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Academy of Operative Dentistry

Award of Excellence

Dr. Thomas J. Hilton

Dr. Thomas J. Hilton

The Award of Excellence of the Academy of Operative Dentistry is given to an individual who has made great contributions to the field of operative dentistry and has achieved a high level of technical skill in its practice. The recipient of the award this year, Dr. Thomas J. Hilton, exemplifies those characteristics.

Tom grew up in Pennsylvania and attended Bucknell University, graduating *Cum Laude* in chemistry in 1974. He then attended the University Of Pennsylvania School Of Dental Medicine and received his DMD degree in 1978.

Following graduation, Tom entered the U.S. Air Force and was stationed at Hahn Air Base in Germany. While at Hahn, Tom sustained a serious injury during a climbing accident and was sent to the regional medical center in Wiesbaden for treatment. It was there that I met Tom, and we began a friendship that has lasted these 37 years. We are fortunate indeed if we can have one friend in a lifetime who can serve as a role model and example of personal integrity and professional accomplishment. I have been blessed with two: Tom, and Dr. Jim Summitt. Jim was the Director of the two-year AEGD program at USAF Medical Center Wilford Hall in those years, and he instilled in both of us the scientific curiosity and drive for clinical excellence that would shape our careers. Tom did manage to mix a little fun into his academic pursuits. While at Wilford Hall, he completed a research project on the effects of flying-induced stress on TMD. Of course, someone had to take multiple rides in the backseat of an F-4 Phantom jet to make the measurements – Tom, of course. Recognizing his aptitude for research, Tom was awarded an Air Force fellowship to study biomaterials at the Oregon Health and Science University School of Dentistry, where he received his MS degree. During his Air Force career, Tom held a number of positions of responsibility in the Air Force, including the directorship of the highly respected USAF Dental Investigative Service. However, I think he would agree that the most meaningful assignment for him was being asked to return to Wilford Hall as Training Officer in the AEGD-2 residency. After 20 years of exemplary service Tom was awarded the Air Force Legion of Merit at his retirement in 1999, an honor extended only to those officers whose achievements over the course of a career have had major impact on the dental service itself.

Teaching and research would become Tom's passion during the next phase of his career. He chose to return to OHSU to join his colleague and friend Dr. Jack Ferracane. His commitment to clinical teaching, research, service and practice was recognized in 2003 by his appointment as Alumni Centennial Professor in Operative Dentistry.

Tom's research interests have been primarily in laboratory and clinical evaluation of dental adhesive systems and resin composite polymerization. In recent years he has been active in practice-based clinical research, establishing the Practice-based Research in Oral Health (PROH) at OHSU along with Jack Ferracane, and co-investigator of the NIH-funded **P**ractice-Based **RE**search **C**onsortium in **E**vidence-based **DENT**istry (PRECEDENT). He has also worked extensively with the National Dental Practice-Based Research Network. His research on cracked teeth, with an emphasis on developing cracked tooth risk assessment techniques, was presented at the 2015 meeting of this academy. Tom's research output has been remarkable. He has published more than 60 articles in peer-reviewed journals, 79 abstracts, and has presented almost 200

presentations at state, national, and international venues. He reviews for many journals, and currently serves on the editorial board of our own journal and others, including the Journal of Dental Research. In addition, Tom was named lead cover author of the fourth edition of Fundamentals of Operative Dentistry: A Contemporary Approach. He had authored chapters of the widely-used textbook since the first edition in 1996.

Over the years, Tom has devoted much time (wife Dea Dea would say too much) to professional leadership and committee work. He is the sort of individual that everyone wants on their working group. His work ethic is unsurpassed, his eye for detail acute, and his observance of deadlines inflexible. It's even better if you can get him to chair your committee and many have recognized this. The section for "service" in his c*urriculum vitae* goes on for pages. Those of you who have served with Tom on the Executive Committee of our own academy can attest to his dedication.

Tom has been the recipient of many honors. He has been awarded Fellowship in both the International College of Dentists and the American College of Dentists. He was named Civilian National Consultant for Dental Materials to the Air Force Surgeon General. In 2015, the International Association for Dental Research presented him with the Ryge-Mahler Award for Clinical Research in Dental Materials. However, after so many years as part of this organization, after braving Chicago weather for so many Februarys, after making so many lifetime friends – I am certain that nothing will mean more to him than this award. He richly deserves to be honored by our Academy with the Award of Excellence.

James C. Broome, DDS, MS

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Clinical Technique/Case Report

Stress-reduced Direct Composites for the Restoration of Structurally Compromised Teeth: Fiber Design According to the "Wallpapering" Technique

S Deliperi • D Alleman • D Rudo

Clinical Relevance

When most of the dentinoenamel complex (DEC) is lost, the "wallpapering" of the residual cavity walls with Leno weaved ultra-high-molecular-weight polyethylene fibers may help to both emulate the crack shielding mechanism of the DEC and absorb the stress from either polymerization shrinkage or occlusal load.

SUMMARY

Purpose: The purpose of this work was to present a restoration technique based on an understanding of the biomechanical properties of the dentinoenamel complex (DEC) and the physical-mechanical properties of the resin-based composite including the stress generated from both polymerization shrinkage and occlusal forces.

Technique Summary: The DEC is a functional interphase that provides crack tip shielding; the DEC should be preserved during restorative procedures. Dentists can design the strategic placement of restorative materials into the cavity to both resist the mode of failure and mimic the performance characteristics of the intact natural tooth. The term "wallpapering" describes a concept of covering the cavity walls with overlapping closely adapted pieces of Leno weaved ultra-high-molecular-weight polyethylene (LWUHMWPE) ribbons. The key for success is that the ribbons are adapted and polymerized as closely as possible against the contours of residual tooth substrate. The resulting thin bond line between the fibers and the tooth structure creates a "bond zone" that is more resistant to failing due to the intrinsic stress and energy absorbing mechanism of the LWUHMWPE ribbons. The formation of defects

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and voids, from which crack propagation may start, is also reduced. The fibers' tight adaptation to tooth structure allows a dramatic decrease of the composite volume between the tooth structure and the fiber, thus protecting the residual weakened walls from both the stress from polymerization shrinkage and the occlusal load.

Conclusion: By using a similar approach, fiber-reinforced stress-reduced direct composite restorations may be performed in the restoration of structurally compromised vital and nonvital teeth.

INTRODUCTION

During a tooth's lifetime, a wide range of overload events may happen, including those from bruxism, trauma (high extrinsic loads), or during dynamic loading (intrinsic chewing strokes in a small area due to a hard foreign body such as a stone or seed). Cracks form within enamel[1] typically without causing catastrophic tooth fracture. The dentin-enamel junction (DEJ) successfully unites two very dissimilar dental materials: the hard and wear-resistant enamel cover-layer and the softer and less mineralized dentin core.[2] The DEJ, or dentinoenamel complex (DEC), is known for its unique biomechanical properties. The DEC is composed of a three-dimensional continuum of gradations of interphases that enable the harmonious transfer of stress between dentin and enamel, two materials having dissimilar elastic properties. The DEC brings these two dissimilar materials into strain harmony and allows them to function together as a tooth. The DEC provides a crack-arresting barrier to the cracks formed in enamel from traversing the enamel-dentin interface and causing catastrophic tooth fractures.[3] These cracks are found to start either at so-called "tufts" (hypocalcified fissures extending outward from the DEC) growing toward the enamel surface or at flaws close to the tooth surface.[3] Latter cracks propagate toward the DEC and are arrested there[4,5] or with limited penetration of the underlying dentin core.[6,7]

Imbeni and others[6] examined how cracks propagate in the proximity of the DEC and also quantified, using interfacial fracture mechanics, the fracture toughness of the DEC region. They reported that the DEJ toughness is approximately 5-10 times higher than enamel but approximately 75% lower than dentin. They also reported that cracks penetrating through the interface tend to reach the (optical) DEC and arrest when they enter the tougher mantle dentin adjacent to the interface. The mantle dentin is a thin material layer close to the DEC that is somewhat softer than the bulk dentin, showing decreased peritubular dentin and tubule density. They explained crack arrest by the gradually increasing toughness from enamel to mantle dentin.[6] Although there is little consensus on the mechanism of crack arrest at the DEC, research definitely agrees that the DEC is a very strong, durable, damage tolerant, and well-bonded interface that is unlikely to fail within healthy teeth despite the formation of multiple cracks within enamel during a lifetime of exposure to masticatory forces.[7-9]

Structurally compromised teeth are teeth exhibiting substantial loss of tooth structure due to previous caries, preexisting restorations, and endodontic procedures. The more structurally compromised the tooth, the lower is the proportion of the residual DEC region in the tooth and the higher is the potential of a catastrophic failure of the residual tooth structure. Cast coverage restorations[10] and large amalgam restorations[11] have been selected for the restoration of endodontically treated teeth for many years. Metal-based restorations and the residual tooth structure behave as two different entities during function because they are not bonded to the residual tooth structure. As a matter of fact, the residual tooth structure is continuously subjected to both occlusal and thermal stresses. Furthermore, the need for mechanical retention or resistance forms, such as boxes, grooves, slots, pins, and posts creates regions of great stress concentrations that dramatically weaken the residual tooth structure and increase the potential for crack formation.[12]

Over the last two decades, new restorative protocols have been proposed to properly use modern adhesive systems and preserve the remaining sound tooth structure.[4,13-15] The goal of these procedures, utilizing either direct or indirect composite restorations, is to maximize the bond and minimize the stress in an attempt to mimic the functional and optical characteristics of the intact natural tooth.[16]

When clinicians select a composite resin restorative material, they need to keep in mind that composite resin is a rigid material; it does not lack of strength or stiffness but lacks of toughness.[17] Toughness is defined as the resistance of a material to the rapid propagation of cracks. Toughness is an inherent property of the material and can be used to predict structural performance.[17]

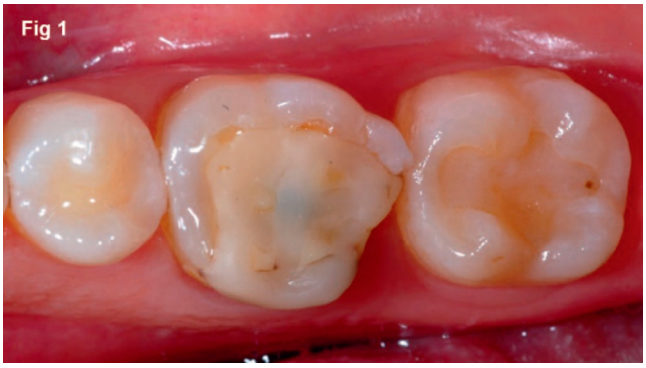

Figure 1. *Preoperative view of tooth #19 showing an incongruous tooth-colored restoration.*

Leno weaved ultra-high-molecular-weight polyethylene (LWUHMWPE) fibers are plasma-treated fibers. LWUHMWPE fiber reinforcement ribbon systems have been introduced in the attempt to increase composite resin toughness, thus increasing both durability and damage tolerance.[17,18] These bondable reinforcement fibers can be closely adapted to the residual tooth structure without requiring additional preparation. The woven fibers have several advantages. The structure of the fiber based on multidirectional yarns and locked nodal intersections creates a great multitude of load paths that redistribute the occlusal forces throughout a greater region of dental restorative composite.[19-21] The higher modulus of elasticity and lower flexural modulus of polyethylene fiber have a modifying effect on the interfacial stresses developed along the cavity walls.[22] Sengun and others[23] reported a fail-safe mechanism for fiber-reinforced restorations compared with restorations without LWUHMWPE fibers. Because fractures generally occur above the cementoenamel junction (CEJ), the remaining tooth structure is restorable, and catastrophic failures are avoided.

