally. We already know that certain factors may affect the bond strength between RMGI and a composite resin.[12] However, the focus of this study is the repair of RMGI using the same material, as an alternative approach.

## CONCLUSION

Given that a RMGI is partially a composite resin, the surface treatment with a dental bonding agent did have a significant positive effect on the bond strength of the repair. We believe that the findings of our study increase the chance of a positive clinical outcome for cases that involve the repair of RMGI with the same material.

### Conflict of Interest

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

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# Enamel Bond Strength of New Universal Adhesive Bonding Agents

DE McLean • EJ Meyers • VL Guillory
KS Vandewalle

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### Clinical Relevance

This laboratory study will facilitate the dental practitioner's decision-making process in selecting an adhesive bonding agent based on the results presented. This study supports the continued use of a two-step self-etch adhesive over the recently introduced universal adhesives.


## SUMMARY

**Purpose: Universal bonding agents have been introduced for use as self-etch or etch-and-rinse adhesives depending on the dental substrate and clinician's preference. The purpose of this study was to evaluate the shear bond strength (SBS) of composite to enamel using universal adhesives compared to a self-etch adhesive when applied in self-etch and etch-and-rinse modes over time.**

**Methods and Materials: Extracted human third molars were used to create 120 enamel specimens. The specimens were ground flat and randomly divided into three groups: two universal adhesives and one self-etch adhesive. Each group was then subdivided, with half the specimens bonded in self-etch mode and half in etch-and-rinse mode. The adhesives were applied as per manufacturers' instructions, and composite was bonded using a standardized mold and cured incrementally. The groups were further divided into two subgroups with 10 specimens each. One subgroup was stored for 24 hours and the second for six months in 37°C distilled water and tested in shear. Failure mode was also determined for each specimen.**

**Results: A three-way analysis of variance (ANOVA) found a significant difference between groups based on bonding agent ($p<0.001$) and surface treatment ($p<0.001$) but not on time ($p=0.943$), with no significant interaction ($p>0.05$). Clearfil SE in etch-and-rinse and self-etch modes had more mixed fractures than either universal adhesive in either mode.**

**Conclusions: Etching enamel significantly increased the SBS of composite to enamel. Clearfil SE had significantly greater bond strength to enamel than either universal adhesive, which were not significantly different from each other.**


*Deidra E McLean, BSC, DDS, MS, Royal Canadian Dental Corps, CFB Trenton, Astra, ON, Canada

Erik J Meyers, DDS, General Dentistry, United States Air Force, San Antonio, TX, USA

Villa L Guillory, DDS, MS, Prosthodontics, United States Air Force, San Antonio, TX, USA

Kraig S Vandewalle, EBM, DDS, MS, General Dentistry, United States Air Force, Lackland Air Force Base, TX, USA

*Corresponding author: Yukon Street, Astra, ON K0K 3W0, Canada; e-mail: deidra.mclean@forces.gc.ca

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## INTRODUCTION

Adhesive dentistry has been around for over 50 years since it was first introduced by Buoncore in 1955.[1]

Since that time, there has been a constant evolution in the field of adhesive dentistry with the progressive introduction of seven generations of adhesive bonding agents.

Adhesive bonding agents must be capable of providing equally effective bonds to both enamel and dentin despite being vastly different structures in terms of composition and natural variability. Enamel's composition is primarily inorganic (86%) hydroxyapatite with 2% organic content, and 12% water; while dentin is composed of 50% inorganic mineral, 30% organic collagen, and 20% water.[2,3] Enamel is a homogenous structure, while dentin is highly variable depending on several factors including age, dentinal tubule number and size, and previous exposure to carious, chemical, or mechanical stimuli.[2]

Other variables that may interfere with adhesive bonding include the creation and removal of a smear layer, as well as its thickness. Dentinal wetness may also affect bond strength if the tooth is left too wet or too dry following acid etching.[4] Matrix metalloproteinases also affect adhesive bonding over time.[5] Other challenges to adhesive dentistry in addition to differences between enamel and dentin include the presence of moisture in the working area, technique sensitivity of the materials, biocompatibility of materials, the requirement for a gap free restorative interface, and the requirement for the bonding agents to rapidly develop high bond strengths.

The basic mechanism of adhesion between tooth structure and adhesive bonding agents is based on an exchange process. Minerals from hard tissue are replaced by resin monomers that effectively create a micromechanical bond.[6] Despite the similarities between adhesives, the composition of these materials and the manner in which they are applied differ. The demand for simpler, more user-friendly and less technique-sensitive adhesives has inspired manufacturers to develop new adhesives at a rapid rate.[7]

Currently, there are four generations of dental adhesives available to dentists including fourth, fifth, sixth, and seventh generation adhesive bonding agents. In addition to the generation classification, there is also an adhesive classification system. This hierarchy classification system includes two major categories of adhesives: etch-and-rinse adhesives and self-etch adhesives. These major categories are further divided into four subtypes: three-step etch-and-rinse, two-step etch-and-rinse, two-step self-etch, and one-step self-etch. The two-step etch-and-rinse and one-step self-etch are also referred to as simplified adhesives because the primer and adhesive are combined. The one-step self-etch adhesives may be further subdivided into "two-component" and "single-component" one-step adhesives.[7] These classification systems and how they relate are demonstrated in Figure 1.

Fourth generation or three-step etch-and-rinse adhesive bonding agents were developed in the early 1990s and are considered multi-step adhesives involving three separate applications including acid etching, application of the primer, followed by application of a separate adhesive. Fifth generation or two-step etch-and-rinse or simplified etch-and-rinse adhesives involve acid etching, followed by the combined application of a primer and an adhesive. The sixth generation or two-step self-etch adhesives involve application of an acidified primer followed by application of the adhesive resin. The one-step self-etch adhesives, also known as the simplified self-etch adhesives, involve application of a combined acidified primer and the adhesive resin in a single step. The two-component one-step self-etch adhesives, which are also sixth generation adhesive bonding agents, separate the active ingredients. Specifically, the functional monomer is separated from water, theoretically providing a longer shelf life, but additional and adequate mixing of both components is required. The single-component one-step adhesives, also known as seventh generation adhesive bonding agents, can be considered as the only true "all in one" adhesives, combining the acidified primer and the adhesive resin and do not require mixing prior to application.[7]

Despite the various generations or adhesive classifications, there are significant differences between adhesive bonding agents even within the same class. For example, self-etch adhesives may vary greatly in their level of acidity. They may have strong, intermediately strong, mild, or ultra-mild acid etchants.[8] Therefore, clinical performance is highly product dependent.

From the literature, Heintze and others[9] conducted a meta-analysis in 2010 that looked at the retention rates of cervical composite restorations bonded with various adhesive-bonding agents. As a result of these numerous clinical studies, it was concluded that the highest retention rates were achieved with the two-step, self-etch adhesive, Clearfil SE Bond (Kuraray, New York, NY, USA), followed closely by the three-step etch-and-rinse adhesive, Optibond FL (Kerr Dental, Orange, CA, USA). Clearfil SE Bond had been shown to produce lower bond strength to enamel, particularly uncut

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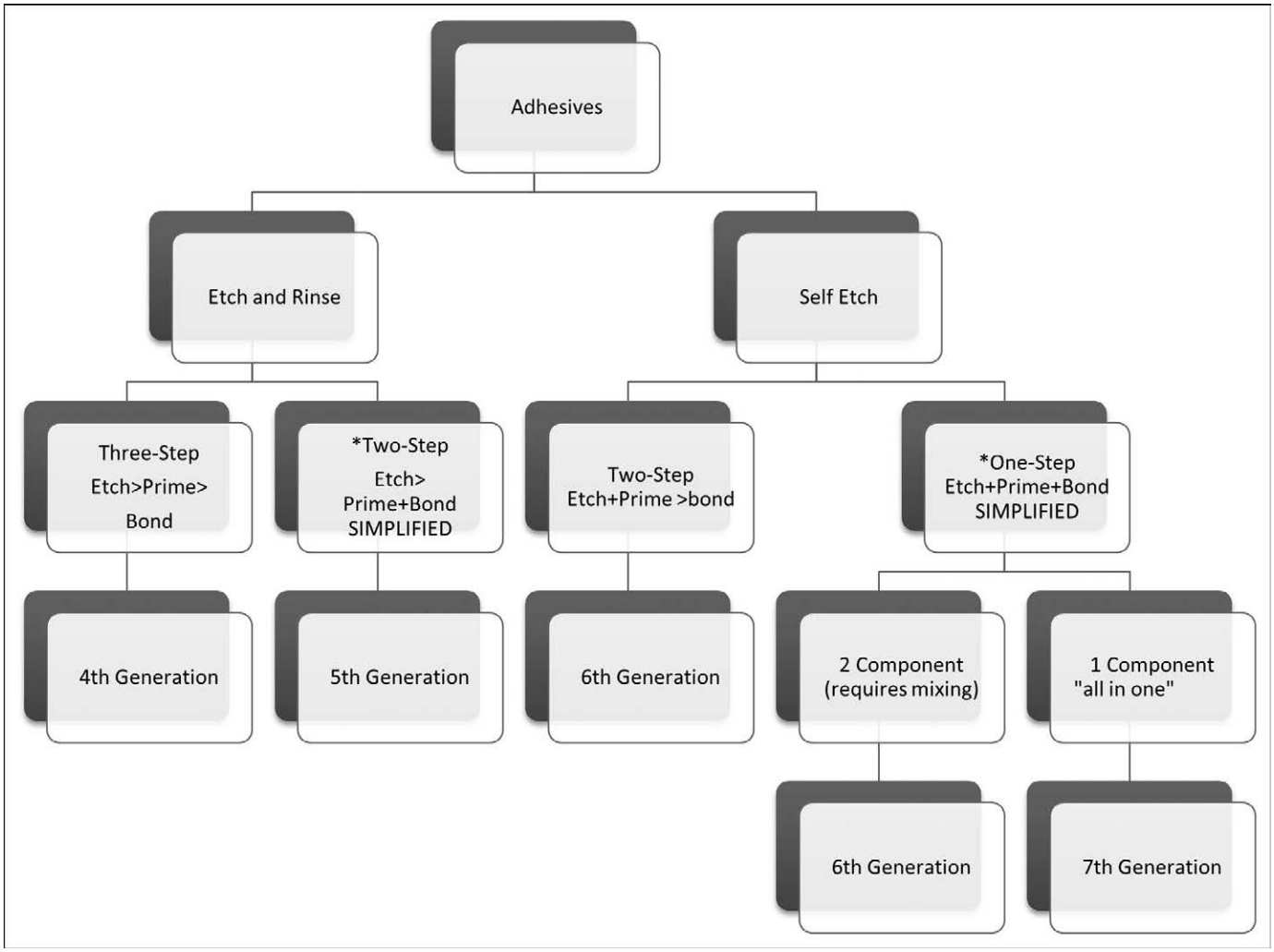


Figure 1. *Classification of adhesive bonding agents.*

enamel, but by selectively etching the enamel with phosphoric acid, Peumans and others[10] demonstrated that retention rates of Class V restorations after five years were 100% as opposed to 98% retentive without the selective etch step, but not statistically different. Studies have also shown significantly less marginal defects and staining with selective etching of enamel.[10,11] Van Meerbeek and others[7] also advocate the use of the selective etch technique using phosphoric acid on enamel because it produces the most durable bond to enamel that effectively seals and protects the more vulnerable bond to dentin against degradation.

A study by Peumans and others[12] that looked at the average annual failure rate of Class V composite resin restorations bonded with various dental adhesives revealed the three-step etch-and-rinse and two-step self-etch bonding agents to be most effective with a 4.8% and 4.7% annual failure rate, respectively. The simplified adhesives, including the two-step etch-and-rinse and one-step self-etch adhesives had the highest annual failure rates of 6.2% and 8.1%, respectively. According to a study by De Munck and others,[13] after approximately three months, all categories of dental adhesives start to exhibit mechanical and morphologic evidence of bond degradation. The three-step etch-and-rinse adhesives were said to remain the "gold standard" in terms of bond durability followed closely by the two-step self-etch adhesives. Any kind of simplification in the clinical application procedure resulted in loss of bonding effectiveness due to hydrolysis and elution of interface components.[13]

In late 2011 and early 2012, 3M ESPE and Bisco introduced two new universal bonding agents. According to the manufacturers, these products can

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Table 1: *Adhesive Agents, Surface Treatments, and Storage Times*

| Dental Adhesive | Immediate Group (24 h) | Aged Group (6 mo) |
|---|---|---|
| Clearfil SE (self-etch) | CF SE 24 hr | CF SE 6 mo |
| Clearfil SE (etch-and-rinse) | CF E&R 24 hr | CF E&R 6 mo |
| Scotchbond Universal (self-etch) | SB SE 24 hr | SB SE 6 mo |
| Scotchbond Universal (etch-and-rinse) | SB E&R 24 hr | SB E&R 6 mo |
| All-Bond Universal (self-etch) | AB SE 24 hr | AB SE 6 mo |
| All-Bond Universal (etch-and-rinse) | AB E&R 24 hr | AB E&R 6 mo |

be used as etch-and-rinse, self-etch, and selective-etch adhesives for bonding to enamel or dentin as well as many indirect restorative surfaces depending on the clinician's preference. Reportedly, neither product requires refrigeration and can be stored at room temperature for two years.

The purpose of this *in vitro* study was to examine the shear bond strength of the new universal bonding agents over time to enamel surfaces when used as an etch-and-rinse and self-etch adhesive compared to a two-step self-etch adhesive used in similar modes. The null hypothesis to be tested was that there would be no significant difference in the shear bond strength of composite to enamel based on type of bonding agent, type of surface treatment, or time.

## METHODS AND MATERIALS

The protocol was approved by the Wilford Hall Ambulatory Surgical Clinic Institutional Review Board. Extracted human permanent third molars were stored in 0.5% chloramine T solution at an average room temperature of 20°C for up to six months before being utilized. The teeth were visually examined and discarded if the enamel had caries or visible fracture lines. The crowns of the teeth were sectioned mesiodistally, then buccal and lingual sections were obtained by sectioning the crowns at the cementoenamel junction using a water-cooled diamond saw (Isomet 5000, Buehler, Lake Bluff, IL, USA). Each enamel specimen was mounted in polyvinylchloride pipe using dental stone and bis-acryl resin. After the stone had set, a small area of the enamel was cut flat using a diamond wheel bur then smoothed using 600-grit silicon-carbide paper.

The enamel specimens were randomly divided into 12 groups with 10 specimens each in order to compare the shear bond strength of different adhesives over time as depicted in Table 1. The adhesives that were compared included Clearfil SE (Kuraray), applied as a two-step self-etch and as a three-step etch-and-rinse adhesive; Scotchbond Universal Adhesive (3M ESPE, St Paul, MN, USA) as a one-step self-etch adhesive and as a two-step etch-and-rinse adhesive, and All-Bond Universal (Bisco, Schaumburg, IL, USA) as a one-step self-etch adhesive and as a two-step etch-and-rinse adhesive. For the adhesives applied with an etch-and-rinse technique, 34% phosphoric-acid gel etchant (Kerr Dental) was applied to the cut enamel for 15 seconds, rinsed with water for 15 seconds, then lightly air dried for three seconds before the application of the adhesive to the flattened enamel specimens as per manufacturer's instructions. The adhesives applied with a self-etch technique were applied directly to the cut enamel surfaces as per manufacturer's instructions. All adhesives were light cured with a light-curing unit (Bluephase 16i, Ivoclar Vivadent, Amherst, NY, USA) for 20 seconds. Irradiance was determined with a radiometer (LED Radiometer, Kerr Dental) and was considered acceptable if greater than 1200 $mW/cm^2$.

Following application of the adhesives, the bonded specimens were placed in a jig (Ultradent Products, South Jordan, UT) and secured beneath a white plastic mold. The bonded area was limited to the 2.4 mm circle determined by the mold. Z250 (3M ESPE) composite resin was applied in three increments to a height of 4 mm. Each increment was polymerized for 20 seconds as recommended by the manufacturer using the light-curing unit. The immediate and aged shear bond strength specimens were stored for 24 hours and six months, respectively, in distilled water at 37°C in a laboratory oven (Model 20GC, Quincy Lab, Chicago, IL, USA).

The shear bond strength of the specimens was tested in shear mode with a customized probe (Ultradent Products) in a universal testing machine (Model 5943, Instron, Norwood, MA, USA) using a crosshead speed of 1.0 mm/min until failure. Shear bond strength in megapascals was calculated from the peak load of failure in Newtons divided by the specimen surface area. The mean and standard deviation were determined per group. Data were analyzed with a three-way analysis of variance (ANOVA) and Tukey

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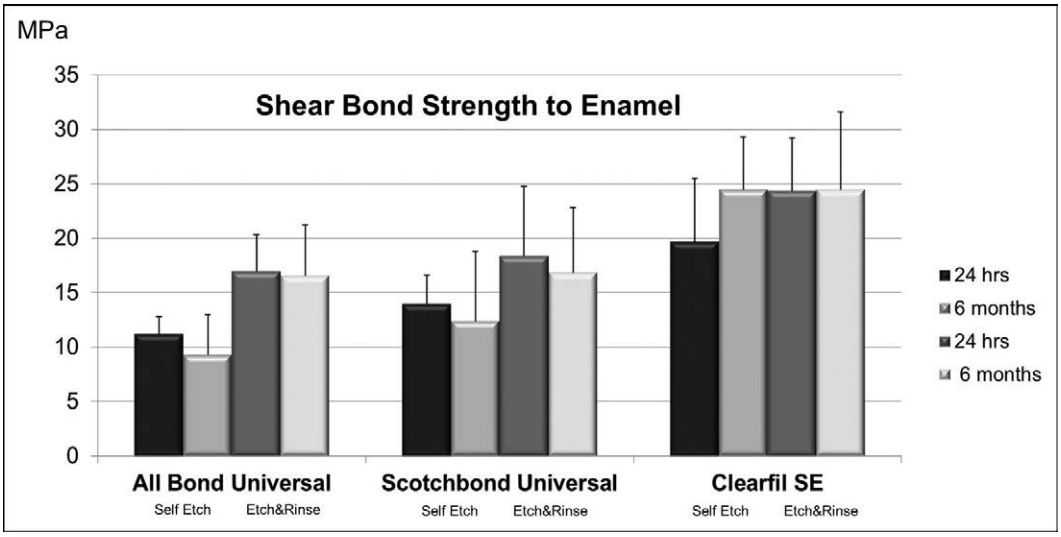


Figure 2. *Mean shear bond strength of adhesive agents applied in self-etch and etch-and-rinse modes over time.*

post-hoc test ($\alpha$=0.05) to evaluate the effects of bonding agent (three-levels), surface treatment (two-levels), and time (two-levels) on the shear bond strength of composite to enamel. Following testing, the specimens were examined under light microscopy at 10× magnification to determine the failure mode as either: 1) adhesive fracture at the adhesive interface, 2) cohesive fracture in the composite, enamel, or dentin, or 3) mixed fracture involving a combined adhesive and cohesive fracture.

## RESULTS

Three-way ANOVA and Tukey post-hoc tests revealed significant differences in the mean shear bond strengths of adhesive agents ($p<0.001$) and surface treatment ($p<0.001$) but not on time ($p>0.05$) and with no significant interaction ($p=0.943$).

In general, the bond strength of composite to enamel was significantly greater using Clearfil SE compared to Scotchbond Universal or All-Bond Universal, which were not significantly different from each other. Etching the enamel significantly improved bond strengths of the universal adhesives compared to self-etching only. Storage time did not significantly affect bond strengths (Figure 2). A high percentage of mixed fractures including dentin corresponded to the higher bond strength values as found with Clearfil SE. The lowest bond strengths and the most adhesive failures occurred with All-Bond Universal followed by Scotchbond Universal in self-etch mode. More mixed fractures were found for both universal adhesives when applied in an etch-and-rinse mode. Storage time did not affect fracture mode (Figure 3).

## DISCUSSION

This *in vitro* study demonstrated that the etch-and-rinse or selective-etch technique is an effective

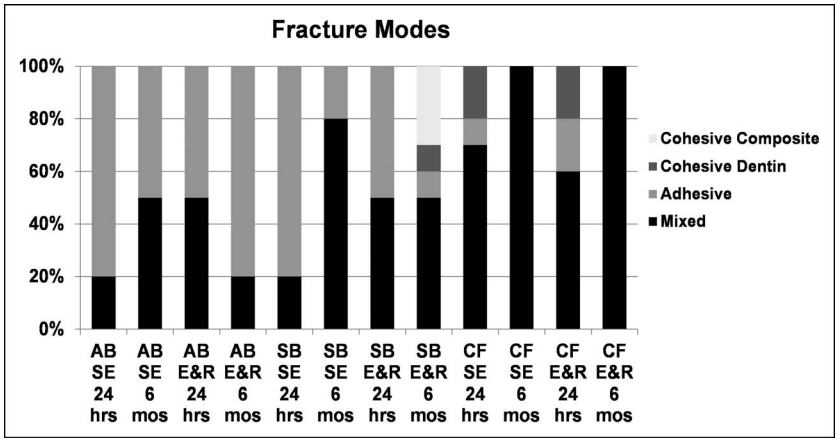


Figure 3. *Fracture mode of adhesive bonding agents over time.*

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approach to achieving more predictable and stable micromechanical bonding of composite to enamel. However, this study also demonstrated that there is considerable variation between dental bonding agents and ultimately that the shear bond strengths produced are largely material dependent.

Surface treatment significantly affected the shear bond strength of composite to enamel for the universal bonding agents; therefore, the null hypothesis that there would be no difference based on surface treatment must be rejected.

From the results of this study, the shear bond strengths of the universal adhesives to enamel were improved when the bonding agents were applied as two-step etch-and-rinse adhesives rather than one-step self-etch adhesives. This was attributed to an improved micromechanical bond being produced with the addition of the etch-and-rinse or selective-etch surface treatment. Etch-and-rinse or selective-etch adhesive systems are characterized by an initial etching step, typically with 32%-37% phosphoric acid, followed by a thorough rinsing procedure that is responsible for the complete removal of the smear layer and selective dissolution of the enamel rods. This creates microporosities in the enamel that are readily penetrated by bonding agents via capillary attraction.[14] Following polymerization, micromechanical interlocking of tiny resin tags within the etched enamel surface provide a strong micromechanical bond to enamel.[15] The alternative self-etch approach only dissolves the smear layer but does not remove it, as there is no rinsing step, leaving the dissolved products to become incorporated within the bonded layer.[16] Furthermore, the degree of demineralization produced by self-etch adhesives depends largely on the acidity or etching aggressiveness of the functional monomer and is material dependent. According to Sunfeld and others,[17] the penetration of the adhesive system may be restricted to the more superficial enamel layers with creation of shorter resin tags when self-etch adhesives are used without a selective-etch step.

Erickson and others[18]also found improved bond strengths with a pre-etch step and attributed this to the degree of etching or the etch morphology achieved. When used without a selective or pre-etch step, even the most acidic of the self-etch adhesives only produced an etch pattern primarily involving the ends of enamel prisms with little effect on the interprismatic regions. The subsequent resin penetration was described as a negative replica of the etch pattern with resin penetrating the etched prisms but not into the interprismatic unetched regions. The weakest acidic self-etch adhesives only achieved a fine pitting of the enamel surface and corresponding fine resin projections. Tay and others[19] also reported differences in the thickness of the enamel hybrid layers depending on the acidity of the adhesive and the resultant aggressiveness of apatite dissolution.

Both Scotchbond Universal (pH=2.7) and All-Bond Universal (pH=3.2) are considered ultramild to mild acidic adhesives; therefore, the additional selective-etch step followed by thorough rinsing logically produced improved micromechanical bonds between the composite resin and the highly mineralized enamel substrate. Nonetheless, neither the acidity of the adhesive agent, thickness of the hybrid layer, nor the length of the resin tags are solely responsible for bonding effectiveness and stability for all adhesives. This study confirmed previous studies and demonstrated that an ultra-mild (pH=2.7) self-etch adhesive, Clearfil SE Bond (Kuraray), was capable of achieving strong bonds to enamel with or without a selective-etch step.[12,20] This was particularly evident for the six-month Clearfil SE groups in which the self-etch group produced the same mean shear bond strength as the etch-and-rinse group. The bonding effectiveness of Clearfil SE is believed to be related to the separation of the acidic monomers in its functional primer from its adhesive agent as well as its specific composition that includes methacryloxydecyl phosphate (MDP). The monomer MDP contains phosphate groups capable of producing ionic chemical bonds with calcium in hydroxyapatite. The universal adhesives are ethanol and water-based adhesive bonding agents and also contain MDP; however, by virtue of the etch, primer and adhesive components being combined, the bond strength may ultimately be reduced. Ultimately, the shear bond strength of a dental bonding agent is material dependent.

Within the limitations of this study, the bond strengths produced by the different adhesive bonding agents were significantly different regardless of storage time and surface treatment. These differences are likely due to the specific chemical composition and formulation of each adhesive bonding agent. The null hypothesis that there would be no significant difference in the shear bond strength of composite to enamel based on type of adhesive bonding agent must therefore be rejected. The universal bonding agents are considered simplified adhesives and specifically as fifth or seventh generation bonding agents depending on their use with or without a selective etch step. As stated previously, one-step self-etch adhesives combine the acidified primer and

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adhesive agents and two-step etch-and-rinse adhesives combine the primer and adhesive and traditionally both have been more acidic and hydrophilic than the two-step self-etch adhesives that separate their acidic primers from the bonding agents. The hydrophilicity of the one-step self-etch adhesives has been stated to be the main disadvantage of these materials. This hydrophilicity leads to decreased bond strengths due to permeability of the adhesive layer and contributes to the hydrolysis of resin polymers and the consequent degradation of tooth-resin bonds over time.[15,21,22]

In terms of failure mode, Al-Salehi and Burke[23] reported that there is a relationship between the bond strength and fracture failure mode. From the results of this study, the higher bond strengths did correlate with greater mixed fractures or cohesive plus adhesive failure modes. Clearfil SE in etch-and-rinse and self-etch modes had more mixed fractures than either All-Bond Universal or Scotchbond Universal in either mode. The universal bonding agents produced more mixed fractures when used in etch-and-rinse mode than self-etch mode, which also correlated with bond strength. Storage time had no effect on failure mode.

From the results of this study, we failed to reject the null hypothesis that there would be no significant difference in the shear bond strength of composite to enamel based on time. Although the bond strengths of these new universal adhesives were found to be inferior, the bond strengths of the materials between 24 hours and six months of water storage were not significantly different; therefore, longer storage times would be needed to determine the effect of bond strength over time.

## CONCLUSIONS

The new universal bonding agents demonstrated higher shear bond strengths to enamel with the added selective-etch step; however, neither adhesive produced shear bond strengths comparable to Clearfil SE, which also produced the most mixed fractures. Storage time did not affect shear bond strengths of any of the materials tested.

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### Human Subject Statement

This study was conducted in accordance with all the provisions of the human subject oversight committee guidelines and policies at Wilford Hall Ambulatory Surgical Center. The approval code for this study was FWH20120082N. This study was conducted at Wilford Hall Ambulatory Surgical Center.

### Conflict of Interest

The views expressed in this article are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, or the United States Government or the Canadian Forces, Department of National Defense, or the Canadian Government. The authors do not have any financial interest in the companies whose materials are discussed in this article.

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# Surface Roughness and Gloss of Actual Composites as Polished With Different Polishing Systems

SA Rodrigues-Junior • P Chemin • PP Piaia
JL Ferracane

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### Clinical Relevance

The choice of polishing system should take into consideration the type of composite used. For actual commercial composites, multistep systems produce lower surface roughness and higher gloss than the one-step system.


## SUMMARY

**Objective: This in vitro study evaluated the effect of polishing with different polishing systems on the surface roughness and gloss of commercial composites. Methods: One hundred disk-shaped specimens (10 mm in diameter × 2 mm thick) were made with Filtek P-90, Filtek Z350 XT, Opallis, and Grandio. The specimens were manually finished with #400 sandpaper and polished by a single operator using three multistep systems (Superfix, Diamond Pro, and Sof-lex), one two-step system (Polidores DFL), and one one-step system (Enhance), following the manufacturer's instructions. The average surface roughness (μm) was measured with a surface profilometer (TR 200 Surface Roughness Tester), and gloss was measured using a small-area glossmeter (Novo-Curve, Rhopoint Instrumentation, East Sussex, UK). Data were analyzed by two-way analysis of variance and Tukey's test (α=0.05). Results: Statistically significant differences in surface roughness were identified by varying the polishing systems ($p<0.0001$) and by the interaction between polishing system and composite ($p<0.0001$). Pairwise comparisons revealed higher surface roughness for Grandio when polished with Sof-Lex and Filtek Z250 and Opallis when polished with Enhance. Gloss was influenced by the composites ($p<0.0001$), the polishing systems ($p<0.0001$), and the interaction between them ($p<0.0001$). The one-step system, Enhance, produced the lowest gloss for all composites. Conclusions: Surface roughness and gloss were affected by composites and polishing systems. The inter-**


*Sinval Adalberto Rodrigues-Junior, DDS, MS, PhD, Health Sciences Postgraduate Program, Universidade Comunitária da Região de Chapecó, Chapecó, Brazil, and School of Dentistry, Universidade Comunitária da Região de Chapecó, Chapecó, Brazil

Priscila Chemin, DDS, School of Dentistry, Universidade Comunitária da Região de Chapecó, Chapecó, Brazil

Priscila Peroza Piaia, DDS, School of Dentistry, Universidade Comunitária da Região de Chapecó, Chapecó, Brazil

Jack Liborio Ferracane, PhD, Division of Biomaterials and Biomechanics, Department of Restorative Dentistry, Oregon Health and Science University, Portland, OR, USA

*Corresponding author: Universidade Comunitária da Região de Chapecó, Área de Ciências da Saúde, Caixa postal 1141, Av. Senador Atílio, Fontana, n. 591-E, Efapi, CEP 89809-000, Chapecó, SC, Brazil; e-mail: rodriguesjunior.sa@unochapeco.edu.br

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**action between both also influenced these surface characteristics, meaning that a single polishing system will not behave similarly for all composites. The multistep systems produced higher gloss, while the one-step system produced the highest surface roughness and the lowest gloss of all.**

## INTRODUCTION

Dental composites have evolved throughout the years, undergoing modifications in composition and properties. The first composites were constituted by hard and large filler particles and were hard to polish. The refinement of the milling and grinding techniques resulted in the microhybrid composites, constituted of particles that vary from 0.4 to 1.0 μm in size and by nanoparticles (ranging from 1 to 100 nm). These composites are considered universal composites and are used for both anterior and posterior restorations.[1] Most recently, nanofill composites were introduced as another category of universal composite, constituted exclusively of nanoscale-sized filler particles.[2,3] Studies involving such composites have reported excellent polishability as well as superior polish retention.[2,4,5]

Recently, dental composites with new monomers have been introduced. One of them, the epoxy-based silorane system, aims to reduce the shrinkage stress resulting from the cure of traditional dimethacrylate-based composites.[6] The silorane-based composite has shown promising results of low shrinkage stress generation and water sorption and good mechanical properties.[6-8]

The surface quality of the restorations influences their clinical performance and affects aspects such as anatomic form, shade, gloss, and the surface roughness[9] with resulting bacterial accumulation. In fact, a threshold surface roughness of 0.2 μm or greater has been suggested as enhancing bacterial accumulation.[10,11] Also, the surface quality influences the speed of water diffusion into the bulk of the material and its subsequent degradation and affects the wear resistance resulting from toothbrushing or occlusal contact from mastication.[9]

Gloss plays a rather important role in esthetic dental restorations since differences in gloss between the restoration and the surrounding enamel are easily detected by the human eye, even when there is color match between the restoration and the tooth structure.[12] Also, a glossy enamel surface is maintained when submitted to mechanical wear, while the glossy surface of composites, which are typically lower initially, tend to further decrease under the same mechanical challenge over time.[12]

Several authors have suggested that the smoothest surface is achieved using a Mylar strip to cover the composite surface during curing.[5,13-18] However, the smooth surface produced by the contact with a Mylar strip results from the formation of a resin-rich superficial layer, less hard in comparison with the material in the bulk but near the surface, despite the inhibition of the contact of the composite surface with oxygen during polymerization by the strip.[14,19] This superficial layer would be more susceptible to changes of the restoration shade when in contact with staining food and beverages and therefore should always be removed by finishing and polishing procedures.[14,16]

Finishing refers to excess removal and gross contouring of the restoration and is usually performed using tungsten carbide finishing burs or diamonds. Polishing, on the other hand, refers to the reduction of surface roughness and removal of scratches generated by the finishing instruments.[16] Polishing also aims to prevent bacterial adhesion, which begins by the adhesion of a salivary pellicle layer on the surface of the tooth or restoration[20] and has been proven to be favored by rough surfaces.[11]

An immense variety of finishing and polishing systems is available in the dental market, involving multistep disks, fine and superfine diamond burs, abrasive disks, and diamond- and silicon-impregnated soft rubber cups.[5,21] Factors such as the flexibility of the back material in which the abrasive is embedded, the hardness of the abrasive, and its grit size all influence the ability of the polishing systems to produce a smooth surface.[13]

However, the polishing performance cannot be credited solely to the polishers. The interaction between polishing systems and composites on surface roughness has been shown to be significant in some studies,[15,17,22] indicating that the systems behave differently depending on the composite polished. This impairs one's ability to choose a single polishing system for all composites. In this sense, Berger and others[23] suggested the use of the polishing system from the same manufacturer as the composite as a safe option since these have produced good results in comparison with other polishers.

Nevertheless, discrepancies in literature reports and the continuous introduction of new polishing systems reveal the demand for new research on the topic. Therefore, the aim of our study was to evaluate

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Table 1: *Composites Used in the Study*

| Composite | Manufacturer | Shade | Organic Matrix | Filler | Light Curing Time (Seconds) | Batch Number |
|---|---|---|---|---|---|---|
| Filtek P90 (microhybrid) | 3M/ESPE, St. Paul, MN, USA | A2 | Silorane | Silica and zirconia—average size of 0.6 μm; 60% in vol., 82% in wt | 20 | N194550 |
| Filtek Z350 XT (nanofill) | 3M/ESPE | A2 | BisGMA, BisEMA, UDMA, TEGDMA | Silica and zirconia (clusters of 0.6-1.4 μm—individual particle size of 5-20 nm); 59.5% in vol., 73.2% in wt[a] | 20 | N186543 |
| Opallis (microhybrid) | FGM Produtos Odontológicos, Joinville, SC, Brazil | EA2 | BisGMA, BisEMA, TEGDMA, UDMA | Silanized barium-aluminum-silicate glass and nanoparticles of silica dioxide (40 nm to 3 μm—average size of 0.5μm); 57%-58% in vol., 78.5%-79.8% in wt | 20 | 031011 |
| Grandio (nanohybrid) | Voco, Cuxhaven, Germany | A2 | | ~84%-85% filler in wt[b] | 20 | 1139078 |

[a] *Rodrigues Junior and others.*[24]
[b] *Beun and others.*[25]

the surface roughness and gloss produced by different polishing systems on different commercial composites, testing the null hypothesis that there would be no difference in surface roughness and gloss, regardless of the composite or polishing system used.

## METHODS AND MATERIALS

One hundred disk-shaped specimens (10 mm in diameter × 2 mm thick) were made using the composites described in Table 1. The specimens were produced by packing the composite into a stainless-steel mold. A Mylar strip was placed over the surface of the uncured specimen and pressed against it with a glass plate in order to extrude the excess material. The specimens were light cured for the time recommended by the manufacturers (Table 1), using an LED light-curing unit (Ultraled, series no. 1010179, Dabi Atlante, Ribeirão Preto, SP, Brazil). The light irradiance was monitored with an LED radiometer (ECEL, series no. 000165, Ribeirão Preto) before usage and ranged between 653 and 663 mW/cm$^2$.

The specimens were removed from the mold immediately after light curing and were stored in distilled water at room temperature for 24 hours in the dark. After the storage period, one side of each specimen was finished on #400 carburundum paper. The specimens were then randomly allocated to each of the five polishing systems (Table 2).