If clinicians can understand the mode of failure of both the composite resin and the weakened residual tooth structure, the strategic fiber insertion against the cavity walls may contribute to avoid failure through a stress distributing and energy absorbing mechanism.

This paper introduces a clinical protocol for the restoration of structurally compromised devitalized teeth using the "wallpapering" of the residual cavity walls with LWUHMWPE fibers to mimic the crack shielding mechanism of the DEC.

CASE PRESENTATION AND CLINICAL TECHNIQUE

A 35-year-old female patient presented with an existing fractured composite resin restoration in a lower molar tooth (#19). The patient's tooth was restored with a direct composite resin and prefabricated carbon fiber posts eight years earlier (Figure 1). The patient reported that failure of both the distal marginal ridge and the disto-lingual cusp occurred two years following the restoration placement.

When a stress-reduced direct composite (SRDC) protocol is selected, six steps need to be followed[24]:

1. Analysis of the occlusion and opposing dentition
2. Cavity preparation and caries removal end points
3. Analysis of residual tooth structure
4. Preparation of the dental substrate to achieve a reliable bond to enamel and dentin
5. Control of polymerization stresses by using appropriate layering and curing techniques, and wallpapering of dentin walls with LWUHMWPE fibers
6. Occlusal force equilibration

Step 1: Analysis of the Occlusion

Analysis of occlusion is required to intercept either areas of occlusal overload or lack of centric stops. A composite mockup may be performed to establish and test a temporary occlusion; it also allows for the determination of the three-dimensional location of fibers within the restorations. It is important that the fibers are not damaged or exposed to the oral cavity by later occlusal adjustments. Preoperative occlusal analysis showed concentration of the occlusal load on the residual facial cusp of tooth #19 and in the distal area close to the fractured marginal ridge (Figure 2). Because of the unbalanced occlusion, a fracture of the remaining wall can occur under mastication due to concentration of the load on the weakened facial cusps. After completing the analysis of occlusion and presenting a treatment plan to the patient for both a direct and indirect restoration, a fiber-reinforced (FR)-SRDC restoration was selected for the restoration of tooth #19.

Step 2: Cavity Preparation and Caries Removal End Points

A rubber dam was placed, and the existing restoration was removed using # 2 and #4 round burs (Brasseler, Savannah, GA, USA). The cavity was prepared in a very conservative manner, removing just the decayed dental tissue and trying to preserve the remaining sound tooth structure according to the basic guidelines for direct adhesive preparations. A caries indicator (Sable Seek, Ultradent Products,

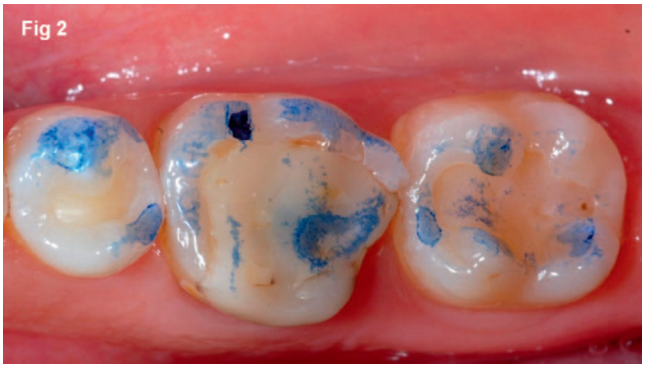

Figure 2. *Before starting anesthesia, occlusion was checked and centric stops were recorded.*

South Jordan, UT, USA) was applied to the dentin; stained nonmineralized and denatured dental tissues were removed with a spoon excavator to an ideal caries removal end point that creates a highly bondable peripheral seal zone.[25] Residual enamel sharp angles and unsupported prisms were smoothed using the Standard Distal (SD) and Standard Ball (SB) partially diamond-tipped ultrasonic tips (EMS, Nyon, Switzerland); the SB instrument was also used to smooth sharp angles located within the dentin. No bevels were placed on either the occlusal or the gingival margins. The main goals of step 2 were to avoid the formation of any sharp line angle on either the prepared enamel or dentin and to preserve the peripheral rim.

Step 3: Analysis of Residual Tooth Structure

Once the preparation was complete, it was determined that both the lingual cusps were missing, the lingual cavosurface margins were located below the gingival level on dentin-cementum, and a very thin area of enamel was preserved on the distal gingival margins (Figure 3). However, the thickness of the residual facial walls greater than 2 mm and the preservation of the entire mesial marginal ridge were considered sufficient to support an FR-SRDC restoration.

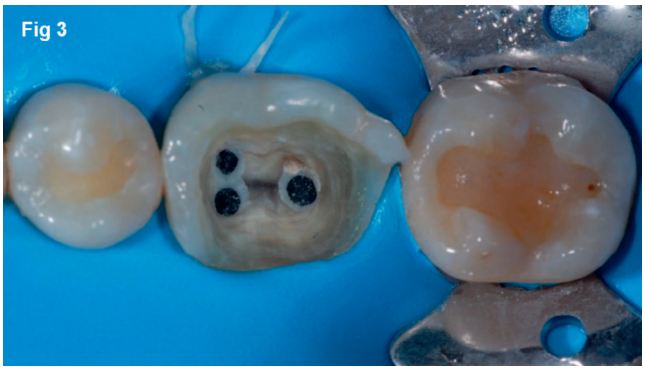

Figure 3. *Cavity preparation was completed using partially diamond tipped ultrasonic tips.*

Step 4: Preparation of the Dental Substrate to Achieve a Reliable Bond to Enamel and Dentin

A circular matrix (OmniMatrix, Ultradent Products) was placed around tooth #19, and lingual and interproximal matrix adaptation was secured by only tightening it; good adaptation to the gingival margin was achieved without using any dental wedge and burnishing the most gingival area of the metal matrix (Figure 4). The tooth was etched for 15 seconds using a 35% phosphoric acid (UltraEtch, Ultradent Products) (Figure 5). The etchant was removed, and the cavity was rinsed with water spray for 30 seconds, being careful to maintain a moist surface. The cavity was disinfected with a 2% chlorexidine antibacterial solution (Cavity Cleanser, Bisco, Schaumburg, IL, USA) (Figure 6).[26] A three-step etch and rinse 40% filled ethanol-based adhesive system (All Bond 3, Bisco) was placed in the preparation; both the primer and the coating resin were gently air thinned and light cured for 20 seconds using an LED curing light (Valo, Ultradent Products) (Figure 7).

Step 5: Control of Polymerization Stresses: Step 5a: Buildup of the Skeleton

The missing peripheral tooth structure was built up via 2-mm wedge-shaped composite increments. Vit-l-escence microhybrid composite resin (Ultradent Products) was used to restore the teeth. Stratification was initiated using multiple 1- to 1.5-mm triangular-shaped (wedge-shaped) increments; apico-occlusal placed layers of A4 shade were used to reconstruct the cervical third of both the lingual and distal surfaces (Figures 8 and 9). At this point, the circular matrix was replaced with a sectional matrix to achieve a more predictable contact point with the second molar tooth.[24] Both the proximal surface and the external shell of the lingual cusp buildups were completed using the Pearl Smoke (PS) enamel shade.

Step 5b: Wallpapering of Dentin Walls With LWUHMWPE Fibers or Ribbond Fibers

Preparation to the wallpapering includes the selection of the correct length and width of the fibers to properly fit into the cavity. A dental probe (Hu-

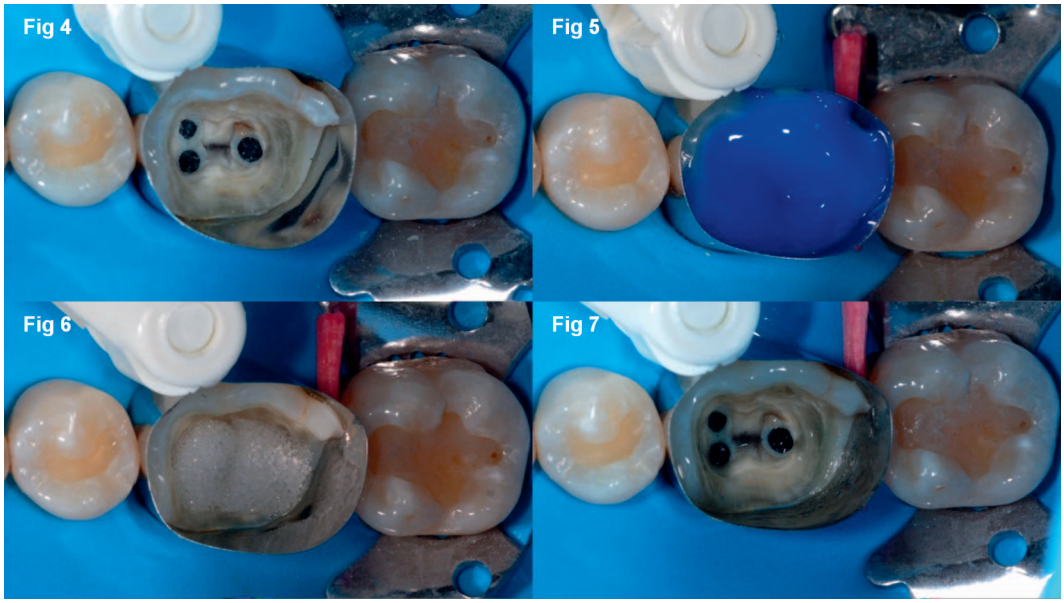

Figure 4. *A circular matrix was placed.*
Figure 5. *Etching was performed using 35% phosphoric acid.*
Figure 6. *A 2% digluconate chlorexidine solution was applied on dentin for 30 seconds.*
Figure 7. *An ethanol-based primer was applied on both enamel and dentin followed by the application of a hydrophobic resin coating.*

Friedy, Chicago, IL, USA) was used to measure the mesio-distal distance and the pulp chamber-coronal length of the cavity. Two 4-mm-wide by 11-mm-long Ribbond fiber pieces (Ribbond THM, Ribbond Inc, Seattle, WA, USA) were wetted with an unfilled resin first (Ribbond Wetting resin, Ribbond Inc).[12,27] After removing the excess resin, fibers were covered with a very thin layer of tacky flowable composite, Ribbond Securing Composite (Ribbond Inc); fibers were C-shaped prior to insertion into the cavity. The first Ribbond fiber was bonded immediately against the lingual wall and cured for 20 seconds (Figure 10). The same procedure was also completed for the second polyethylene fiber which was placed on the facial wall and cured for 20 seconds. The Ribbond pieces overlapped one another at the proximal surfaces, with each piece stopping at an imaginary DEJ line on the top and folding down onto the axial-pulpal floor line angle on the bottom at both the facial, lingual and proximal walls (Figure 11). Being bondable reinforcement fibers, they could be closely adapted to the residual tooth structure. The fibers' tight adaptation to tooth structure was the key to decreasing the composite volume between the tooth structure and the fiber; thus, stress from polymerization shrinkage could be prevented on the residual weakened walls. In cases of visible cracks, structural weakness of the pulp chamber floor, or patients with parafunction, another piece of Ribbond may be prepared in the same manner and bonded closely against the pulpal floor. In a similar clinical scenario, one more piece of Ribbond may be placed

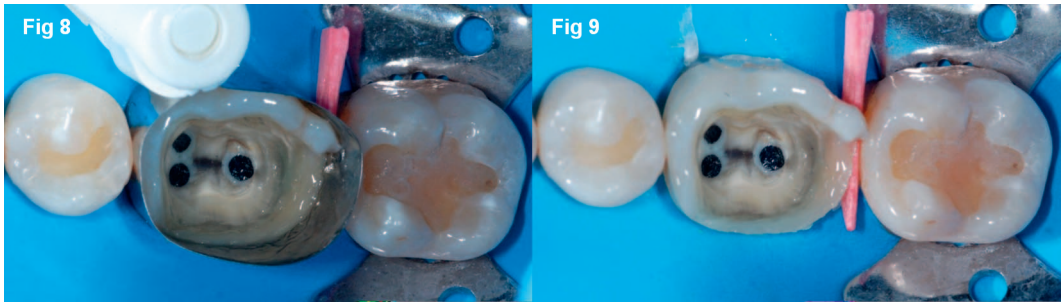

Figure 8. *Multiple 1- to 1.5-mm triangular-shaped (wedge-shaped) increments were used to reconstruct the cervical third of both the lingual and distal surfaces.*
Figure 9. *The circular matrix was replaced with a sectional matrix and the peripheral enamel skeleton was built up first using wedge-shaped increments.*

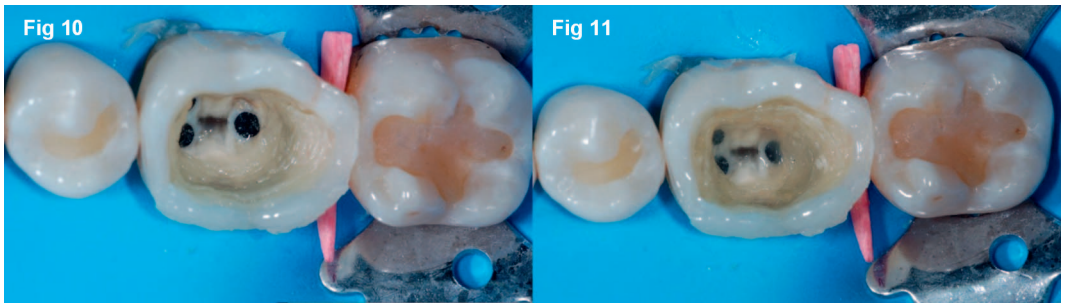

Figure 10. *The first C-shaped polyethylene fiber is bonded immediately against the lingual wall and cured for 20 seconds.*
Figure 11. *The same procedure is also completed for the second C-shaped polyethylene fiber, which is placed as close as possible to the contour of the facial wall.*

1.5 mm below the occlusal surface to assure an additional distributing and energy absorbing mechanism.

Step 5c: Dentin and Occlusal Surface Buildup

Stress reduction during the early polymerization of the dentin bonding system (the first 3-30 minutes) was very important.[13,28] By the time the skeleton buildup and fiber application were completed, time had been given for the bond to dentin to mature before it was connected to the next layers of composite resins.[16] Stratification of dentin was started by placing a 1- to 1.5-mm even layer of A3.5 flowable composite (PermaFlo, Ultradent Products) on the dentin floor, which was followed by the application of dentin wedge-shaped increments strategically placed to only two bonded surfaces, decreasing the cavity configuration or C-factor ratio (Figures 12 and 13). The C-factor is defined as the ratio between bonded and unbonded cavity surfaces; increasing this ratio also increases the stress from polymerization shrinkage.[29] Due to the stress absorbing effect of the Ribbond fibers,[22] 2-mm-thick dentin layers of composite resin can be placed into contact with the Ribbond fibers. For the same reason, single increments of PS enamel shade were applied to one cusp at a time; each cusp was cured separately, achieving the final primary and secondary occlusal morphology (Figure 14). To reduce stress from polymerization shrinkage, the authors utilized a previously described polymerization technique, based on a combination of pulse (enamel) and progressive (dentin) curing technique through the tooth.[16] The pulse curing protocol was adopted for the proximal and occlusal enamel buildup polymerization; it was accomplished by using a very short curing time (one or two seconds) per each increment. The progressive curing technique was used for the polymerization of the dentin increments; it was performed by placing the light tip in contact with

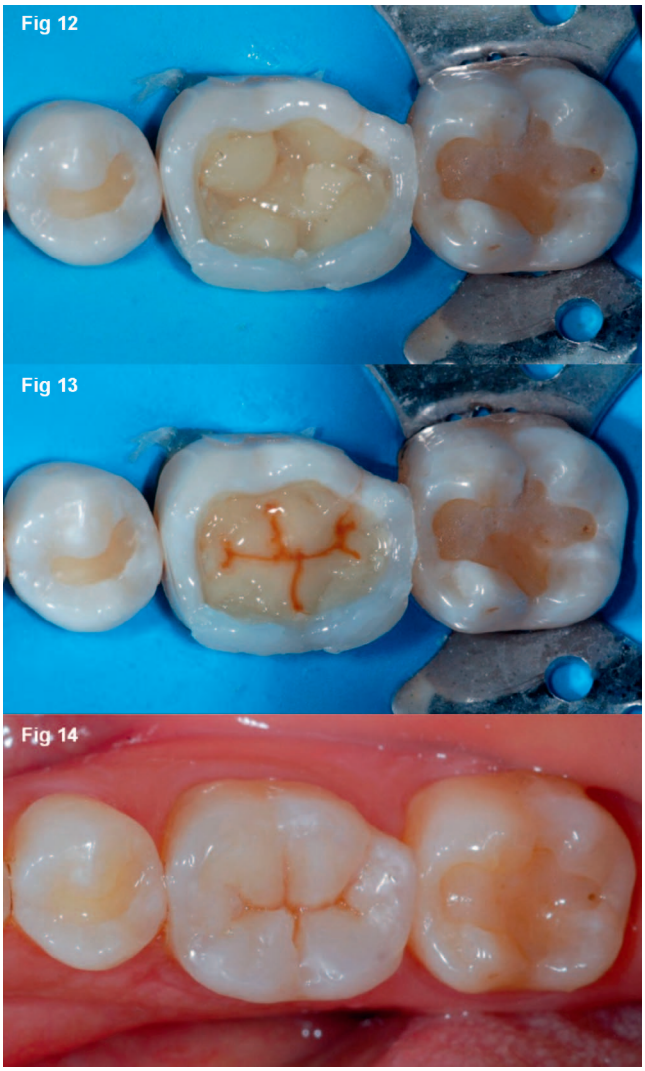

Figure 12. *Dentin stratification was performed by using wedge-shaped increments of composite dentin shades.*
Figure 13. *A brown composite tint was placed at the end of dentin stratification.*
Figure 14. *Restoration was completed with the application of PS enamel shade to each cusp to develop cusp ridges and supplemental morphology.*

Table 1: *Recommended Photocuring Times and Intensities for Proximal and Occlusal Enamel and Dentin*

Buildup location	Polymerization technique	Intensity (mW/cm^2)	Time (s)
Proximal enamel	Pulse (P)	800	2 (P) + 20[a]
Dentin	Progressive (Pr)	800	20[a]
Occlusal enamel	Pulse (P)	800	1 (P) + 20[a]

[a] *"Curing through": 20 seconds per each surface (lingual, facial, and occlusal surface).*

the external cavity walls to start the polymerization through the wall (indirect polymerization) at a lower intensity (Table 1). Areas of undercuts in the cavity are very common when adopting an ultra-conservative preparation protocol. A progressive curing protocol assured composite resin polymerization in hidden areas of the cavity and reduced stress. Final polymerization was then provided at a higher intensity and extended curing time. Initial occlusal and proximal adjustment of the restoration was performed using #7404 and #7902 carbide burs (Brasseler). The patient was recalled after 48 hours to complete the occlusal adjustment and perform the final polishing.

Step 6: Occlusal Force Equilibration

Occlusion was verified, avoiding excessive load on the residual facial cusp and creating a centric stop in the composite restoration at the distal area of the tooth-restoration complex. The centric stops located on the tooth structure and composite resin are of the same intensity; they do not differ from the ones on the adjacent teeth (Figure 15). A "verticalization" of occlusion is adopted to avoid overloading of either the restored or residual cusps during both centric and eccentric movements. Centric stops are preferably located at the center of the tooth to assure a prevalence of axial loads on the tooth restoration complex and avoid excessive lateral forces.