Each polishing point was used only once with a low-speed hand piece (KaVo, Joinville, SC, Brazil). The polishing procedure was performed by a single operator, according to the manufacturer's instructions:

1. Diamond Pro (multistep system)
   - Step 1—Coarse grit: coarse disk dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds
   - Step 2—Medium grit: medium disk dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds
   - Step 3—Fine grit: fine disk dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds
   - Step 4—Superfine grit: superfine disk dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds
2. Superfix (multistep system)
   - Step 1—Coarse grit: coarse disk dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds
   - Step 2—Medium grit: medium disk dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds
   - Step 3—Fine grit: fine disk dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds
   - Step 4—Superfine grit: superfine disk dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds

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Table 2: *Polishing Systems*

| Polishing System | Steps | Manufacturer | Composition | Approximate Average Particle Size (μm)[a] | Batch Number |
|---|---|---|---|---|---|
| Diamond Pro | 4 | FGM Produtos Odontológicos, Joinville, SC, Brazil | Polyester (PET), adhesive, abrasive, rubber silicone | Dark blue = 180<br>Medium blue = 100<br>Light blue = 25<br>White = 15 | 041111 |
| Superfix | 4 | TDV Dental Ltda., Pomerode, SC, Brazil | Polyethylene terephthalate, aluminum oxide, synthetic rubber resin, polyvinyl chloride, metal, water-based pigments | Dark green = 200<br>Light green = 100<br>Yellow = 30<br>White = 20 | 0812/1011 |
| Polidores DFL | 2 | DFL, Rio de Janeiro, RJ, Brazil | | Yellow = 40<br>White = 12 | — |
| Enhance | 1 | Dentsply, Petrópolis, RJ, Brazil | Tripolymer (methyl methacrylate-butadiene-styrene), silanized pirolitic sílica, uretane dimethacrylate, canforoquinone, N-methyl dietanolamine, aluminum oxide | 30 | — |
| Sof-Lex Pop On | 3[b] | 3M/ESPE, Sumaré, SP, Brazil | Polyester and aluminum oxide | Dark orange = 30<br>Light orange = 30<br>Yellow = 5 | 1123800210 |

[a] *From scanning electron microscope image analysis.*
[b] *The coarse grit of Sof-Lex Pop On was not used because it produced a coarse, uneven surface.*

3. Polidores DFL (two-step system)
   - Step 1—Coarse grit: coarse disk (yellow) dry for 20 seconds, rinse and dry with water/air syringe for 6 seconds
   - Step 2—Fine grit: fine disk (white) dry for 20 seconds, rinse and dry with water/air syringe for 6 seconds
4. Enhance (one-step system)
   - Step 1—Light pressure for 40 seconds, rinse and dry with water/air syringe for 6 seconds
5. Sof-Lex Pop On (multistep system):
   - Step 1—Medium grit: medium disk (orange) dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds
   - Step 2—Fine grit: fine disk (light orange) dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds
   - Step 3—Superfine grit: superfine disk (yellow) dry for 15 seconds, rinse and dry with water/air syringe for 6 seconds

The average surface roughness (Ra, μm) was measured with a surface profilometer (TR 200 Surface Roughness Tester, TIME Group, Pittsburgh, PA, USA) using a tracing length of 2 mm and a cutoff of 0.8 mm to maximize filtration of surface waviness. Three tracings were made on each specimen in a wheel spoke arrangement, and the average was calculated.

Gloss was measured using a small-area glossmeter (Novo-Curve, Rhopoint Instrumentation, East Sussex, UK), with a square measurement area of 2 × 2 mm and 60-degree geometry. The glossmeter was calibrated each time against the supplied black, reflective standard. Gloss was expressed in gloss units. A jig was designed to place the specimen over the aperture in the same place each time, and four measurements were made by rotating the specimen 90 degrees around its center. The average of the four measurements was determined. Initial gloss measurements were made of the surface created with #400 paper, but these were always essentially zero. Surface roughness and maximum gloss data were tabulated and analyzed by two-way analysis of variance and Tukey's multiple comparison test ($\alpha$=0.05).

The polishing systems were gold sputter coated for 45 seconds in a Denton Vacuum Desk II (Denton Vacuum Inc, Moorestown, NJ, USA) at a current of 45 mA and a vacuum of 50 mTorr. The specimens were observed with scanning electron microscopy (SEM) using secondary electron mode (Jeol, JSM-5910, Jeol Ltd, Tokyo, Japan). SEM micrographs at 200× and 500× were made of the polishing systems, and an estimate of the average particle size was obtained by comparing the size of several of the particles to the measurement bar in the SEM images.

## RESULTS

The results of surface roughness of the composites polished with the different polishing systems are presented in Table 3. Two-way analysis of variance

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Table 3: *Average Surface Roughness Values in μm and Standard Deviation (±SD) for the Composites and Polishing Systems Evaluated*

| Composite | Polishing System | | | | |
|---|---|---|---|---|---|
| | Diamond Pro | Superfix | Polidores DFL | Enhance | Sof-Lex |
| Grandio | 0.229 ± 0.046 Aa | 0.211 ± 0.035 Aa | 0.387 ± 0.044 Aa | 0.444 ± 0.260 Aa | 0.693 ± 0.293 Ba |
| Filtek P90 | 0.422 ± 0.076 Aa | 0.486 ± 0.217 Aa | 0.412 ± 0.171 Aa | 0.686 ± 0.243 Aa | 0.347 ± 0.101 Aa |
| Filtek Z350 | 0.298 ± 0.068 Aa | 0.306 ± 0.104 Aa | 0.587 ± 0.236 Aa | 0.705 ± 0.162 Ba | 0.619 ± 0.131 Aa |
| Opallis | 0.285 ± 0.146 Aa | 0.268 ± 0.054 Aa | 0.388 ± 0.113 Aa | 0.759 ± 0.067 Ba | 0.458 ± 0.097 Aa |

*Capital letters refer to statistical groupings in the row, and small letters refer to statistical groupings in the column. Different letters indicate statistical differences between groups (p<0.05).*

revealed that there was no significant difference in surface roughness between the composites evaluated ($p$=0.077). On the other hand, statistically significant differences were found between polishing systems ($p$<0.0001) and from the interaction between composites and polishing systems ($p$=0.001). The surface roughness produced by the polishing systems for each composite was ranked as follows: for Grandio → Diamond Pro = Superfix = Polidores DFL = Enhance < Sof-Lex Pop On; for Filtek P90 → Diamond Pro = Superfix = Polidores DFL = Enhance = Sof-Lex Pop On; and for Filtek Z350 and Opallis → Diamond Pro = Superfix = Polidores DFL = Sof-Lex Pop On < Enhance (Figure 1). Pairwise multiple comparisons showed significantly higher surface roughness for Grandio when polished with the Sof-Lex Pop On system and of Filtek Z350 and Opallis when polished with the Enhance polishing system (Table 3).

The results of gloss are presented in Table 4. Two-way analysis of variance identified statistically significant differences in gloss between the composites ($p$<0.0001) and the polishing systems ($p$<0.0001) and from the interaction between composites and polishing systems ($p$<0.0001). The gloss produced by the polishing systems for each composite was ranked as follows: for Grandio and Filtek P90 → (Sof-Lex Pop On = Diamond Pro = Superfix) > (Polidores DFL = Enhance) and for Filtek Z350 and Opallis (Sof-Lex Pop On = Polidores DFL = Superfix) > (Diamond Pro = Enhance) (Table 4).

Figures 3 and 4 present the abrasive surface of the polishing systems used in the study. The estimated average of the abrasive particle size, based on the magnitude bar of the micrographs, is presented in Table 2. All the flexible abrasive disk systems used were constituted by aluminum oxide abrasive particles with different average size and shape, as revealed by the SEM micrographs (Figures 3A-H and 4A-C). Superfix and Diamond Pro presented irregular-shaped particles in the coarse disk ranging from 180 to 200 μm. The following grits of Diamond Pro presented round-shaped abrasive particles, similar to Sof-Lex though bigger. The medium, fine, and superfine disks of Superfix contained irregular filler particles, close to the average size of the disks of Diamond Pro. Micrographs of Enhance revealed a relatively flat surface covered by approximately 30-μm-sized abrasive particles (Figure 4D), while the

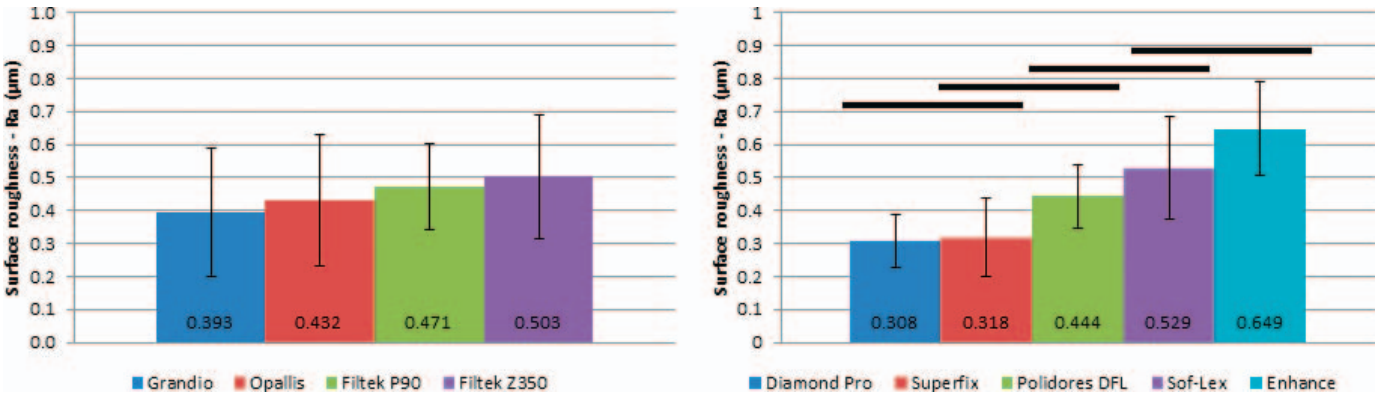


Figure 1. *Surface roughness did not differ significantly for the composites evaluated (left). Surface roughness produced by the different polishing systems varied (right). Black bars indicate that there is no statistically significant difference between groups.*

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Table 4: *Average Gloss Values and Standard Deviation (±SD) for the Composites and Polishing Systems Evaluated*

| Composite | Polishing System | | | | |
|---|---|---|---|---|---|
| | Diamond Pro | Superfix | Polidores DFL | Enhance | Sof-Lex |
| Grandio | 29.40 ± 6.8 Aa | 42.66 ± 3.94 Ab | 22.74 ± 5.49 Bb | 22.74 ± 9.98 Ba | 44.44 ± 3.43 Aa |
| Filtek P90 | 48.26 ± 10.91 Aa | 47.46 ± 18.89 Ab | 34.7 ± 11.20 Ba | 33.36 ± 14.79 Ba | 56.12 ± 3.49 Aa |
| Filtek Z350 | 31.32 ± 4.27 Ba | 41.32 ± 4.82 Ab | 52.4 ± 10.12 Aa | 14.02 ± 4.82 Ba | 59 ± 6.12 Aa |
| Opallis | 36.20 ± 10.04 Ba | 68.22 ± 3.26 Aa | 48.08 ± 7.47 Aa | 27.44 ± 4.18 Ba | 50.36 ± 8.83 Aa |

*Capital letters refer to statistical groupings in the row, and small letters refer to statistical groupings in the column. Different letters indicate statistical differences between groups ($p<0.05$).*

two-step Polidores DFL showed irregular particles of about 40 and 12 μm in the coarse and the fine disks, respectively (Figure 4E,F).

## DISCUSSION

Most restorative procedures involving composites involve curing against a polyester or metallic strip that aids in the insertion of the composite layers. Regardless of the evidence that the surface in contact with the strip is usually smoother, gross contouring is required to better define the anatomy, and the scratch reduction and smoothening during initial polishing is required to achieve a highly polished, light-reflective, enamel-like surface.[21] An adequate surface polishing contributes to the restoration longevity by reducing the surface roughness, stain accumulation, and gingival inflammation and minimizing wear.[23] Also, polishing should produce high gloss in order to mimic the natural tooth structure in esthetic restorations, as increasingly demanded by patients.[26]

Proper polishing involves a complex combination of factors related to the restorative material, the restoration anatomy, the polishing system, and the operator's ability. Manual polishing was chosen in this study because it better simulates the clinical conditions. Jones and others[27], though, have shown that operator-dependent factors, such as force, speed, and application time, vary widely from one operator to another. Also, Heintze and others[28] revealed that surface roughness and gloss strongly rely on force and polishing time. Based on their results, composites achieve roughness values lower than the 0.2-μm threshold only after 60 seconds of polishing. Time was the only operator-dependent variable controlled in this study, achieved by using a digital chronometer held by a second person, and reached 60 seconds for only two polishing systems (Diamond Pro and Superfix). Even so, the roughness values in these groups were still higher than 0.2 μm, varying from 0.211 to 0.486 μm.

Jung and others[29] demonstrated the influence of the operator's experience on polishing. Studies conducted to simulate clinical polishing procedures rely on the operator's manual ability and might result in high variability. Other studies, in contrast, reveal no connection between the quality of the surface polishing and the operator's clinical

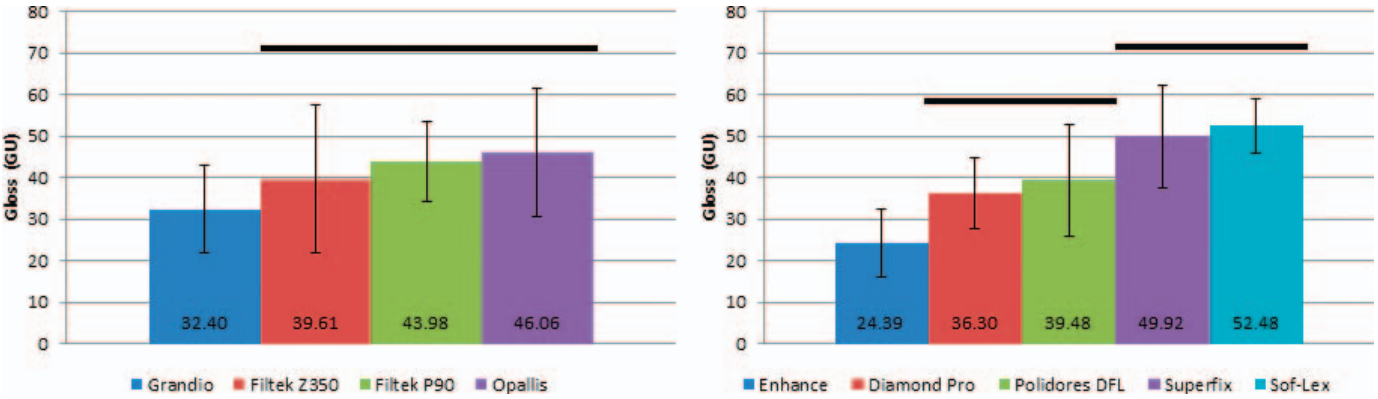


Figure 2. *Gloss of the composites evaluated (left). Gloss produced by the polishing systems studied (right). Black bars indicate that there is no statistically significant difference between groups.*

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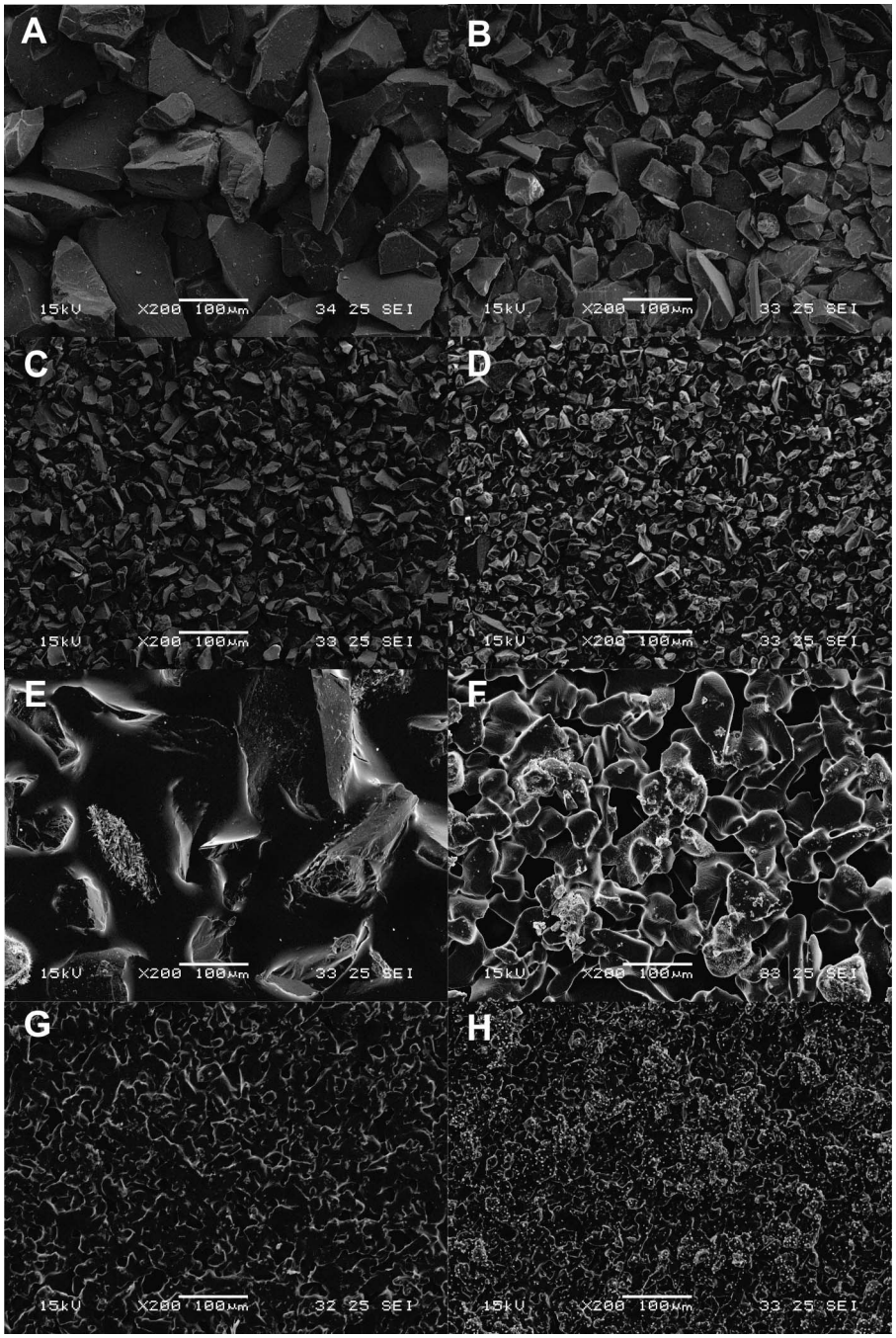


Figure 3. *Micrographs of the polishing systems (200×). (A): Superfix coarse. (B): Superfix medium. (C): Superfix fine. (D): Superfix superfine. (E): Diamond Pro coarse. (F): Diamond Pro medium. (G): Diamond Pro fine. (H): Diamond Pro superfine.*

experience. Zimmerli and others[30] observed no significant differences in Ra values produced by operators of different age and experience levels. However, it is worth noting that the less experienced operator had, at least, 6 years of experience in dental practice. In the present study, a single third-year graduate student performed all the polishing procedures, using the different polishing systems.

The operator's ability to handle the polishing procedure also depends on the flexibility of the backing material to which the abrasive is dispersed[13,15,16] since it represents a mechanism for compensating the force applied during polishing.[28] The one-step Enhance system and the two-step Polidores DFL system are rubber-based instruments, meaning that the abrasive particles are dispersed in a rubber-like elastic matrix, constituted

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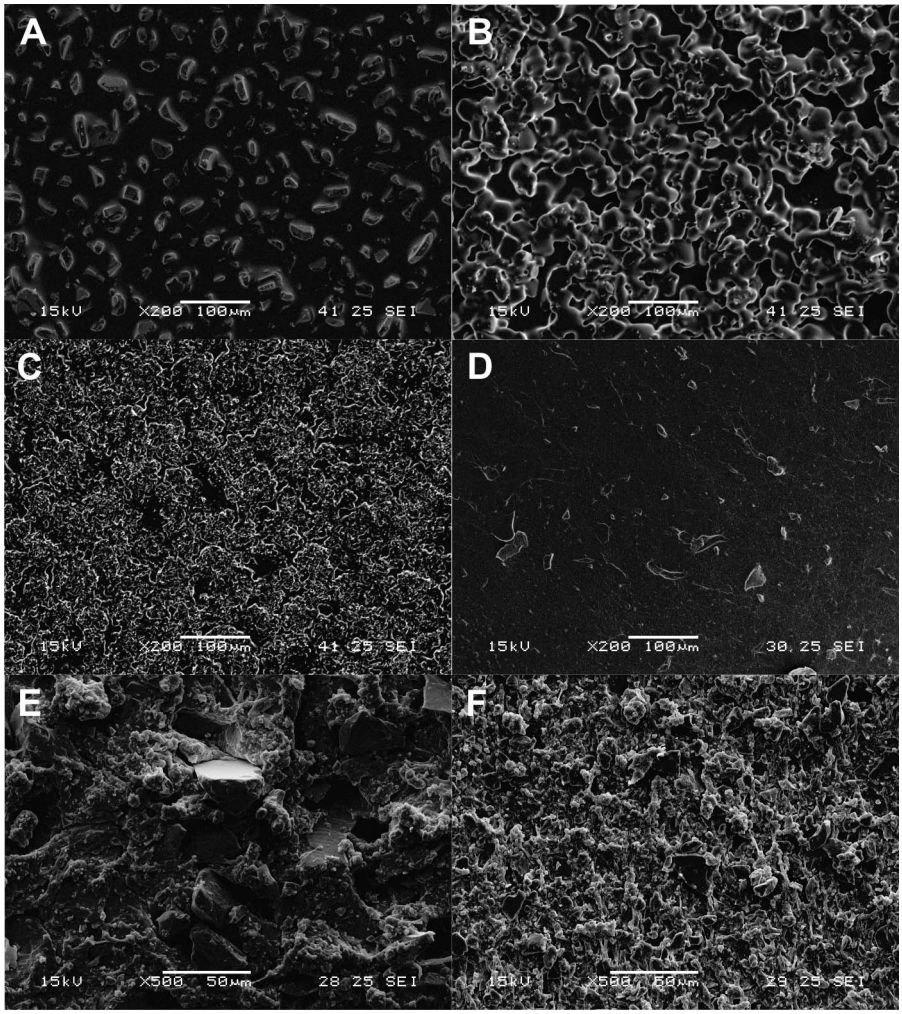


Figure 4. *Micrographs of the polishing systems (200× and 500×). (A): Sof-Lex medium. (B): Sof-Lex fine. (C): Sof-Lex superfine. (D): Enhance. (E): Polidores DFL coarse. (F): Polidores DFL fine.*

by a synthetic elastic polymer (Enhance) or silicon (Polidores DFL).[21] In this sense, these systems are stiffer and do not deflect with the applied force as easily as the flexible disks do.[28]

On the other hand, these systems are produced in variable shapes that include disks, cups, and points, helping to polish curved surfaces with the adequate anatomic contour of the tooth.[15] Those employed in this study were disk shaped, so the flat surface of the specimens could be uniformly polished regardless of the polishing system used. While the two-step system produced similar roughness results to the two multistep systems, the one-step system produced the highest surface roughness with Filtek Z350 and Opallis (Table 3; Figure 1). Both systems were used for 40 seconds, which was the least polishing time in the study. Therefore, it is likely that the difference between the one-step and the two-step systems might have been caused by a sequential decreasing particle size in the latter, leading to results similar to the multistep systems. For instance, the average particle size depicted in micrographs of the two-step system was about 40 μm for the coarse grit and 12 μm for the fine grit (Table 2). Other studies, though, have employed Enhance (30 μm average particle size) as a pretreatment for PoGo (about 10-15 μm average particle size, according to da Costa and others[5]), considering it, therefore, as a two-step system, and have obtained lower surface roughness than other polishing procedures[31] and even similar to the one produced by the Mylar strip, depending on the composite polished.[15]

There was no significant difference among the multistep flexible disk systems for most composites. The exception was the Sof-Lex Pop On system, which produced the highest surface roughness on Grandio (Table 3). This system differs from the other disk systems by having more flexible disks that have a

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metallic center through which they are attached to the mandrel and that demand tilting during polishing of a flat surface. However, the high flexibility of the disk might produce uneven surfaces when the applied force is high. This effect might have been higher for Grandio, which presented significantly higher surface roughness after polishing with this system (Table 3). Although in absolute terms Diamond Pro and Superfix produced lower surface roughness comparing to Sof-Lex Pop On, the difference was not significant, and the general surface roughness results with multistep systems reinforce the idea that sequential application of decreasing particle size disks is a desirable approach to produce a smooth surface in restorations.

Wear, according to Turssi and others,[32] could be defined as "the progressive loss of substance resulting from a mechanical interaction between two contacting surfaces, which are in relative motion." In this sense, the polishing procedure of dental restorations is an intentional, selective, and controlled wear of the restorative material surface, produced mostly through two-body abrasion,[21,32] aiming to attain a smooth, glossy surface. As such, polishing occurs as a function of a tribological process that involves structural aspects (eg, the materials in contact and contact geometry), interaction conditions (eg, the loads and the contact duration), and environmental conditions (eg, the surface chemistry, topography, and temperature).[32]

Aspects related to the composites that might affect polishing are the hardness of the filler relative to the abrasive, the filler content, shape, size and orientation, the filler hardness relative to the matrix, the degree of conversion of the matrix, and the stability of the silane coupling between the fillers and the resin matrix.[13,32] Among all these factors, the polishability and the surface roughness of composites has been shown as a function of the filler particle size,[1,5,33] with larger particles increasing surface roughness. Marghalani[33] also identified a significant influence of the filler shape on surface roughness, suggesting that irregular-shaped particles produce rougher surfaces. Based on our results, one could assume that compositional differences between the composites tested significantly influenced the surface roughness results (Table 3).

It has been shown that the mechanical behavior of composites is strongly dependent on the filler packing fraction, which in turn varies as a function of the average filler particle size.[34] The filler wt% of the composites was based on information provided by the manufacturers, except that for Filtek Z350 and Grandio, which was determined elsewhere through thermogravimetric analysis.[24,25] Grandio presented slightly lower filler wt% (84%) than that reported by the manufacturer (87%),[25] while Filtek Z350 presented up to 73% filler by weight.[24] According to Sabbagh and others,[35] these data discrepancies should be viewed with caution, as they might result from weighing the particles after the silane treatment. The filler percentage reported ranged from 73% to 84% to 85%, hence an 11% to 12% difference. Some studies have suggested that the microstructural arrangement of the filler plays an important role in the mechanical behavior of the composites regardless of the filler wt%.[36]

With the exception of the silorane-based composite Filtek P90, meant for posterior restorations, the other composites studied also contain nanosized filler particles that range from 20 to 40 nm and tend to fill the spaces in between the larger particles, therefore protecting to some extent the soft matrix from abrasion.[16] This particle size distribution could explain the lack of substantial difference in surface roughness for these composites and suggests a homogeneous behavior of these materials, closer to the microfill and nanofill composites, in spite of the presence of large particles.[5]

Based on the clustered arrangement of the nanofill particles in Filtek Z350, a wear mechanism has been suggested in which the clusters break off instead of plucking out the entire particle.[2,4,17] This mechanism is believed to be responsible for the polishability and polish retention of this composite.[2] Other composites containing nanosized filler particles, such as Opallis and Grandio, do not seem to present this mechanism. Janus and others[17] classify composites that contain both macro- and nanosized filler particles as nanofilled hybrid composites and reveal that these materials present voids from plucked-out filler particles after finishing. This would be consistent with the fact that the larger particles in Opallis and Grandio are cohesive particles and not clusters of smaller particles, as found in Z350, and therefore are not capable of breaking up but rather are plucked from the surface, leaving voids. Surface roughness of the silorane-based composite was comparable to the other dimethacrylate-based composites (Table 3; Figure 1). This was consistent with its similar filler composition to the other microhybrid composite, Opallis.

Gloss is an optical phenomenon defined by the amount of light rays that are reflected by a material

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surface at nearly the same angle as they hit the surface.[5,37] Based on this, one would expect gloss to present an inverse relationship with the surface roughness since the higher the surface roughness, the higher the degree of diffuse reflection of light, affecting gloss negatively.[19,26,34] This relationship, though, has not been so obvious in the literature.[26,28] Heintze and others[28] observed that the negative correlation between surface roughness and gloss varies during the polishing procedure and is not necessarily higher at the end of the procedure or similar for all composites.

Light reflectance is influenced by microstructural features of the material, namely, the mean size, shape, and index of refraction of the filler, and the viscosity and index of refraction of the matrix and the homogeneity of the filler-matrix complex.[5,12] According to Lefever and others,[12] the higher the filler size and the lower the homogeneity of the filler-matrix complex, the lower the light reflectivity of the composite material. Opallis and Filtek P90 presented average filler sizes of 0.5 and 0.6 μm, respectively, which were close to the size of the nanofill clusters in Filtek Z350. Grandio's filler phase, on the other hand, is constituted by irregular-shaped particles,[25] which has been shown to impair the production of a smooth, reflective surface compared with round-shaped filler particles.[34]

Gloss results revealed a significant interaction between composite and polishing system. Pairwise comparison revealed that there was no significant difference between Grandio and Filtek P90 when polished with Diamond Pro, Superfix, and Sof-Lex Pop On. A similar behavior was observed when Filtek Z350 and Opallis where polished with Superfix, Polidores DFL, and Sof-Lex Pop On. In absolute terms, the highest overall gloss was produced by Opallis when polished by Superfix; however, its gloss performance was lowered when polished with Diamond Pro and Enhance, reinforcing the trend of variability of the surface properties of actual composites with variation of the polishing systems. Among the multistep systems, Sof-Lex Pop On and Superfix were able to produce the highest gloss in all composites (Table 4). Low gloss values were produced by the one-step system Enhance for all composites (Table 4), which might be explained by the lowest polishing time used with this system.[28,37] Similar ranking was observed when Grandio and Filtek P90 were polished with Polidores DFL and Enhance and Filtek Z350 and Opallis were polished with Diamond Pro.

Differences in abrasive particle sizes, shapes, and distribution have been pointed out as influencing surface roughness and gloss[5] and could be observed through the micrographs (Figures 3 and 4). In absolute terms, the one-step system Enhance, which produced the highest roughness and the lowest gloss, presented the largest abrasive particles dispersed into a urethane elastic matrix.[21] Abrasives from the superfine disks of Diamond Pro and Superfix averaged 15 and 20 μm, respectively, while Sof-Lex showed the smallest abrasive particle (approximately 5 μm). One's expectation was that the smoother and glossier surfaces would be produced by the polisher with the smallest abrasive particle,[5] and Sof-Lex produced the glossiest surface, followed by Superfix, which also produced high gloss, similar to Sof-Lex, despite differences in abrasive grain size and shape (Figures 3 and 4). It has been stated that, besides size and shape of the abrasive, its binding to the matrix and the type and flexibility of the matrix might also influence the polishing efficiency.[5]

## CONCLUSION

The null hypothesis was rejected since surface roughness and gloss were affected by the composites and polishing systems studied. In addition, a single polishing system did not produce equivalent surface characteristics for all composites. Although each polishing system produced similar roughness on the four composites evaluated, there were some differences in relation to surface gloss. The multistep systems produced the highest gloss on Grandio and Filtek P90 but not on Filtek Z350 and Opallis.

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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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# Cytotoxic Effects of Hydrogen Peroxide on Human Gingival Fibroblasts In Vitro

M Furukawa • Jr K-Kaneyama • M Yamada
A Senda • A Manabe • A Miyazaki

### Clinical Relevance

This study aimed to elucidate the cytotoxic effects of an in-office bleaching agent on cultured human gingival fibroblasts.

## SUMMARY

**In-office bleaching is a popular treatment in modern esthetic dentistry. However, bleaching agents sometimes accidentally adhere to the gingiva and peripheral tissues, even when applied by well-trained dentists. This can lead to transient pain and whitish changes in the gingiva. Although these symptoms disappear within several hours, the effects of bleaching agents on gingiva have not been well described in the literature. The present study aimed to elucidate the cytotoxic effects of a bleaching agent on cultured human gingival fibroblasts (HGFs).**

**We performed a comprehensive analysis of the toxic effects of in-office bleaching agents on gingiva using cultured HGFs and DNA microarray. Survival rates of HGFs decreased with increases in the concentration of hydrogen peroxide, which became significant at concentrations of $1.5 \times 10^{-3}$% or higher at every time point. Concentrations lower than $1.5 \times 10^{-3}$% did not affect survival rates of HGFs. Cytotoxicity of hydrogen peroxide was significantly weakened by the addition of vitamin E. Stimulation by in-office bleaching agents triggered the proinflammatory cytokine tumor necrosis factor (TNF)–α cascade in gingival fibroblasts. As the TNF-α cascade can be inhibited by vitamin E additives, treatment with vitamin E may protect gingival fibroblasts against the toxic effects of an in-office bleaching agent.**

**The present results suggest that local administration of vitamin E to gingiva before in-**

*Masae Furukawa, DDS, PhD, assistant professor, Department of Clinical Cariology and Aesthetic Dentistry, School of Dentistry, Showa University, Tokyo, Japan

Joo-ri Kim-Kaneyama, PhD, associate professor, Department of Biochemistry, School of Medicine, Showa University, Tokyo, Japan

Mitsuyoshi Yamada, DDS, PhD, assistant professor, Department of Operative Dentistry, School of Dentistry, Aichi Gakuin University, Nagoya, Japan

Akira Senda, DDS, PhD, professor and chair, Department of Operative Dentistry, School of Dentistry, Aichi Gakuin University, Nagoya, Japan

Atsufumi Manabe, DDS, PhD, professor and chair, Department of Clinical Cariology and Aesthetic Dentistry, School of Dentistry, Showa University, Tokyo, Japan

Akira Miyazaki, MD, PhD, professor and chair, Department of Biochemistry, School of Medicine, Showa University, Tokyo, Japan

*Corresponding author: 2-1-1 Kitasenzoku, Ohta-ku, Tokyo, 1458515, Japan; e-mail: masae@dent.showa-u.ac.jp

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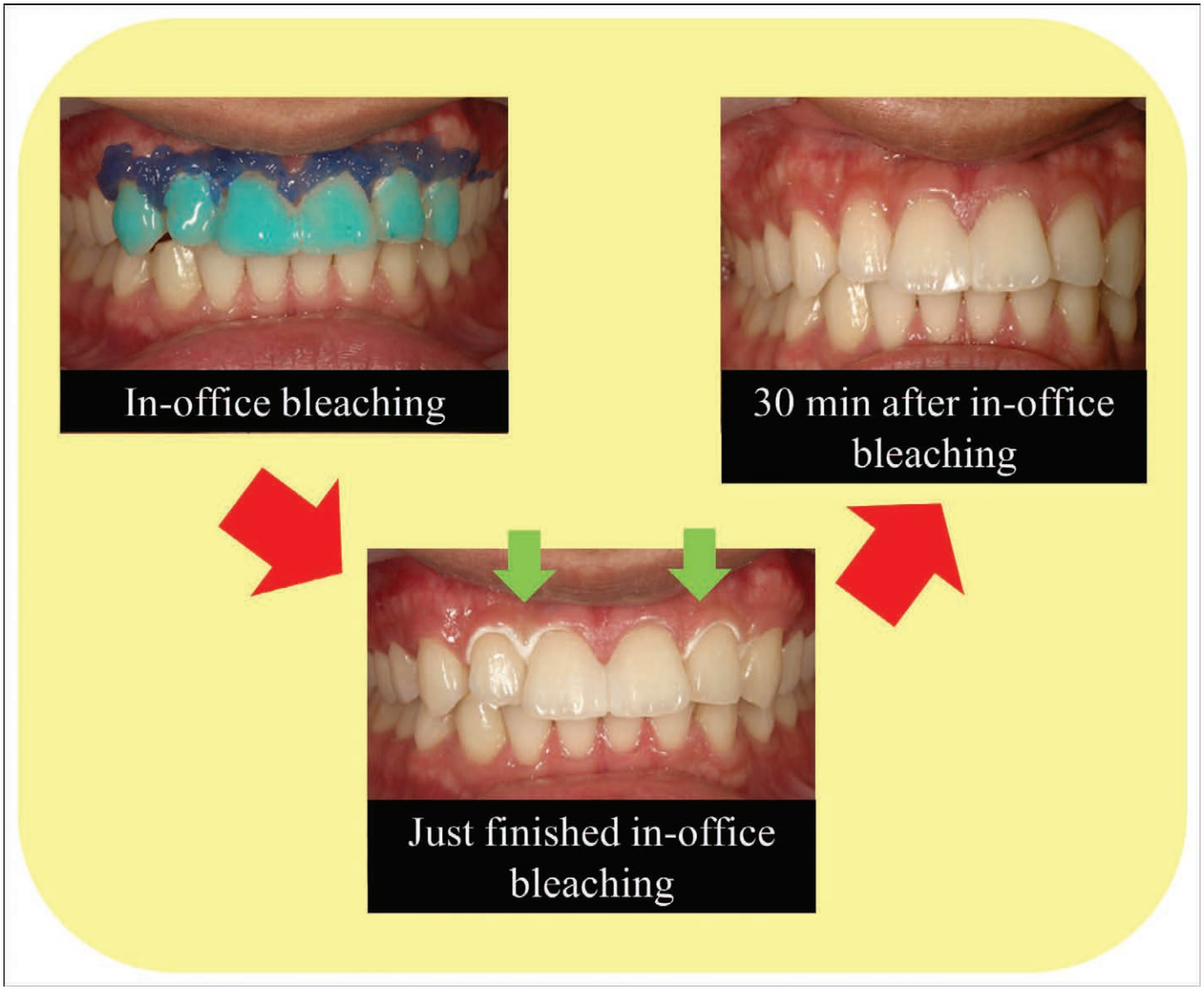


Figure 1. *Toxic effects of in-office bleaching agent on gingiva. Despite the application of petroleum jelly to protect the gingiva when performing in-office bleaching, the in-office bleaching agent sometimes comes into contact with the gingiva, as shown in the photo. When this occurs, the gingiva whitens and pain results, but the gingiva returns to its original state within 30 minutes and pain subsides.*

**office bleaching may be useful for preventing gingival irritation due to accidental adhesion of a bleaching agent.**

## INTRODUCTION

As patients' needs for dental treatment shift from traditional treatment including caries and dentures to esthetic treatment, an increasing number of people are visiting the dental office with hopes for whiter teeth. Acceptable vital tooth-whitening techniques include the "at-home"[1,2] and "in-office"[3,4] techniques. Over-the-counter (OTC) bleaching products are sold as cosmetics and are freely available through stores, pharmacies, and the Internet.[5]

Tooth sensitivity and gingival irritation are two of the most common side effects during/after at-home, in-office, and OTC bleaching.[6-11] Although in-office bleaching is a particularly popular method for bleaching, the bleaching agent may sometimes come into contact with the patient's gingiva or oral mucosa during the in-office bleaching procedure, even if the gingiva is protected with a light-cured resin or rubber dam and bleaching is performed by an experienced dentist (Figure 1). This may result in temporary whitening and pain in the gingiva or oral

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mucosa, but the pain subsides within a few hours and the whitened spot eventually regains its original color.[11,12] This has also been reported with at-home bleaching.[13-16] However, this phenomenon has yet to be studied from a molecular biology perspective, and there are no studies reporting the prevention or treatment of gingival irritation. Therefore, when patients experience gingival irritation during at-home bleaching, dentists usually recommend to stop bleaching, change the concentration of the bleaching agent, or change bleaching materials.[17] We thus set out to determine the effects of in-office bleaching agents on the gingiva *in vitro*. We examined the following four points using cultured human gingival fibroblasts (HGFs) to investigate the toxicity of in-office bleaching agents on gingiva. 1) HGFs were stimulated with hydrogen peroxide ($H_2O_2$), and cytotoxicity was examined by Alamar blue staining. In clinical practice, it is commonly believed that vitamin E cream can be applied to gingiva that has come into contact with an in-office bleaching agent to relieve the signs, but there is no evidence to support this notion. To test this belief, 2) under these conditions, the protective effects of vitamin E on cytotoxicity were examined. 3) Phalloidin staining was then performed in order to examine the changes in cell morphology under each condition. 4) To comprehensively analyze the cell response triggered in HGFs by in-office bleaching agent stimulation, DNA microarray profiling was performed to assess changes in gene expression induced by $H_2O_2$ and vitamin E addition.