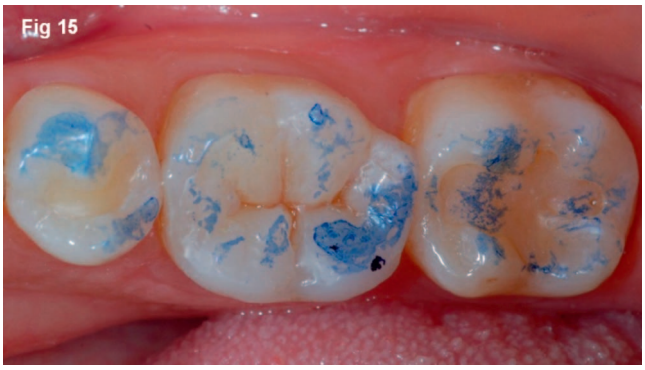

Figure 15. *Occlusal view of the final restorations after occlusion checking.*

Figure 16 shows a schematic representation of the wallpapering technique.

DISCUSSION

Endodontically treated teeth have been restored using indirect porcelain-bonded restorations and indirect/semidirect resin-bonded composite restorations.[13,30]

The stress generated from polymerization shrinkage and the lack of adequate protocols have discouraged many clinicians from selecting a direct technique for the restoration of structurally compromised vital and devitalized teeth for many years. However, SRDC restorations have been proposed as a valid alternative to indirect resin-bonded composite restorations.[4] Spreafico and others[15] reported no difference in the clinical performance of semidirect and direct class II composite resin restorations after a 3.5-year evaluation period. Deliperi and Bardwell[31] reported no failure for class II direct cusp-replacing resin-bonded composite restorations after two years of clinical service using both a bonding preservation and stress-reducing protocol. When adopting the same restorative protocol, they also reported equal results for large-size three-surface SRDC restorations over a two-year period.[32] These clinical studies were performed on vital teeth with thickness of the residual cavity walls greater than 2 mm.

Lately, increasing attention has been focused on the proper utilization of LWUHMWPE fibers (Ribbond Inc) for the direct restoration of structurally compromised endodontically treated teeth.[12,27,33] Although only a few clinical case reports and a pilot study have been published in the literature, fiber-reinforced restorations performed very well in different laboratory tests.[5-9,22] LWUHMWPE Ribbond fibers increase the flexural strength and fracture toughness of composite resin restorations. Due to the fiber design based on a dense network of locked nodal intersections, they also serve as a crack stopping mechanism; the locked stitch interwoven fibers prevent rapid crack growth and change the direction that ultimately dissipates the strain. Belli and others[5] reported that the placement of LWUHMWPE Ribbond fibers against the dentin walls increased the fracture strength and decreased the cusp movement under loading of root filled molars with Mesial Occlusal Distal (MOD)

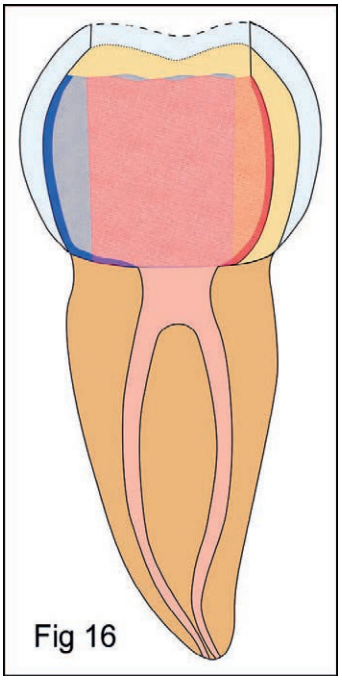

Figure 16. *Schematic representation of the fiber laydown protocol showing the wallpapering of both the facial and lingual surface; fibers stop at an imaginary DEJ line in the coronal area and fold down onto the axial-pulpal floor at the cervical area.*

cavities.[34] In a study published in 2006, Belli and others described that Ribbond increased the microtensile bond strength and lowered the C-factor effect.[35]

The former laboratory studies were performed placing the composite into the cavity without following a stress-reducing protocol; both the maturation of the bond[36,37] and the strategic layering/curing protocol may further reduce stress concentration on the residual cavity walls.[4,24] In the first three to five minutes following the polymerization of the adhesive system, early bond strength to enamel was reported to be twice as strong as the early bond strength to dentin[38]; this trend changes dramatically after a five-minute period as the late bond strength to dentin may be even higher than the one to enamel.[39]

Combining composite stratification with wedge-shaped increments and polymerization with a low-intensity approach is also mandatory to reduce stress in the restoration. Multiple wedge-shaped increments are placed trying to contact no more than two bonded cavity walls; the technique allows the decrease of stress from polymerization shrinkage by reducing the composite mass (per increment) and transforming the high C-factor configuration into multiple low C-factor configurations.[4,24] The application of small increments allows the clinician to influence the C-factor at a micro level (micro C-factor) by maximizing the unbonded free surfaces each time a single composite increment is placed. In addition to this sophisticated stratification technique, a combination of progressive and pulse curing polymerization is used on dentin and enamel, respectively, to further decrease the stress from polymerization shrinkage.[4] By adopting a similar soft-start curing protocol, physical and mechanical properties of composite resin may also be improved; more time is available for composite flow into the direction of the cavity walls, resulting in stress release during polymerization shrinkage and increased crosslinking. The quality of the polymer network, which is not equivalent to the degree of conversion, is influenced by the modified curing scheme. A research study[40] corroborated previous findings[41,42] supporting that polymerization protocols based on low intensity and increased curing time result in longer polymer chain formation; conversely, frequency of crosslinking increases using higher intensity and short curing times, which leads to multiple short polymer chains formation and reduced degree of cure.

The literature suggests that cracking along the DEC occurs very rarely.[5-9] The DEC seems to be a very well- and strongly bonded interface that provides crack tip shielding. Preserving the DEC during cavity preparation as much as possible is the first rule each restorative dentist should follow.

Interestingly, Bechtle and others[9] reported that crack arrest occurs only if cracks approach the DEC from the enamel side. If cracks are induced from the dentin side, samples fractured after elastic and some amount of plastic deformation. This in vitro finding does have clinical significance.

During occlusal loading, vertical loading creates lateral forces against the cavity walls (the Poisson effect); the lateral forces create a tensile force across the pulpal floor that may be responsible for the initiation of a crack on the residual cavity walls. Due to the composite resin's intrinsic lack of toughness, a catastrophic failure may occur if structurally compromised teeth are restored with a resin bonded composite only.[34,43,44] The wallpapering of the residual cavity walls with the Ribbond polyethylene fibers is intended to diminish the possibility of a failure while preserving the residual sound tooth structure. When a failure occurs, it happens in a safe mode due to the energy absorbing mechanism and stress distribution effect of the fibers; the damage on

the tooth-restoration complex is minimal and can be easily repaired because it occurs above the CEJ.[23] The intrinsic characteristic of the fiber network and the correct fiber insertion into the cavity walls may help clinicians to push the envelope with direct restorations; if a stress-reduced approach is adopted, direct restorations may be extended to structurally compromised vital and devitalized teeth without requiring cusp coverage of residual weak walls. However, the thinner the remaining cavity walls, the higher the risk for a catastrophic failure of the tooth to occur. Structurally compromised teeth with residual cavity walls thinner than 2 mm lack of the major portion of the DEC on both the occlusal, proximal, and lateral walls. The lack of the functional shielding mechanism of the DEC and the composite resin's intrinsic lack of toughness are two of the reasons that pushed clinicians to cover the residual weak cusps with bonded onlay restorations.[13,14] By adapting the LWUHMWPE Ribbond ribbons as closely as possible against the internal contours of the residual tooth substrate, it is possible to both replicate and reinforce the crack shielding mechanism of the DEC. The Leno weaved Ribbond provides multiple load paths that distribute the stresses over a greater region. Because of this greater stress distribution effect, stress from either polymerization shrinkage or occlusal load can be better controlled, and thin cavity walls can be preserved. Like the DEC, which enables the dentin and enamel to function in strain harmony together, the Ribbond liner bonded immediately against the cavity walls, which enabled the tooth substrate and the restorative composite to also function in strain harmony.

Bechtle and others[9] also tested notched rectangular-shaped enamel-dentin bending bars with the dentin side under tension. They observed that crack propagation occurred simultaneously within dentin and enamel; the two cracks may either coalesce or stay separate at the DEC. In the latter case, they reported that the DEC formed an unbroken ligament between cracks, which kept the two fractured parts together (DEC bridging).

The ideal cavity preparation for resin bonded composite is a "saucer-shaped" configuration[45]; however, this is not always possible. A notched dentin sample represents an in vitro tool to replicate a very common clinical scenario. The areas of the cavity prepared for mechanical retention or resistance form (boxes, grooves, slots) do have acute internal line angles. These regions of greatly increased stress intensities challenge the crack tip shielding mechanism of the DEC. The use of ultrasonic tips definitely helps to smooth the sharp line angles; however, covering these areas with Ribbond fibers is recommended to avoid stress concentration on these areas and avoid the formation of cracks either on the pulpal floor and axial walls.

Cavity preparation, material design, and occlusal equilibration represent different stages of the restorative procedure to achieve stress reduction and assure the longevity of the tooth-restoration complex.

CONCLUSION

The SRDC protocol allows clinicians to not only create minimally invasive preparations but preserve the remaining sound tooth tissues in structurally compromised teeth. Avoiding the creation of sharp angles during cavity preparation minimizes the increase in stress intensities in both the remaining tooth structure and the restorative composite; allowing some time for dentin bond maturation and designing the strategic placement of restorative materials into the cavity to both resist cracks and mimic the performance characteristics of the intact natural tooth are the fundamentals to complete stress-reduced direct composite resin restorations in structurally compromised teeth.[46]

Long-term clinical studies are required to confirm the superiority of this protocol over traditional restorative strategies.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Sardinia Dental Teaching Center.

Conflict of Interest

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

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Clinical Research

Comparison of Effectiveness and Sensitivity Using Two In-Office Bleaching Protocols for a 6% Hydrogen Peroxide Gel in a Randomized Clinical Trial

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Clinical Relevance

Simplifying the bleaching procedure by an abbreviated protocol saves materials and can decrease the risk of gel contact with soft tissues surrounding the teeth. The effectiveness of the low-concentration hydrogen peroxide gel was maintained via this outpatient technique.

SUMMARY

Objective: The aim of this blinded and randomized clinical trial was to compare two application protocols (one 36-minute application vs three 12-minute applications). We then assessed the effectiveness of the bleaching and any increase in sensitivity that was induced by bleaching via a split-mouth design.