The following null hypotheses were tested: 1) HGFs were subjected to inflammation by $H_2O_2$ exposure, (2) damaged HGFs were stimulated with a proinflammatory cytokine, and (3) damaged HGFs were restored by vitamin E supplementation.

## METHODS AND MATERIALS

### Examination of Cytotoxicity

HGFs (ScienCell, Carlsbad, CA, USA) were grown in a gingival fibroblast medium (ScienCell) supplemented with 5% inactivated fetal bovine serum (FCS; ScienCell) until the cells formed a single layer at 37°C in the presence of 5% carbon dioxide on a 10-cm dish coated with poly-L-lysine (poly-L-lysine–coated 100-mm dish; Iwaki, Tokyo, Japan). In the present study, fibroblasts after four to six population doublings (4-6 PD fibroblasts) were used in experiments. To measure cytotoxicity (Figure 2), HGFs were seeded onto 96-well multiplates (poly-L-lysine–coated 96-well micro plate; Iwaki) with $1 \times 10^5$ cells/well and were incubated for 24 hours, after which cells were stimulated by $H_2O_2$ (Wako) at the concentrations shown in Figure 3, and were allowed to react for 90 seconds, 5 minutes, 10 minutes, 30 minutes, or 60 minutes. Next, Alamar Blue (Invitrogen, Carlsbad, CA, USA) was added, and cultures were incubated for a further 24 hours (Figure 4) and then measured at 540 nm with an absorbance microarray scanner (VersaMax; Molecular Devices, Tokyo, Japan).

### Effects of Vitamin E Under $H_2O_2$ Stimulation

During 15% $H_2O_2$ stimulation, 50, 100, 125, 250, and 375 μM of (±)-α-tocopherol (Wako; "vitamin E") was added to cultures and was allowed to react for 90 seconds. Alamar blue was then added and cytotoxicity was examined as described above.

### Examination of Changes in Cell Morphology

Cultured HGFs were seeded onto six-well dishes, and a control group, a 15% $H_2O_2$ group, and a 15% $H_2O_2$ + 250 μM vitamin E group were each allowed to react for 90 seconds. After incubation, cultures were fixed in 10% formalin and cells were stained with TRITC-labeled phalloidin.

### Examination of Changes in Expression of Various Genes by Microarray

Using samples under the same stimulation conditions as described in the previous section, microarray (Takara Bio Dragon Genomics Center, Mie, Japan) was used to examine which genes in the $H_2O_2$ stimulation-induced gene cluster had expression suppressed by addition of vitamin E.

### Statistical Analysis

Data were expressed as means ± standard deviation for the multiple experiments. Student *t*-tests were used for concentration of $H_2O_2$ and vitamin E with cell viability ($p<0.05$).

## RESULTS

### Cytotoxicity

Almost no cytotoxicity from $H_2O_2$ was seen in the low-concentration stimulation group ($1.5 \times 10^{-3}$% or less) at any duration other than 60 minutes. However, 60-minute stimulation resulted in cytotoxicity at all concentrations. Stimulation at 15% $H_2O_2$, which is similar to the concentration used in clinical practice, showed marked cytotoxicity, lowering cell survival by half at 90 seconds ($p<0.05$), which is the shortest time period examined (Figure 3).

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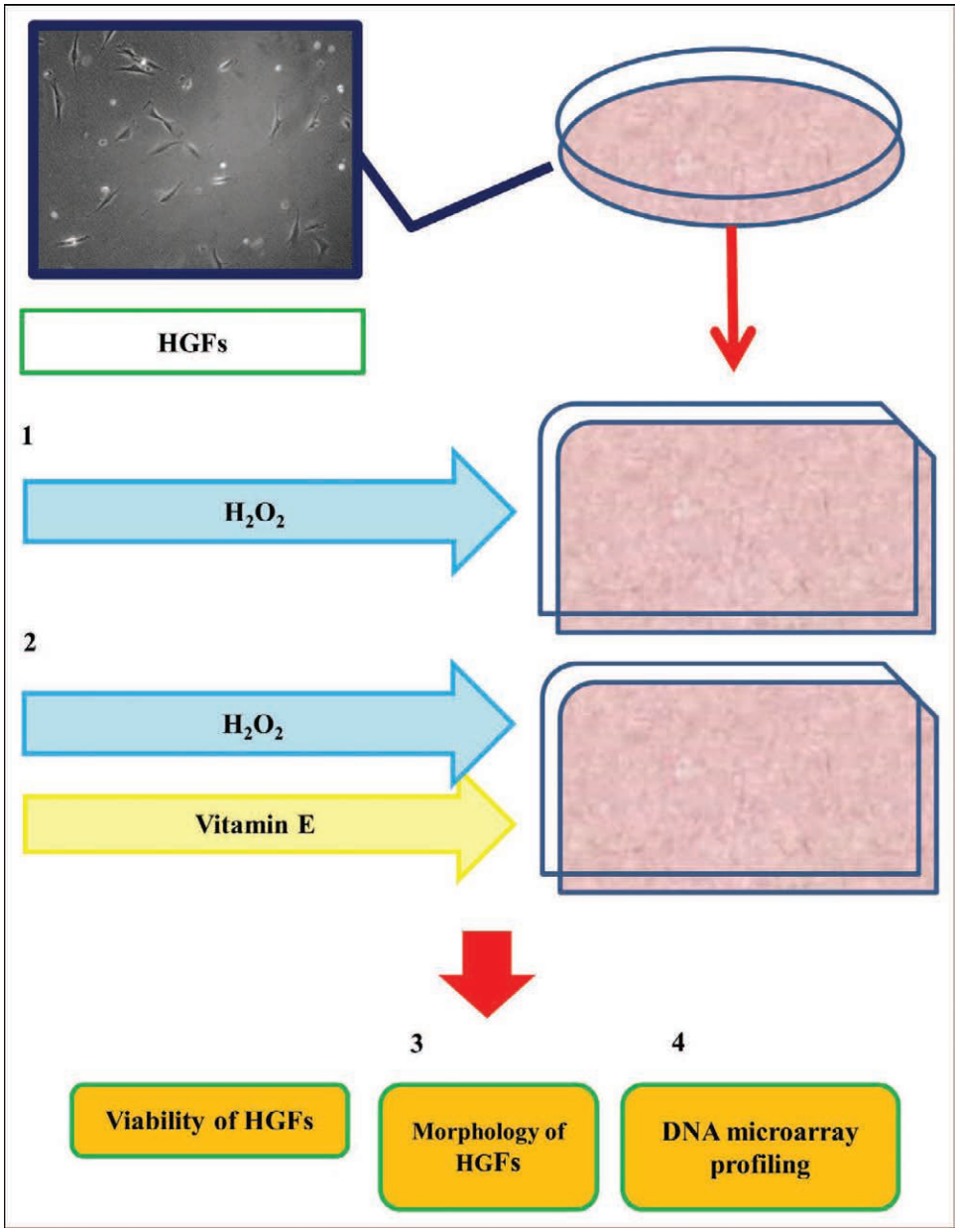


Figure 2. *Experimental methods. Human gingival fibroblasts were used to analyze cell survival rate, cellular morphological changes, and changes in gene expression* in vitro *after $H_2O_2$ stimulation and/or addition of vitamin E.*

### Examination of Cytotoxicity Suppression by Vitamin E

As HGF cytotoxicity from $H_2O_2$ stimulation was demonstrated in the results of the Cytotoxicity section above, we then examined whether addition of vitamin E as an antioxidant acts to suppress cytotoxicity from $H_2O_2$. As an experimental condition, 15% $H_2O_2$ was used, as it resulted in the most severe cytotoxicity effects in HGFs in the Cytotoxicity section, and it is the concentration closest to that used in clinical practice. A stimulation duration of 90 seconds was used, as this is when patients begin to feel pain when the in-office bleaching agent contacts the gingiva. Similarly to the Cytotoxicity section above, 50, 100, 125, 250, and 375 μM vitamin E was added to cultured HGFs. Cell survival, which was cut in half by $H_2O_2$ stimulation, recovered to control levels with the addition of 250 μM vitamin E (Figure 5).

### Observation of Changes in Cell Morphology

Cultured HGFs are normally spindle shaped (Figure 6a). The cell morphology of HGFs exposed to $H_2O_2$ stimulation changed dramatically (Figure 6b). Specifically, the spindle-shaped fibroblasts became circular after $H_2O_2$ stimulation (Figure 6b, arrow). Addition of vitamin E suppressed the rounding of

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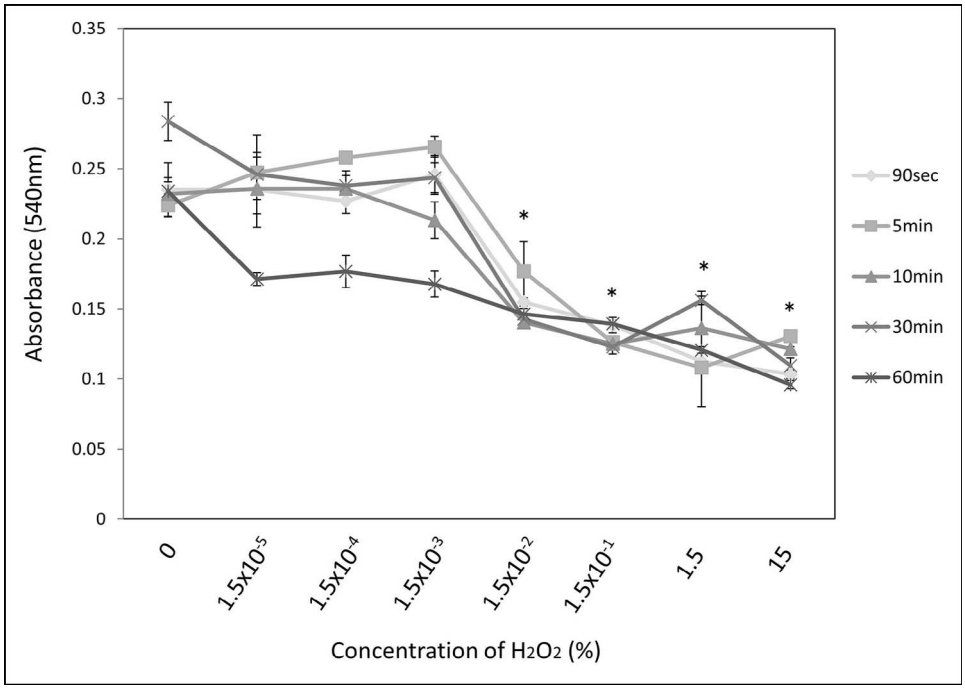


Figure 3. *Gingival fibroblast cytotoxicity after $H_2O_2$ stimulation. Cytotoxicity is low with $H_2O_2$ concentrations of 1.5 × $10^{-3}$% or higher. The significance of the differences was assessed by* t*-test (*$p<0.05$*).*

cell morphology caused by $H_2O_2$ stimulation (Figure 6c).

### Examination of Changes in Gene Expression by Microarray

Using the same stimulation samples as in the section above, microarray was used to analyze the gene clusters that were suppressed from the addition of vitamin E among the gene cluster induced by $H_2O_2$ stimulation. The results confirmed that $H_2O_2$ stimulation induced the expression of genes associated with the proinflammatory cytokine TNF-α cascade (Table 1). Expression of these genes was suppressed with vitamin E addition. These results demonstrate that $H_2O_2$ stimulation response includes triggering the proinflammatory cytokine TNF-α cascade and that addition of vitamin E suppresses the response of this cascade (Figure 7).

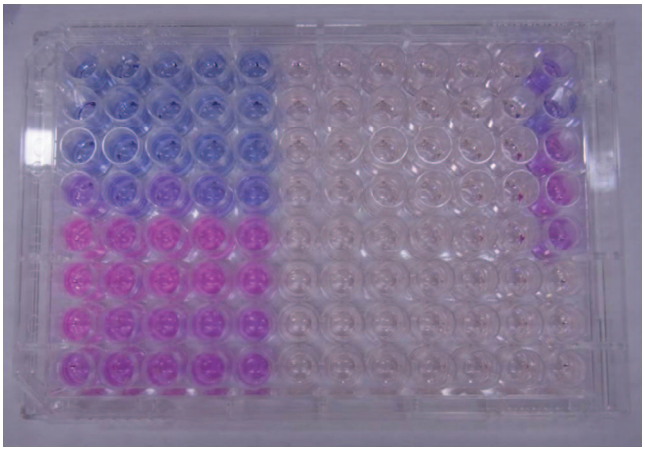

Figure 4. *Alamar blue staining. Red indicates high cytotoxicity.*

## DISCUSSION

We conducted *in vitro* examinations of signs such as whitening of the gingiva and pain that may result from tooth whitening. No studies to this effect in HGFs have been reported. These signs may arise from at-home bleaching agents[6,7,9] but mostly result from in-office bleaching agents[11,12] and OTC products.[10]

Many studies have been carried out on tooth hypersensitivity from in-office or at-home bleaching agents.[6-15] From *in vitro* studies, it has been concluded that whitening agents histologically penetrate the dentin[18] and do not damage the pulp.[19,20] In current dental practice, pain incurred during the procedure is generally treated with medication or fluoride or by stopping the procedure.[21-25] Several studies examining plaque control as an index have shown that whitening agents reduce plaque on the gingiva and reduce gingival inflammation.[26,27] Hydrogen peroxide and carbamide peroxide have been used for debridement during endodontic therapy,[28] in mouth rinses to reduce plaque in individuals with gingivitis,[29,30] and for treatment of periodontal diseases.[31]

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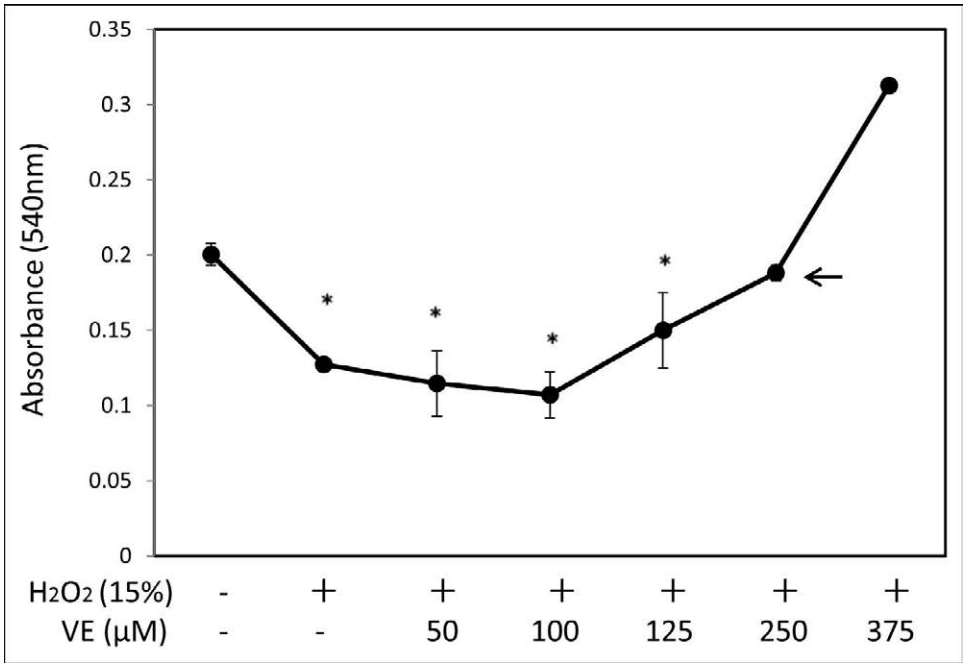


Figure 5. *Examination of cytotoxicity suppression by vitamin E. Various concentrations of vitamin E were added to human gingival fibroblasts treated with the $H_2O_2$ concentration obtained in cytotoxicity tests ($1.5 \times 10^{-3}$%). After a 90-second reaction time, Alamar blue was used to test cytotoxicity. Cell survival, which was cut in half by $H_2O_2$ stimulation, recovered to control levels with the addition of 250 μM of vitamin E. The significance of the differences was assessed by* t*-test (*$p<0.05$*).*

Gingival fibroblasts are known to be affected by $H_2O_2$.[32,33] However, studies have shown that fibroblasts in other parts of the body are more strongly affected.[34] In the present study, highly concentrated $H_2O_2$ caused inflammation in gingival fibroblasts and had toxic effects, with marked changes in cell morphology. One study found that higher concentrations of $H_2O_2$ caused senescence-like changes in cells.[33] It has long been known from *in vivo* animal experiments that $H_2O_2$ causes acute inflammation and even edema in skin.[35-40] Simon and others[41] have shown $H_2O_2$ to cause necrosis in human fibroblasts. Cells are protected by various types of enzymes and other substances and manage the immune system that responds to inflammation.[42,43] Inferring from the above studies, it is possible that $H_2O_2$ penetrates the cell membrane to cause damage, so that enzymes cannot do their job.

The $H_2O_2$ stimulation duration in the present study was 90 seconds. In our own experience in actual clinical practice, patients begin to complain of pain about 90 seconds after the in-office bleaching agent contacts the gingiva. At the same time, the gingiva whitens, and 60 minutes after touching the gingiva, both the pain and the whitening have completely disappeared (Figure 1).

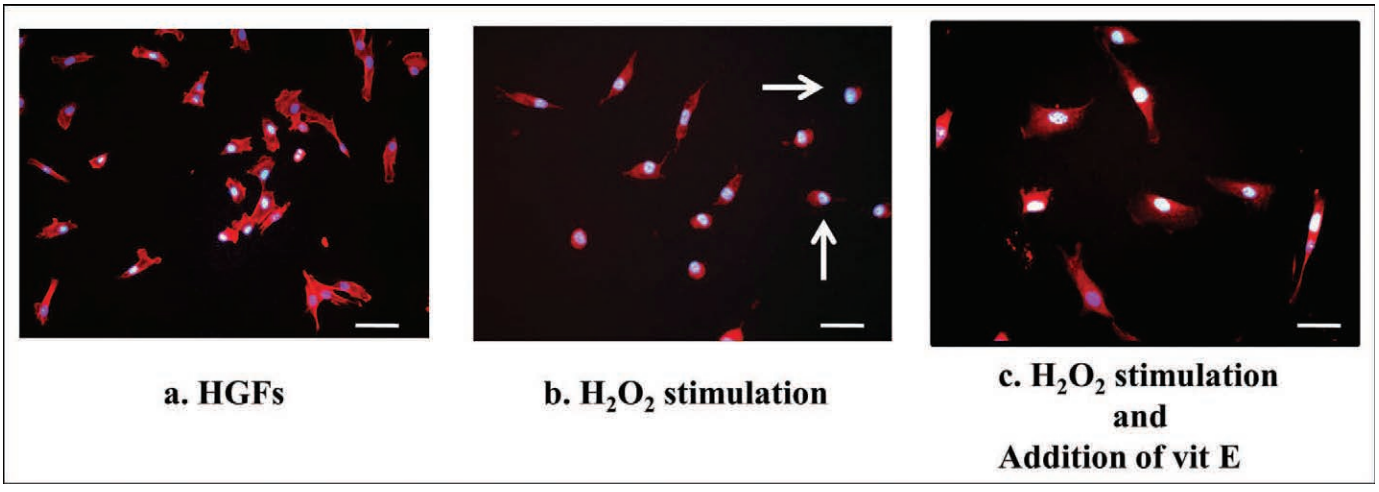


Figure 6. *Changes in cell morphology (phalloidin staining). Cultured human gingival fibroblasts were divided into (a): control group, (b): 15% $H_2O_2$ group, and (c): 15% $H_2O_2$ + 250 μM vitamin E group and were allowed to react for 90 seconds. Scale bars = 30 μm.*

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Table 1: *Gene Clusters Induced by $H_2O_2$ Stimulation and Suppressed by Addition of Vitamin E. Average of* log2 expression ratio *(Test/control).*

| Name of Gene | Function | $H_2O_2$ | Vitamin E |
|---|---|---|---|
| Homo sapiens cation channel, sperm associated 2 (CATSPER2) | Protein binding | 2.844 | −2.838 |
| | Voltage-gated calcium channel activity | | |
| Homo sapiens calsyntenin 3 (CLSTN3) | Calcium ion binding | 2.04 | −2.681 |
| Homo sapiens deleted in liver cancer 1 (DLC1) | Protein binding | 1.68 | −1.55 |
| Homo sapiens ephrin-B3 (EFNB3) | Axon guidance | 1.68 | −1.81 |
| Homo sapiens Fc receptor-like 5 (FCRL5) | Receptor activity association to diseases | 3.84 | −3.3 |
| Homo sapiens growth hormone 1 (GH1) | Cytokine-cytokine receptor interaction | 1.90 | −2.053 |
| Homo sapiens guanine nucleotide binding protein (G protein) | GTPase activity | 1.745 | −1.669 |
| Homo sapiens kinesin family member 26A (KIF26A) | ATP binding | 1.99 | −4.39 |
| Homo sapiens Kruppel-like factor 6 (KLF6) | DNA binding | 1.776 | −1.55 |
| Homo sapiens nuclear receptor subfamily 1, group H, member 4 (NR1H4) | Protein binding | 3.054 | −2.50 |
| Homo sapiens olfactory receptor, family 2, subfamily H, member 1 (OR2H1) | G-protein coupled receptor activity | 2.629 | −2.23 |
| | olfactory receptor activity | | |
| Homo sapiens olfactory receptor, family 3, subfamily A, member 4 pseudogene (OR3A4P) | Odorant receptor | 2.132 | −1.76 |
| Homo sapiens outer dense fiber of sperm tails 4 (ODF4) | Unknown | 2.4745 | −2.015 |
| Homo sapiens phosphate regulating endopeptidase homolog, X-linked (PHEX) | Zinc ion activity | 1.562 | −1.53 |
| | Aminopeptodase activity | | |
| | Metalloendopeptise activity | | |
| Homo sapiens phospholipase C, zeta 1 (PLCZ1) | Calcium ion binding | 3.205 | −2.879 |
| Homo sapiens POU class 4 homeobox 1 (POU4F1) | DNA binding transcription factor activity | 2.968 | −2.618 |
| Homo sapiens RUN domain containing 3B (RUNDC3B), transcript variant 1 | Unknown | 2.647 | −1.962 |
| Homo sapiens scavenger receptor cysteine rich domain containing (5 domains) (SSC5D) | Scavenger receptor activity | 1.518 | −1.862 |
| Homo sapiens small nucleolar RNA, C/D box 88C (SNORD88C) | Unknown | 2.088 | −2.51 |
| Homo sapiens small nucleolar RNA, H/ACA box 30 (SNORA30) | Unknown | 1.82 | −1.92 |
| Homo sapiens transcription factor 20 (AR1) (TCF20) | DNA binding | 1.58 | −1.70 |
| Homo sapiens transmembrane protein 236 (TMEM236) | Unknown | 2.72 | −2.24 |
| Homo sapiens tumor necrosis factor (ligand) superfamily, member 10 (TNFSF10) | Cytokine-cytokine receptor interaction | 1.920 | −2.07 |
| Homo sapiens tumor necrosis factor receptor superfamily, member 19 (TNFRSF19) | Cytokine-cytokine receptor interaction | 2.268 | −1.62 |
| Homo sapiens tumor necrosis factor receptor superfamily, member 4 (TNFRSF4) | Cytokine-cytokine receptor interaction | 2.667 | −2.454 |
| Homo sapiens V-set and transmembrane domain containing 2 like (VSTM2L) | Protein binding | 1.518 | −1.790 |
| Homo sapiens zinc finger and BTB domain containing 22 (ZBTB22) | DNA binding, zinc ion binding | 2.018 | −1.981 |
| Netrin G1 | Protein binding | 2.057 | −1.864 |

Recent studies have reported gingival irritation after at-home bleaching.[13,14] Kirsten and others[16] reported that patients experienced gingival irritation from at-home bleaching both immediately after the procedure and up to 45 days following treatment. On the other hand, no participants reported gingival sensitivity at 6 months posttreatment with 10% carbamide peroxide.[44] They performed observations at 3, 6, and 47 months posttreatment, but we believe that gingival irritation is acute and spontaneous. During at-home bleaching, patients were not monitored by a dentist; thus, gingival irritation could not be established with certainty.

Ghalili and others[15] found that gingival irritation disappeared in 10 minutes using OTC products. Al

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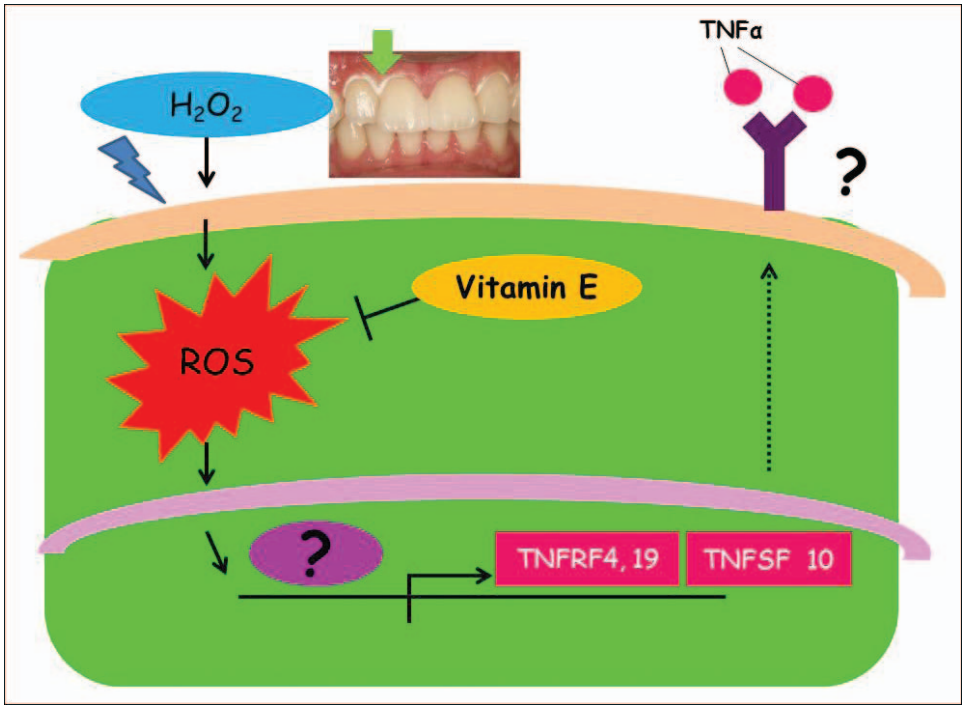


Figure 7. *Inflammatory response pathway from $H_2O_2$ stimulation via TNF-α.*

Shethri and others[12] reported that hypersensitivity and gingival irritation disappeared within two days after in-office bleaching. Previous investigations have shown that 15% of patients reported gingival irritation after in-office bleaching, but it was possible to safely control contact of the bleaching gel with the gingival margin by using light-cured gingival dams.[45] Therefore, it is difficult to compare previously reported gingival irritation results with our observations. The cytotoxic effects produced *in vitro* cannot be immediately applied *in vivo*.

$H_2O_2$ is commonly used in dentistry for tooth bleaching and cleaning, among other purposes. $H_2O_2$ is a form of active oxygen that can cause oxidative damage to fatty acids, biological membranes, and DNA. Therefore, humans are endowed with defenses against $H_2O_2$, and vitamin E is particularly effective at preventing damage by $H_2O_2$.[46,47]

As demonstrated by microarray, the genes induced by $H_2O_2$ stimulation and suppressed by the addition of vitamin E are shown in Table 1. Among the genes listed in Table 1, TNFSF10 belongs to the tumor necrosis factor (TNF)–α ligand superfamily. TNFRSF4 and TNFRSF19 belong to the TNF-α receptor superfamily (TNFRSF). Other authors have examined the gingiva and proinflammatory cytokines. Firat and others[27] examined two types of in-office bleaching agent and one type of at-home bleaching agent to examine periodontal tissue inflammation indices such as gingival index, plaque index, bleeding on probing, and gingival crevicular fluid and found that interleukin (IL)–1"β expression increased with in-office bleaching but that there was no change in the expression of IL-10. However, there have been no reports to date on TNFRF4, TNFRF19, or TNFSF10.

In inflamed tissue, macrophages and other cells of the innate immune system synthesize TNF-α,[48-50] a proinflammatory cytokine, to fight off infection and treat tissue damage. This TNF-α then binds to cell surface receptors and induces the production of other cytokines, triggering and maintaining inflammation. In this study, it is possible that the in-office bleaching agent that came into contact with the gingiva triggered a cellular response through the inflammatory cascade via TNF-α (Figure 7). This cellular response may be suppressed by addition of vitamin E.

When using vitamin E in clinical practice, most dental offices in Japan currently use commercially available vitamin E cream. A recent study has shown that vitamin E added to cells that have been stimulated by $H_2O_2$ restores the cell membrane.[47] Such reports suggest that application of vitamin E to gingiva that has come into contact with an in-office bleaching agent could restore the cell membrane in areas that have been damaged. The results of the present study demonstrate that applying vitamin E

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to the gingiva for protection prior to in-office bleaching is useful for suppressing cytotoxicity in HGFs caused by the in-office bleaching agent. The results also support a method for responding to unpleasant signs arising from tooth whitening. Moreover, while host defense mechanisms may be able to protect the tissues and cells from the toxic effects of $H_2O_2$ in in-office bleaching agents, the effects of longer clinical contact between gingival tissues and $H_2O_2$ remain uncertain.

The three null hypotheses were accepted. HGFs exhibited inflammation after $H_2O_2$ exposure, damaged HGFs expressed proinflammatory cytokines, and damaged HGFs were restored by vitamin E supplementation.

## CONCLUSIONS

1. $H_2O_2$ affected HGFs and induced proinflammatory cytokines.
2. Supplemental vitamin E is able to repair HGFs.
3. Pretreatment with vitamin E supplementation before in-office bleaching can help to reduce gingival irritation.
4. Further studies are necessary to develop a vitamin E cream to protect the gingiva.

### Human Subjects Statement

This study was conducted at the Department of Clinical Cariology and Aesthetic Dentistry, Showa University, School of Dentistry in Tokyo, Japan.

### 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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# Marginal Leakage of Class V Composite Restorations Assessed Using Microcomputed Tomography and Scanning Electron Microscope

C Rengo • C Goracci • G Ametrano
N Chieffi • G Spagnuolo • S Rengo
M Ferrari

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### Clinical Relevance

Microcomputed tomography (micro-CT) provides uninterrupted inspection of the tooth-restoration interface and detection of the deepest leakage point. As a nondestructive technique, micro-CT allows further testing of the specimen to relate sealing ability with other clinically relevant properties of the restorative materials.

*Carlo Rengo, DDS, PhD, Department of Dental Materials and Fixed Prosthodontics, Tuscan School of Dental Medicine, University of Florence and Siena, Siena, Italy

Cecilia Goracci, DDS, PhD, Medical Biotechnologies, University of Florence and Siena, Siena, Italy

Gianluca Ametrano, PhD, Department of Oral and Maxillofacial Sciences, University of Napoli "Federico II," Napoli, Italy

Nicoletta Chieffi, DDS, Department of Dental Materials and Fixed Prosthodontics, University of Florence and Siena, Siena, Italy

Gianrico Sagnuolo, DDS, PhD, Department of Oral and Maxillofacial Sciences, University of Napoli "Federico II," Napoli, Italy

Sandro Rengo, MD, DMD, Department of Oral and Maxillofacial Sciences, University of Napoli "Federico II," Napoli, Italy

Marco Ferrari, MD, DMD, PhD, Department of Dental Materials and Fixed Prosthodontics, University of Florence and Siena, Siena, Italy

*Corresponding author: Viale Bracci, Siena, 53100 Italy; e-mail: carlorengo@alice.it

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## SUMMARY

**Objective: The aim of the study was to compare in Class V composite restorations marginal leakage measurements obtained with microcomputed tomography (micro-CT) and scanning electron microscopy (SEM) observations.**

**Methods: Class V cavities were prepared on 10 human molars and restored using Optibond FL (Kerr, Orange, CA, USA) and Premise Flowable (Kerr). Sealing ability was evaluated by assessing silver-nitrate penetration depth along enamel and dentin margins. Leakage was quantified using a scoring system. Micro-CT analysis provided 502 cross-sectional images for each tooth. Microleakage evaluation was performed first on three cross-sections corresponding to the sections examined by SEM, then on all 502 of the obtained micro-CT images. SEM observations were performed first at 20× magnification, then, if showing a zero score, at 80× magnification. Enamel and dentin microleakage scores assigned to corre-**

**sponding sections through micro-CT and SEM (20×) were compared (Wilcoxon signed-rank test, α=0.05).**

**Results: No statistically significant difference in leakage scores emerged between micro-CT and 20×-magnification SEM. Eight tooth sections that were given a zero score under SEM at 20× magnification showed to be infiltrated at the higher magnification (80×). For five teeth a higher score was assigned following scanning of 502 cross-sections than based on the observation of three sections.**

**Conclusions: Micro-CT presents as a valid, nondestructive *in vitro* method to quantitatively evaluate marginal leakage of adhesive restorations.**

## INTRODUCTION

Marginal leakage is considered a major cause of restoration failure, being responsible for over 50% of replacement procedures.[1,2] Polymerization shrinkage, factors related to adhesive procedures, or inadequate tooth isolation are some of the factors that may cause a failure in adapting the material to the cavity walls,[3] leading to frequently described clinical outcomes such as postoperative sensitivity, secondary caries, marginal tooth discoloration, or pulp pathology.[4-6] Increasing the marginal adaptation of restorations and their durability is one of the main efforts of modern adhesive dentistry.[7] The sealing ability of adhesive materials, which is a property of clinical relevance, can be assessed *in vitro* with microleakage tests.[4,8,9] Most of these methods require the use of a tracer or a dye and cutting the tooth into a series of sections to visualize the extent of staining along the tooth-restoration interface with scanning electron microscopy (SEM), light microscopy, or digital imaging. The depth of dye penetration along the margin can be measured or graded with a scoring system.[4,8,9] A shortcoming of these tests is that they provide a two-dimensional (2D) and semiquantitative evaluation of leakage because interfacial staining is visualized on a limited number of tooth slabs, and some tooth structure is inevitably lost with sectioning.[10] Furthermore, tooth sectioning is a time-consuming and destructive procedure that prevents further testing of the specimen.

Over the last decade the use of x-ray microcomputed tomography (micro-CT) has had considerable development in dental research, finding numerous applications, as described in a recent review.[11] Micro-CT is a nondestructive method that, starting from a series of 2D images, produces a three-dimensional (3D) reconstruction of the observed specimen.[12,13] Lately, the technique has been proposed for the evaluation of marginal leakage in adhesive restorations and pit and fissure sealing.[7,13-19] However, micro-CT has been compared with the conventional section method only in the assessment of marginal leakage at the interface between enamel and pit and fissure sealant.[15] Therefore, the objective of the present study was to comparatively evaluate the two techniques in the evaluation of Class V composite resin restorations. Specifically, the aim was to test the null hypothesis that leakage measurements obtained with micro-CT and with the section method do not differ significantly.

## METHODS AND MATERIALS

### Sample Preparation

Ten sound human molars were stored in 0.5% chloramine-T solution under refrigeration (4°C) and used within one month after extraction. The preparation of Class V cavities was performed on the buccal aspect of each tooth, using a cylindrical diamond bur (305L, Intensiv, Switzerland) mounted on a high-speed handpiece under abundant water cooling. The cavities were round, about 1.5 mm deep and 4 mm in diameter. Cavities were restored using the three-step etch-and-rinse adhesive Optibond FL (Kerr, Orange, CA, USA) and the resin composite Premise Flowable (Kerr). Light curing was performed with a halogen curing device (VIP, Bisco Inc, Schaumburg, IL, USA; 600 mW/cm$^2$). Table 1 reports chemical composition, batch numbers, and instructions for use of the materials. After storage in distilled water at 37°C for one day, teeth were prepared for microleakage testing.[19] The entire tooth surface was covered with two layers of fast-setting nail varnish within 1 mm of the bonded interface. In order to allow the varnish to dry, the specimens were left undisturbed for one day. Then, teeth were immersed in a 50% weight/weight silver-nitrate aqueous solution for 24 hours at room temperature. The silver-impregnated teeth were thoroughly rinsed with distilled water, placed into a photo-developing solution for eight hours (Dental X-Ray Developer, Kodak Co, Rochester, NY, USA), and again abundantly rinsed with water. Teeth were to be viewed first with micro-CT, then under SEM. On each tooth crown three indentations, 1 mm in depth (Figure 1), were made using a water-cooled, slow-speed Isomet saw (Buehler, Lake Bluff, IL, USA). At each indentation a micro-CT scan was performed.

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Table 1: *Chemical Composition, Batch Numbers, and Instructions for Use of the Tested Materials*

| Material | Type | Lot (Batch) No. | Manufacturer | Composition | Application Mode |
|---|---|---|---|---|---|
| Gel Etchant | 37.5% phosphoric acid | 3213200 | Kerr, Orange, CA, USA | 37.5% orthophosphoric acid, silica thickener | Apply for 15 seconds; rinse with water for 15 seconds; gently air dry for a few seconds. |
| Optibond FL | Light-cure total-etch adhesive bonding system | Primer: 3215398 Adhesive: 3215399 | Kerr, Orange, CA, USA | Primer: HEMA, GPDM, PAMM, ethanol, water, photo initiator; Adhesive: TEGDMA, UDMA, GPDM, HEMA, bis-GMA, filler, photo initiator | Apply primer with light scrubbing motion for 15 seconds; gently air dry 5 seconds; apply adhesive; light application of air; light cure for 20 seconds. |
| Premise Flowable | Light-cured, flowable composite | 3433707 Shade: A3 | Kerr, Orange, CA, USA | Prepolymerized filler (PPF), barium glass, silica filler, ethoxylated bisphenol-A-dimethacrylate, TEGDMA, light-cure initiators and stabilizers, organophosphate dispersant | Apply 2-mm-thick layer; light cure for 20 seconds. |

*Abbreviations: HEMA, hydroxyethylmethacrylate; GPDM, glycerol phosphate dimethacrylate; PAMM, phthalic acid monoethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; bis-GMA, bisphenol A glycidyl methacrylate.*

## Micro-CT

Micro-CT scanning was performed using SkyScan 1072 (SkyScanb.v.b.a., Aartselaar, Belgium) with the following settings: 10 W, 100 kV, 98 μA, a 1-mm thick aluminum plate, 15× magnification, 4.9-second exposure time, 0.45° rotation step. The acquisition procedure involved several 2D lateral projections of the specimens during a 180° rotation around the vertical axis. The digital data were processed using a reconstruction software (NRecon V1.4.0; SkyScan).

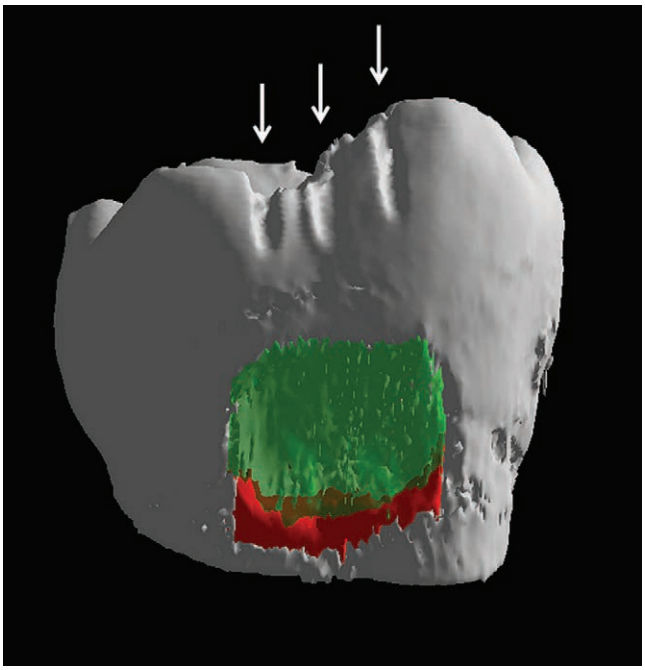

Figure 1. *Image of a restored and stained tooth obtained through 3D reconstruction of micro-CT scans. Silver nitrate infiltration (red) is visible along the cervical margin and wall of the composite resin restoration (partially transparent green). Indentations were made (arrows) to provide landmarks for longitudinal sectioning of the crown into slabs that were then viewed under SEM.*

*3D Reconstruction*—The software also provided axial cross-sections of each tooth with a pixel size of 19.1 × 19.1 μm. The distance between each cross-section was 38 μm. Images of 502 slices per tooth were acquired. After cone-beam reconstruction, the raw data were converted to 16-bit grayscale picture files with a resolution of 1024 × 1024 pixels. The cross-sectional images were imported into a 3D visualization software package (Mimics software; Materialize, Leuven, Belgium).

## Microleakage Assessment

Two experienced investigators (C.R., G.S.) independently evaluated microleakage at the enamel (occlusal) and the dentin (cervical) margins on SEM micrographs and micro-CT images (Figures 2-4).

For microleakage assessment, the exact length of the entire enamel-composite and dentin-composite interfaces as well as the length of the stained interface were measured in pixels using the image analysis software Digimizer V.3.0.0 (MedCalc Software, Mariakerke, Belgium). The percentage of microleakage was thus calculated: (Length of stained interface / Total interfacial length) × 100 and converted to a scoring system[20] from 0 to 4, as presented in Table 2. Scoring differences between the evaluators were discussed until a final score was reached by consensus.

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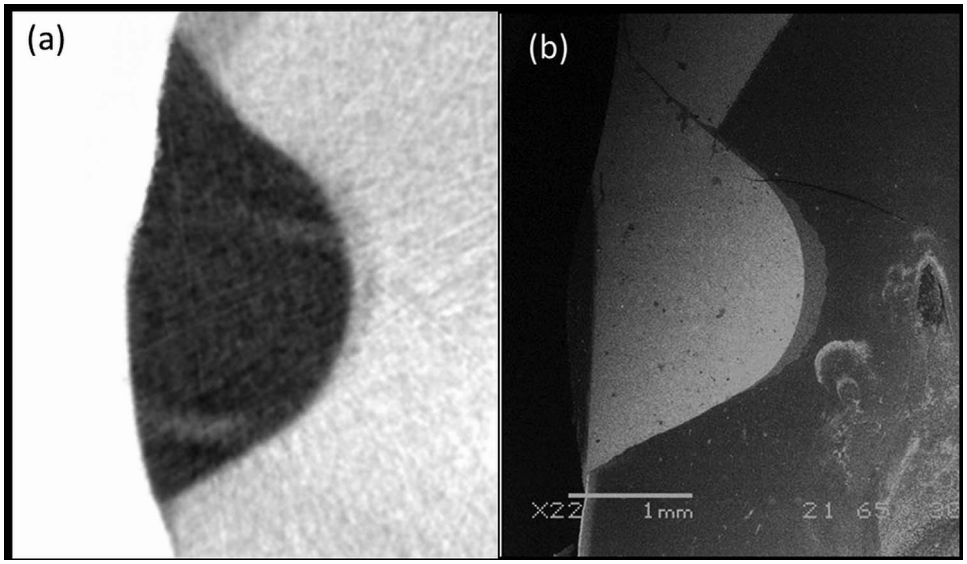


Figure 2. *(a): Micro-CT scan of a tooth showing margins free from dye infiltration and (b): corresponding section observed under SEM.*

### Scanning Electron Microscopy

Starting from the indentations preliminarily cut in the crown as landmarks (Figure 1), each tooth was sectioned longitudinally with the saw in a buccolingual direction to provide four slabs with three specular surfaces. For each pair of slabs offering specular surfaces, only one slab selected at random was processed for SEM observations of interfacial morphology. The slabs were polished with a series of silicon carbide papers of increasing grit from 600 to 1200 under water irrigation and subsequently treated with silica-free 32% phosphoric acid gel (Uni-Etch, Bisco) for 60 seconds. A 120-second deproteinization using 2% sodium hypochlorite solution was followed by dehydration of the specimens in an ascending series of aqueous ethanol solutions to absolute ethanol. The specimens were then dried using hexamethyldisilazane (Carlo Erba, Rodano, Italy). Each specimen was observed under the SEM JSM-6060LV (JEOL, Tokyo, Japan) at different magnifications. Specimens were first viewed at 20×, which was the highest possible magnification in relation to the total field of view of the tooth-restoration interface,[15] and scores were assigned. Specimens that received a zero score were further observed at a higher magnification (80×) to detect any silver nitrate infiltration that was perhaps invisible at 20×.

### Statistical Analysis

Separate statistical analyses were applied to data recorded at the enamel (occlusal) and at the dentin (cervical) site. The Wilcoxon signed-rank test was applied to compare microleakage scores assigned to corresponding sections on micro-CTs and 20×-magnification SEM images. Calculations were handled

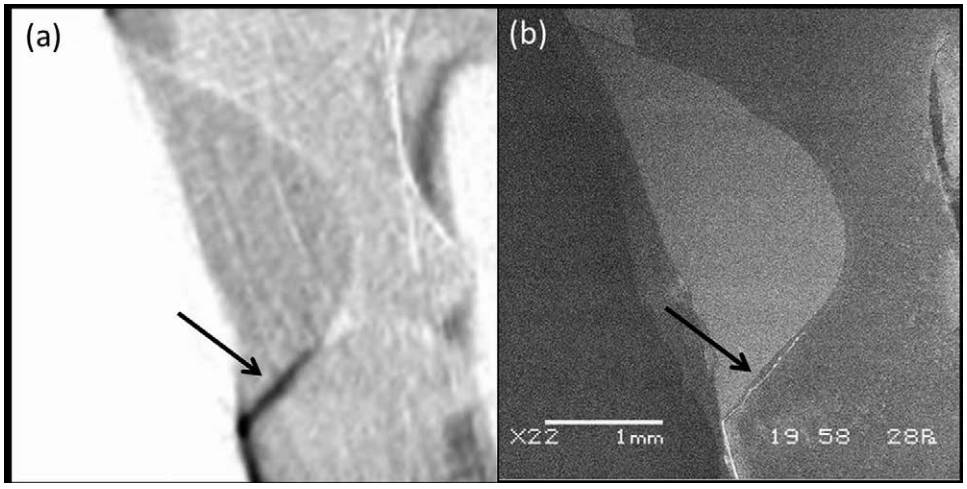


Figure 3. *(a): Micro-CT scan of a tooth showing dye penetration along the dentin-restoration interface and (b): corresponding section observed under SEM.*

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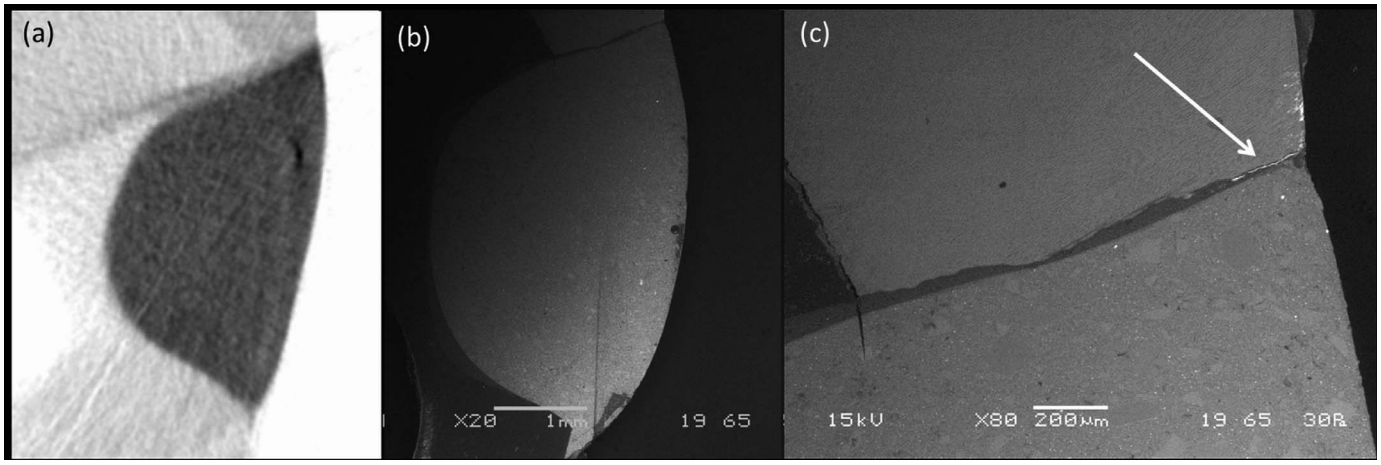


Figure 4. *(a): Micro-CT scan and (b): corresponding SEM micrograph at 20× magnification of a tooth apparently free of infiltration, but (c): showing silver nitrate deposition at the enamel-restoration margin when observed under SEM at 80× magnification.*

by SPSS software, version 18.0 (SPSS Inc, Chicago, IL, USA).

## RESULTS

Microleakage scores assigned to SEM micrographs and to micro-CT images of corresponding tooth sections are presented in Table 3. Silver nitrate infiltration was detected as white stains along the material-tooth interface (Figures 2-4). Agreement between 20×-magnification SEM and micro-CT in assessing leakage was observed for the great majority (93.3%) of the observed specimens, both at the enamel and at the dentin margin (Figure 3). Table 4 reports the descriptive statistics of microleakage scores. The statistical analysis revealed that scores assigned using micro-CT images and 20× SEM micrographs did not differ significantly either at the enamel ($p$=0.5) or at the dentin ($p$=1.0) site (Table 4).

Eight tooth sections that were given a zero score at 20× magnification showed staining along up to one-third of the interface at the higher magnification (score 1, Table 3). For three of these sections such discrepancy occurred both at the enamel and at the dentin site.

With specific regard to micro-CT analysis, for five teeth (No. 1, 4, 6, 9, and 10 of Table 3) a higher score was assigned following scanning of 502 cross-sections than based on the observation of three sections. The difference emerged also in comparison with SEM observations. For most of those teeth (No. 4, 6, 9, 10 of Table 3), scanning of all the cross-sections allowed to spot a score 1 leakage that was indeed undetected when viewing only three sections or SEM images. For one tooth (No. 1 of Table 3), scans of all the cross-sections revealed that the dye had penetrated along the interface deeper than it had appeared from the observation of only three sections or of SEM micrographs.

Table 2: *Score System to Quantify Microleakage at the Enamel-Composite and Dentin-Composite Interfaces*

| SCORE | MICROLEAKAGE |
|---|---|
| 0 | Absence of infiltration |
| 1 | Infiltration up to 1/3 of the interface |
| 2 | Infiltration up to 2/3 of the interface |
| 3 | Infiltration higher that 2/3 of the interface |

## DISCUSSION

Lately, micro-CT has been proposed as a nondestructive method to assess three-dimensionally the sealing ability of adhesive materials.[7,12-16,21] With the purpose of validating the new technique, in the present study the ability of micro-CT to investigate leakage in Class V restorations was comparatively assessed with reference to the commonly used section method.[8] The finding that microleakage measurements produced by micro-CT and 20×-magnification SEM did not differ significantly leads to acceptance of the formulated null hypothesis. Moreover, the high agreement noted between the two methods points out that micro-CT is as reliable as low-magnification SEM in assessing interfacial leakage of adhesive restorations. The 20× magnification was selected as adequate for viewing the entire tooth-restoration interface. However, such magnification in eight of 30 sections failed to reveal leakage that was instead visible when the specimens were observed at 80× (Figure 4c). This is in agreement with Chen & others[15] who advocated

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Table 3: *Microleakage Scores of the Tooth Sections Observed Under SEM and With Micro-CT*

| Tooth No. | Section | SEM 20× | | SEM 80× | | Micro-CT | | Micro-CT 502 Sections (Highest Score Recorded Per Tooth) | |
|---|---|---|---|---|---|---|---|---|---|
| | | Enamel | Dentin | Enamel | Dentin | Enamel | Dentin | Enamel | Dentin |
| 1 | 1 | 0 | 1 | 0 | N/E | 0 | 1 | | (2) |
| | 2 | 0 | 1 | 0 | N/E | 0 | 1 | | |
| | 3 | 0 | 1 | 0 | N/E | 0 | 1 | | |
| 2 | 1 | *0* | *0* | *1* | *1* | 0 | 0 | | |
| | 2 | **3** | **1** | N/E | N/E | **0** | **0** | | |
| | 3 | **4** | 1 | N/E | N/E | **0** | 1 | | |
| 3 | 1 | 0 | 1 | 0 | N/E | 0 | 1 | | |
| | 2 | 0 | 1 | 0 | N/E | 0 | 1 | | |
| | 3 | 0 | 2 | 0 | N/E | 0 | 2 | | |
| 4 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | (1) |
| | 2 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| | 3 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 5 | 1 | 0 | 1 | 0 | N/E | 0 | 1 | | |
| | 2 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| | 3 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 6 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | (1) | |
| | 2 | *0* | *0* | *1* | *1* | 0 | 0 | | |
| | 3 | *0* | 0 | *1* | 0 | 0 | 0 | | |
| 7 | 1 | 0 | 1 | 0 | N/E | 0 | 1 | | |
| | 2 | *0* | 0 | *1* | 0 | 0 | 0 | | |
| | 3 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 8 | 1 | 0 | 1 | 0 | 0 | 0 | 1 | | |
| | 2 | *0* | 1 | *1* | N/E | 0 | 1 | | |
| | 3 | *0* | *0* | *1* | *1* | 0 | 0 | | |
| 9 | 1 | 1 | 0 | N/E | 0 | 1 | 0 | | (1) |
| | 2 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| | 3 | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 10 | 1 | 0 | 1 | 0 | N/E | 0 | 1 | (1) | |
| | 2 | 0 | *0* | 0 | *1* | 0 | 0 | | |
| | 3 | 0 | ***0*** | 0 | *1* | 0 | **1** | | |

*Abbreviation: N/E, not evaluated.*
*[a] The sections that were assigned a 0 score in 20×-magnification micrographs were observed also at 80×. The italic character labels sections that were given a higher score at 80× than at 20× magnifications. The bold character labels sections that were assigned different scores under SEM and with micro-CT. Values in brackets indicate the highest score recorded in the tooth when 502 micro-CT scans were performed.*

Table 4: *Descriptive Statistics of Microleakage Scores Assessed on Three Sections Per Tooth With 20× SEM and Micro-CT[a]*

| Substrate | Technique | n | Median | 25% | 75% |
|---|---|---|---|---|---|
| Enamel | 20× SEM | 30 | 0.000 | 0.000 | 0.000 |
| | Micro-CT | 30 | 0.000 | 0.000 | 0.000 |
| Dentin | 20× SEM | 30 | 0.000 | 0.000 | 1.000 |
| | Micro-CT | 30 | 0.000 | 0.000 | 1.000 |

*[a] No statistically significant difference emerged within each substrate between the two methods.*

that the ability to increase image magnification is an advantage of SEM over micro-CT. Conversely, in micro-CT, magnification depends upon the distance between the specimen and the x-ray source and therefore upon the specimen size: The smaller the distance, the larger the magnification and the greater the detail of the image.[7] On the other hand, micro-CT has the benefit of obtaining an uninterrupted inspection of the interface that enables detection of the deepest leakage point.[7,15] As a matter of fact, in the present study, for half of the teeth, scanning all the micro-CT cross-sections led to a higher leakage score than that determined from

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the assessment of three sections or of SEM specimens. Such occurrence may reasonably be related to the "hit-or-miss" characteristic of the section method[18] which provides only partial information. Particularly in microscopic investigations, the loss of tooth structure with slicing makes the inspection of the tooth-restoration interface inevitably defective. The thickness of the cutting blade itself limits the number of sections that can be obtained. Consequently, restorations of clinically relevant size and shape may yield very few specimens for observations.[18] Moreover, the section method precludes further testing of the specimen and therefore the possibility to relate leakage to other properties of the materials, such as shrinkage, elastic, or mechanical behavior, that might be predictive of the clinical outcome of the restoration.[18]

No conclusive evidence has yet been provided regarding the ideal concentration of silver nitrate and the optimal time of tooth immersion for micro-CT observations. Eden and others[7] stated that a four-hour immersion in a 50% silver nitrate solution allowed accurate and reliable measurements of microleakage in Class II composite resin restorations performed *in vivo*. Later, Chen and others,[21] having compared the outcome of three-hour and four-hour immersion in 50% silver nitrate solution, suggested the shorter time interval, although admitting that repeating the experiment with a larger sample size would be advisable. In the present study specimens were immersed in 50% silver nitrate solution for 24 hours. This is indeed the commonly followed protocol for the observation under SEM, to which the same specimens were to be subsequently subjected. Eden and others[7] reported that after a 14-hour immersion in 50% silver nitrate of primary teeth that had been restored *in vivo*, not only interfacial staining but also silver nitrate scattering in adjacent caries-affected dentin was observed. Differently from the mentioned study,[7] in the present investigation only caries-free teeth were included. This may explain why, despite the longer immersion in the staining solution, no dye diffusion within the dentin underlying the adhesive interface was seen in this study's specimens.

In previous micro-CT investigations of pit and fissure sealants, the occurrence of "background noise" due to x-ray scattering from the tooth surface and sometimes interfering with visualization of sealant and enamel was reported.[15,16,21] No such difficulty was experienced in the present study, possibly in relation to the use of a flowable resin composite, which is a higher density material than a sealant. Moreover, in this study micro-CT images could be colored for a clearer differentiation of the substrates and the stain (Figure 1).

Beside pit and fissure sealants, micro-CT has also been used to assess marginal leakage of Class II restorations in primary molars[7] and standardized cylindrical cavities in permanent molars.[18] In the present study, noncarious Class V adhesive restorations were chosen for testing, given that they have been considered ideal for assessing bonding effectiveness for several reasons.[22] First, preparation of Class V cavities is minimal and their restoration is relatively easy, thereby reducing technique-sensitivity and operator-related variability. Additionally, different from sealants, Class V cavities have margins located both in enamel and in dentin. Moreover, Class V lesions have a relatively small configuration factor; consequently, the mechanical properties of the composite resin used are less influential, and the bonding potential of the adhesive determines the outcome of the restoration to a greater extent.[22]

It should also be considered that in the available studies validating micro-CT against microscopy,[15,18] for experimental purposes, it was decided to prevent the development of a bond to the dental substrate by omitting the etching procedure and the adhesive application. It could be argued, however, that such experimental conditions did not reflect the clinical situation. In contrast, in the present investigation, the bonding agent was applied before composite resin layering, as done in the clinical setting.

As far as the cost of image production is concerned, micro-CT devices are more expensive than the equipment for the conventional section method. However, it should be considered that micro-CT has applications in other research fields beside leakage testing.[18,23-28]

In conclusion, in the present *in vitro* investigation, micro-CT was demonstrated to be similarly adequate to 20×-magnification SEM at assessing marginal leakage of Class V composite resin restorations. The possibility to adjust magnification to gain a more detailed view of the interface was precluded with micro-CT, because the magnification depended upon the distance between the x-ray source and the specimen. On the other hand, SEM allowed leakage evaluation only in the plane of sectioning and with inevitable loss of information at the site of cutting. Conversely, micro-CT enabled a 3D mapping of the tooth-restoration interface and detection of the deepest leakage level. It was plausibly due to the hit-or-miss characteristic of

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the section method that for five of 10 tested teeth, a higher leakage score was determined from the observation of all the micro-CT scans than when only three tooth sections were viewed either with micro-CT or under SEM.

It could be argued that, in order to fully assess the potential of the techniques and whether their outcomes were similar, the whole range of scores should have been covered, whereas a prevalence of low scores was reported in the present investigation. The predominance of low scores, however, was related to the reliability of the tested adhesive.[22,29-31] Also, in previous microleakage studies Optibond FL (Kerr) demonstrated limited infiltration both at the enamel and at the dentin margins.[20,32-34] In the current investigation, Optibond FL (Kerr) was chosen because it has been considered as the gold-standard bonding agent in dental adhesion.[22,29,30]

Whenever a lack of statistically significant between-group differences is observed, the power of the test and the sample size should be considered as a possible alternative explanation for the finding. Nevertheless, it should be pointed out that the sample size used in the present investigation, 30 specimens per group, was chosen with reference to a previous study,[35] where it was reported that such a sample size was adequate to find statistically significant differences in microleakage between two groups, with a power of 80% and a significance level of 0.05.

Finally, it is known that the correlation between the findings of *in vitro* leakage tests and clinical outcomes has recently been debated[36,37] and that the comparison of results among studies is questionable due to the great variability in investigating methods.[22] It is also agreed that, although *in vitro* tests retain the potential to promptly provide some useful information on materials' properties, the ultimate evidence on the sealing ability of adhesive restorations should be gathered through long-term clinical trials.

## CONCLUSIONS

The finding that scores recorded with the two compared techniques did not differ significantly indicated that micro-CT was as reliable as low-magnification SEM in assessing interfacial leakage of adhesive restorations. Being a nondestructive method, micro-CT has the added value of allowing further testing of the specimen for the assessment of other clinically relevant properties of the materials besides sealing ability.

### Conflict of Interest

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

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## ERRATUM

In the article, QD Alomari, et al (*2015*) The Effect of Combining Radiographs and DIAGNOdent With Visual Examination on Detection and Treatment Decisions of Noncavitated Occlusodentinal Caries. Operative Dentistry: May/June 2015, Vol. 40, No. 3, pp. 313-321, the name of the second author, Muawia A. Qudeimat, was misspelled. It should appear as MA Qudeimat.

Operative Dentistry apologizes for the error.

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# Enamel and Dentin Microhardness and Chemical Composition After Experimental Light-activated Bleaching

E Klaric • M Rakic • I Sever
O Milat • M Par • Z Tarle

### Clinical Relevance

With regard to enamel and dentin microhardness and chemical composition, bleaching lights can be safely used. The adverse effects of peroxide concentration and gel acidity could be reversed after a two-week amorphous calcium phosphate and artificial saliva treatment.

## SUMMARY

**Objectives: To evaluate 1) the influence of five bleaching agents (with additional light activation) on enamel and dentin surface microhardness and chemical composition and 2) the remineralizing potential of artificial saliva and amorphous calcium phosphate (ACP).**

**Methods and Materials: The study was conducted on 125 human third molars dissected into quarters for separate enamel and dentin measurements. The bleaching process was performed with 38% and 25% hydrogen peroxide (HP) and 30%, 16%, and 10% carbamide peroxide (CP) gels two times for 15 minutes each time. All bleaching gels were tested alone and in combination with ZOOM2, light-emitting diode (LED), organic LED, and femtosecond laser. A total of 25 bleaching combinations (n=10) were evaluated. Microhardness was measured by a Vickers diamond. Chemical analysis was performed using energy-dispersive X-ray spectroscopy.**

**Results: Bleaching agents used in the absence of light activation caused a significant reduction in enamel and dentin surface microhardness ($p<0.001$), ranging from 8% for 16% CP to 40% for 25% HP. The effects of different light activations were negligible. After two-week treatment with ACP and artificial saliva, maximum deviation from baseline microhardness**

*Eva Klaric, PhD, DMD, School of Dental Medicine, Department of Endodontics and Restorative Dentistry, Zagreb, Croatia

Mario Rakic, PhD, Institute of Physics, Laboratory for Femtosecond Spectroscopy, Zagreb, Croatia

Ivan Sever, MSc, Institute for Tourism, Zagreb, Croatia

Ognjen Milat, PhD, Institute of Physics, Zagreb, Croatia

Matej Par, DMD, private practice, Zagreb, Croatia

Zrinka Tarle, PhD, DMD, professor, School of Dental Medicine, Department of Endodontics and Restorative Dentistry, Zagreb, Croatia

*Corresponding author: Gunduliceva 5, Zagreb, 10000, Croatia; e-mail: eklaric@sfzg.hr

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**was just 3%. Such treatment increased the concentrations of calcium, phosphorus, and fluorine.**

**Conclusions: An increase in peroxide concentration and gel acidity negatively affected microhardness and concentrations of calcium and phosphorus in enamel and dentin. ACP and artificial saliva stimulated the remineralization of hard tissues.**

## INTRODUCTION

Tooth bleaching is considered the easiest and most cost-effective procedure for treating tooth discoloration. To date, studies on the effects of peroxide- and carbamide-based products on dental ultrastructure remain inconclusive. While some authors have reported no adverse effects, others have claimed that the use of such products can be associated with many side effects, which include enamel and dentin surface alterations such as reduction in surface or subsurface microhardness, reduction in calcium and phosphate ratios with loss of organic components from treated tooth surfaces,[1-3] or tooth sensitivity, which could be reduced with remineralizing agents.[4,5] Bleaching agents have an effect on the chemical and morphological structure of hard dental tissues, while hydrogen peroxide (HP) has the ability to produce highly reactive peroxide and superoxide ions. The main reaction of the bleaching process is oxidation. Reduction of microhardness or changes in the chemical structure are primarily the result of the oxidation process in the enamel and dentin organic and inorganic substances.[6] However, it is highly probable that the low pH of the bleaching agents can also lead to chemical and structural changes in dentin, which was demonstrated for internal dental bleaching.[7] The postoperative sensitivity is usually related to the small microscopic enamel defects and subsurface pores and the ability of bleaching agents, typically hydrogen peroxide, to penetrate into the pulp and cause an irritating effect in the pulp cells. This can sometimes lead to mild reversible pulpitis, which can be manifested as tooth hypersensitivity and intermittent spontaneous pain.[8]In the postbleaching period, the presence of saliva, fluorides, or other remineralizing solutions, such as amorphous calcium phosphate (ACP), may provide a balance between the remineralization and demineralization processes.[9] ACP is a direct precursor of biologic apatite in the biomineralization process. It can release calcium and phosphate ions and maintain a supersaturated mineral environment, which can in turn reduce demineralization and improve remineralization of dental hard tissues.[10]

For acceleration and more effective bleaching, different light sources may be used, such as a quartz-tungsten-halogen lamp, plasma lamps, light-emitting diode (LED), and halogen or laser lights.[11] In this study, bleaching was activated by a ZOOM2 (Discus Dental, Culver City, CA, USA) light source and the following experimental light sources: LED (LED Engin Inc, San Jose, CA, USA) with a center wavelength of 405 nm (LED405), white organic LED (OLED) (PPML, Pescara, Italy), and femtosecond laser (Millenia, Spectra-Physics, Santa Clara, CA, USA). OLED was never tested as a light source for tooth bleaching, but it is often used for widespread illumination.[12] Its organic semiconductor is situated between two electrodes. The organic films consist of a hole-injection layer, a hole-transport layer, an emissive layer, and an electron-transport layer. When voltage is applied to the OLED cell, the injected positive and negative charges recombine in the emissive layer and create electroluminescent light. The experimental setup of the femtosecond laser used in this study consisted of a green pump laser at 532 nm (Millenia, Spectra-Physics, Santa Clara, CA, USA) and a Tsunami oscillator (Ti:sapphire laser, Spectra-Physics, Santa Clara, CA, USA) which generated femtosecond pulses. The function of the pump laser was to stimulate the crystal Ti:sapphire in the oscillator, which in turn generated femtosecond pulses of a wavelength in the range of 700-950 nm. In this study, we used the central wavelength of approximately 770 nm. Moreover, light-accelerated bleaching may lead to considerable heat production,[13] which can cause pulp irritation or even necrosis.

Surface and chemical changes after the bleaching treatment may be evaluated by microhardness tests, scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS).[2,3] Surface microhardness measurement is used to determine the mechanical properties of dental hard tissues.[14] SEM and EDS are typically used to characterize the chemical composition after bleaching.

The purposes of this *in vitro* study were to evaluate the effects of peroxide concentration and acidity of bleaching agents and the effects of experimental light sources on the tooth surface and to assess the remineralizing potential of artificial saliva and ACP. The research hypotheses (H) were as follows: H1: Bleaching agents of different concentrations and acidities lead to a significantly different

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Table 1: *Bleaching Agents Evaluated in the Study*

| Product | Manufacturer | LOT Number | Active Bleaching Agent | Percent |
|---|---|---|---|---|
| ZOOM2 | Discus Dental, Culver City, CA, USA | 12 311007 | Hydrogen peroxide | 25 |
| Boost | Ultradent, South Jordan, UT, USA | B66KD | Hydrogen peroxide | 38 |
| Viva Style 30 | Ivoclar Vivadent, Schaan, Liechtenstein | B8HW7 | Carbamide peroxide | 30 |
| Viva Style 16 | Ivoclar Vivadent, Schaan, Liechtenstein | RL3318 | Carbamide peroxide | 16 |
| Viva Style 10 | Ivoclar Vivadent, Schaan, Liechtenstein | ML3364 | Carbamide peroxide | 10 |

decrease in enamel and dentin surface microhardness. H2: Bleaching agents of different concentrations and acidities have different demineralization effects on enamel and dentin. H3: Additional light activation leads to a significant decrease in enamel and dentin surface microhardness. H4: Additional light activation leads to further demineralization of enamel and dentin. H5: Posttreatment with ACP and artificial saliva restores surface microhardness and stimulates remineralization of enamel and dentin.