Methods and Materials: Thirty patients were treated. One group had a half arch of teeth treated with a traditional application protocol (group A: 3 × 12 minutes for two sessions). The other received an abbreviated protocol (group B: 1 × 36 minutes over two sessions). Two sessions were appointed with a two-day inter-

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val between them. The tooth color was registered at each session, as well as one week and one month after completing the treatment via a spectrophotometer. This measured L*, a*, and b*. This was also evaluated subjectively using the VITA classical A1-D4 guide and VITA Bleachedguide 3D-MASTER. Tooth sensitivity was registered according to the visual analogue scale (VAS) scale. Tooth color variation and sensitivity were compared between groups.

Results: Both treatments changed tooth color vs baseline. The $\Delta E^* = 5.71 \pm 2.62$ in group A, and $\Delta E^* = 4.93 \pm 2.09$ in group B one month after completing the bleaching (p=0.20). No statistical differences were seen via subjective evaluations. There were no differences in tooth sensitivity between the groups. The absolute risk of sensitivity reported for both groups was 6.25% (p=0.298). The intensity by VAS was mild (p=1.00).

Conclusions: We used hydrogen peroxide (6%) that was light activated with a hybrid LED/laser and two different protocols (one 36-minute application vs three 12-minute applications each for two sessions). These approaches were equally effective. There were no differences in absolute risk of sensitivity; both groups reported mild sensitivity.

INTRODUCTION

Tooth bleaching is the treatment of choice for dental discoloration.[1] Color enhancement increases esthetics and self-esteem.[2] However, bleaching can also produce adverse effects such as tooth sensitivity—this is an important problem.[3]

In-office bleaching sessions usually include three applications of peroxide gel for effectiveness.[4] This includes multiple steps for the dentist and the patient, which can generate discomfort and be time consuming. There is currently no rationale for this protocol in the literature. Caneppele and others recently published an *in vitro* study showing no differences in color change when comparing different abbreviated protocols (1×40 minutes) using in-office bleaching gels.[5] Matis and others showed that, after one hour of contact, 10% and 22% hydrogen peroxide gels still have an activity of 30%.[6] This means that a prolonged application should have the same bleaching effect as three short applications. This would use less material, save time, and minimize irritation. Reis and others showed that prolonged application of 35% hydrogen peroxide was as effective as three applications of 15 minutes with a risk of absolute sensitivity of 15% with mild or moderate intensity.[7] There are no reports of clinical trials with low concentration (6%) gels catalyzed with a LED lamp or laser light for prolonged application. It is important to study the effectiveness and adverse effects related to the bleaching procedure.

De Paula and others showed that a reduction in intervals between in-office bleaching sessions with conventional concentrations had no statistically significant differences on sensitivity and effectiveness.[8] However, this trial also shows a risk of absolute sensitivity of 60% in treated patients. Thus, this problem still lacks a solution.

There is a recent tendency to reduce the concentration of hydrogen peroxide for in-office bleaching. The active ingredients are catalyzed with titanium dioxide nanoparticles and a hybrid (LED/laser). This allows 6% to still be effective with a reduced risk of sensitivity.[9-11] This could reduce the intervals between sessions, abbreviate the protocol, and produce less sensitivity.

The objective of this trial was to compare two application protocols: 1) one application of 36 minutes and 2) three applications for 12 minutes. Both protocols were applied twice. We then assessed their effect on the effectiveness of bleaching and bleaching-induced sensitivity via a split-mouth design. The null hypothesis was that there is no difference in the effectiveness and bleaching-induced sensitivity from the abbreviated application protocol vs the traditional protocol.

METHODS AND MATERIALS

The Ethics Committee of the local Faculty of Dentistry approved this clinical study. The study was conducted between July 2015 and October 2015. It was registered on the Clinical Trials Registry (#NCT02603354) and was conducted according to the Consolidated Standards of Reporting Trials Statement[12] (Figure 1) and Helsinki Declaration of 1975 (revised in 2000).

Thirty volunteers were selected and received a gingival debridement to remove supragingival deposits when appropriate. There was then a coronal cleaning with a soft nylon cup and pumice paste to remove stains. Subsequently, all patients were instructed in a standardized tooth brushing technique. They were given a toothbrush and toothpaste for the period of treatment and evaluation. They also signed a written informed consent.

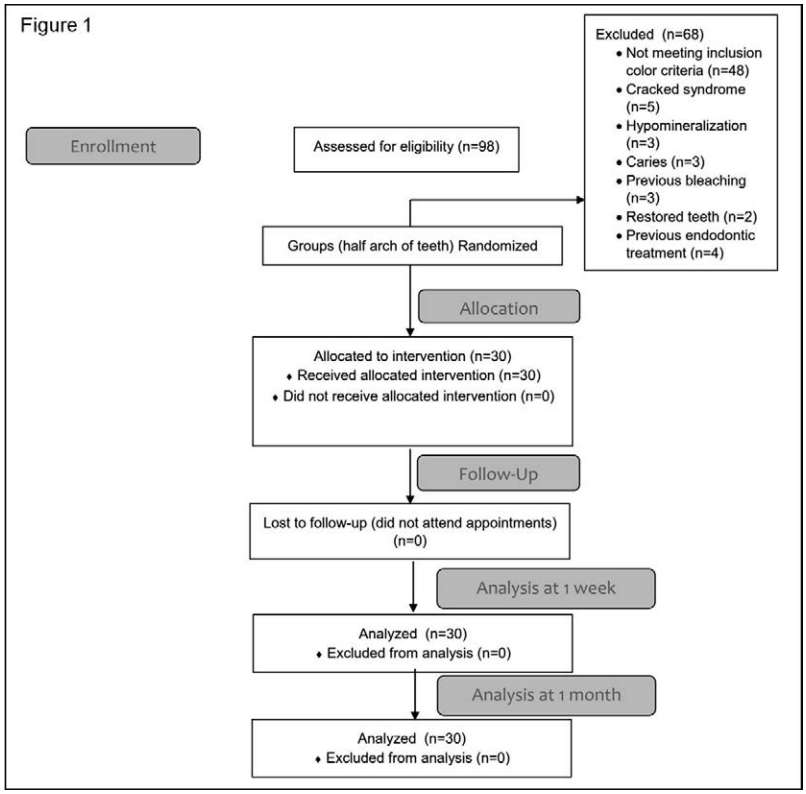

Figure 1. *CONSORT flow diagram.*

This was a randomized, blinded (evaluator) trial using a split-mouth design[13] (one half arch was treated by the abbreviated protocol [1 × 36 minutes for two sessions] and the other by the traditional application protocol [3 × 12 minutes for two sessions]). This was followed with nonprobability sampling. The patients were invited to participate in the study through posters posted around the city or recruited from participants in other studies in the same department who were contacted by email or phone.

A total of 98 patients were examined in a dental chair to confirm that they met the inclusion criteria. The patients included in this study were over 18 years old and had two central incisors shade A2 or darker determined with a value-oriented shade guide. The exclusion criteria were patients who were pregnant or lactating; had moderate or severe fluorosis, tetracycline stains, orthodontic treatment, periodontal disease, orofacial tumors, trauma, or tooth malformation; or were taking analgesic, anti-inflammatory, or antibiotic drugs. Patients with restored anterior teeth, previous bleaching procedures, cervical lesions, or dental pain were also excluded.

Two trained operators (restorative dentistry professors) performed the bleaching treatments. A third participant that did not have contact with the patients conducted the randomization. The group allocation (half arch of teeth) was performed by random drawing using Microsoft Excel 2010 (Microsoft, Redmond, WA, USA) from coding assigned to each participant. There were two experimental groups. Group A was the control, and a hydrogen peroxide bleaching compound 6% (Lase Peroxide Flex 6%, DMC Equipamentos, São Carlos, SP, Brazil) was applied to the maxillary group of teeth with a conventional protocol application (three rounds of 12 minutes per two sessions). Group B was the experimental group. The other maxillary half arch of teeth was treated with the same peroxide gel (6%) but with a reduced application protocol (one round for 36 minutes over two sessions). Both groups were bleached with the same compound catalyzed by titanium oxide nanoparticles and activated with a blue hybrid light and an infrared laser during the complete times.

Sample Size Calculation

The primary outcome was color alteration (ΔE). Previous studies[2,14-16] showed that the use of in-office bleaching agents containing hydrogen peroxide with or without LED/laser light leads to a ΔE* value of 2.0-7.0 after two bleaching sessions. We wanted an

80% chance of detecting significance at 5%. The increase in the primary outcome measurement was 5.5 in the control group and 4.5 in the experimental group. This difference was determined by a preliminary pilot study considering the maximum SDs obtained. Thus, a power calculation showed that 25 participants were required for sufficient statistical power. Due to the higher dropout rate seen in our previous two studies, we included 20% more patients: this led to 30 patients in each group.[17]

Bleaching Protocol

In each session, volunteers received prophylaxis with pumice powder and water. The gingival tissue was protected with a light-cured resin gum barrier applied according to the manufacturer's instructions (Lase Protect, DMC Equipamentos). The bleaching agent was prepared by mixing hydrogen peroxide and thickening compounds according to the manufacturer's instructions (three peroxide drops for one drop of thickener). The resulting gel was distributed uniformly on the maxillary teeth (right or left). A total of eight teeth between the second premolars were bleached for each patient. In each bleaching session, the bleaching gels were applied three times for 12 minutes each (group A). The other group received one treatment of 36 minutes (group B) per session. In each application, the surface of the gel was light activated with continuous irradiance using LED/laser light with a total power of 1800 mW for photocatalysis (Whitening Lase Plus, DMC Equipamentos). This is specific equipment for in-office dental bleaching. It presents six LEDs (470 ± 15 nm, 300 mW each), generating 1800 mW of power, and three infrared laser diodes (810 nm, 200 mW each), generating 600 mW of power. This irradiates a total area of 8.5 cm^2 with an intensity of 300 mW/cm^2. Irradiation was the same for the two groups with a protocol of one minute of irradiation and one-minute rest during the entire process (the total irradiation time for each group was 36 minutes). Two bleaching sessions were completed, and the interval between the sessions was two days.

Efficacy Evaluation

Objective Evaluation—Two calibrated evaluators were used to measure the tooth color for baseline immediately after the first and second day, as well as one week and one month after the last session. The color evaluation was obtained from a 6-mm area located in the middle third of the labial surface of the left and right central incisors. To standardize this evaluation, an impression of the maxillary arch was collected to make a guide for high-putty silicone (Zetaplus, Zhermack, Badia Polesine, Rovigo, Italy). A window was created on the labial surface in the middle third of the central incisor using a device with well-formed borders and a 3-mm radius corresponding to the reflectance of a reliable spectrophotometer (VITA Easyshade Compact, VITA Zahnfabrik, Bad Säckingen, Germany).[18] The shade was determined using parameters L*, a*, and b*. The color alteration after each session was given by the differences between the values obtained at the session and the baseline (ΔE*). The ΔE* was calculated using the following formula: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.

Subjective Evaluation—Subjective evaluation protocol used standarized light conditions (same place, time, patient positions, natural light source, all assessments were between 10:00 AM and 3:00 PM). There were two evaluators (κ interoperator = 0.85), and the color competition had 50:50% acceptability threshold (ISO TR 28642-2011). The viewing geometry, object-observer distance, visual angle, and background color were held constant between subjects. The 16 tabs of the shade guide (VITA classical A1-D4, Vita Zahnfabrik) were arranged from the highest (B1) to the lowest (C4) value, and the 15-tab shade guide was used (VITA Bleachedguide 3D-MASTER, Vita, Vita Zahnfabrik) to assess the color. Although the VITA classical A1-D4 scale is not linear in the truest sense, we treated the changes as continuous with linear ranking as done in several dental bleaching clinical trials. Two calibrated evaluators (κ=0.85) recorded the shade of the maxillary central left and right incisors at baseline with the same periods as the objective evaluation.

We checked the color in the middle third area of the labial surface of the anterior central incisor according to the American Dental Association guidelines.[19] The color changes were calculated from the beginning of the active phase through the individual recall times by the change in the number of shade guide units (ΔSGU). This occurred toward the lighter end of the value-oriented list of shade tabs. In the event that the operators disagreed about color matching, a consensus was reached prior to dismissing the patient. The perceptibility threshold used for this study was 3.3 ΔE* units.[20]

Tooth Sensitivity Evaluation Induced by Bleaching

Tooth sensitivity (TS) was characterized via frequency and intensity with a self-completed form and clinical evaluation during the session and immedi-

Table 1: *Baseline Color Features of Volunteers*

Group	VITA classical A1-D4			VITA Bleachedguide 3D-MASTER		
	Median value	Minimum	Maximum	Median value	Minimum	Maximum
Group A	9 (A3)	5 (A2)	14 (C3)	8 (2.5M2)	7 (2M2)	12 (4.5M2)
Group B	9 (A3)	5 (A2)	12 (A3.5)	8 (2.5M2)	7 (2M2)	10 (3.5M2)
Mann-Whitney test *p* value	0.763			0.238		
	L*		a*		b*	
	Average value	SD	Average value	SD	Average value	SD
Group A	81.94	4.47	−0.96	1.07	24.66	3.40
Group B	82.33	4.07	−1.02	1.48	24.72	2.70
t-test *p* value	0.714		0.869		0.945	

ately after via the visual analogue scale (VAS). For the VAS, the investigators instructed the participants to place a line perpendicular to a 10-cm-long line with zero at one end indicating "no TS" and the other end indicating "unbearable TS." The occurrence was analyzed according to whether sensitivity was reported. The volunteers were instructed to fill out a form for each bleaching session and for the following days between sessions in case of sensitivity in any of the bleached teeth at any time.

Statistical Analysis

After verifying the normality of the data distribution and the homogeneity of the variance-covariance matrix, the efficacy of the treatments was evaluated with respect to color alteration (ΔE and ΔSGU) and analyzed by the Mann-Whitney test for between-group comparisons. The proportion of patients with sensitivity between groups was compared with a *Z*-test. The statistical analyses were performed using SPSS 23.0 (SPSS Inc, Chicago, IL, USA) with α=0.05.

RESULTS

Baseline Characteristics

Of the 98 patients, 30 were enrolled. The sample consisted of 14 women (53.3%) and 16 men (46.7%), with average ages of 27.63 ± 7.1 years for men and 26.71 ± 9.3 years for women. The entire cohort was 27.24 ± 8.0 years. Features of color at baseline are shown in Table 1.

Spectrophotometer Data

Color changes measured via ΔE*, ΔL*, Δa*, and Δb* from the baseline are shown in Table 2. There was not a significant ΔE* difference according to the Mann-Whitney test between the two groups at all times ($p>0.30$; values shown in Table 3).

Shade Guide Data

Color changes measured subjectively expressed by VITA classical A1-D4 and VITA Bleachedguide 3D-MASTER ΔSGU units are shown in Table 4. For both scales, there was no significant difference between the different evaluations ($p>0.10$).

Occurrence and Intensity of Sensitivity

The absolute risk of sensitivity reported for both groups was 6.25% (n=2 [same patients in both groups]). There was no statistically significant difference when comparing the proportions of patients by the *Z*-test ($p=0.298$). The intensity of the sensitivity by the VAS scale ($\hat{X} = 0.15 \pm 0.61$) and maximum value per patient immediately after sessions ($\hat{X} = 0.18 \pm 0.88$) were mild, and there

Table 2: *ΔL*, Δa*, and Δb* of Each Group Between Baseline and Each Checkpoint*

	ΔL*				Δa*				Δb*			
	First session	Second session	Week control	Month control	First session	Second session	Week control	Month control	First session	Second session	Week control	Month control
Group A	1.31 ± 3.40	2.04 ± 4.09	2.79 ± 3.55	2.22 ± 3.25	0.07 ± 1.24	−0.38 ± 0.83	−1.00 ± 1.04	−1.08 ± 0.73	0.03 ± 2.77	−1.75 ± 3.57	−3.45 ± 2.85	−3.72 ± 2.98
Group B	0.67 ± 4.14	1.64 ± 4.33	1.35 ± 4.17	1.94 ± 2.59	−0.09 ± 1.13	−0.25 ± 1.33	−0.94 ± 1.29	−0.77 ± 1.03	0.01 ± 2.46	−1.30 ± 3.05	−3.69 ± 2.37	−3.38 ± 0.42
t-test *p* value	0.503	0.708	0.149	0.717	0.593	0.630	0.846	0.188	0.981	0.587	0.721	0.620

Table 3: *Color Change, Expressed as ΔE*, of Each Group Between Baseline and Each Checkpoint*

	ΔE*			
	First session	**Second session**	**Week control**	**Month control**
Group A	3.76 ± 2.93	5.06 ± 3.26	5.83 ± 2.71	5.71 ± 2.62
Group B	4.13 ± 2.89	5.03 ± 3.01	5.98 ± 2.27	4.93 ± 2.09
t-test *p* value	0.617	0.864	0.950	0.207

was no statistically significant difference between groups (p=1.00).

DISCUSSION

This trial assessed the influence of an abbreviated protocol (single application of 36 minutes per two sessions) of hydrogen peroxide of low concentration (6%) compared with traditional application of three applications for 12 minutes per two sessions with 72 minutes of total contact gel time per tooth. Dentists generally look to streamline their procedures and use of consumables. A simplified protocol would have less gel contact on the soft tissues surrounding the teeth. This avoids possible adverse effects or patient discomfort. There is no clear argument in the literature regarding a 3 × 12-minute application or other protocols for in-office gels.

Chen and others showed that the time to achieve effectiveness of hydrogen peroxide during the bleaching process should be more than 20 minutes of contact[21] in an *in vitro* trial. The strips and/or toothpaste that contain low concentrations (6%) of hydrogen peroxide come with instructions for prolonged use—even several hours (>20) to achieve effective color change. These manufacturers argue that because of its low concentration, cell damage and postoperative problems are less likely.[22]

The results show no difference in effectiveness and sensitivity reported by patients with the two protocols. This means one less step in clinical practice for the dentist; the null hypothesis was accepted. This split-mouth[23] experimental design is a good model to compare experimental and control groups under similar conditions. This controls for confounding variables like habits, diet, and hygiene.

We found no difference between the two groups. Both groups had a change in E of about 5 units—this is considered effective bleaching.[24] These results agree with Martin and others who describe a change of 5 E* units with 6% gel in the office,[2] but with different application protocols: 1) three sessions with two applications, each for 12 minutes; 2) two sessions with three applications for 12 minutes, and 3) two sessions with one application of 36 minutes. All of these approaches are equally effective.

Traditionally, the use of bleaching gels in trays for home bleaching uses contact times of more than two hours with higher concentrations of hydrogen peroxide than in this clinical trial. This is because the effectiveness of the gel is reduced after only two hours of contact.[25] However, the difference in using a gel at home or a gel technique in-office is that the tray allows a reservoir to maintain a more humid environment. The gel used in the in-office technique is exposed to the environment and seemingly loses water faster. This is the argument used by manufacturers to recommend applications of 15 minutes. Kwon and others used a linear low-density polyethylene wrap to prevent dehydration of the gel during a prolonged bleaching session without replenishment of the whitening gel.[26] This trial demonstrates that continuous use of hydrogen peroxide gel for 36 minutes of low peroxide concentration gel activated by light LED/laser is effective but with an abbreviated application protocol.