## METHODS AND MATERIALS

### Specimen Preparation

A total of 125 freshly extracted intact human third molars were cleaned and stored in 1% chloramine solution. The root portions of the teeth were sectioned with a slow-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) approximately 2 mm below the cementoenamel junction and stored in deionized water. Tooth crowns were dissected into quarters and embedded in acrylic resin (AcryFix Kit; Struers, Balerrup, Denmark). For enamel measurements, specimen vestibular surfaces were polished using water-cooled carborundum discs (Water Proof Silicon Carbide Paper, 4000 grit; Buehler, Dusseldorf, Germany) and 1.0-μm, 0.3-μm, and 0.05-μm micropolish powder (Buehler) to expose a standardized area of 3 × 3 mm. After the crowns were dissected, lingual parts of the crowns with inner exposed dentin were used for dentin measurements. Dentin samples were polished in the same way as enamel samples.

### Bleaching Procedure

The tooth surface was bleached with either 25% HP, 38% HP, 10% carbamide peroxide (CP), 16% CP, or 30% CP gel two times for 15 minutes each time (Table 1). Bleaching gel was applied in a 2-mm-thick layer and removed after 15 minutes using a Heidemann spatula. The tooth surface was rinsed with deionized water and dried with compressed air and cotton tissues before applying another layer of bleaching gel to the surface. Bleaching was performed in two modes: with bleaching gel alone (ie, without the light activation) and with bleaching gel activated by one of the following light sources: ZOOM2 light source, LED405, OLED, and femtosecond laser (Table 2).

A total of 500 specimens were divided into two main (enamel/dentin) groups and 25 subgroups (ie, treatment groups) defined by 25 different bleaching combinations, differing in bleaching gel and/or light source treatments (Table 3). The specimens were randomly assigned to each treatment group (n=10) separately for enamel and dentin measurements. Before randomization, they were aligned and appointed a unique identification number. Simple randomization of 250 specimens into 25 treatment groups was done in the procedure PROC PLAN of the SAS System software (SAS Institute Inc, Cary, North Carolina, USA). During the bleaching, each specimen was placed on a cotton pellet soaked in artificial saliva. Afterwards, enamel and dentin surfaces were cleaned and dried. ACP gel (ACP Relief, Discus Dental, Culver City, CA, USA) was applied to the surface of half of the specimens for 20 minutes every day for 14 days. After each ACP gel application, teeth

Table 2: *Light Sources Evaluated in the Study*

| Product | Manufacturer | Type of Light | Power, mW/cm$^2$ |
|---|---|---|---|
| ZOOM2 | Discus Dental, Culver City, CA, USA | Mercury metal halide light λ 350-400 nm | 2.000 |
| LED | LED Engin Inc, San Jose, CA, USA | LED λ 405 nm | 400 |
| PPML OLED KIT engineering prototype | PPML, Pescara, Italy | OLED λ 400-760 nm | 200 |
| Millenia | Spectra-Physics, Santa Clara, CA, USA | Femtosecond laser λ 770 nm | 800 |

*Abbreviations: LED, light-emitting diode; OLED, white organic light-emitting diode.*

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Table 3: *Treatment Group Assignments with Corresponding Group Labels*

| Bleaching Gel/Light Source Tested | Light Source (Application Time: 2 × 15 min) | | | | |
|---|---|---|---|---|---|
| | None (Control) | ZOOM2 | LED405 | OLED | Femtosecond Laser |
| Bleaching gel (application time: 2x15 minutes) | | | | | |
| 25% HP | 25HPnone | 25HPZOOM | 25HPLED | 25HPOLED | 25HPfemt |
| 38% HP | 38HPnone | 38HPZOOM | 38HPLED | 38HPOLED | 38HPfemt |
| 10% CP | 10CPnone | 10CPZOOM | 10CPLED | 10CPOLED | 10CPfemt |
| 16% CP | 16CPnone | 16CPZOOM | 16CPLED | 16CPOLED | 16CPfemt |
| 30% CP | 30CPnone | 30CPZOOM | 30CPLED | 30CPOLED | 30CPfemt |

*Abbreviations: CP, carbamide peroxide; HP, hydrogen peroxide.*

were soaked in artificial saliva (to protect the specimens from dehydration) and stored at 37°C in order to simulate intraoral conditions (Cultura Incubator, Ivoclar Vivadent, Schaan, Liechtenstein). The artificial saliva was replaced daily. The other half of the specimens were kept in deionized water for 14 days, which was also replaced daily.

The pH of the bleaching gel was measured using a pH/Ion meter (Pinnacle 555 pH/Ion meter, Corning, Tewksbury, MA, USA), which was initially calibrated. Each bleaching gel was placed in 30-mL graduated plastic cups. The pH electrode was immersed inside the gel to allow uniform contact with the electrode tip. The bleaching gel was in contact with the pH electrode for 20 minutes at room temperature (24°C). The electrode was thoroughly washed between samples. ZOOM2 gel had a pH of 3.20 and BOOST gel had a pH of 6.75, while all VivaStyle gels had a pH of 7.00.

For this experiment 500 mL of Glandosane spray (Fresenius KABI, Cheshire, UK) was used. Since its pH value is 5.23 (Pinnacle 555 pH/Ion meter, Corning), it was mixed with 67.3 g of 1% NaOH solution (Kemika, Zagreb, Croatia) using the magnetic stirrer with a hot plate (Cole Parmer, East Bunker Court, Vernon Hills, USA) to obtain a neutral pH of 7.0.

### Microhardness and EDS Measurements

Microhardness was determined using a Vickers diamond (Leitz Miniload2 Microhardness Tester, Leitz, Germany) at a load of 100*g* applied for 10 seconds. The Vickers hardness indentations were performed on the central area of each specimen, with a distance of 100 μm between them, and were averaged.

Three specimens in each group were randomly selected and analyzed using EDS (JSM 7000F, JEOL, Japan). The specimens were dried and fixed on aluminum stubs. Elemental analysis and precise chemical characterization of a sample were performed at 10,000 keV.

Measurements were performed just before and immediately after the bleaching and after two-week storage in artificial saliva and ACP or deionized water.

### Statistical Analysis

Analysis was performed separately for enamel and dentin. Descriptive statistics were presented graphically in the form of medians and interquartile ranges (IQs). Microhardness data were positively skewed and were thus log-transformed prior to further analysis. A mixed effects analysis of variance model was applied. Random effect parameters represented general variability among specimens due to the heterogeneous characteristics of teeth related to their color, composition, size, and shape. Different treatment levels were specified as fixed effects. Covariance structure among repeated measurements on the same specimen was further modeled by selecting appropriate residual covariance structure based on minimizing the information criterion (AIC). A normal probability plot indicated that model residuals were approximately normally distributed.

Log-transformed data were transformed back to the original scale for reporting, so differences between treatment groups were expressed as ratios of geometric means, interpreted as a percent change in surface microhardness. The significance level was set at 0.05. *p*-values were adjusted for multiple comparisons according to the Bonferroni-Holm method. Analysis was performed in SAS 8.2 (SAS Institute Inc, Cary, North Carolina, USA).

## RESULTS

### Microhardness Analysis

Median baseline enamel microhardness was 37.81 (IQ range: 37.01-38.92), and dentin microhardness

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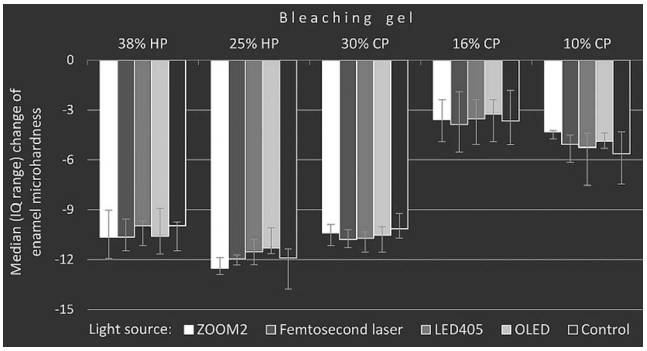


Figure 1. *Effects of different bleaching treatments on enamel surface microhardness.*

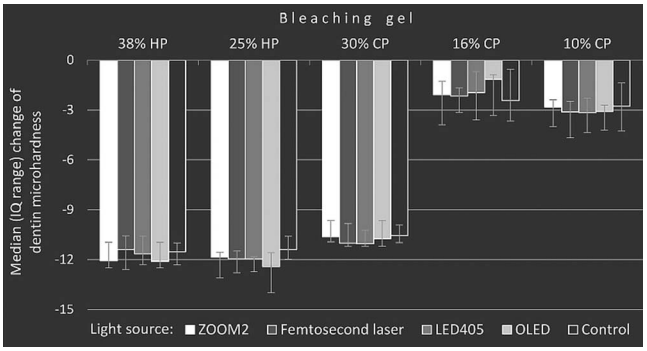


Figure 2. *Effects of different bleaching treatments on dentin surface microhardness.*

measured 29.70 (IQ range: 28.55-30.29). As suggested in Figures 1 and 2, the type of light source or light activation itself was not a significant factor for microhardness change during bleaching. The effects of 25HPZOOM, 25HPLED, 25HPOLED, and 25HPfemt were on average similar (not significantly different, $p>0.05$) to those of the 25HPnone treatment group. The same was observed for comparisons of 38HPZOOM, 38HPLED, 38HPOLED, and 38HPfemt with 38HPnone, as well as for comparisons of 10CPZOOM, 10CPLED, 10CPOLED, and 10CPfemt with 10CPnone; for comparisons of 16CPZOOM, 16CPLED, 16CPOLED, and 16CPfemt with 16CPnone; and for comparisons of 30CPZOOM, 30CPLED, 30CPOLED, and 30CPfemt with 30CPnone. Therefore, the analysis focused on the effects of different bleaching gels, which are described in more detail below.

A significant microhardness decrease was observed during applications of all bleaching gel types (Table 4; $p<0.001$ in all bleaching gel groups—25HPnone, 38HPnone, 10CPnone, 16CPnone, and 30CPnone). Enamel surface microhardness was most severely affected during bleaching with 25% HP, which on average caused a 31% decrease in enamel microhardness (corresponding to a geometric mean ratio of 0.69). Treatment with 30% CP and 38% HP resulted in 28% and 27% decreases, respectively. On the other hand, adverse effects of 16% CP and 10% CP were substantially smaller, with an average decrease of 9% and 15%, respectively. A similar pattern was observed with dentin. Bleaching treatment with 25% HP had the largest detrimental effect, demonstrating an average 40% decrease in dentin surface microhardness. Somewhat smaller decreases of 38% and 36% were observed during bleaching with 38% HP and 30% CP, respectively. Again, bleaching with 16% CP and 10% CP was less detrimental, resulting in 8% and 11% average decreases in dentin microhardness, respectively.

The restoration of surface microhardness was not stimulated by two-week storage in deionized water (Table 4). Microhardness remained at a similar level, on average deviating less than 6% and 4% from the enamel and dentin microhardness values recorded immediately after the bleaching process. However, using artificial saliva and ACP as a postbleaching treatment medium restored the enamel and dentin microhardness. After two-week storage in this medium, enamel and dentin microhardness values were significantly higher ($p<0.001$ in all bleaching gel groups—25HPnone, 38HPnone, 10CPnone, 16CPnone, and 30CPnone) than immediately after the bleaching and approximately equal to the baseline values for all bleaching gels tested. Although differences between baseline microhardness values were still significant for some bleaching treatments, the maximum average drop from baseline values was just 3% in enamel and 1% in dentin.

## EDS Analysis

Various structural chemical elements were detected in enamel and dentin—calcium (Ca), carbon (C), fluorine (F), magnesium (Mg), oxygen (O), phosphorus (P), and sodium (Na). Detected concentrations of Mg and Na were generally low (median values below 1.0). Summary statistics for concentrations of other elements are presented in Figures 3 and 4. Bleaching had an almost negligible effect on concentrations of C, O, and F, regardless of the bleaching gel applied. However, different effects of bleaching gels were apparent for concentrations of Ca and P. While concentrations of these elements remained at approximately the same level after bleaching with 10% CP and 16% CP, gels containing higher concentrations of peroxide—30% CP, 25% HP, and 38% HP—noticeably reduced the concentrations of Ca and P in

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Table 4: *Microhardness Summary Statistics for Different Treatments of Enamel and Dentin*[a]

| Bleaching Gel | Treatment | | | |
|---|---|---|---|---|
| | Baseline G. Mean (95% CI) | Bleaching G. Mean (95% CI) | Deionized Water G. Mean (95% CI) | Artificial Saliva + ACP G. Mean (95% CI) |
| Enamel | | | | |
| 38% HP | 38.0 (37.73-38.28) A | 27.7 (27.46-28.03) B | 26.9 (26.55-27.21) C | 37.5 (36.82-38.29) A |
| 25% HP | 38.1 (37.76-38.49) A | 26.2 (26.03-26.43) B | 26.4 (26.08-26.69) B | 37.0 (36.57-37.42) C |
| 30% CP | 38.2 (37.93-38.51) A | 27.7 (27.57-27.85) B | 27.6 (27.03-28.09) B | 38.1 (37.52-38.77) A |
| 16% CP | 37.9 (37.63-38.21) A | 34.3 (33.91-34.79) B | 34.5 (34.05-34.97) B | 37.2 (36.74-37.71) C |
| 10% CP | 36.8 (36.50-37.15) A | 31.4 (31.07-31.77) B | 29.6 (29.15-29.97) C | 35.6 (35.36-35.80) D |
| Dentin | | | | |
| 38% HP | 30.9 (30.52-31.19) A | 19.0 (18.89-19.19) B | 18.9 (18.80-19.04) B | 30.6 (30.26-30.94) A |
| 25% HP | 30.3 (30.01-30.58) A | 18.2 (18.01-18.34) B | 18.5 (18.24-18.67) C | 29.9 (29.63-30.17) D |
| 30% CP | 29.2 (28.97-29.46) A | 18.7 (18.56-18.88) B | 19.1 (18.94-19.33) C | 29.2 (28.74-29.56) A |
| 16% CP | 29.2 (28.97-29.39) A | 26.9 (26.48-27.33) B | 27.9 (27.60-28.28) C | 29.3 (28.81-29.70) A |
| 10% CP | 28.2 (27.94-28.51) A | 25.0 (24.79-25.25) B | 24.1 (23.85-24.36) C | 28.1 (27.65-28.43) A |

*Abbreviations: ACP, amorphous calcium phosphate; CI, confidence interval; CP, carbamide peroxide; HP, hydrogen peroxide.*
[a] *Geometric means and 95% confidence intervals presented in the table; different letters in each row represent statistically significant differences among treatments.*

enamel and dentin. Performances within these two groups were similar, regardless of the specific gel applied. Therefore, only the effects of bleaching with 10% CP and 38% HP are presented in Figures 3 and 4.

Artificial saliva and ACP treatment clearly affected enamel and dentin concentrations of C, O, F, Ca, and P. After two-week storage in this medium, the concentration of C in enamel increased, and its concentration in dentin decreased. The same treatment increased the concentrations of Ca, F, and P and reduced the concentration of O in both enamel and dentin.

## DISCUSSION

Light-activated bleaching enhances the decomposition of HP for improved bleaching results. When the bleaching agent is induced by light, some amount of its energy is absorbed, and the result is heat production, which can be observed as a possible side effect during this type of tooth bleaching. Light sources may have photothermal effects, which are then associated with the chemical effect of the bleaching materials.[11] Although the light sources used in this study can lead to temperature rise[13] or cause possible pulp damage or tooth hypersensitivity,[4] this study demonstrated that the tested light sources do not stimulate a reduction in enamel and dentin surface microhardness or a change in their chemical composition. Araujo and others[15] assessed microhardness change after application of 35% HP in combination with LED, halogen lamps, and an argon laser and concluded that the application of bleaching gel leads to a decrease in microhardness after 14 days, while the light activation does not affect this value. Gomes and others[16] demonstrated that the use of 35% HP gel alone leads to a

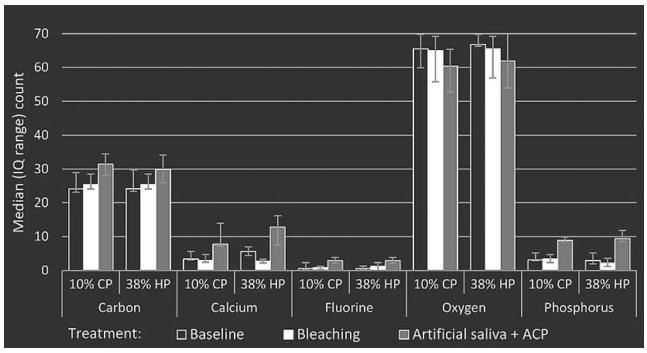


Figure 3. *Effects of bleaching (10% CP and 38% HP presented) and artificial saliva/ACP treatments on enamel concentrations of carbon, calcium, fluorine, oxygen, and phosphorus.*

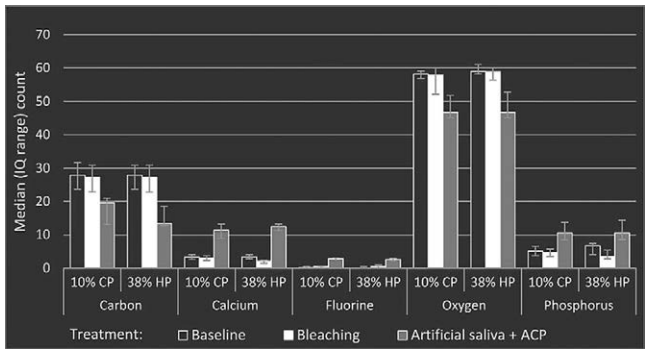


Figure 4. *Effects of bleaching (10% CP and 38% HP presented) and artificial saliva/ACP treatments on dentin concentrations of carbon, calcium, fluorine, oxygen, and phosphorus.*

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microhardness reduction of 15%, while light activation by plasma arc and LED light source did not contribute significantly to a reduction of microhardness. Marcondes and others[17] have shown that HP gel combined with an LED light source or Nd:YAG laser causes no greater decrease in enamel surface microhardness, while Zhang and others[18] indicated that LED and diode laser alone do not affect enamel microhardness. Majeed and others[19] showed that 38% HP with and without light activation led to a similar reduction of dentin microhardness. The data for OLED and femtosecond laser are not available in the existing literature, but the general findings of this study are in line with the current research.

On the other hand, bleaching gels affect the chemical and morphological structure as a result of oxidation of the organic and inorganic substances in enamel and dentin.[20] This indicates that both enamel and dentin are permeable to reactive oxygen species released by decomposition of HP and CP. A number of studies[1,15,19,21] assessed the relationship between concentrations of HP or CP and the changes in enamel and dentin microhardness. In this study, HP and CP gels showed significant and detrimental effects on enamel and dentin surface microhardness. The bleaching treatments containing higher concentrations of peroxide—25% HP, 38% HP, and 30% CP—demonstrated a significantly greater reduction in enamel and dentin microhardness than did 10% CP and 16% CP. Furthermore, application of 25% HP gel, which had a lower pH value (pH=3.20) than 38% HP (pH=6.75) and all of the CP gels (pH=7.00), led to the largest decrease in surface microhardness. The acid pH measured for 25% HP was below the critical level for enamel, which is between 4.5 and 5.5 and can cause hard tissue demineralization. Demineralization could be attributed to the low concentrations of calcium and phosphate ions and high concentrations of sodium and chloride ions in bleaching gels, which can cause undersaturation with respect to hydroxyapatite.[2] Thirty-eight percent HP and 30% CP bleaching gels had pHs over this critical value, but they also caused hard tissue demineralization. This suggests that the acidic properties of the bleaching agent also contribute to the change in the mineral content of hard dental tissues. High acidity may cause a further decrease of microhardness.[22,23] Sun and others[24] demonstrated that neutral 30% HP had the same efficiency in tooth bleaching and caused fewer deleterious effects on enamel than did acidic 30% HP. Therefore, it can be concluded that bleaching agents with greater concentrations and acidity are potentially more harmful for the surface microhardness and can produce more alterations of the enamel and dentin structure and reduce their microhardness.

The buffering potential and remineralization effect of saliva are well documented.[9,10,21] Furthermore, ACP gels alone or in combination with casein phosphopeptide or nano-carbonate apatite are known factors for the remineralization of hard dental tissues.[25-28] Our results indicate that postbleaching treatment with artificial saliva and ACP significantly increases and restores enamel and dentin surface microhardness, which was not demonstrated for postbleaching treatment in deionized water. This result is in accordance with the findings of De Abreu and others.[29] Two-week storage in artificial saliva with everyday ACP treatment showed that both agents have potential remineralization effects and cause possible mineral precipitation of calcium and phosphate ions. In addition, this treatment can improve the surface microhardness of the hard dental tissues. Therefore, such treatment should be used after the bleaching process. Separating the remineralization effects of artificial saliva alone from those of artificial saliva in combination with ACP is one interesting venue for future research.

The ACP Relief gel used in this study contained sodium fluoride. da Costa and others[30] found no microhardness recovery after treatment with sodium fluoride gel or nanohydroxyapatite materials, indicating that the observed effect of microhardness recovery recorded in this study could be attributable to the ACP activity. On the other hand, Borges and others[22] suggested that application of fluoride gel in combination with calcium and artificial saliva enhances the microhardness of bleached enamel. Lewinstein and others[31] reported a significant reduction in enamel microhardness after the bleaching, which recovered after the application of 0.05% fluoride solution. Furthermore, Chen and others[9] noted that fluoridated bleaching agents produced less demineralization of surface morphology and microhardness, while the addition of fluoride did not impede the whitening effect. Therefore, HP in combination with hydroxyapatite, fluoride, and calcium could reduce microhardness loss and tooth sensitivity.[32-35]

The bleaching process, regardless of whether or not it was light activated, led to relevant changes in the chemical composition of enamel and dentin. A greater decrease in the concentrations of Ca and P was observed during treatment with gels containing

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higher concentrations of peroxide—30% CP, 25% HP, and 38% HP, in comparison to 10% CP and 16% CP. In addition, a possible decrease in ion concentrations can be attributed to the lower pH values of bleaching gels such as 25% HP (pH=3.20) and 38% HP (pH=6.75). Acidic properties of the bleaching gels can lead to a reduction in the mineral content of enamel and dentin. While Amaral and others[36] concluded that bleaching gels do not alter concentrations of Ca and P on the enamel surface, Rotstein and others[37] found that the ratio of Ca to P significantly decreased after using 30% HP and 10% CP. McCracken and Haywood[38] noted that six-hour exposure to CP can result in an average Ca loss of 1.06 $\mu g/mm^2$. However, this loss is clinically negligible. For comparison, one glass of cola drink causes a Ca loss of about 1 $\mu g/mm^2$. Cakir and others[39] suggested that 10%, 20%, and 35% CP decrease the levels of Ca, P, and K and increase the amount of Na, F, and O. Such transient loss of minerals is considered reversible after a few days in artificial saliva, fluoride-containing gels, or other means of remineralization. Gjorgievska and Nicholson[40] and others have shown that application of remineralizing paste after bleaching with 16% CP leads to an increase in the quantity of Ca and F. This study showed that 14 days of artificial saliva and daily ACP treatment increased the concentrations of Ca, P, and F ions. The precipitation of salivary components, such as calcium and phosphates, either from artificial saliva or from remineralizing preparation could contribute to a significant recovery of hard dental tissue after bleaching.[41,42] The observed effect can be seen as a potential caries-protective action.

This study has some limitations. Under these clinical conditions, the tooth surface is protected by the saliva and enamel pellicle, so after bleaching the demineralized enamel and dentin can undergo possible remineralization and recalcification. For a simulation of clinical conditions, the samples were stored in artificial saliva before and for two weeks after the bleaching treatment. However, variations in salivary composition throughout the day cannot be simulated in an experimental setup.[25,43] Nevertheless, this *in vitro* study has demonstrated that artificial saliva and ACP treatment can improve surface microhardness, microstructure, and chemical composition as well as enhance the remineralization of the hard dental tissues by providing calcium and phosphate ions. For full and prompt recovery of enamel and dentin, such treatment is strongly recommended.

## CONCLUSION

The microhardness and chemical composition of the hard dental tissues were adversely affected by the concentration of peroxide in the bleaching agent and its acidity while applied experimental light sources had a negligible effect. Postoperative treatment with ACP and artificial saliva restored microhardness and positively affected concentrations of Ca, F, and P in enamel and dentin. With regard to enamel and dentin microhardness and chemical composition, the bleaching lights could be safely used.

### Acknowledgement

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### Human Subjects Statement

The use of extracted human teeth was approved by the Research Ethics Committee of the School of Dental Medicine, University of Zagreb, Croatia. This study was conducted at the School of Dental Medicine, Department of Endodontics and Restorative Dentistry, in Gunduliceva 5 in Zagreb, Croatia.

### Conflict of Interest

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

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# Visual Acuity and Experience with Magnification Devices in Swiss Dental Practices

M Eichenberger • P Perrin • S T Ramseyer
A Lussi

### Clinical Relevance

Many dentists are not aware of their visual handicaps. Magnification devices should be used to compensate for individual or age-related visual deficiencies.


## SUMMARY

**Objectives: The aims of the present study in Swiss dental practices were 1) to provide an update on the prevalence of different magnification devices, 2) to examine the relationship between self-assessed and objectively measured visual acuity, and 3) to evaluate the visual performance of dentists in the individually optimized clinical situation of their respective practices.**

**Methods and Materials: Sixty-nine dentists from 40 randomly selected private practices (n=20, <40 years; n=49, ≥40 years) participated in the study. A questionnaire was provided to evaluate the self-assessed near visual acuity and the experience with magnification devices. The objective near visual acuity was measured under standardized conditions on a negatoscope. The clinical situation, including the use of habitual optical aids, was evaluated with visual tests on a phantom head.**

**Results: A total of 64% of the dentists owned a dental loupe: 45% Galilean loupes, 16% Keplerian loupes, and 3% single lens loupes. In total, 19% of the questioned dentists owned a microscope in addition to the loupes. The correlation between the self-assessed and the objective visual performance of the dentists was weak (Spearman rank correlation coefficient=0.25). In the habitual clinical situation, magnification devices ($p$=0.03) and the dentist's age ($p$=0.0012) had a significant influence on the visual performance.**

**Conclusions: Many dentists were not aware of their visual handicaps. Optical aids such as loupes or microscopes should be used early enough to compensate for individual or age-related visual deficiencies.**


Martina Eichenberger, Dr Med Dent, University of Bern, Department of Preventive, Restorative and Pediatric Dentistry, Bern, Switzerland

*Philippe Perrin, Dr Med Dent, University of Bern, Department of Preventive, Restorative and Pediatric Dentistry, Bern, Switzerland

Simon T Ramseyer, Dr Med Dent, University of Bern, Department of Preventive, Restorative and Pediatric Dentistry, Bern, Switzerland

Adrian Lussi, Dr Med Dent, Dipl Chem, professor, University of Bern, Department of Preventive, Restorative and Pediatric Dentistry, Bern, Switzerland

*Corresponding author: Freiburgstrasse 7, Bern, 3010, Switzerland; e-mail: philippe.perrin@zmk.unibe.ch

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## INTRODUCTION

Magnification devices are used in many medical professions[1,2] as well as in dentistry.[3-5] Ergonomic benefits,[6-8] better diagnostic capability,[9] and enhanced quality of therapy[10] are potential benefits of the use of magnification devices. Although the body of scientific evidence supporting the impact of magnification on the dentist's performance is weak,[11-16] it seems obvious that good vision is crucial in dentistry, as it is in other medical professions in which vision is important. The influence of presbyopia on the dentist's visual performance is discussed in the literature,[17-19] but scientific studies with objective and discriminatory near vision tests are rare.[20-23] The results of miniaturized visual tests, validated for discriminatory testing of near visual acuity at a dental working distance, showed large variability in the natural vision of dentists (independent of their age) and an important deficiency due to presbyopia for dentists who are ≥40 years old.[21,22] Visual deficiencies could easily be compensated for with magnification devices.[21,22] These studies were performed in the standardized conditions of a dental school. The findings of highly variable natural vision and age-related visual deficiencies were corroborated in a group of dentists in their respective private practices.[23] It is unknown if private practitioners are aware of any existing visual deficiencies and if the individually optimized setting of their own private practices can support good visual performance over the course of the dentist's professional life.

The aims of the present study, which was performed in Swiss dental practices, were 1) to assess the prevalence of different magnification devices, 2) to examine the relationship between self-assessed and objectively measured visual acuity, and 3) to evaluate the visual performance of dentists in the individually optimized conditions of their respective practices.

## METHODS AND MATERIALS

Forty private practices were randomly selected from the register of the local dental association in two regions of Switzerland. Multiple dentists from the same practice were included in the study. Dentists were included in the study if they were part of an active private practice and were less than 65 years old. A total of 69 dentists from 40 practices participated in the study (n=20, <40 years; n=49, ≥40 years).

A questionnaire was designed to investigate the dentists' experience with magnification devices and the motivation to use them (Figure 1). A self-assessment of the visual performance as dental professionals was evaluated on a modified visual analog scale (VAS score '0' = very poor; VAS score '5' = very good) (Figure 1).

The visual acuity of the test persons was measured in their respective private practices. Miniaturized visual tests with E-optotypes, as described and validated in previous studies,[21,22] were used. The distance between the three bars of the smallest E-optotype that could be read corresponded to the smallest detectable dimension.[24] Tests with a range from 0.01 to 0.12 mm were used. The smallest recognized dimension was registered (eg, 0.04 mm) and converted into the reciprocal value (eg, 25 $mm^{-1}$) to obtain a positive association between the value and visual acuity.[22] For the standardized near vision test, the transparent tests were fixed behind a fenestrated piece of black cardboard and mounted on a negatoscope. The natural visual performance was measured at a distance of 300 mm. These measurements were compared with the self-assessment in the questionnaire.

For the clinical visual test in the dentist's respective patient setting, the visual tests were fixed in distal cavities of maxillary molars and premolars of a phantom head, as described by Eichenberger and others[22] (Figure 2). The head was positioned on the patient's dental chair (Figure 3), and the dentists could individually choose the working distance, the light source, and the use of magnification aids, according to the usual setting of their daily work. The influence of age (<40 years or ≥40 years) and magnification device (Galilean or Keplerian loupes) on this visual performance was statistically analyzed. Microscopes were not included in the analysis, as the dentists indicated that they used microscopes only in certain fields of dentistry (eg, for endodontics).

The wearing of individual eyeglasses was allowed during the entire testing procedure. The smallest line of the visual tests that could be read, the eye-object distance, and the respective magnification devices that were used were recorded.

For statistical analysis, the software program R version 2.14.1 (http://www.r-project.org/) was used. The significance level was set at $\alpha = 0.05$. Descriptive statistics were carried out to determine medians, ranges, and standard deviations for the two age groups under standardized visual test conditions and under the optimized individual clinical situation. As data were metric, a nonparametric two-way analysis

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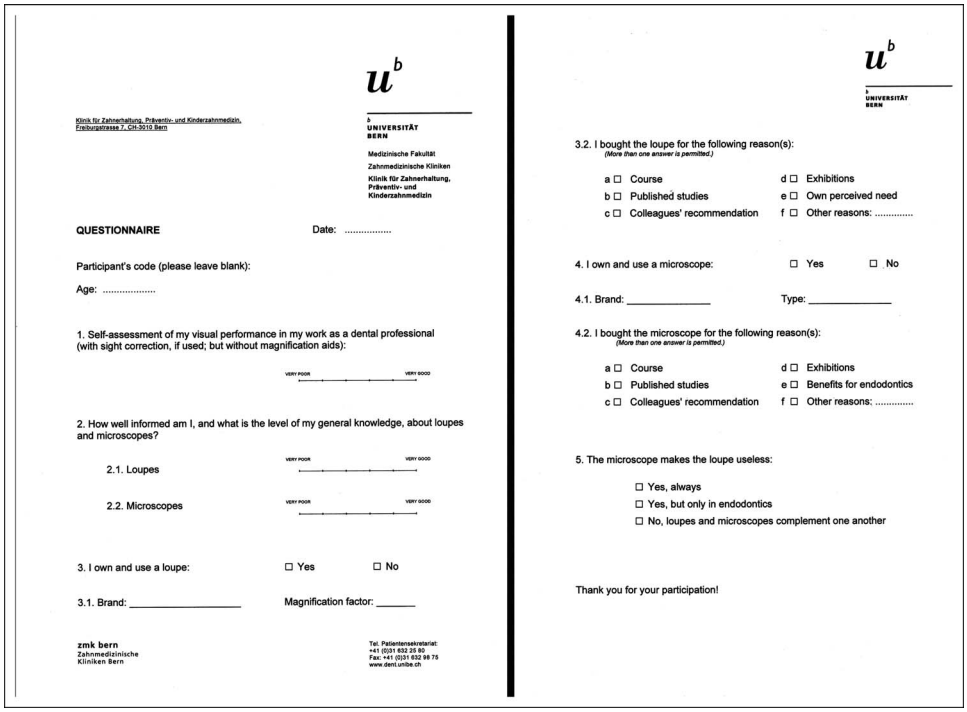

Klinik für Zahnerhaltung, Präventiv- und Kinderzahnmedizin,
Freiburgstrasse 7, CH-3010 Bern

UNIVERSITÄT BERN

Medizinische Fakultät
Zahnmedizinische Kliniken
Klinik für Zahnerhaltung, Präventiv- und Kinderzahnmedizin

**QUESTIONNAIRE** Date: .....

Participant's code (please leave blank):

Age: .....

1. Self-assessment of my visual performance in my work as a dental professional (with sight correction, if used; but without magnification aids):

VERY POOR — VERY GOOD

2. How well informed am I, and what is the level of my general knowledge, about loupes and microscopes?

2.1. Loupes (VERY POOR — VERY GOOD)

2.2. Microscopes (VERY POOR — VERY GOOD)

3. I own and use a loupe: ☐ Yes ☐ No

3.1. Brand: _____ Magnification factor: _____

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3.2. I bought the loupe for the following reason(s):
*(More than one answer is permitted.)*

a ☐ Course
b ☐ Published studies
c ☐ Colleagues' recommendation
d ☐ Exhibitions
e ☐ Own perceived need
f ☐ Other reasons: .....

4. I own and use a microscope: ☐ Yes ☐ No

4.1. Brand: _____ Type: _____

4.2. I bought the microscope for the following reason(s):
*(More than one answer is permitted.)*

a ☐ Course
b ☐ Published studies
c ☐ Colleagues' recommendation
d ☐ Exhibitions
e ☐ Benefits for endodontics
f ☐ Other reasons: .....

5. The microscope makes the loupe useless:

☐ Yes, always
☐ Yes, but only in endodontics
☐ No, loupes and microscopes complement one another

Thank you for your participation!

Figure 1. *A questionnaire investigated the dentists' experience with magnification devices and the motivation to use them. A self-assessment of the visual performance as dental professionals was additionally evaluated on a modified visual analog scale.*

of variance, followed by exact Wilcoxon rank-sum tests, was used to analyze the influence of age and magnification devices on the best clinical situation. The Jonckheere-Terpstra test followed by exact Wilcoxon rank-sum tests evaluated if the visual performance improved with increasing magnification.[25] The *p*-values were adjusted as a result of multiple comparisons using the Bonferroni-Holm correction.[26] The Spearman rank correlation coefficient was used to detect a relationship between the dentist's self-assessment and the visual performance under standardized visual test conditions.