Table 4: Delta SGU by VITA classical A1-D4 and VITA Bleachedguide 3D-MASTER Guides of Both Groups at Each Checkpoint

	VITA classical A1-D4				VITA Bleachedguide 3D-MASTER			
	First session ΔSGU median (min/max)	**Second session ΔSGU median (min/max)**	**Week control ΔSGU median (min/max)**	**Month ΔSGU median (min/max)**	**First session ΔSGU median (min/max)**	**Second session ΔSGU median (min/max)**	**Week control ΔSGU median (min/max)**	**Month ΔSGU median (min/max)**
Group A	4 (−1/11)	6 (2/13)	6 (−1/10)	6 (−1/10)	2 (0/5)	3 (1/6)	2.5 (0/6)	2 (0/5)
Group B	5 (0/9)	6 (0/11)	6 (0/9)	5.5 (0/10)	3 (1/6)	4 (1/7)	3 (0/6)	2 (−1/6)
Mann-Whitney test *p* value	0.808	0.669	0.898	0.580	0.395	0.522	0.401	0.773

Effectiveness was measured subjectively on two scales—the VITA classical A1-D4 and VITA Bleachedguide 3D-MASTER guide. There were no differences between the two protocols on either scale. The VITA classical A1-D4 scale is a simple scale to use and is popular among dentists. However, the distribution of colors is not symmetrical according to the dimensions of color. Thus, the validity of this instrument to capture the variation in teeth is not completely reliable. For this, we also decided to use the VITA Bleachedguide 3D-MASTER Guide scale that is distributed more uniformly according to brightness. This allows greater consistency and reproducibility to measure changes during tooth bleaching.[27] However, we emphasize the split-mouth design. There is a high risk of bias in measuring a neighboring tooth because the human eye cannot distinguish between fewer than two E* units. This could affect data collection[28]—a problem described by Martin and others in a similar study.[2]

Matis and others[25] showed that about 30% of the hydrogen peroxide remained active after 30 minutes. This suggests that the 6% gel with activated LED/laser light applied for 36 minutes is equal to the group with three applications of 12 minutes. The chemical reaction catalyzed by the blue light LED uses titanium dioxide as a semiconductor nanoparticle to catalyze the formation of hydroxyl radicals from hydrogen peroxide. On the other hand, the infrared laser can also hyperpolarize nerve pain and generate immediate sensitivity control. This is reflected in the low rates of sensitivity and intensity induced by bleaching.[15] One of the gel change justifications for the bleaching session is because of the pH of the bleaching gel.[29] With the passage of time, a greater quantity of free radicals will be released. Thus, there is a pH decrease.[30] It is known that more demineralization will occur when the pH of the bleaching gel becomes more acidic.[31] This mineral loss disrupts the enamel prisms and increases the possibility of sensitivity—often described as "zinger" pain.[32]

The intervals studied here were shorter than the commonly used seven days. De Paula and others[8] showed that there was no impact on the postbleaching sensitivity by shortening intervals to two days between sessions. Our results are consistent with the results of this trial because there was no difference. It is also important to consider that postbleaching sensitivity is dependent on the concentration of hydrogen peroxide gel. Trial reports using gels of 35% or even 15% show higher absolute values and intensity of postbleaching sensitivity than our results.[7,10,24,33,34] However, we must consider that the low values of sensitivity in this work are due to activating the bleaching gel by hybrid infrared LED/laser.[15] There must be an immediate influence on nerve fibers (ΔA) by the action of hyperpolarization of the laser. This mediates the sensitivity and should not have been influenced by the studied application protocols. The sensitivity and intensity values were low relative to the literature.[7,24,35-40]

The actual role of the blue light (cold lamp) is unknown. It could be a catalyst for a chemical reaction. Other data show that the use of light does not increase the effectiveness of in-office bleaching.[3] Titanium oxide is a semiconductor under blue light and theoretically catalyzes the formation of hydroxyl radicals from hydrogen peroxide.[41] The exact role by which light or titanium oxide nanoparticles catalyze the mechanism of action remains unclear. In the literature, 6% hydrogen peroxide gels should be applied for >20 hours for effective bleaching.[36] In this trial, there was a contact of only 72 minutes. This assumes that the light (LED/Infrared Laser) is the catalyst for the chemical reaction. The effect of the infrared laser clearly influenced the values of absolute risk and intensity of sensitivity induced by bleaching. It coincides with Martin and others,[2] who described a 36.6% risk of mild sensitivity from bleaching vs the 6.25% shown here. Some authors correlate the presence of an acute inflammatory reaction in dental pulps of human bleached teeth with gel in high concentrations (>35%). This suggests that some released mediators of the inflammatory reaction such as bradykinin[42] or substance P[43]—probably due to the low concentrations of peroxide—are lower, and their effect is a nonexistent mediator of the sensitivity reported by patients.

The limitations of this study include the impossibility of blinding the operators and patients regarding the change of technique. However, the calibrated blinding of evaluators was very strict. The split-mouth design is an excellent tool[39] because it allows different groups within the same patient. Thus, each patient is his or her own control. This eliminates confounding variables. The major problem of this design is a potential leakage of the treatment effect from one site to another called the carry-across effect, in this study potentially reflected as a bias in sensitivity assessment.[23]

CONCLUSIONS

There was no significant difference in tooth whitening with 6% hydrogen peroxide bleaching gel in one

36-minute application compared with the traditional three 12-minute applications. There were no differences in sensitivity noted for these two protocols.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the University of Chile. The approval code for this study is: 2015-01b.

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

Stress-strain Analysis of Premolars With Non-carious Cervical Lesions: Influence of Restorative Material, Loading Direction and Mechanical Fatigue

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Clinical Relevance

Stress-strain analysis confirmed that non-carious cervical lesions (NCCLs) restored with resin-based composite or combining resin-based composite with a ceramic laminate have a more favorable biomechanical behavior. Removal of occlusal interferences reduces stress-strain pattern in the cervical tooth structure and NCCL restorative material.

SUMMARY

Noncarious cervical lesions (NCCLs) are characterized by a loss of dental structure at the cementoenamel junction (CEJ) caused by stress, biocorrosion, and attrition. Variations in occlusal loading can promote different stress and strain patterns on the CEJ. Restoration of NCCLs is part of lesion management; however, there is still no conclusive restorative protocol for NCCLs. This study aimed to evaluate the stress and strain distribution of

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maxillary premolars with NCCLs according to three factors: 1) restorative technique; 2) direction of occlusal loading; and 3) mechanical fatigue. Three-dimensional (3D) finite element analysis (FEA) and strain gauge testing were used to assess stress and strain, respectively. 3D-FEA orthotropic, linear, and elastic models were generated: sound tooth (SO); unrestored NCCL; or NCCL restored with glass ionomer; flowable composite resin; nanofilled composite resin (CR); lithium disilicate ceramic; and nanofilled composite resin core associated with a lithium disilicate laminate (CL). A 150-N compressive static load was applied in two conditions: axially in both cusps (Al); and at a 45° angle to the long axis of the tooth applied to the palatine cusp (Ol). For the experimental tests, specimens were treated as described previously, and one strain gauge was attached to the buccal surface of each tooth to record tooth strains before and after cyclic loading (200,000 cycles, 50 N). FEA showed that the association of NCCL and Ol resulted in higher stress values. CR and CL restorations showed the closest biomechanical behavior to SO for both loading types. Loaded Al or Ol specimens showed higher strain values after mechanical fatigue. Lower stress and strain were observed with Al when compared with Ol. The restoration of NCCLs with composite resin only or associated with ceramic laminates seems to be the best approach because the results for those groups were similar in biomechanical behaviors to sound teeth.

INTRODUCTION

The dental structures in the cervical region are more vulnerable to wear because enamel is very thin at this site, and cementum and dentin are not very resistant.[1] This specific tissue loss is a common finding, reported in up to 60% of patients, and is most prevalent in maxillary posterior teeth, mainly premolars.[2,3] Cervical wear is classified as a noncarious cervical lesion (NCCL), which is a pathological process characterized by loss of dental hard tissues near the cementoenamel junction (CEJ).[4] Multiple factors can be associated with this process, such as stress (abfraction: parafunction and traumatic occlusion), friction (wear: toothbrush/dentifrice abrasion), and biocorrosion (chemical, biochemical, and electrochemical degradation: extrinsic and intrinsic acids).[4]

The multifactorial etiology of NCCLs results in varying and unclear management protocols that are still controversial.[5] The treatment of NCCLs may be multidisciplinary, including occlusal analysis and adjustment or replacement of lost dental structures, associated with instructing patients about oral habits, which is essential to the success of rehabilitating NCCLs.[6] Distinct occlusal loading conditions can lead to changes in the stress distribution patterns at the cervical region.[7,8] The stress concentration and fatigue process may result in rupture of brittle structures, such as enamel,[8,9] while also favoring gaps at the interface of restorative materials and dental structures, with possible restorative failures.[10] However, simple restoration of cervical lesions does not treat the etiological factors.[11]

Loss of dental structures either by caries, fractures, coronal preparations, or noncarious wear is a key factor for altering the biomechanical behavior of teeth.[12,13] Therefore, restorative materials that present mechanical properties similar to dental tissues can be advantageous for repairing NCCLs and restoring the stress-strain pattern of sound teeth.[13] Although several studies have analyzed and described restorative protocols for NCCLs,[10,13,14] the literature is still missing deeper investigations considering the effect of different materials and restorative techniques for these lesions. However, consensus exists regarding the use adhesive materials with mechanical and optical properties similar to tooth structures,[6] which can improve biomechanical behavior[13] and esthetics and reduce hypersensitivity.[11]

Composite resins,[6] glass ionomer cements,[10] and flowable resins[14] are the materials most commonly used for replacing lost enamel and dentin at the cervical region. Nevertheless, the survival rate of these restorations can be influenced by chemical degradation and attrition,[15,16] a reduction in hardness,[16] stress concentration during mastication,[17,18] shrinkage stress,[19,20] deficiencies in enamel margins,[21] color mismatch,[21] and reduced adhesion to sclerotic dentin, which is usually present at the base of NCCLs.[22,23]

Although composite resins present good biomechanical behaviors due to their ability to mimic dentin,[13] enamel properties are more closely mimicked by ceramic materials.[24] The advances of indirect restorative materials has resulted in satisfactory bond strengths among glass ceramics, composite resin cements, and dental structures.[25,26] The introduction of strengthened glass ceramics with notable optical properties and good survival

rates[27,28] has allowed for their use in areas subjected to high mechanical efforts demanding esthetics.[28] In addition, glass ceramics present excellent surface smoothness.[29] This aspect is relevant due to the high frequency (69.5%) of NCCLs in which the gingival wall angle is at gingival or subgingival levels[30] because the proximity of restorations to the periodontal margin can increase gingival bleeding, insertion loss, and gingival recession.[31] However, well-finished restorations and smoother materials have been shown to accumulate less plaque and remain free of periodontal pathologies.[32]

Thus, the purpose of this study was to evaluate the stress and strain distribution of maxillary premolars with NCCLs using three-dimensional (3D) finite element analysis (FEA) and strain gauge testing according to three factors: 1) restorative technique; 2) direction of occlusal loading; and 3) mechanical fatigue. The hypotheses tested was that the biomechanical behavior of premolars with NCCLs is not affected by restorative materials with different elastic moduli, occlusal loading direction, or cyclic loading.

METHODS AND MATERIALS

Finite Element Analysis

3D finite element linear elastic analysis was performed using anatomically based geometric representations for pulp, dentin, enamel, periodontal ligament, and cortical and medullar bones.[33] Fourteen models were generated (Rhinoceros 3D software, Rhinoceros, Miami, FL, USA) simulating sound tooth (SO); unrestored buccal saucer-shaped NCCLs (UN); and NCCLs restored with resin-modified glass ionomer (GI); flowable composite resin (FR); conventional nanofilled composite resin (CR); lithium disilicate glass ceramic (LD); and conventional nanofilled composite resin core associated with a 0.5-mm lithium disilicate glass ceramic laminate (CL).