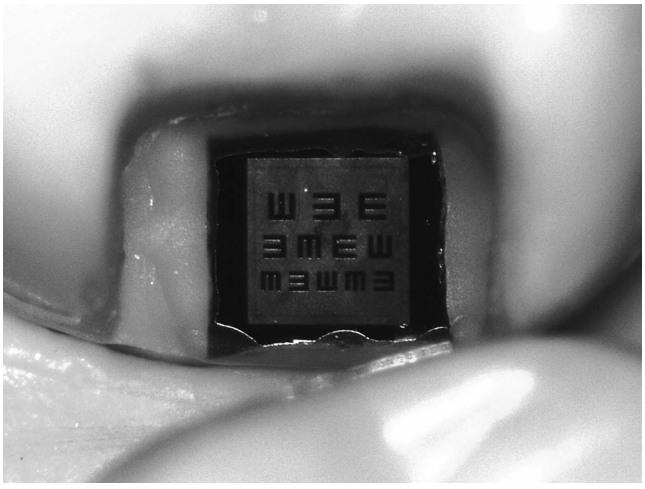

Figure 2. *One of four visual tests in distal cavities of maxillary premolars and molars.*

## RESULTS

The questionnaire revealed that 64% of the dentists owned a magnifying loupe system: 45% owned Galilean loupes (G), 16% Keplerian loupes (K), and 3% single-lens loupes (SL). Among the loupe users, 13 dentists (19% of all participating dentists) were using a microscope (M) in certain fields of dentistry (eg, endodontics). These dentists were convinced that loupes and microscopes complement one another, meaning that both magnification devices have advantages in certain clinical situations.[27] The decision to buy and use magnification aids was mainly influenced by colleagues (34%) or by the subjective need for better visual performance (33%). Less important reasons were courses (16%), exhibitions (11%), or scientific studies (6%). A total of 38% of all participating dentists reported a further need for information concerning magnification devices in dentistry.

The results of the self-assessment on a VAS (0-5) showed a median value of 4 and a full range of 0 to 5. Eleven dentists assessed a score of $\leq 2.5$, 58 dentists scored $>2.5$. The Spearman rank correlation coeffi-

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Figure 3. *The phantom head was positioned on the patient's dental chair to simulate the clinical situation.*

cient revealed only a weak positive correlation between these self-assessed values and the objective visual test on the negatoscope (correlation coefficient: 0.25). Twenty-two dentists (32% of all participating dentists) with a sufficient or good self-assessed value (>2.5) showed an objective visual acuity below the median of the group (<13.74 $mm^{-1}$) (Figure 4).

The results of the clinical visual test with the phantom head are presented in Figure 5. In the dentists' respective clinical settings, the visual performance with eventual optical aids showed a significant impact of the dentist's age ($p$=0.0012) and the magnification device ($p$=0.03) (Figure 5). When comparing the different clinical conditions without regard for the dentist's age, the best visual performance was achieved with Keplerian loupes, followed by Galilean loupes and by natural vision (all $p \leq 0.0006$). When comparing the different age groups, the post hoc tests detected a significant difference only for natural visual acuity with a free choice of distance (NVf; $p$=0.02). No significant differences were found between the dentists who were younger or older than 40 years when they were using loupes (Figure 5).

## DISCUSSION

Two-thirds of the questioned private practitioners owned a magnification device. This rate is higher than those measured in similar studies, which reported values of 9%,[28] 34%,[26] 44%,[29] and 53.7%.[3] The low percentage reported in the study of Forgie and others[28] might be explained by the date of the survey, as the use of magnification devices has been growing during the last few years.[3-5] One study[4] found 86% of senior dental students routinely using magnifying loupes. This group received basic information concerning magnification systems during the first-year curriculum. This high value could support the effect of basic education as motivation to use magnification devices. Forgie and others[28] reported a strong association between the use of magnification devices and a course about magnification or a practice partner using magnification. The main motivating factors in the present study were the influence of friends and the subjective feeling of needing better vision, but specifically not courses, studies, or the manufacturers marketing at an exhibition. However, the latter three might inform the 38% looking for more information about magnification devices in dentistry.

Remarkable in this context is the uneven distribution of the different loupe systems. We tested dentists in two regions of Switzerland. All Keplerian loupes were found in one region, while the Galilean loupes of the other region were nearly all (16 out of 21) from the same manufacturer. This could confirm the importance of personal relations and recommendations, as indicated in the questionnaire, but this also reflects a lack of knowledge in the dental community.

The self-assessment revealed that most of the dentists were convinced of their good visual performance. However, the Spearman correlation coefficient showed only a weak statistical correlation between the subjective feeling and the objective measurement. This suggests the importance of regular visual tests while one is practicing dentistry. It is clinically relevant that 22 dentists (32%) assessed their vision to be sufficient or good (VAS >2.5), while the objective value was below the median visual performance of all dentists (<13.74 $mm^{-1}$). These dentists were not aware of their visual deficiency.

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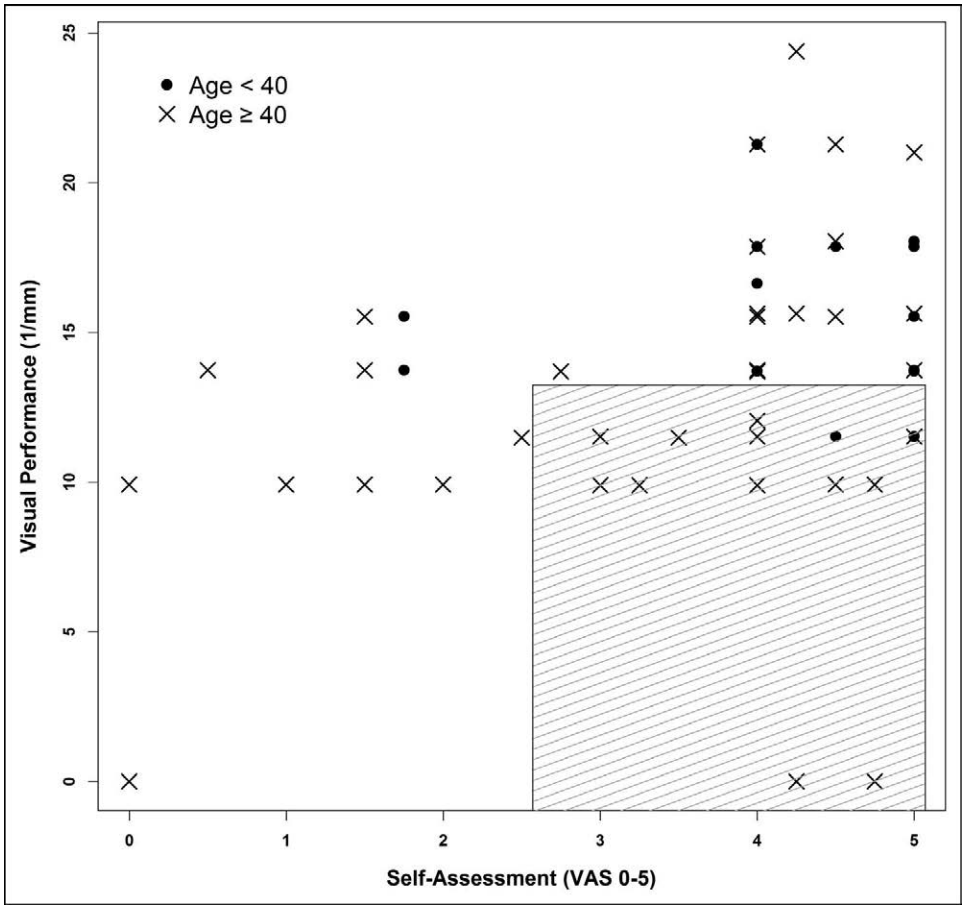


Figure 4. *Objective and self-assessed near visual acuity. The correlation between the self-assessment and objectively measured visual acuity was weak. Notice the 22 dentists (32%) with a sufficient self-assessed score (VAS >2.5) but with poor objective visual acuity (gray rectangle). Twenty of these 22 dentists were ≥40 years old.*

The visual performance in the individually optimized clinical situation of the daily practice is, from a clinical point of view, the relevant parameter to discuss vision of dental professionals. A phantom head with miniaturized visual tests in distal cavities of upper molars offers a validated method for this purpose.[22] It is the nature of an individual clinical setting that neither the working distance nor the light source or the used optical aids are standardized, but rather are individually adapted by the respective dentist. The influence of the light source was not part of this study and should be evaluated in further research under standardized conditions.

Significant differences between the two age groups could only be found for NVf. Younger dentists can profit from natural magnification by reducing the eye-object distance, with a linear relationship between distance and magnification. This is routinely used as a controlling distance and is biologically not possible for older dentists as a result of their presbyopia.[18,19,22] Loupes, with their fixed and ergonomic focal distance, inhibit the natural magnification for young dentists as well. The weak magnification of typical Galilean loupes (2.5×) could compensate for the loss of natural magnification, but it could not significantly improve the visual performance in the group of young dentists. As a consequence, Galilean loupes offer ergonomic rather than optical benefits for young dentists and compensate for presbyopic deficiencies in older dentists. This is illustrated by the equivalence of the visual performance in dentists ≥40 years old with Galilean loupes (G) compared to the performance of dentists <40 years old with natural vision at a free distance (NVf). A former study[22] including Keplerian loupes with a magnification factor of 4.3× showed a significant improvement in the visual acuity independent of the dentist's age. The impact of Keplerian loupes could not be further analyzed in the present study as a result of an insufficient number of young dentists using this type of loupe.

The microscopes were not included in our measurements because none of the dentists used it routinely for the majority of his or her dental work. Nevertheless, it is well known that microscopes enable a highly superior visual performance and

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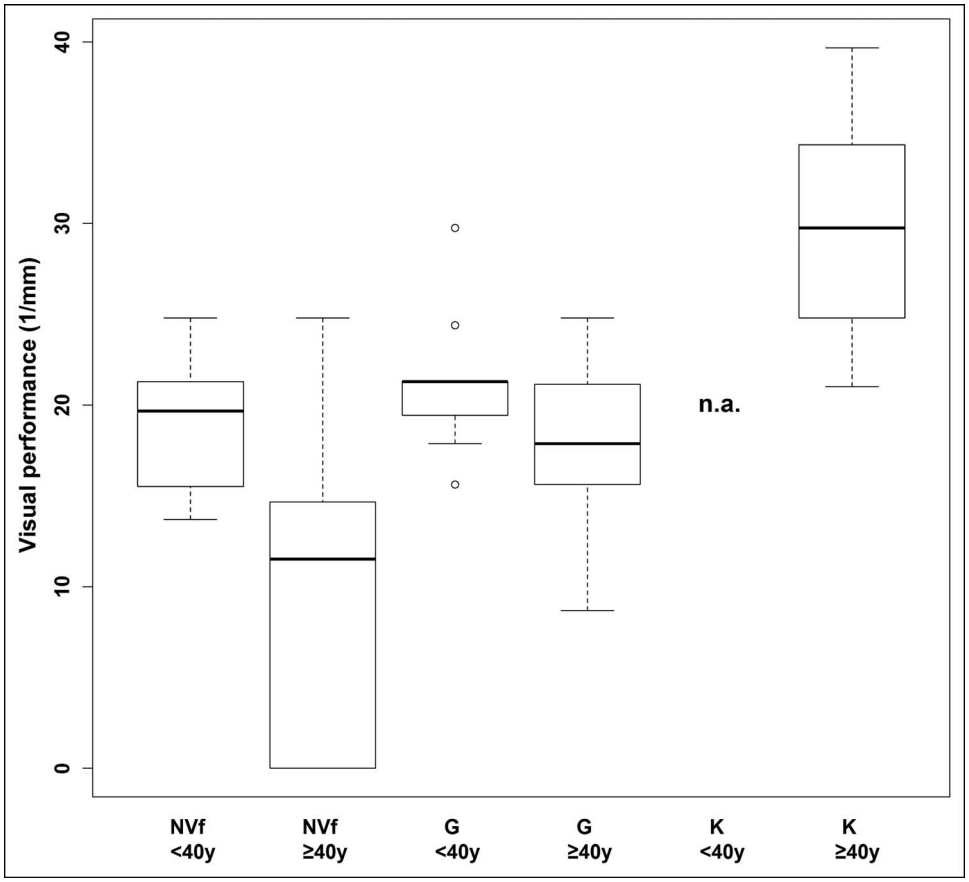


Figure 5. *The visual performance in the dentists' respective clinical settings ($mm^{-1}$). Dentists younger than 40 years of age showed a significantly better visual performance for NVf compared to dentists who are 40 years or older ($p=0.02$). When using magnification devices, no significant differences could be shown. Notice the similar visual acuity of NVf <40 years and G ≥40 years. NVf = natural visual acuity with a free choice of distance; G = Galilean loupe; K = Keplerian loupe; y = years; n.a. = not available, as only one dentist <40 years old was using a Keplerian loupe.*

could have the potential to solve visual challenges in dentistry.[30-32]

We stated in the introduction that there exists a lack of knowledge about the impact of vision and magnification on the performance of dentists. The results of this study confirm the need for further research in this field.

## CONCLUSIONS

Self-assessment cannot replace regular optical near vision tests. A wide range of near visual acuity can be found in the individual clinical conditions of a dental private practice. The visual performance is influenced by magnification devices as well as by the dentist's age. The benefit of Galilean loupes for most of the younger dentists is ergonomic rather than optical. For older dentists, Galilean loupes can compensate for presbyopic deficiencies. The question still remains whether good visual performance affects clinical tasks in different specialties, and this topic should be the subject of further studies.

### Acknowledgement

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### Human Subjects Statement

This study was conducted at the AMK Universität in Bern, Switzerland under the auspices of the Kantonale Ethikkommission Bern.

### Conflict of Interest

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

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# Retention of Root Canal Posts: Effect of Cement Film Thickness, Luting Cement, and Post Pretreatment

A Sahafi • AR Benetti • S Flury
A Peutzfeldt

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### Clinical Relevance

When treating oversized post spaces, posts should be adhesively bonded in the root canal. That is, the posts should be luted with resin cement and adhesion ensured not only to the dentin of the root canal but also to the post.


## SUMMARY

**The aim of this study was to investigate the effect of the cement film thickness of a zinc phosphate or a resin cement on retention of untreated and pretreated root canal posts. Prefabricated zirconia posts (CosmoPost: 1.4 mm) and two types of luting cements (a zinc phosphate cement [DeTrey Zinc] and a self-etch adhesive resin cement [Panavia F2.0]) were used. After removal of the crowns of 360 extracted premolars, canines, or incisors, the root canals were prepared with a parallel-sided drill system to three different final diameters. Half the posts did not receive any pretreatment. The other half received tribochemical silicate coating according to the manufacturer's instructions. Posts were then luted in the prepared root canals (n=30 per group). Following water storage at 37°C for seven days, retention of the posts was determined by the pull-out method. Irrespective of the luting cement, pretreatment with tribochemical silicate coating significantly increased retention of the posts. Increased cement film thickness resulted in decreased retention of untreated posts and of pretreated posts luted with zinc phosphate cement. Increased cement film thickness had no influence on retention of pretreated posts luted with resin cement. Thus, retention of the posts was influenced by the type of luting cement, by the cement film thickness, and by the post pretreatment.**


*Alireza Sahafi, DDS, PhD, private practice, Kongens Lyngby, Denmark

Ana R Benetti, DDS, PhD, Section for Oral Rehabilitation, Dental Materials, and Oral Diagnostics, Department of Odontology, University of Copenhagen, Copenhagen, Denmark

Simon Flury, DDS, Department of Preventive, Restorative, and Pediatric Dentistry, School of Dental Medicine, University of Bern, Bern, Switzerland

Anne Peutzfeldt, DDS, PhD, DrOdont, Department of Preventive, Restorative, and Pediatric Dentistry, School of Dental Medicine, University of Bern, Bern, Switzerland

*Corresponding author: Lyngby Hovedgade 17, DK-2800 Kongens Lyngby, Denmark; e-mail: astandklinik@gmail.com

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## INTRODUCTION

Posts are often inserted into endodontically treated teeth with minimal remaining tooth substance to

provide retention and stability for a core.[1] Several studies[2-4] have investigated the clinical survival and failure of teeth restored with posts and cores. Failure modes include loss of retention of the post, fracture of the root, and fracture of the post or the core, with loss of retention of posts reported as one of the most frequent types of failure.[1-4] In their review of failure modes of teeth restored with posts luted adhesively with resin cement, Rasimick and others[4] found debonding loss to account for 37% of all reported failures. These authors[4] also calculated pooled odds of 2.3% that a restoration will fail due to debonding, compared with the 4.3% calculated for posts luted with zinc phosphate cement or glass ionomer cement.

The retention of posts is influenced by numerous factors related to the post, the luting cement, as well as the cement-post and cement-dentin interactions.[1-8] Focusing on the type of luting cement, several studies[9-14] found superior retention of posts luted with resin cement compared with posts luted with zinc phosphate cement. One explanation is the superior strength of resin cements over zinc phosphate cement,[10,15] another, the ability of resin cements to supplement micromechanical retention by adhesive bonding, thereby reinforcing the restored tooth.[13,16]

Regarding the interface between the resin cement and the post, several pretreatments of the post have been proposed to improve adhesion. These pretreatments include sandblasting with alumina particles, tribochemical silicate coating followed by application of a silane, etching with hydrofluoric acid, and coating with primers. The tribochemical silicate coating system CoJet (3M ESPE, Seefeld, Germany) has been found to significantly improve bonding of resin cement.[16-19] This system uses silicate-coated alumina particles to sandblast the post surface prior to the application of silane and resin cement. Sandblasting produces high spot heat, which, together with the blasting pressure, results in the welding of the silicate layer onto the post surface. Subsequent silanization enhances the bond strength of resin cement to the silicated surface.[19]

Retention of posts depends not only on bonding the resin cement to the post but also on bonding the resin cement to the dentin of the root canal. This latter bonding is influenced by the dentin surface area, the presence of a smear layer on the dentin surface after post space preparation,[20-22] the surface conditioning of dentin, the adhesive system, and the type of resin cement.[7,23-25]

Finally, not only the type of luting cement but also the film thickness of the cement may influence the retention of posts. A uniform cement film thickness between 25 and 50 μm has generally been accepted for luting of fixed prosthetic restorations.[26] Optimal and uniform thickness of the cement requires precise fit of the post in the post space. Due to the irregularities of the root canal morphology or the tapered endodontic preparations, obtaining a precisely fitting post and a uniform film thickness would often require extensive post-space preparation and lead to weakening of the remaining tooth structure. To the knowledge of the present authors, no studies have investigated the effect of film thickness on retention of posts using zinc phosphate cement. However, increasing the film thickness of zinc phosphate cement has been shown to decrease the retention or the resistance to loading of inlays or crowns.[26-28] In contrast, numerous studies have investigated the effect of film thickness on the retention of posts luted with resin cements; however, the results of these studies are conflicting. Whereas some studies[29,30] report no influence of film thickness, one study[31] found retention to be lower with a thick film (oversized post space) compared with a thin film (precisely fitted post space). Another study[32] found retention to be higher with a thick cement film (oversized post space), and yet other studies[33-35] found greater film thicknesses (oversized post space) to increase retention, provided the film thickness was not too great. One reason for the contradicting conclusions could be that the quality of the adhesion of the resin cement to the post and to the dentin varied among the studies, reflecting that different posts, resin cements, and adhesive systems were used. Considering the superior mechanical strength of resin cements over zinc phosphate cement and their adhesive potential, it seems likely that the retention of posts luted with resin cement is less sensitive to variations in film thickness. However, none of the studies carried out so far have considered the role of adhesion when investigating the influence of the cement film thickness on the retention of posts. Therefore, the aim of this *in vitro* study was to test the following hypotheses: 1) When posts, untreated or pretreated, are luted with zinc phosphate cement, thus being without any adhesive bonding, retention of the posts decreases with increasing film thickness of the cement and 2) when untreated posts are luted with resin cement, thus being without adhesive bonding to the surface of the post, retention of the posts decreases with increasing film thickness of the cement, but 3) when pretreated posts are luted with resin cement, thus being

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Table 1: *Post, Luting Cements, and Pretreatments Used*

| Post | Luting Cements | Pretreatment Steps |
|---|---|---|
| Zirconia posts:<br>CosmoPost<br>Ivoclar Vivadent,<br>Schaan, Liechtenstein | Zinc phosphate cement:<br>DeTrey Zinc<br>Dentsply DeTrey,<br>Konstanz, Germany | No pretreatment |
| | | Tribochemical silica coating (CoJet):<br>CoJet-Sand (particle size=30 μm)<br>3M ESPE, Seefeld, Germany<br>Silane coating:<br>ESPE Sil<br>3M ESPE, Seefeld, Germany |
| | Self-etch adhesive resin cement:<br>Panavia F2.0<br>Kuraray,<br>Okayama, Japan | No pretreatment |
| | | Tribochemical silica coating (CoJet):<br>CoJet-Sand (particle size=30 μm)<br>3M ESPE, Seefeld, Germany<br>Silane coating:<br>ESPE Sil<br>3M ESPE, Seefeld, Germany |

adhesively bonded to both the dentin and the surface of the post, retention is not affected by the film thickness of the cement.

## METHODS AND MATERIALS

### Retention of Prefabricated Zirconia Posts

Prefabricated zirconia posts and two types of luting cements (a zinc phosphate cement [DeTrey Zinc, Dentsply DeTrey, Konstanz, Germany] and a self-etch adhesive resin cement [Panavia F2.0, Kuraray, Okayama, Japan]) were used in the present study. Half the posts did not receive any pretreatment. The other half was pretreated by tribochemical silicate coating according to the manufacturer's instructions. This treatment system consisted of air abrasion with an intraoral sandblasting device (Dentoprep, Rønvig, Daugaard, Denmark) at 3 bar for 15 seconds using 30-μm silicate-coated particles (CoJet-Sand, 3M ESPE), followed by silane application. The investigated posts, pretreatment, and luting cements are listed in Table 1 along with their respective manufacturers.

A total of 360 extracted single-rooted human premolars, canines, or incisors were kept in an antimicrobial medium (0.5% chloramine-T) after extraction. The teeth were extracted for therapeutic reasons. The clinical crown of each tooth was removed perpendicularly to the long axis with a low-speed diamond saw, leaving at least 10 mm of root length. The roots were randomly distributed into 12 experimental groups, each consisting of 30 roots. Roots were prepared with a parallel-sided drill system (Edenta, Au, Switzerland) to three different diameters (1.40, 1.55, or 1.80 mm). For all roots, the length of the prepared root canal was 5 mm. The precise length of post-space preparation was ensured by a "stop" of resin composite luted 5 mm from the top of the drill. Following preparation, the canal was rinsed with deionized water for 60 seconds and dried with paper points (No. 45 Top Dent, Upplands Väsby, Sweden).

The posts were luted "upside down" to ensure that the entire luted part of these parallel-conical posts was parallel-sided. In the case of luting with Panavia F2.0, the walls of the root canals were treated with the corresponding primer (ED Primer II, Kuraray) according to the manufacturer's directions. Both luting cements were mixed according to the manufacturer's recommended procedure and applied inside the root canal with the aid of needle tubes (AccuDose NeedleTubes, Centrix, Shelton, CT, USA). Following luting of the posts with Panavia F2.0, the resin cement was light-cured for 20 seconds (Bluephase LED light-curing unit, Ivoclar Vivadent, Schaan, Liechtenstein) using the high-power program (>1200 mW/cm$^2$).

All specimens were allowed to set for 15 minutes and then were stored in water at 37°C for seven days. The retention of the prefabricated zirconia posts was tested by the pull-out method. The specimens were placed in a jig that fixed the root and the nonluted part of the post in a universal testing machine (model 5566, Instron Ltd, High Wycombe, UK). The posts were then extracted from the roots at a crosshead speed of 1 mm/min. The direction of the tensile loading was parallel to the long axis of the luted post. All luting and testing procedures were carried out by the same operator.

### Determination of Film Thickness

The diameter of three randomly selected posts was measured by a digital micrometer (model ID-U1025, Mitutoyo, Kawasaki, Japan). Three measurements

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Table 2: *Retention (N) of Untreated or Pretreated Posts Luted With Zinc Phosphate Cement (DeTrey Zinc) or Resin Cement (Panavia F2.0) of Three Different Film Thicknesses*

| Luting Cement | Cement Film Thickness | Untreated | | | Pretreated | | |
|---|---|---|---|---|---|---|---|
| | | 65 μm | 124 μm | 259 μm | 65 μm | 124 μm | 259 μm |
| DeTrey Zinc | Mean | 116.1 | 89.1 | 72.3 | 150.6 | 110.3 | 105.5 |
| | SD | 40.6 | 33.2 | 30.5 | 45.8 | 40.0 | 52.5 |
| | Median | 114.4 | 89.0 | 74.0 | 144.1 | 101.8 | 84.0 |
| | Maximum | 222.6 | 162.1 | 141.0 | 234.2 | 197.8 | 220.1 |
| | Minimum | 45.8 | 26.1 | 21.8 | 67.5 | 48.7 | 37.7 |
| Panavia F2.0 | Mean | 151.7 | 123.9 | 112.8 | 240.8 | 255.6 | 225.0 |
| | SD | 41.2 | 40.6 | 36.7 | 54.9 | 66.4 | 67.8 |
| | Median | 155.2 | 128.5 | 113.3 | 237.8 | 252.4 | 215.1 |
| | Maximum | 230.4 | 200.7 | 179.8 | 367.1 | 397.5 | 365.7 |
| | Minimum | 78.5 | 45.8 | 26.3 | 133.1 | 132.0 | 105.0 |

per post were made, and a final mean diameter of 1.370 mm (±0.007 mm) was calculated. In 15 additional roots, the canals were prepared with the burs intended for posts with diameters of 1.40, 1.55, and 1.80 mm, respectively. The diameter of each prepared root canal was measured under a light microscope (model M420, Leica, Heerbrugg, Switzerland) at 32× magnification. Per bur size, five canals were prepared and six measurements were made per canal. The following mean values and standard deviations of the root canal diameter were calculated from the preparations performed with the bur for a 1.4-mm post: 1.500 ± 0.047 mm; bur for a 1.55-mm post: 1.618 ± 0.029 mm; and bur for a 1.8-mm post: 1.888 ± 0.051 mm. Film thickness was then calculated as follows:

$$\text{Film thickness} = (\text{diameter of root canal} - \text{diameter of post})/2$$

The calculations resulted in the following three film thicknesses: 65, 124, and 259 μm.

### Statistical Analysis

The retention data were statistically analyzed with a nonparametric aligned rank transformation (ART) analysis of variance (ANOVA)[36] followed by a Bonferroni-Holm correction for multiple testing. The ART ANOVA was followed by exact Wilcoxon rank sum tests without correction for multiple testing. Consequently, the *p*-values of the latter tests must be interpreted in an explorative context. To measure the correlation between film thickness and retention, Spearman rank correlation coefficients were calculated. All statistical analyses were performed with R version 2.12.1 using the extension package exactRankTests (The R Foundation for Statistical Computing, Vienna, Austria; http://www.r-project.org). A global level of significance of $\alpha=0.05$ was applied.

## RESULTS

The retention of the prefabricated zirconia posts, untreated or pretreated, are described in Table 2 and visualized in Figure 1.

The ANOVA showed a significant effect of luting cement ($p<0.0001$), pretreatment ($p<0.0001$), and film thickness ($p<0.0001$). In addition, a significant interaction between luting cement and pretreatment ($p<0.0001$) was observed.

Irrespective of the luting cement, the pretreatment with tribochemical silicate coating significantly increased the retention of prefabricated zirconia posts (DeTrey Zinc: $p=0.0001$; Panavia F2.0: $p<0.0001$). Moreover, luting with Panavia F2.0 resulted in higher retention of the zirconia posts than did luting with DeTrey Zinc, irrespective of the surface treatment (untreated posts: $p<0.0001$; pretreated posts: $p<0.0001$).

When untreated posts were luted with DeTrey Zinc, the film thickness of 65 μm resulted in significantly higher retention than did the film thicknesses of 124 μm ($p=0.007$) or 259 μm ($p<0.0001$). Furthermore, the film thickness of 124 μm resulted in significantly higher retention than the film thickness of 259 μm ($p=0.03$). When posts luted with DeTrey Zinc were pretreated by tribochemical silicate coating, the film thickness of 65 μm still resulted in higher retention than did the film thicknesses of 124 μm ($p=0.004$) or 259 μm ($p=0.001$). Here, though, no significant difference

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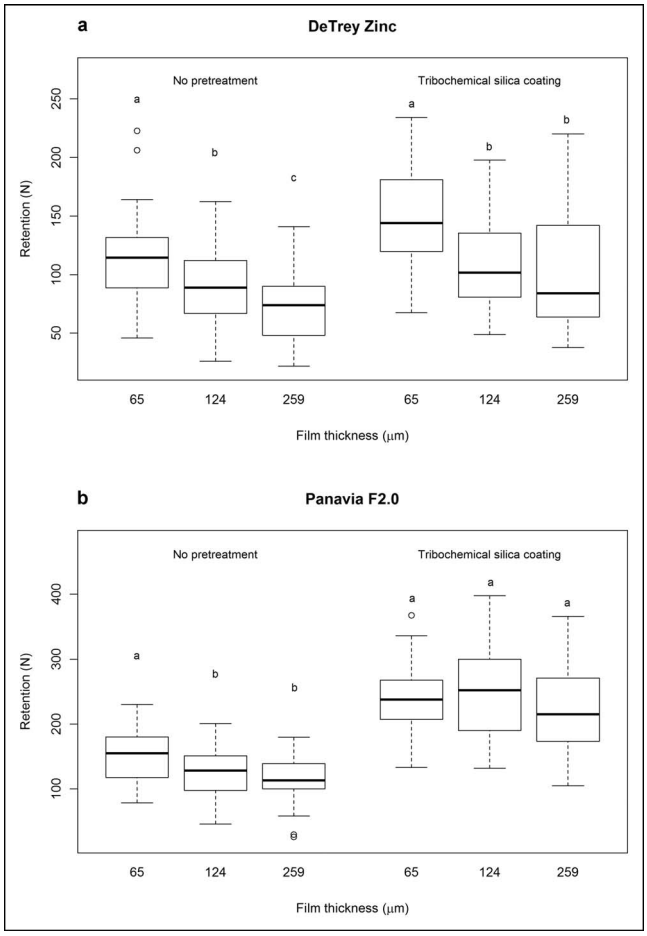


Figure 1. *Retention of untreated or pretreated posts luted with (a): zinc phosphate cement (DeTrey Zinc) or (b): resin cement (Panavia F2.0) of three different film thicknesses. Within each of the four groups of comparison (DeTrey Zinc, no pretreatment; DeTrey Zinc, tribochemical silica coating; Panavia F2.0, no pretreatment; Panavia F2.0, tribochemical silica coating), identical letters indicate no statistically significant difference (*p*>0.05).*

in the retention of posts was present between the film thicknesses of 124 μm and 259 μm ($p$=0.4232).

When untreated posts were luted with Panavia F2.0, a film thickness of 65 μm also resulted in significantly higher retention than the film thicknesses of 124 μm ($p$=0.02) or 259 μm ($p$=0.0006). No significant difference was observed on the retention of posts for film thicknesses of 124 μm and 259 μm ($p$=0.2524). When posts luted with Panavia F2.0 were pretreated by tribochemical silicate coating, no significant difference in their retention was present for the three investigated cement film thicknesses (65 vs 124 μm: $p$=0.37; 65 vs 259 μm: $p$=0.20; 124 vs 259 μm: $p$=0.08).

Moderate negative correlations between the film thickness and retention existed among the three groups of untreated posts luted with DeTrey Zinc (Cor(Spearman)=–0.4475). The same correlation was observed for pretreated posts luted with DeTrey Zinc (Cor(Spearman)=–0.3410), and among the untreated posts luted with Panavia F2.0 (Cor(Spearman)=–0.3596). Conversely, a very weak correlation was observed for pretreated posts luted with Panavia F2.0 (Cor(Spearman)=–0.1242).

## DISCUSSION

The present study evaluated the effect of cement film thickness, luting cement, and post surface pretreatment on the retention of endodontic posts luted in root canals of extracted human premolars, canines, and incisors. This retention is a complex expression of a multitude of factors including the type of luting cement, the bonding of the luting cement to the post and to the dentin, the mechanical properties of the luting cement and of the post, as well as the surface structure and shape of the post. In addition, studies[20,37,38] have demonstrated that surface design (eg, grooved, roughened, serrated, or threaded) influences the retention of posts. In order to eliminate any such influence, the present study was conducted on a smooth post.

When posts were luted with zinc phosphate cement, increased cement film thickness resulted in decreased retention of the posts irrespective of whether the posts had been pretreated. This finding leads to acceptance of the first hypothesis and is in agreement with previous studies,[26-28] although these studies dealt with retention of inlays and crowns and not with root posts. One explanation may be sought in the mechanical properties of zinc phosphate cement. Traditionally, mechanical retention, such as that ensured by zinc phosphate cement, has been thought to depend on three factors: the roughness of the retentive surfaces, the strength of the luting cement, and the compressibility of the cement.[39] Furthermore, the thicker the cement film, the more compressible it will be and the lower the expected retention of the luted restoration.[39] The fact that the tensile and compressive strengths of zinc phosphate cement are inferior to the strength of resin cements[28,40-45] suggests that zinc phosphate cement is much less capable of resisting the forces transmitted to the luting cement during the retention test, thus resulting in premature crack formation of the luting cement and in dislodgement of the posts at a lower loading force. Another explanation may be sought in the resistance of zinc phosphate cement to crack formation and crack propagation. As explained by Wiskott and others,[28] all materials contain defects

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randomly located within their microstructure. Under load, each defect is likely to initiate crack growth. The thicker the material, the more numerous the defects, the higher the probability of crack initiation, and hence the reduced resistance of thicker cement layers.[28]

When untreated posts were luted with Panavia F2.0, increased cement film thickness resulted in decreased retention of the posts. This finding is in agreement with previous studies[31,28] and leads to acceptance of the second hypothesis. The fact that film thickness had a significant effect on retention despite the use of a resin cement may be explained by the just mentioned negative influence of increased cement film thickness on the resistance of the cement to crack formation.[28] An additional explanation is inadequate adhesion between the resin cement and the untreated zirconia posts.[7,8,16,18] Although the resin cement may have adhered well to the dentin, poor adhesion to the post resulted in a situation similar to the one found when zinc phosphate cement was used (eg, where retention was obtained by micromechanical interlocking and not by adhesive bonding). It should be noted that for these untreated posts, the resin cement resulted in higher retention than did the zinc phosphate cement, due to the superior strength of the resin cement.

Pretreatment with tribochemical silicate coating resulted in increased retention of the posts regardless of the type of luting cement. This finding is in accordance with previous studies.[8,18,46] The tribochemical silicate coating uses silicate-coated alumina particles to sandblast the surface prior to application of silane.[19] Sandblasting resulted in increased roughness and surface area, and the high spot heat produced, together with the blasting pressure, resulted in the welding of the silicate layer onto the surface of the post. The subsequent silanization has been found to enhance the bond strength of resin cement to the treated surface.[47] The positive effect of tribochemical silicate coating on posts luted with zinc phosphate cement may be ascribed to the increased roughness of the post surface and thus to increased mechanical interlocking of zinc phosphate cement with the surface of the post.[46]

Whereas increased cement film thickness reduced the retention of untreated posts, film thickness did not influence the retention of pretreated posts luted with resin cement. This leads to the acceptance of the third hypothesis. The tribochemical silicate coating system designed for adhesive bonding of resin cement to restorations has been found to promote effective bonding between resin cements and various types of prefabricated posts.[6-8,16-18,48,49] The positive effect that pretreatment of the posts had on the retention of the posts when these were luted in oversized post spaces (ie, with cement film thickness of 124 μm or 259 μm) may be assumed to derive from efficient adhesion of Panavia F2.0 to the posts.[16-18,48,49] Precise and uniform fit of the post in the prepared space may very often be impossible to obtain clinically if extensive, root-weakening preparation of the root canal is to be avoided. Consequently, the use of prefabricated posts inevitably results in variations in the post space and thus in the thickness of the cement film. To optimize retention in these cases and in cases of evidently oversized post spaces, the present results suggest that posts should be luted adhesively (ie, with a resin cement that effectively bonds to the post as well as to the dentin).

Numerous studies[50-55] have reported posts of relatively low elastic modulus, such as fiber-reinforced resin composite posts, luted adhesively with resin cements to show good performance and high retention, and posts with "dentinlike" elastic modulus have been favored over posts of high elastic modulus, such as titanium or ceramic posts. However, as reported by Theodosopoulou and Chochlidakis[56] in their systematic review, few studies included a control group[57] or they varied several factors at once, for example, the type of post and the luting cement (fiber-reinforced post luted with resin cement vs metal post luted with zinc phosphate cement)[53,58] or the retention principle (a smooth fiber-reinforced post vs a metal screw).[55] It is noteworthy that the one study[57] that did include a control group found no difference in the two-year clinical performance of a titanium post and a glass fiber-reinforced composite post using a self-etch adhesive resin cement. Given that the present study found the type of luting cement and adhesive bonding to influence the retention of the posts, it may be that the previously reported favorable results obtained with low-modulus posts over high-modulus posts were caused by adhesive bonding of the luting cement to the root canal dentin as well as to the post, rather than by the lower elastic modulus of the post *per se*. Further studies are warranted to clarify the correlation between elastic modulus of posts and adhesion.

## CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

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- Increased cement film thickness resulted in decreased retention of the untreated posts luted with either zinc phosphate cement or resin cement.
- Increased film thickness of resin cement did not influence the retention of posts pretreated with tribochemical silicate coating.
- Pretreatment with tribochemical silicate coating generally improved the retention of posts regardless of the type of luting cement.

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#### Human Subjects Statement

This research complied with the Act on Research Ethics Review of Health Research Projects (from June 14, 2011), the National Committee on Health Research Ethics of Denmark.

#### 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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# Effect of Base and Inlay Restorative Material on the Stress Distribution and Fracture Resistance of Weakened Premolars

ACO Souza • TA Xavier • JA Platt
ALS Borges

### Clinical Relevance

Considering the increase in esthetic restorative treatments and the increased emphasis on more conservative cavity preparations, it is timely to assess the influence of the type of direct base and indirect inlay material on the stress distribution in endodontically treated premolars with weakened cusps.

## SUMMARY

**The purpose of this study was to evaluate the influence of direct base and indirect inlay materials on stress distribution and fracture resistance of endodontically treated premolars with weakened cusps. Forty healthy human premolars were selected; five were left intact as controls (group C+), and the others were subjected to endodontic treatment and removal of buccal and lingual cusp dentin. Five teeth were left as negative controls (group C–). The remaining 30 teeth were divided into two groups according to the direct base material (glass ionomer [GIC] or composite resin [CR]). After base placement, each group was subjected to extensive inlay preparation, and then three subgroups were created (n=5): no inlay restoration (GIC and CR), restored with an indirect composite resin inlay (GIC+IR and CR+IR), and restored with a ceramic inlay (GIC+C and CR+C). Each specimen was loaded until fracture in a universal testing machine. For finite element analysis, the results showed that the removal of tooth structure significantly affected fracture resistance. The lowest values were presented by the negative control**

*Ana Carolina de Oliveira Souza, DDS, MS, Department of Dental Materials and Prosthodontics, Institute of Science and Technology, Universidade Estadual Paulista, São José dos Campos, SP, Brazil

Tathy Aparecida Xavier, PhD, Department of Dental Materials, School of Dentistry, Universidade de of São Paulo, São Paulo, SP, Brazil

Jeffrey A Platt, DDS, MS, Department of Restorative Dentistry, Indiana University School of Dentistry, Indiana University Purdue University Indianapolis, Indianapolis, IN, USA

Alexandre Luiz Souto Borges, DDS, MS, PhD, Department of Dental Materials and Prosthodontics, Institute of Science and Technology, Universidade Estadual Paulista, São José dos Campos, SP, Brazil

*Corresponding author: Av. Eng. Francisco José Longo, 777 Jd. São Dimas, Universidade Estadual Paulista, São José dos Campos, SP 12245-000, Brazil; e-mail: carol.odonto@yahoo.com.br

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**group, followed by the restored and based groups (not statistically different from each other) and all lower than the positive control group. In finite element analysis, the stress concentration was lower in the restored tooth compared to the tooth without restoration, whereas in the restored teeth, the stress concentration was similar, regardless of the material used for the base or restoration. It can be concluded that the inlay materials combined with a base showed similar behavior and were not able to regain the strength of intact tooth structure.**

## INTRODUCTION

Because structural strength depends on the quality and integrity of a tooth's anatomic form, the quantity of a sound tooth available to retain and support the restoration is a fundamental clinical concern.[1] Moisture content and stiffness of teeth with an endodontic access were not found to be different from those of untreated vital teeth[2,3]; however, the loss of strategic tooth structure as a result of caries and subsequent restorative procedures severely compromise the strength of these teeth. An extensive mesio-occluso-distal (MOD) cavity plus endodontic access, which is a common clinical finding, results in a reduction of tooth stiffness and fracture strength of approximately 60%.[4,5] This loss of strength constitutes a restorative challenge in terms of restoring fracture resistance.

Some studies claim that inlay restorations can provide the required protection and ensure clinical success of the restoration while providing the maximum preservation of tooth structure.[6] Adhesive restorations are advantageous since macroretentive design is no longer a prerequisite when an adequate amount of tooth surface is available for bonding.[7] Clinicians are particularly interested in using preparations with minimal or no macroretentive properties.[8]

Glass ionomer cement (GIC) has a long history of use in dentistry due to its ability to bond chemically to tooth structure and its cariostatic effect from fluoride release[9-11] in addition to antibacterial activity.[12] Moreover, GIC has little contraction during the setting reaction, and its coefficient of thermal expansion is close to that of tooth structure.[13] These factors have been considered important to the success of effective bonding to tooth structure and provide justification for the cement's use as a support material under weakened enamel.

Many studies have shown the benefit of using composite resin to support weakened tooth structure because it provides reinforcement to the cusps, increasing the strength of the remaining tooth structure.[5,14,15] Generally, the physical and mechanical properties of the material used influence the behavior of the restored tooth under testing conditions.[16] Thus, the purpose of this study was to evaluate, *in vitro*, the influence of the type of base and inlay restorative material on fracture resistance and stress distribution in weakened teeth.

## METHODS AND MATERIALS

### Fracture Resistance Test

The brand name, manufacturer, and basic description of the materials used in this study are listed in Table 1. Forty fresh and caries-free maxillary premolars, extracted for orthodontic reasons, were selected. The teeth had the occlusal surface area and the angle formed by the two cusps measured in a digital X-ray image by the software UTHSCSA ImageTool for Windows, version 3.0 (University of Texas Health Science Center at San Antonio, San Antonio, TX, USA), and those having values above or below 10% of the average were excluded.

For the simulation of 0.3-mm-thick periodontal ligament, each root was immersed in melted wax up to the demarcation line 2 mm apical to the cemento-enamel junction (checked by a digital caliper). Roots were placed in a self-cured polyurethane resin up to the demarcation line in a polyvinyl chloride cylinder (20-mm height $\times$ 18-mm diameter). After curing, the wax was carefully removed from the root using hot water so that the wax could be replaced by polyether impression material[17] (Impregum F, 3M ESPE, St Paul, MN, USA). The root was then stored in distilled water in a refrigerator at 5°C.

The teeth were randomly separated in eight groups, according to direct base and inlay materials, as illustrated in Figure 1. First, an MOD opening was made with refrigerated #3131 diamond burs (KG Sorensen, Cotia, SP, Brazil) at high speed, positioned perpendicular to the long axis of the tooth, 1 mm below the cemento-enamel junction, through the aid of an adapted optical microscope. The marks left by the contact between a 10-mm-diameter steel ball and the occlusal face of the teeth guided the buccal-lingual width of the cavities (0.5 mm inner to the marks). For the removal of the cusp dentin, a 2.5-mm-diameter spherical diamond bur (#3017, KG Sorensen) was introduced until its rod limit into the buccal and lingual walls of the cavity (Figure 2).

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Table 1: *Brand Name, Manufacturer, and Basic Description of the Materials Used*

| Material | Manufacturer | Characteristic | Lots |
|---|---|---|---|
| IPS e.max Press | Ivoclar Vivadent | Ceramic lithium disilicate | N30995 |
| Variolink II | Ivoclar Vivadent | Resin cement | M44477<br>L46354 |
| Monobond Plus | Ivoclar Vivadent | Silanizing agent | M15219 |
| SignumCeramis | Heraeus | Composite resin indirect | 010131 |
| Vidrion F | SS White | Glass ionomer cement | 0080911<br>0080811 |
| Impregum-F | 3M ESPE | Impression material polyether based | 421709<br>420540 |
| Resina F16 | Axon | Polyurethane resin | SKRP-4560 |
| Express | 3M ESPE | Silicon | 1127000396 |
| GuttaPercha | Dentsply | Gutta-percha | 578692D |
| AH Plus | Dentsply | Endodontic sealer | 1106001642 |
| Condac 37 | FGM | Phosphoric acid 37% | 150911 |
| CondacPorcelana | FGM | Hydrofluoric acid 10% | 110311 |

To perform the endodontic treatment, spherical diamond burs (#1014, KG Sorensen) and cone-drill Endo Z (Maillefer Instruments SA, Ballaigues, Switzerland) were used for the access opening. The canals were prepared with rotary instruments (Bio Race, FKG Dentaire, La Chaux-de-Fonds, Switzerland) according to the manufacturer's recommendations. Subsequently, the canals were dried with paper points and filled by insertion of gutta-percha with AH Plus sealer (Dentsply, Petrópolis, Brazil).

After endodontic treatment, with the exception of group C–, direct bases were placed using composite resin (CR, Z350, shade A2, 3M ESPE) or GIC (Vidrion F, SS White, Rio de Janeiro, Brazil). For the composite resin, the teeth were etched with 37% phosphoric acid (Condac 37, FGM, Joinville, Brazil) for 15 seconds, followed by the application of the adhesive Single Bond (3M ESPE), and photoactivated for 20 seconds (Poly Wireless, 1100 mW/cm$^2$, KAVO, Ind. Com Ltda, Joinville, Brazil). The light curing unit irradiance was verified using a radiometer (Kondortech, São Carlos, Brazil). The composite was placed in 2-mm-thick increments, each photoactivated for 40 seconds. For the GIC, mixing occurred according to the manufacturer's recommendations, and the cement was injected into the cavity with a syringe in an attempt to minimize the number of gaps and voids. To avoid dehydration, a surface layer of Single Bond adhesive was placed and light cured for 20 seconds. The specimens were stored in distilled water at 37°C for 24 hours. Then the specimens were again brought to the adapted optical microscope for MOD cavity preparation.

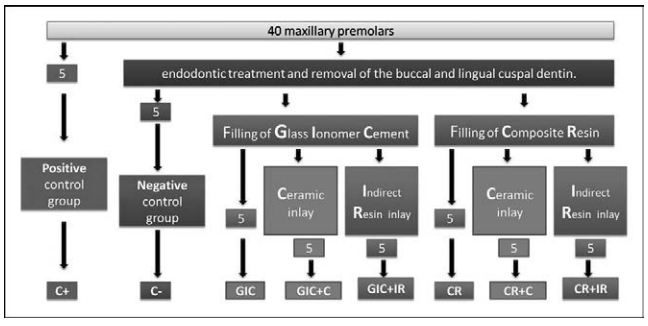


Figure 1. *Scheme of methodology.*

The incline of the pulpal and axial walls of the cavities was 6 degrees, and the inner corners were rounded, as characteristic of diamond bur #3131. Each diamond bur was used for five preparations under refrigeration. The occlusal and proximal boxes of groups were standardized to a width of 3 mm and a depth of 2 mm using a diamond bur (2 mm), as illustrated in Figure 2.

All teeth were impressed with addition silicone (Express, 3M ESPE) and sent to a laboratory to manufacture indirect restorations from two types of material: composite resin (Signum Ceramis, Heraeus, Frankfurt, Germany) and lithium disilicate

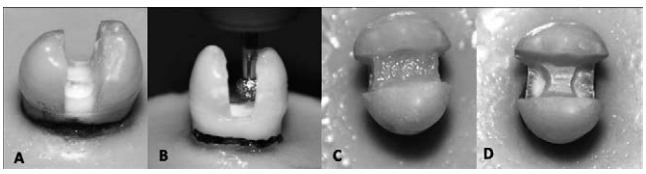


Figure 2. *Sequence of preparation. (A): Mesio-occluso-distal (MOD) preparation and root canal access. (B): Weakened cusps. (C): Fill. (D): Preparation finalized.*

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Table 2: *Mechanical Properties of Materials Used in Finite Element Analysis*

| Material | Longitudinal Elastic Modulus (MPa) | Poisson's Ratio | Reference |
|---|---|---|---|
| Stainless steel | 200,000 | 0.30 | Ansys Library 13.0 |
| Dentin | 17,600 | 0.31 | Reinhardt and others[27] |
| Enamel | 48,000 | 0.30 | Holmes and others[28] |
| Axson F16 Polyurethane | 3,600 | 0.30 | Owner characterization |
| Ligament | 68.9 | 0.45 | Holmes and others[28] |
| IPS e.max Press | 91,000 | 0.24 | Albakry and others[29] |
| Z350Resin | 10,000 | 0.24 | Manufacturer data |
| Glass ionomer cement | 7,300 | 0.30 | Agnihotri and others[30] |
| Signum Ceramis | 4,854 | 0.30 | Manufacturer data |

ceramic (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein).

All teeth were etched with 37% phosphoric acid for 15 seconds and washed with a water jet for 15 seconds, and the excess water was removed with absorbent paper. Then two layers of Single Bond adhesive were applied and cured following the manufacturer's instructions.

The inlays were ultrasonically cleaned (Ultrasound E15H, Elma, South Orange, NJ, USA) with distilled water for 15 seconds and air-dried for 30 seconds. The ceramic surface was etched with 10% hydrofluoric acid gel for 20 seconds, rinsed thoroughly, and dried with paper towels. A silane-bonding agent (Monobond Plus, Ivoclar Vivadent) was applied for 60 seconds.

The inlays were coated with resin cement (Variolink II, Ivoclar Vivadent) and seated in the preparation under 750 g of pressure, and the excess cement was removed. The inlays were cured, allowed to set for 10 minutes, and then stored in distilled water at 37°C for 24 hours.

The specimens were loaded on the enamel surface until fracture using a 10-mm-diameter sphere in a universal testing machine (EMIC, DL200MF, Test Equipment and Systems Ltd, Pinhais, Brazil) at a crosshead speed of 1 mm/min. The data were analyzed by Levene's test, one-way analysis of variance, and Tukey's test with a significance level of 5% and also by checking the power of the test. The fragments were analyzed under a stereomicroscope at a magnification of 20× to examine the types of fracture.

### FEA

The complete tooth structure and polyurethane resin were modeled with Rhinoceros 4.0 software (McNeel North America, Seattle, WA, USA) within the BioCad (CTI Campinas, Brazil) protocol, and the STP file was imported into Ansys 13.0 (ANSYS Inc, Houston, TX, USA) for the FEA. All materials were considered isotropic, homogeneous, and linear and were represented by the elastic modulus and Poisson's ratio (Table 2). The contact regions between the structures were considered perfectly bonded, and the mesh was built with about 260,000 nodes and 160,000 elements for all models. The polyurethane base was considered fixed in the three axes, and the sphere was displaced by 0.02 mm parallel to the tooth principal axis.

## RESULTS

Five samples were enough to find a maximum difference in the mean of 25 N among the groups with an 80% power of the test using Minitab (version 16) software. The means and standard deviations (in kgf) of the fracture resistance for each group are presented in Table 3. The lowest values were presented by groups C– (347 N), GIC (292 N), and CR (390 N), followed by the inlay groups (GIC+C, GIC+IR, CR+C, and CR+IR), which showed no statistical difference between them. All groups showed lower values than the positive control group C+ (1932 N).

The types of failure are given in percentages and are presented in Figure 3. All groups had a higher prevalence of type V fracture in the lingual cusp, except for the C+ group, which showed a higher percentage of type II fracture.

In the FEA, group C+ had a greater concentration of tensile stress in the central sulcus and also a larger gradient difference in the lingual cusp, especially in the middle and occlusal third (Figure 4). In group C, a tensile stress developed in the cervical region. In the buccal wall, the gradient increased significantly compared with group C+, indicating the increase of tensile stress in this

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Table 3: *Means and Standard Deviations of Fracture Resistance (N).*

| Experimental Groups | Mean | Standard Deviation | Homogeneous Groups | | | % Reduction of Fracture Resistance Compared to Healthy Group (C+) |
|---|---|---|---|---|---|---|
| C+ | 1,932 | ±95.6 | A | | | 0 |
| GIC+C | 618 | ±78.7 | | B | | 68.02 |
| CR+C | 582 | ±40.0 | | B | | 69.87 |
| CR+IR | 544 | ±119.4 | | B | C | 71.82 |
| GIC+IR | 544 | ±83.6 | | B | C | 71.85 |
| CR | 390 | ±85.8 | | | C D | 79.81 |
| C– | 347 | ±75.8 | | | D | 82.02 |
| GIC | 292 | ±106.8 | | | D | 84.91 |

[a] *Different letters indicate statistically different values.*

region. The presence of direct base (GIC and CR) considerably reduced the stress concentration in relation to group C– (Figure 4), and these stresses were higher in the group based with glass ionomer.

The inlay groups showed a strengthening of tooth structure, where the values of tensile stress were smaller than in group C–. In the gingival wall, it is possible to see a concentration of compressive stress due to the stability provided by the restoration. It may be noted that with the resin inlay, there was a slight decrease in tension in all tooth structure when compared to ceramic inlay, and groups based with resin had lower tensile values compared to groups

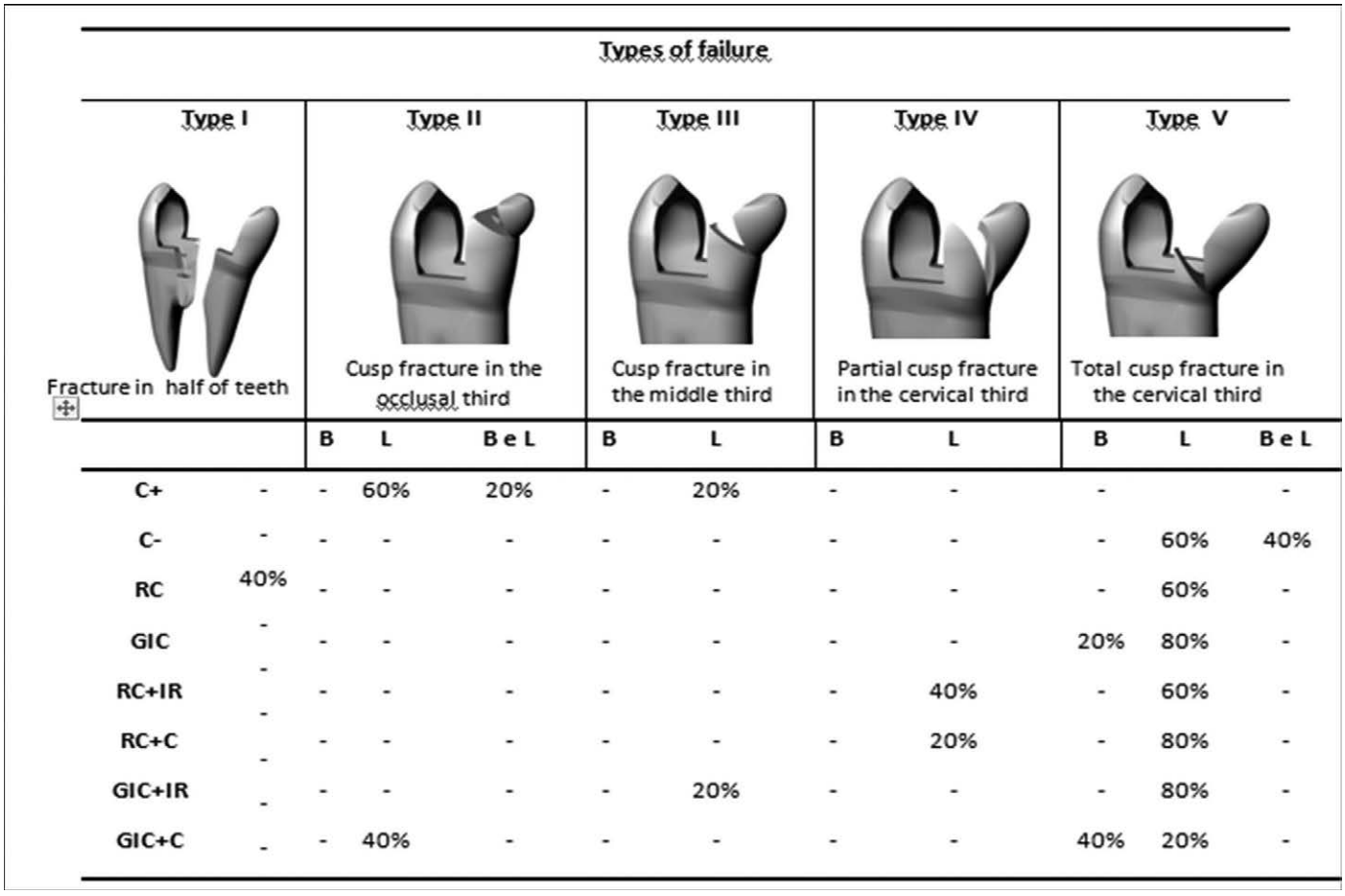


Figure 3. *Mapping of fractures and values of total percentage of fracture in each group.*

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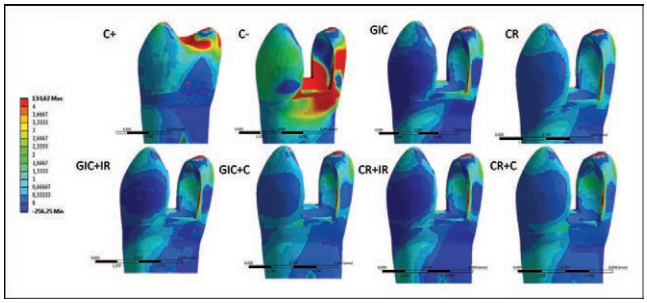


Figure 4. *Maximum principal stress distribution in experimental groups.*

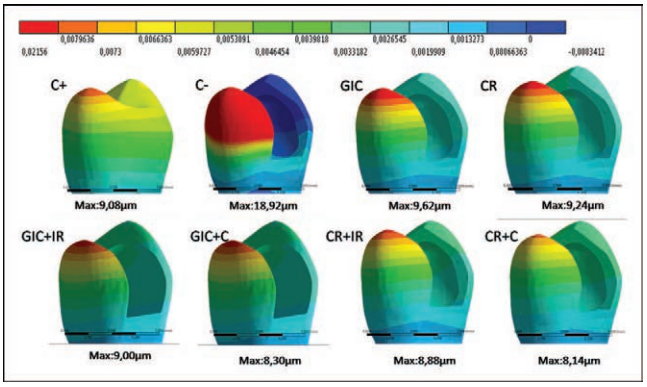


Figure 5. *Total displacement in all groups.*

based with glass ionomer, especially at the gingival wall.

The displacement fields can be seen in Figure 5, which shows that group C+ (9.1 µm) was more rigid than group C– (18.9 µm). For groups without inlays (GIC and CR), a smaller displacement was seen with resin basing (9.24 µm) than with glass ionomer basing (9.62 µm), but both types of materials presented values of maximum displacement near the cavity limits that were much smaller than those presented by group C–.

When glass ionomer basing was simulated, the displacement of the cusps was greater with indirect resin (GIC+IR; 9 µm) compared with the ceramic (GIC+C; 8.3 µm). The basing with resin led to slightly lower values of displacement for both inlays, and for CR+C, the values remained lower (8.1 µm) in relation to indirect resin inlays (CR+IR; 8.7 µm).

## DISCUSSION

Premolar teeth were chosen for this study because of their greater tendency for cusp deflection when subjected to occlusal stress. In clinical investigations, the upper premolars have shown a higher fracture tendency than lower premolars[18] and are the posterior teeth with the highest incidence of fracture.[19,20] The reasons for such findings are probably their location in areas of intense chewing load in combination with lower amounts of structure when compared to molars. Thus, there is a greater disproportion between crown/root structure and its cervical constriction.

The load application can be at three different points: on the restorative material, the interface, or the enamel. The results depend on this point of load application.[21] In the present study, application on the tooth was made to investigate the worst flexural condition for the remaining cusps, subjecting the axial adhesive interface to the highest intensity of stress. In such conditions, the sphere-tooth contact worked as a wedge between the buccal and lingual cusps in the nonrestored teeth, reducing the mean values of load at fracture and promoting more catastrophic fracture types.

A previous study[6] found that performing an extensive inlay preparation did not cause a statistically significant decrease in fracture load when compared to conservative preparations. The restorative material had an influence in the extensive inlays, where the higher elastic modulus ceramic material (103.55±15.84 kgf) resulted in a fracture load greater than inlays made of resin (65.42±10.15 kgf). Therefore, it was decided to test whether these materials would have an effect in the situation of greater tooth fragility, in which the extensive MOD preparation was also associated with weakened cusps and endodontic treatment.

The propagation of the fracture within dentin is dependent on the shape, composition, and properties of the restorative material adjacent to the fracture.[3] The higher the elastic modulus of the restorative material, the less the dental structures deform under the same stress.[21]

For direct bases, GIC and composite resin were selected. According to Yap and others,[22] the GIC has properties that make it very useful as a base material. Some of these features include the coefficient of linear thermal expansion being near to that of tooth structure, the promotion of chemical coupling to enamel and dentin, and the release of fluoride ions. However, the present study found that all the specimens filled with GIC showed fractures with larger tooth portions (type V fractures). A lower elastic modulus (7300 MPa) and lower bond strength to dentin and resin cement compared with composite resin may be factors that explain how this material resulted in fractures with poorer clinical prognosis.

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Both types of direct base, in spite of not significantly changing the resistance values when compared with teeth without base or inlay (C–), could impact the crack that propagated, resulting in the more favorable type of fracture for these groups.

The FEA indicated a much greater concentration of stresses on the lingual cusp in both unprepared and prepared teeth. These results are in agreement with our experimental test, which found a higher frequency of fracture in the lingual cusps, probably due to their anatomy.

The presence of extensive MOD preparation, weakened cusps, and endodontic treatment (group C–) increased the stress concentration within the tooth structure (Figure 4). This stress developed markedly in the cervical region, near the gingival wall of the preparation, which explains the pattern of fracture that produced the largest loss of tooth (100% of the fractures were type V, involving cusps from the gingival wall to the cemento-enamel junction; Figure 3). But when direct bases and adhesive inlays were performed, there was strengthening of dental structures. Ceramic inlays with a direct resin base showed better stress distribution (Figure 4), which can also be related to the smallest displacement of this group (8.1 μm; Figure 5). As the adhesive interfaces were simulated with a perfect bonded contact, the material with higher stiffness resulted in a smaller displacement of the cusps, generating lower stress concentration, consistent with the increased fracture load seen in the laboratory tests.

The combination of experimental test and FEA supported that the approximations made in the modeling of the structures were effective for analyzing complex structures, such as the case of treated endodontic teeth. However, in spite of all the advantages of this method, it should be kept in mind that the accuracy of the results, among other factors, depends on the degree of simplification of the geometries involved, the quality of multibody interaction, and the accumulation of mathematical approaches. It is possible that the FEA has overestimated the difference between group C– and the other groups because in the experimental test there were nonlinear phenomena, such as crack propagation, failure in the interface, the presence of preexisting cracks, accommodation of a spherical tip within the teeth, and other behaviors of the structures that were not simulated in the analysis.

The defects created in the weakened cusps and endodontic treatment led to a decrease of 82.02% in the fracture load compared to the group of healthy teeth (Figure 3) and 38.8% when compared to the MOD without weakened cusps or endodontic treatment (569.18 N) obtained by Costa and others.[6] This research did not test covered cusp groups because the aim was to preserve as much tooth structure as possible applying adhesive material techniques. However, this extreme condition did not result in the reinforcement ability of ceramic restorations. It is likely that in smaller defects, the method for assessing the resistance would have greater sensitivity to detect significant differences between the performances of the materials.

Scotti and others[23] showed that cuspal coverage should provide improved fracture resistance in maxillary premolars, especially when the residual wall thickness is less than 2 mm. In our study, the remaining wall thickness was 2 mm, so the effect of weakening was highlighted. The restorations without cuspal coverage, when subjected to axial loads produced by dental contact, induce a wedge effect, leading to a deflection of the cusps. This becomes more critical in a posterior tooth where there is the loss of major dental structures, especially the marginal ridges, enamel ridges, and the roof of the pulp chamber.[24]

Nevertheless, we must emphasize that the applied loads used in this study were of a static nature in contrast to the repeated dynamic loads teeth are exposed to in the oral cavity. Typically, the dynamic simulation is used when one wants to analyze fatigue or crack propagation. In our study, we tried to relate the FEA with the experimental test, in which a static load was applied. In the case of static loading, we can verify stress concentrations and correlate them to the fracture pattern. Also, with the stress concentration in the static analysis performed by FEA, it is possible to correlate it with the maximum fracture load obtained in the experimental test: the group with the highest stress concentration in FEA should fail under lower forces in the *in vitro* test. In this study, thermocycling was not carried out before the failure tests because we tried to isolate the effect of mechanical properties of the materials on the resistance of the tooth/restoration complex, creating the fewest possible number of defects in the adhesive interface, which could interfere in the experimental results. With thermocycling, the materials are expected to present different coefficients of linear thermal expansion, producing defects at the interface. Also, the results obtained by the experimental tests could be better correlated with the FEA findings, in which a perfect

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bonded adhesive interface was simulated. In future work, we suggest performing thermocycling, more closely simulating the oral environment, to clarify the influence of temperature changes on the fracture load of these materials.

A critical factor in evaluating the results of tests with extracted teeth is to know how they may have been impacted by extraction.[25] This fact, together with the variability of dental anatomy itself, makes it difficult to standardize the study and may explain the high values of standard deviation. Studies should be conducted to improve understanding of the multiple variables existing when using natural objects with the aim of decreasing the variability of results and promoting an increase in the power of statistical tests to detect significant differences.

## CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

- Sound teeth showed higher fracture load than groups having endodontic treatment and weakened cusps.
- Direct bases without inlays were not able to increase the values of fracture, independent of the type of material.
- Using direct bases and inlays increased the values of fracture load but resulted in lower values compared to healthy teeth, independent of the combination of materials.
- No difference was found in fracture load of teeth according to the types of direct base material (GIC or composite resin) or inlay (ceramic or indirect resin).

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### Human Subjects Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies. The approval code for this study is 046/2011-PH/CEP.

### 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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# Systematic Review and Meta-Analysis of Randomized Clinical Trials on Chemomechanical Caries Removal

HHH Hamama • CKY Yiu • MF Burrow
NM King

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### Clinical Relevance

The sodium hypochlorite–based (Carisolv) chemomechanical caries removal method is more time consuming than the enzyme-based (Papacarie) chemomechanical caries removal method.


## SUMMARY

**Objectives: The aim of this review was to assess the methodologies used in previously published prospective randomized clinical trials on chemomechanical caries removal and to conduct a meta-analysis to quantify the differences in the excavation time between chemomechanical and conventional caries removal methods.**

**Methods: An electronic search was performed using Scopus, PubMed, EBSCO host, and Cochrane Library databases. The following categories were excluded during the assessment process: non-English studies published before 2000, animal studies, review articles, laboratory studies, case reports, and nonrandomized or retrospective clinical trials. The methodologies of the selected clinical trials were assessed. Furthermore, the reviewed clinical trials were subjected to meta-analysis for quantifying the differences in excavation time between the chemomechanical and the conventional caries removal techniques.**

**Results: Only 19 randomized clinical trials fit the inclusion criteria of this systematic review. None of the 19 reviewed trials completely fulfilled Delphi's ideal criteria for quality assessment of randomized clinical trials. The meta-analysis results revealed that the shortest mean excavation time was recorded for rotary caries excavation (2.99±0.001 minutes), followed by the enzyme-based chemomechanical caries removal method (6.36±0.08 minutes) and the the hand excavation method (atraumatic restorative technique; 6.98±0.17 minutes). The longest caries excavation time was**


Hamdi HH Hamama, BDS, MDS, PhD, Clinical Assistant Professor, Aesthetic and Restorative Dentistry, Faculty of Dentistry, Mansoura University, Egypt

*Cynthia KY Yiu, BDS, MDS, PhD, clinical professor, Paediatric Dentistry and Orthodontics, Faculty of Dentistry, University of Hong Kong, Hong Kong SAR, China

Michael F Burrow, BDS, MDS, PhD, MEd, MRACDS (Pros), FRACDS, professor and chair of Biomaterials, Melbourne Dental School, The University of Melbourne, Australia.

Nigel M King, Winthrop professor, School of Dentistry, Oral Health Centre of Western Australia, University of Western Australia, Perth, Australia

*Corresponding author: Prince Philip Dental Hospital, 34 Hospital Rd., Hong Kong SAR, China; e-mail: ckyyiu@hkucc.hku.hk

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**recorded for the sodium hypochlorite-based chemomechanical caries removal method (8.12±0.02 minutes).**

**Conclusions: It was found that none of the current reviewed trials fulfilled all the ideal requirements of clinical trials. Furthermore, the current scientific evidence shows that the sodium hypochlorite-based (Carisolv) chemomechanical caries removal method was more time consuming when compared to the enzyme-based (Papacarie) chemomechanical and the conventional caries removal methods. Further prospective randomized controlled clinical trials evaluating the long-term follow-up of papain-treated permanent teeth are needed.**

## INTRODUCTION

Evidence-based dentistry is "the integration and interpretation of the current research evidence, combined with personal experience, which allows clinicians and academic researchers to make judgments, decisions and improve their clinical practice."[1] Systematic reviews and meta-analytical studies are considered the highest level of evidence that support evidence-based decision making, which is the "formalized process of using a specific set of skills for identifying, searching for and interpreting clinical and scientific evidence, so that it can be used at the point of care."[2]

Chemomechanical caries removal is considered one of the most conservative and convenient caries removal methods.[3] Early studies reported that the sodium hypochlorite–based (NaOCl) chemomechanical caries removal method was a time-consuming process and that its effectiveness was questionable.[4,5] However, this caries removal method has been further developed in the past 10 years by modifying the chemical formula of the original chemomechanical caries removal agents (eg, new Carisolv gel) as well as the introduction of new generations of chemomechanical caries removal agents (eg, enzyme-based agents, such as Papacarie and Biosolv).

Many randomized clinical trials have been performed to evaluate the effect of chemomechanical caries removal agents on excavation time, pain level, and the long-term success rate of restored cavities.[6-12] However, these trials have shown great variability among their objectives, methodologies, and results. In the literature, very few narrative reviews have focused on chemomechanical caries removal methods.[4,5,13] Until now, there appears to be no systematic review or meta-analytical study that has evaluated the clinical trials using chemomechanical caries removal methods. Hence, the evidence supporting the use of the chemomechanical caries excavation method as a useful and efficacious technique remains weak.

The aim of this review was to assess the methodologies used in previously published prospective randomized clinical trials on chemomechanical caries removal and to conduct a meta-analysis for quantifying the difference in the excavation time between the chemomechanical and conventional caries removal methods. The key questions of this systematic review were these: Did the methodologies followed in the previously published studies fulfill the ideal requirements of the prospective randomized clinical trial, and is the chemomechanical caries removal method a time-consuming process compared with the other conventional caries removal methods?

## METHODS AND MATERIALS

### Protocol Development and Eligibility Criteria

The protocol of this systematic review was designed following the guidelines of the Preferred Reporting Items Systematic Review and Meta-Analysis statement.[14] The clinical trials selected for the current systematic review were at least two-arm prospective randomized clinical trials (RCT) and written in English. For all the selected RCT on NaOCl-based chemomechanical caries removal agents, the modified Carisolv gel (5% NaOCl) has been used. For the selected RCT on the enzyme-based chemomechanical caries removal agents, either papain-based (Papacarie) or trypsin-based (Biosolv) agents have been used. The two chemomechanical caries removal methods were then compared with a conventional hand excavation atraumatic restorative technique [ART] method) or rotary caries removal methods.

### Information Source and Search

An electronic search was done by one of the authors (HH) using the following databases: Scopus, PubMed, EBSCO host, and Cochrane Library. The online searching was conducted following this web-search criteria: "Chemomechanical Caries Removal" or "Chemomechanical Caries Excavation" or "Papacarie" or "Papain" or "Carisolv" or "Biosolv." Moreover, a parallel hand search was done through nonelectronic journals (eg, the *American Journal of Dentistry*, the *European Archives of Paediatric Dentistry*, and the *Brazilian Journal of Oral Sciences*).

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### Study Selection, Exclusion Criteria, and Article Assessment Process

Assessment of the clinical trial depended on title, abstract, and full text (if needed). All articles found by both electronic and hand searching were collected onto one sheet and printed as three copies that were distributed among the three authors. Each author, individually, checked the eligibility criteria for each study, and the agreement of at least two authors was essential for exclusion/inclusion of the clinical trial for the systematic review. The selected trials were discussed and selections matched in face-to-face meetings. The following categories were excluded during the assessment process: non-English studies published before 2000, animal studies, review articles, laboratory studies, case reports, and nonrandomized or retrospective clinical trials (RCT; Figure 1). Moreover, all the RCT, which were performed using either the Caridex or the old version of the Carisolv (2.5% NaOCl) chemomechanical caries removal agent, were excluded.

The objectives, results, and conclusions of each clinical trial were summarized and evaluated. Furthermore, the methodology of each RCT was assessed, based on the Delphi's ideal criteria for quality assessment of randomized clinical trials, from different aspects, namely, randomization, sample size, type of subjects, rubber dam application, study design, ethical- related issues, degree of blindness of the evaluation process, performance of an intraexaminer calibration test, methodology of clinical examination and long-term clinical follow-up.

### Meta-Analysis of Mean Caries Excavation Time

The majority of the RCT evaluated the time taken for caries excavation by chemomechanical (test group) and conventional (control group) caries removal methods. For each caries excavation method, the sample size and the mean caries excavation time (minutes) were extracted from the studies and subjected to a meta-analysis using Comprehensive Meta-Analysis software, version 2 (Biostat, Englewood, NJ, USA), at the 95% confidence interval. The meta-analysis of this systematic review followed the statistical model of Borenstein[15], which has been designed for comparing the meta-analysis outcomes of different groups within the same study. Thus, the results of the meta-analysis were subjected to a further one-way analysis of variance, followed by the Tukey *post hoc* multiple comparison test using GraphPad Instat software, version 3.10 (GraphPad Software, Inc, San Diego, CA, USA). This additional step was performed to quantify the difference in the excavation time between the conventional and the chemomechanical caries removal methods.

## RESULTS

The initial search through the Science Direct database resulted in 157 articles being identified and was then followed by a subsequent search of the three other databases in addition to the manual searching. This added a further article; hence, the total number of originals screened was 158. Twelve articles were excluded because they were not written in English. Moreover, another 45 studies were excluded because they utilized either Caridex or the old version of the Carisolv gel. From the remaining 101 articles, nine were animal based, three were reviews, and 52 were laboratory studies, all of which were excluded. Another six studies were excluded because they utilized chemomechanical caries removal agents for other purposes not related to caries excavation, such as treatment of oral ulcers,[16] periodontal therapy,[17,18] cleaning of organic debris prior to application of pit and fissure sealants,[19] plaque removal,[20] and root canal irrigation.[21] From the remaining 31 clinical studies, three were case reports, and seven were nonrandomized clinical trials and were therefore excluded. Two clinical trials were further excluded, because they were conducted on special needs patients.[22,23] Finally, 19 randomized clinical trials fit the inclusion criteria of this systematic review. The detailed study selection procedure is illustrated in Figure 1.

The selected clinical trials had the following geographic distribution; nine were performed in Europe (47%), four in India (21%), three in South America (17%), and one each in Pakistan (5%), the United States (5%), and Egypt (5%). Fourteen clinical trials were performed using the NaOCl-based (Carisolv) agent (74%), while three RCT used papain-based (Papacarie; 15.5%). Moreover, two clinical trials by Bohari and others[12] and Kochhar and others[24] compared NaOCl-based (Carisolv) and papain-based (Papacarie) chemomechanical caries removal agents (10.5%). Fourteen of the 19 clinical trials were conducted on deciduous teeth (74%), while the remaining five were conducted on permanent teeth (26%). Only the study by Peric and others[25] compared the effect of chemomechanical caries removal agents on both dentitions (5%). Long-term clinical follow-up was performed in only seven of the clinical trials (37%) fulfilling the search criteria, while the remaining 12 clinical trials

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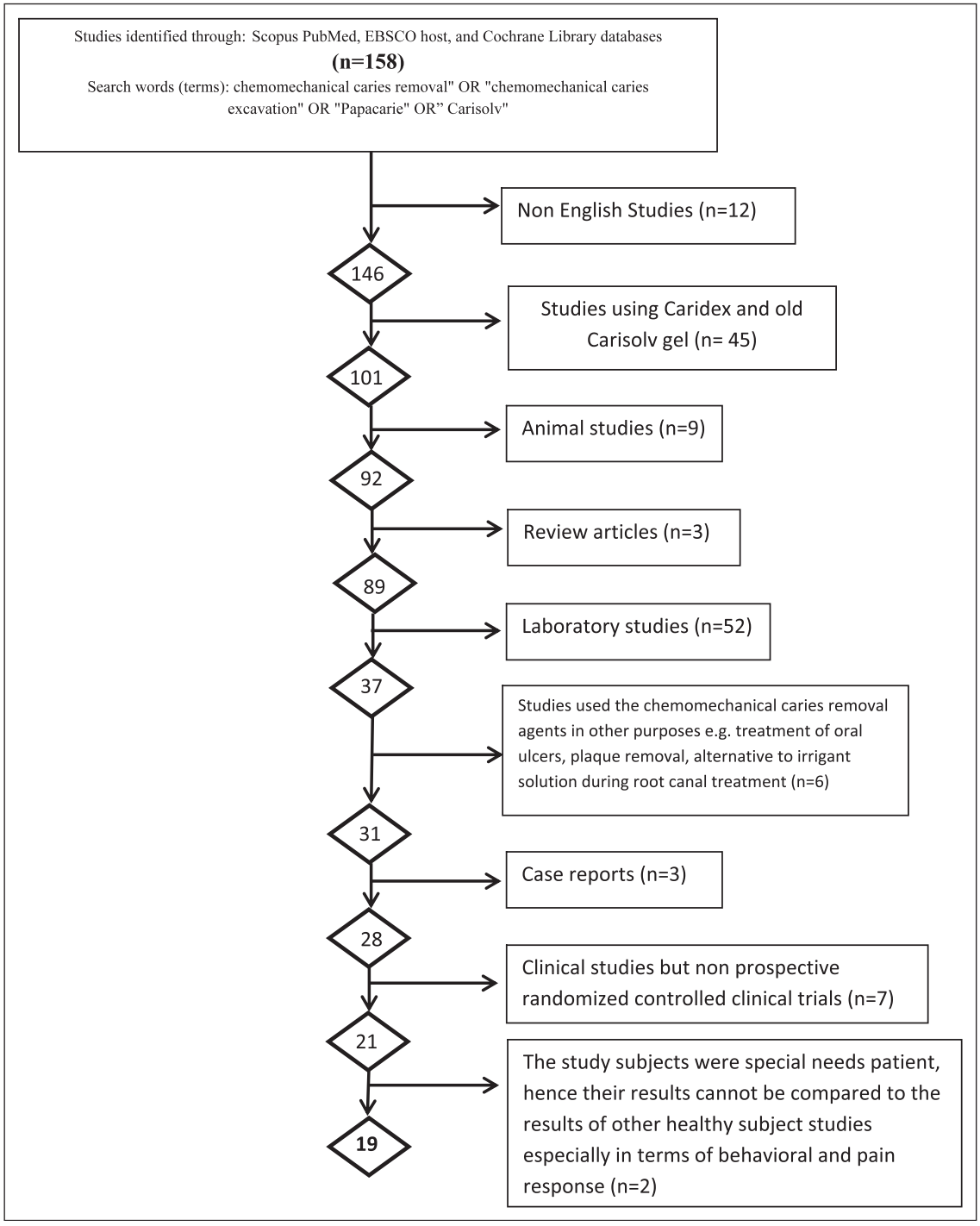


Figure 1. *Flow chart of the study selection procedure.*

published only the immediate short-term outcomes (Table 1).

The assessment of the methodologies used in the reviewed clinical trials is summarized in Table 2. Thirteen of the 19 clinical trials (68%) clearly mentioned the randomization protocols of their study population, while the remaining six did not. Two clinical trials[12,26] out of the 19 did not mention the total number of the study population, and only one clinical trial, by Topaloglu-Ak and others,[10] showed that a sample size calculation method had been used. Furthermore, two clinical trials out of the 19 did not equalize the number of subjects in both

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Table 1: *Summary of the Search Characteristic Findings of the Clinical Trials*

| | | | | | | |
|---|---|---|---|---|---|---|
| Geographic Distribution | Europe 9 (47%) | India 4 (21%) | South America 3 (17%) | Egypt 1 (5%) | Pakistan 1 (5%) | United States 1 (5%) |
| Chemomechanical caries removal agent | Carisolv 14 (74%) | | Papacarie 3 (15.5%) | | Carisolv and Papacarie 2 (10.5%) | |
| Tooth subject | Deciduous 14 (74%) | | Permanent 5 (26%) | | Deciduous and permanent 1 (5%) | |
| Follow-up | Performed 7 (37%) | | | Not performed 12 (63%) | | |
| Study design | Split-mouth 10 (53%) | | Parallel 5 (26%) | | Not clearly mentioned 4 (21%) | |

the test and the control groups.[8,27] The majority (15 RCT [79%]) of the clinical trials mentioned the inclusion/exclusion criteria; however, the remaining four (21%) did not clearly present the eligibility criteria for subject selection. Among the selected clinical trials, seven (37%) used a rubber dam during the restorative procedures, and nine (47%) did not, while the remaining three (16%) failed to clearly mention the isolation method used.

Among the reviewed clinical trials, 10 (53%) used a split-mouth design, five (26%) used a parallel design, and four (21%) did not clearly mention the design. Eleven (58%) of the 19 clinical trials indicated that ethical approval was obtained from an appropriate institute, while the remaining eight studies did not clearly indicate the ethical-related issues. Three trials did state, however, that signed informed consents from the child's parents or guardians were obtained.[6,9,28]

As shown in Table 2, the final evaluation was performed by one investigator in six (32%) clinical trials and by two investigators in 5=five (26%) clinical trials. Furthermore, the remaining eight studies (42%) did not clearly mention the number of investigators who participated in the study. An intraexaminer calibration test was performed in all of the two-investigator clinical trials, except the study by Bergmann and others.[29] The blinding of the evaluators to the treatment method was reported in only four trials (21%), while the remaining trials did not clearly describe the methods of keeping the principal investigator(s) blinded to the groups tested. Eighteen (94%) of the 19 clinical trials used conventional visual and tactile methods for evaluation of the excavated surfaces. In addition to the tactile and visual methods, caries detector dyes were used in two (11%) trials,[24,30] microbial analysis was used in another two (11%) trials,[7,11] and the DIAGNOdent (KaVo Dental, Biberach, Germany) was used in one (5%) trial.[12] Among the 13 clinical trials that recorded the behavioral responses of the patients before, during, and after treatment, six (46%) trials[11,12,24,30-32] used standardized scales (eg, Face-Leg-Activity-Cry-Consolability [FLACC] scale, Sound, Eyes and Motor [SEM] scale, Visual Analogue and Verbal scale, Frankl scale,[33] and Wong Baker Faces Pain scale). Conversely, the remaining seven trials used questionnaires and subjective methods to record the behavioral and pain responses of the study subjects.[6,8,9,25,27,29,34]

The results of the meta-analysis of the mean caries excavation time for rotary, ART, NaOCl-based (Carisolv), and papain-based (Papacarie) gels are shown in Figures 2 through 5, respectively. According to statistical model by Borenstein and others,[15] results of one-way analysis of variance and the Tukey *post hoc* test revealed that the shortest estimated mean excavation time was recorded during rotary caries excavation (2.99±0.001 minutes), followed by the papain-based (Papacarie) chemomechanical caries removal method (6.36±0.08 minutes) and the hand excavation method (ART; 6.98±0.17 minutes). The longest caries excavation time (8.12±0.02 minutes) was recorded for the NaOCl-based (Carisolv) chemomechanical caries removal method (Table 3).

## DISCUSSION

Those clinical trials that used Caridex and the old version of Carisolv (0.25% NaOCl) were excluded due to limited clinical usage of these chemomechanical caries removal agents as well as their non-availability in the dental market.[5] Furthermore, their relative long excavation time may have affected the meta-analysis results.

Initially, it was decided to restrict the search to the long-term follow-up trials. However, after screening all long-term follow-up trials, it was found that most of them reported no significant difference between the immediate and the long-term follow-up outcomes; thus, all the immediate follow-up trials were

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Table 2: *Evaluation of the Methodologies Used in the Clinical Trials Reviewed*[a]

| Study | Sample Size | Inclusion and Exclusion Criteria | Application of Rubber Dam | Study Design |
|---|---|---|---|---|
| Bohari and others[12] | 120 primary molars | ✓ | ✓ | Parallel |
| Kochhar and others[24] | 120 primary molars | ? | ✓ | Parallel |
| Singh and others[11] | 80 primary molars | ✓ | ✓ | Split-mouth |
| Bussadori and others[26] | 14 permanent molars | ✓ | X | ? |
| Kotb and others[32] | 74 primary molar | ✓ | X | Split-mouth |
| Peric and others[25] | 120 primary and permanent molars | ✓ | X | Parallel |
| Topaloglu-Ak and others[10] | 327 primary molars | ✓ | X | Parallel |
| Subramaniam and others[41] | 40 primary molars | ? | ✓ | Split-mouth |
| Barata and others[35] | 100 permanent molars | ✓ | X | Split-mouth |
| Hosein and Hasan[28] | 60 permanent molars | ✓ | ? | Split-mouth |
| Pandit and others[30] | 150 primary molars | ? | X | ? |
| Kirzioglu and others[34] | 56 primary teeth | ✓ | X | Split-mouth |
| Peters and others[27] | 50 primary teeth | ✓ | X | Parallel |
| Lozano-Chourio and others[31] | 80 primary teeth | ✓ | ✓ | Split-mouth |
| Bergmann and others[29] | 92 primary molars | ? | X | Split-mouth |
| Fure and Lingstrom[8] | 202 permanent molars | ✓ | ✓ | ? |
| Azrak and others[7] | 42 primary molars | ✓ | ? | Split-mouth |
| Kavvadia and others[9] | 92 primary molars | ✓ | Not in all the cases | ? |
| Kakaboura and others[6] | 90 permanent molars | ✓ | ? | Split-mouth |

[a] *✓ yes; X, no; ?, not clearly stated.*
*Abbreviations: A, rotary caries removal; B, hand excavation (atraumatic restorative technique [ART]); C, sodium hypochlorite (NaOCl)-based (Carisolv) chemomechanical caries removal; D: papain-based (Papacarie) chemomechanical caries removal; E, laser caries removal; FLACC, Face-Leg-Activity-Cry-Consolability scale; MH, Mount and Hume classification of the contact area; SEM, Sound, Eyes and Motor scale; USPHS, US Public Health Service; VAV, Visual Analogue and Verbal scale; WBFP, Wong Baker Faces Pain scale.*

included in the current review.[8,10,25,26,29,34,35] The two clinical trials by Carrillo and others[22] and Guare Rde and others,[23] which were conducted on special needs subjects, were excluded because their results should not be compared with the healthy subjects, particularly in terms of analyzing behavioral and pain responses.

It was noticed that around 50% of the clinical trials were performed in Europe, which could be attributed to the early availability of Carisolv in this region. Most of the trials (~85%) utilized the NaOCl-based (Carisolv) chemomechanical agent due to its popularity and knowledge of its existence. Conversely, only a few clinical trials used the papain-based (Papacarie) chemomechanical caries removal agent due to its recent introduction to the market.[11,12,24,26,32]

None of the 19 reviewed trials completely fulfilled Delphi's ideal criteria for quality assessment of randomized clinical trials, which was published in

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Table 2: Extended.

| Study | Study Groups | | Obtaining Ethical Approval and Signed Informed Consent | No. of Investigators | Blindness of the Investigator |
|---|---|---|---|---|---|
| | Control | Test | | | |
| Bohari and others[12] | A | C, D, and E | ✓&✓ | ? | ? |
| Kochhar and others[24] | A | B, C, and D | ✓&? | ? | ? |
| Singh and others[11] | A | D | ✓&✓ | 1 | ? |
| Bussadori and others[26] | X | D | ✓&✓ | ? | ? |
| Kotb and others[32] | A | D | ?&? | 1 | ? |
| Peric and others[25] | A | C | ✓&✓ | 1 | ✓ |
| Topaloglu-Ak and others[10] | B | (B+C) | ✓&✓ | 2 | ✓ |
| Subramaniam and others[41] | A | C | ?&? | ? | ? |
| Barata and others[35] | B | C | ✓&✓ | 2 | ✓ |
| Hosein and Hasan[28] | A | C | ?&✓ | 1 | ? |
| Pandit and others[30] | A | B and C | ?&? | ? | ? |
| Kirzioglu and others[34] | B | C | ✓&✓ | 1 | ? |
| Peters and others[27] | A | C | ✓&✓ | 1 | ? |
| Lozano-Chourio and others[31] | A | C | ✓&✓ | 1 for assessment of caries removal<br>2 for assessment of cavity entrance size | ✓ |
| Bergmann and others[29] | A | C | ✓&✓ | 1 investigator per center | ? |
| Fure and Lingstrom[8] | C (0.25% NaOCl) Carisolv gel | C (0.5% NaOCl) Carisolv gel | ✓&✓ | ? | ? |
| Azrak and others[7] | A | C | ?&? | ? | ? |
| Kavvadia and others[9] | A | C | ?&✓ | ? | ? |
| Kakaboura and others[6] | A | C | ?&✓ | ? | ? |

[a] ✓ *yes; X, no; ?, not clearly stated.*
*Abbreviations: A, rotary caries removal; B, hand excavation (atraumatic restorative technique [ART]); C, sodium hypochlorite (NaOCl)-based (Carisolv) chemomechanical caries removal; D: papain-based (Papacarie) chemomechanical caries removal; E, laser caries removal; FLACC, Face-Leg-Activity-Cry-Consolability scale; MH, Mount and Hume classification of the contact area; SEM, Sound, Eyes and Motor scale; USPHS, US Public Health Service; VAV, Visual Analogue and Verbal scale; WBFP, Wong Baker Faces Pain scale.*

1998.[36] The trial by Topaloglu-Ak and others[10] is the only clinical trial that fulfilled most of Delphi's criteria: the sample size calculation method, the number of operators and investigators, and blindness of the investigators to the treatment was clearly stated. However, the detailed randomization and teeth isolation methods were not clearly reported, which may have affected the follow-up results of the resin composite restorations. The trial by Barata and others[35] was the only clinical trial that clearly described the detailed randomization method. All the reviewed trials were at least two-arm clinical trials, except the study by Bussadori and others,[26] which was a one-arm trial. This trial was included in the current review because it was the only clinical study that has been conducted on permanent teeth using enzyme-based (Papacarie) with a long-term follow-up of 24 months. However, this trial was not included in the meta-analysis because it is a one-arm trial and the caries excavation time was not evaluated.

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Table 2: Extended.

| Study | Intraexaminer Calibration Test | Clinical Examination Criteria | Use of Standardized Scales to Record Patient Response | Follow-Up |
|---|---|---|---|---|
| Bohari and others[12] | ? | Tactile and DIAGNOdent | ✓ FLACC | X |
| Kochhar and others[24] | ? | Ericson scale and caries detector dye | ✓ VAV | X |
| Singh and others[11] | NA | Erickson's criteria and microbial evaluation | ✓ WBFP | X |
| Bussadori and others[26] | ? | Visual and tactile methods | NA | 6, 12, and 24 mo |
| Kotb and others[32] | NA | Tactile | ✓ SEM | X |
| Peric and others[25] | NA | Tactile | X Questionnaire | 1 wk, 6 mo, and 12 mo |
| Topaloglu-Ak and others[10] | ✓ | MH[42] | NA | 6, 12, and 24 mo |
| Subramaniam and others[41] | ? | Tactile | NA | X |
| Barata and others[35] | ✓ | ? | NA | 12 mo |
| Hosein and Hasan[28] | NA | Tactile | NA | X |
| Pandit and others[30] | ? | Ericson scale and caries detector dye | ✓ VAV | X |
| Kirzioglu and others[34] | NA | Tactile | X Questionnaire | 3, 6, 9, and 12 mo using USPHS Ryge[43] criteria |
| Peters and others[27] | NA | Tactile | ? | X |
| Lozano-Chourio and others[31] | NA ✓ | Tactile | ✓ Frankl and others[33] scale | X |
| Bergmann and others[29] | ? | Tactile | X Questionnaire | 6 mo |
| Fure and Lingstrom[8] | ? | Tactile | X Questionnaire | 1 yr |
| Azrak and others[7] | ? | Tactile and microbial evaluation | NA | X |
| Kavvadia and others[9] | ? | Tactile | Subjective scale | X |
| Kakaboura and others[6] | ? | Tactile | X Questionnaire | X |

[a] ✓ *yes; X, no; ?, not clearly stated.*
*Abbreviations: A, rotary caries removal; B, hand excavation (atraumatic restorative technique [ART]); C, sodium hypochlorite (NaOCl)-based (Carisolv) chemomechanical caries removal; D: papain-based (Papacarie) chemomechanical caries removal; E, laser caries removal; FLACC, Face-Leg-Activity-Cry-Consolability scale; MH, Mount and Hume classification of the contact area; SEM, Sound, Eyes and Motor scale; USPHS, US Public Health Service; VAV, Visual Analogue and Verbal scale; WBFP, Wong Baker Faces Pain scale.*

The sizes of the test and control groups in the clinical trials by Fure and Lingstrom[8] and Peters and others[27] were not equal in size, which may have affected the accuracy of the statistical analysis. Also, the results of the multi-comparison analysis of the study by Bohari and others[12] were not well presented in the manuscript. All reviewed trials used conventional visual and tactile sensation for the evaluation of the excavated surfaces. In addition to conventional diagnostic means, Bohari and others[12] used the DIAGNOdent for quantifying the results. Furthermore, the calibration method of the DIAGNOdent was clearly described in the study. Pandit and others[30] and Kochhar and others[24] used caries detector dyes for verifying complete caries removal; however, the use of caries detector dyes may give false-positive results.[37-39] These false results were attributed to the nonspecificity of the dye to damaged collagen fibers of the infected dentin and resulted in staining of demineralized caries-affected

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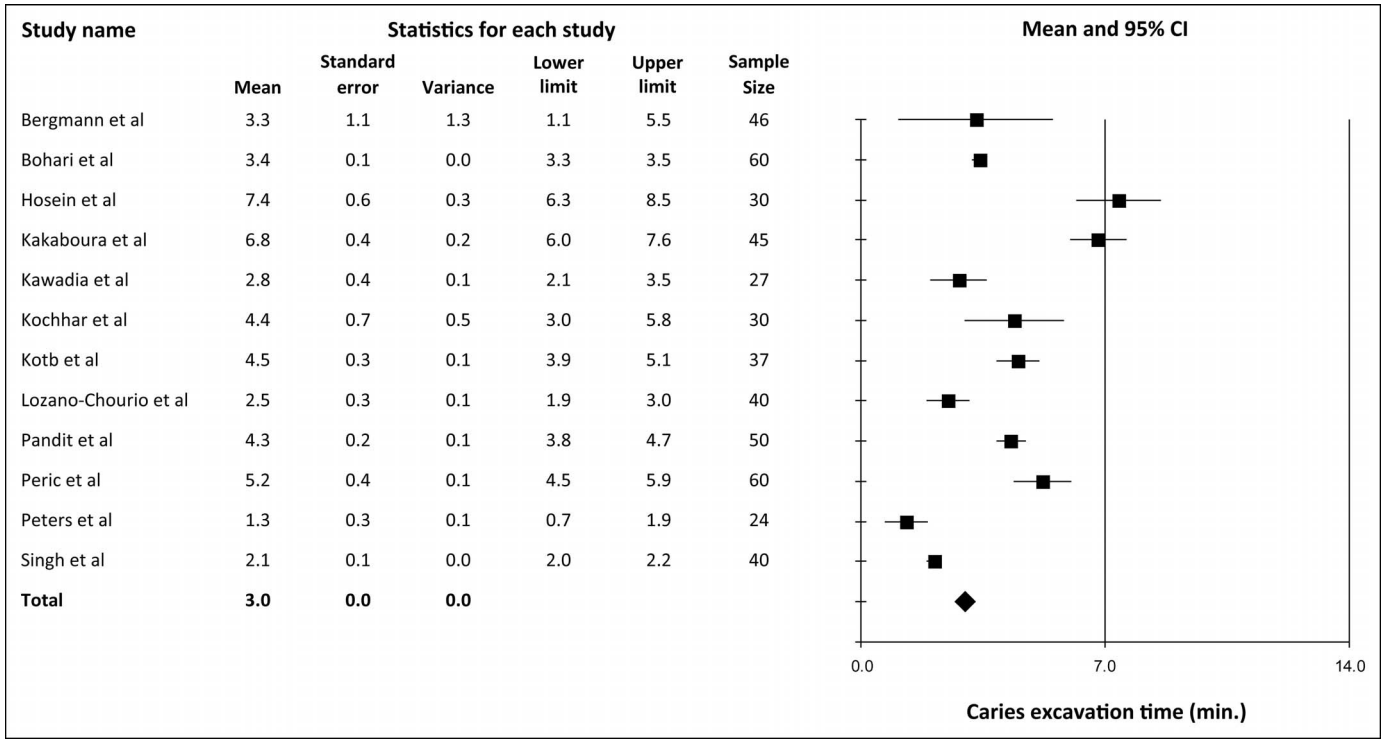


Figure 2. *Meta-analysis results of the mean excavation time for the conventional rotary caries removal method.*

dentin.[37,38,40] Also, microbial analysis of pre- and postexcavation samples were used in two trials for assessment of the efficacy of the caries removal process.[7,11]

Some trials used subjective patient behavioral and pain response assessment methods (questionnaires).[6,8,9,29,34] Conversely, the other trials[11,12,24,30-32] used standardized scales (eg, FLACC, SEM, Visual Analogue and Verbal scale, Frankl scale,[33] and Wong Baker Faces Pain scale), increasing the reliability of their results due to their unbiased outcomes. The majority of trials[8,10-12,24,25,27,29,31,34,35] obtained ethical approval from their respective institutes or at least signed informed consent by the child's parent or guardian.[6,9,28] However, four trials[7,30,32,41] did not clearly mention any ethical related issues, which may have affected the confidence toward their methodologies and results.

Only seven of the 19 trials performed long-term follow-up of the treated cases.[8,10,25,26,29,34,35] The trial by Topaloglu-Ak and others[10] was the only one that described the long-term follow-up (6, 12, and 24 months), which was performed under standard illumination conditions (battery-powered headlight).

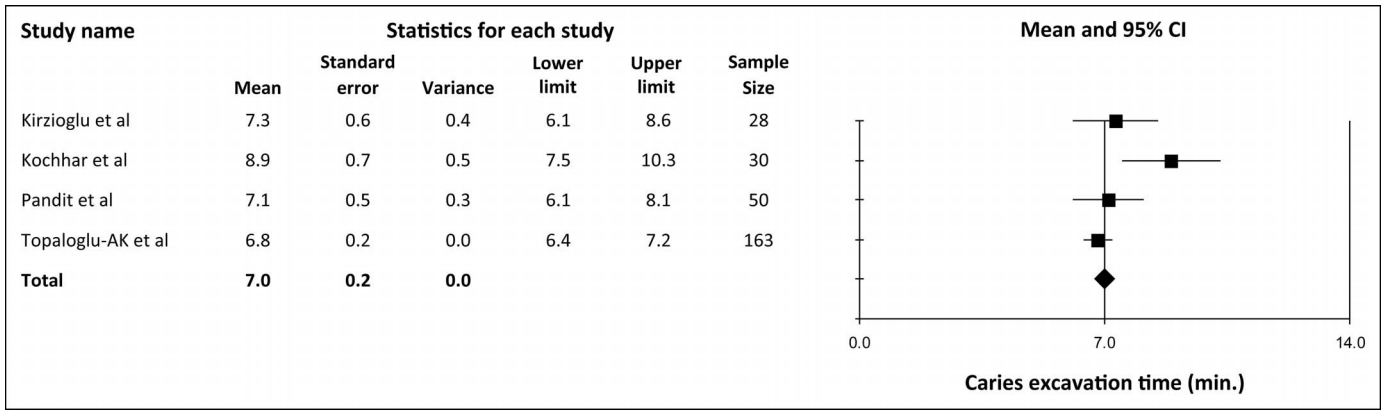


Figure 3. *Meta-analysis results of the mean excavation time for the hand excavation (ART) caries removal method.*

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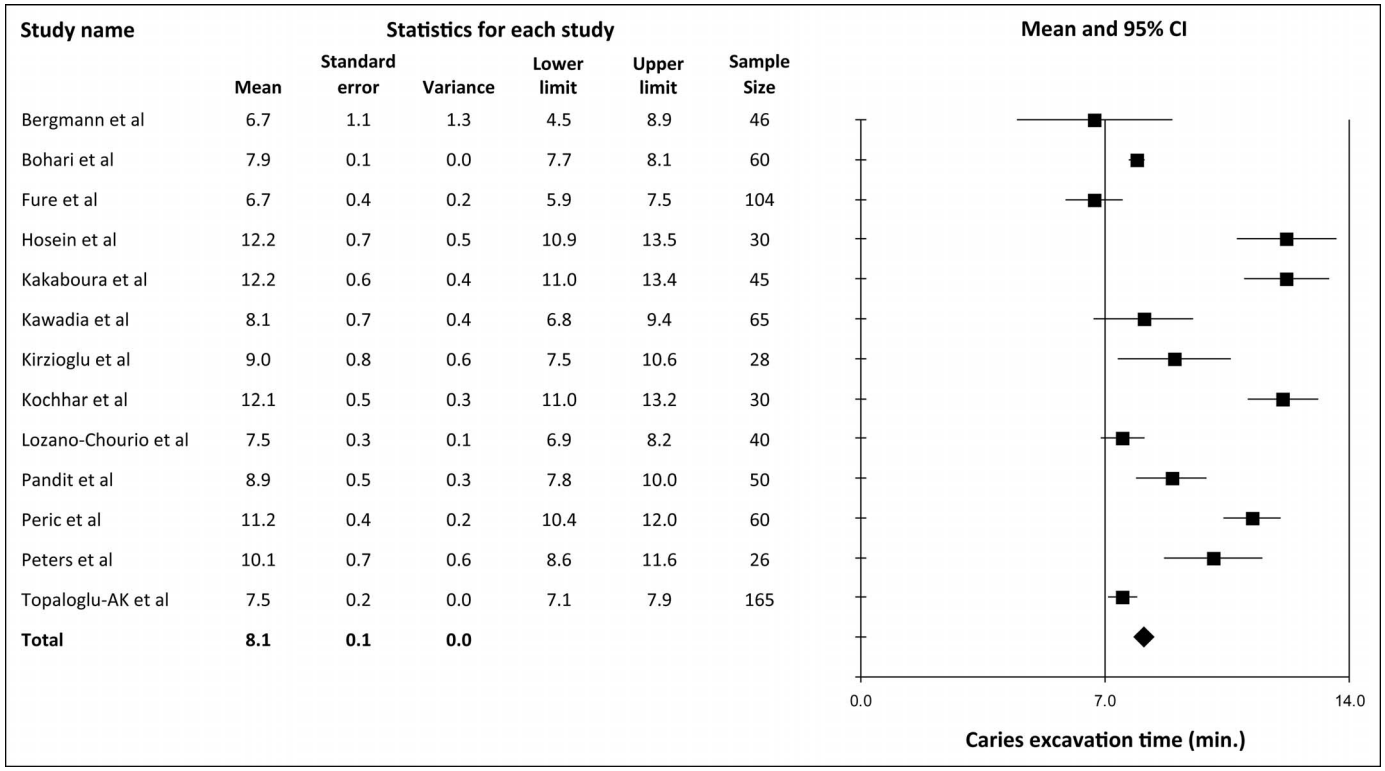


Figure 4. *Meta-analysis results of the mean excavation time for NaOCl-based (Carisolv) chemomechanical caries removal method.*

Bergmann and others[29]and Peric and others[25] did not restore the treated cavities with the same restorative material (amalgam, glass ionomer, or resin composite were used), which may have impacted the follow-up results. Bussadori and others[26] followed up the restored cavities radiographically using periapical radiograph films. However, the method of standardizing the radiographic procedures during the different follow-up sessions was not clearly stated.

The majority of trials reported that NaOCl-based (Carisolv) chemomechanical caries removal is an effective method for caries removal and is more comfortable for patients compared with conventional hand or rotary caries excavation methods. However, most studies also reported that it is a time-consuming method, which is supported by the meta-analysis results. Moreover, trials conducted on papain-based chemomechanical agent (Papacarie) concluded that the enzyme-based chemomechanical caries removal method is considered a viable alternative to the conventional caries removal method and is less time consuming than the NaOCl-based method.

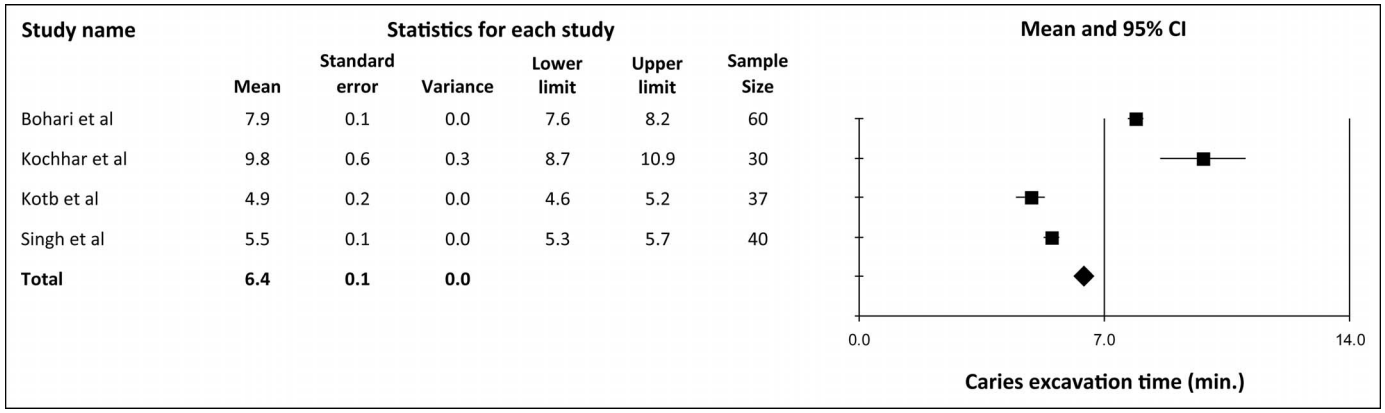


Figure 5. *Meta-analysis results of the mean excavation time for enzyme-based (Papacarie) chemomechanical caries removal method.*

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Table 3: *Analyses of Caries Excavation Time*

| Caries Removal Method | N (Study) | ET Mean ± SD (min)* |
|---|---|---|
| Rotary | 12 | 2.99 ± 0.001 A** |
| Hand excavation (ART) | 4 | 6.98 ± 0.17 C |
| NaOCl-based (Carisolv) chemomechanical caries removal | 13 | 8.12 ± 0.02 D |
| Papain-based (Papacarie) chemomechanical caries removal | 4 | 6.36 ± 0.08 B |

** Results are based on a one-way analysis of variance and the Tukey* post hoc *test of the meta-analysis data following the statistical model of Borenstein and others.*[15]
*** Groups identified by different superscripts were significantly different at* $p<0.05$.
*Abbreviations: ART, atraumatic restorative technique; ET, excavation time; NaOCl, sodium hypochlorite; SD, standard deviation.*

The results of the current review revealed that the number of long-term follow-up clinical trials using Papacarie and conducted on permanent teeth was few in the literature. Therefore, further trials are needed to strengthen the scientific evidence.

### Limitations of the Study

The currently available NaOCl-based (Carisolv) and enzyme-based (Papacarie) chemomechanical caries removal agents originate from non–English-speaking countries, Sweden and Brazil, respectively. Thus, the excluded non-English articles, particularly those written in Swedish and Portuguese, may contain useful information about the clinical usage of both chemomechanical caries removal agents.

## CONCLUSIONS

It was found that none of the current reviewed trials fulfilled all the ideal requirements of clinical trials. Furthermore, the current scientific evidence shows that the NaOCl-based (Carisolv) chemomechanical caries removal method was more time consuming when compared to enzyme-based (Papacarie) chemomechanical and conventional caries removal methods. Further prospective randomized controlled clinical trials evaluating the long-term follow-up of papain-treated permanent teeth are needed.

#### Conflict of Interest

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

*(Accepted 1 August 2014)*

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