The models were exported to the processing analysis software (ANSYS 12.0, Ansys Workbench 12.0.1, Canonsburg, PA, USA) using the Standard for the Exchange of Product Data (STEP) format (Figure 1). The following steps were performed in this software: preprocessing (definition of mechanical properties, volumes, connection types, mesh for each structure, and boundary conditions), processing (data calculation), and postprocessing (analysis of results by stress distribution criteria). All dental structures and restorative materials were considered homogeneous and linearly elastic. Enamel and dentin were considered orthotropic and the other structures isotropic (Table 1).[10,34-40]

After testing the mesh conversion to define the appropriate mesh refinement level, volumes corresponding to each structure were meshed with the controlled and connected elements. The meshing process involved division of the studied system into a set of small discrete elements defined by nodes. Solid quadratic tetrahedral elements of 10 nodes were used (Figure 2A). The mesh conversion test was initiated using the software automatic meshing and was continued by gradually decreasing the size of the elements. For each test stage, the results were generated by equivalent stress criterion (von Mises) to verify the higher stress values of dentin. The mesh was considered satisfactory when, even reducing the dimension of elements, the higher stress levels were similar to the results observed with the previous mesh refinement. The number of elements used varied depending on the different volumes, so that the final model accurately represented the original geometry (Table 2). Due to the adhesive properties of the restorative materials used, restorations were bonded to dental structures by considering a mesh connection with dentin and enamel.

The boundary conditions consisted of developing a displacement/restriction model using load application. Loading of models (150 N) was applied to specific surfaces previously defined in the computer-aided design software, as follows: Axial loading (Al) was equally distributed on both cusps, simulating homogeneous contact distribution (Figure 2B); oblique loading (Ol) simulated occlusal interference on the palatine cusp of the model,[8] with the load applied at a 45° angle to the long axis of the tooth (Figure 2C). Models were restrained at the base and lateral surfaces of cortical and trabecular bone to avoid displacement (Figure 2D). Stress distribution analysis was performed using equivalent stress criterion (von Mises). After complete stress analysis in all structures, the results were plotted in transparency, except for enamel, dentin, and restorative materials, for better visualization. The FEA strain values of the restorative materials were also measured on all nodes corresponding to the surfaces where the strain gauge was fixed on the laboratory strain gauge test to allow comparisons.

Strain Gauge Test and Cyclic Loading

For the strain gauge test, 25 intact human maxillary single-rooted premolars, free of cracks and defects, were selected (gathered following an informed

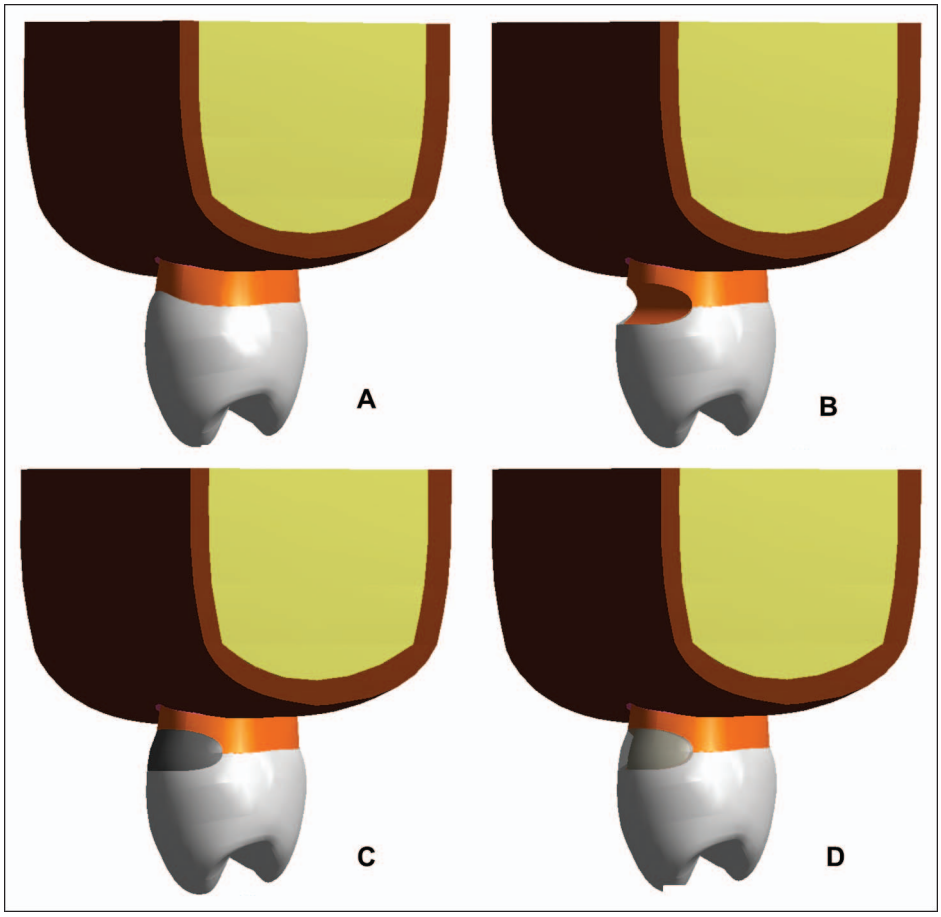

Figure 1. *Volume for the different CAD models. (A): Sound tooth. (B): Unrestored NCCL. (C): NCCL restoration with single material. (D): NCCL restored with two materials.*

Table 1: *Mechanical Properties of Orthotropic and Isotropic Structures*

Structure	Orthotropic Structures		
	Longitudinal	Transversal	Z
	Elastic Modulus (MPa)		
Enamel[34]	73,720	63,270	63,270
Dentin[34]	17,070	5610	5610
	Shear Coefficient (MPa)		
Enamel[34]	20,890	24,070	20,890
Dentin[34]	1700	6000	1700
	Poisson Ratio (v)		
Enamel[34]	0.23	0.45	0.23
Dentin[34]	0.30	0.33	0.30
	Isotropic Structures		
	Elastic Modulus (MPa)	Poisson Ratio (v)	
Flowable resin[35]	5300	0.28	
Glass ionomer[10]	10800	0.30	
Lithium disilicate[36]	65000	0.23	
Pulp[37]	2.07	0.45	
Periodontal ligament[38]	68.9	0.45	
Cortical bone[39]	13,700	0.30	
Medullar bone[39]	1370	0.30	
Hybrid composite resin[40]	22,000	0.27	

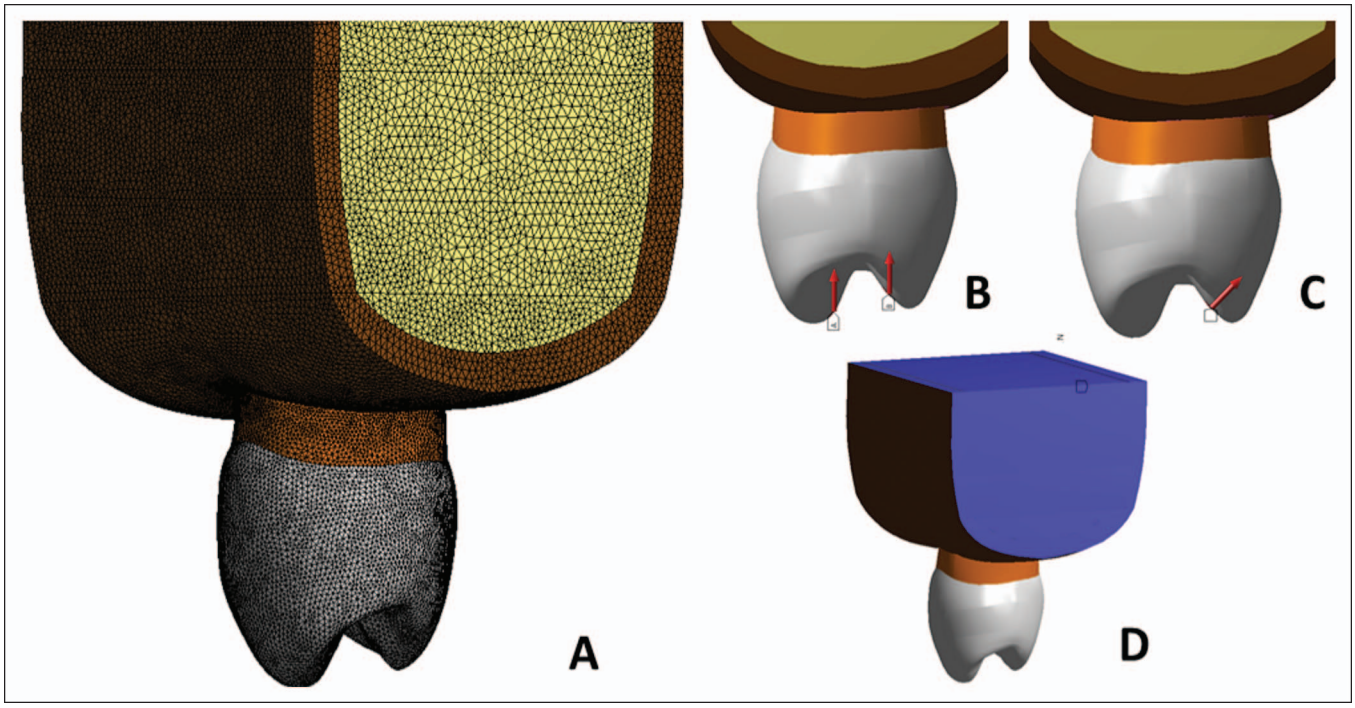

Figure 2. *3D-FEA model generation and boundary conditions. (A): Meshing of the models. (B): Axial loading. (C): Oblique loading. (D): Displacement restriction (null displacement in the blue area).*

consent approved by the Committee for Ethics in Research: No. 539.002). Teeth of similar size and shape were selected by crown dimensions after measuring the bucco-lingual and mesio-distal widths in millimeters, allowing a maximum deviation of 10% from the determined mean, providing greater standardization of samples. The roots of the specimens were covered with a 0.2-mm layer of polyether-based impression material (Impregum Soft, 3M ESPE, St Paul, MN, USA) and embedded in a polystyrene resin cylinder, simulating the periodontal ligament and alveolar bone, respectively.[41]

One strain gauge (PA-06-038AB-120LEN, Excel Sensors, São Paulo, SP, Brazil) was positioned parallel to the long axis at the buccal surface of the tooth, 2.0 mm above the CEJ. The base material of the gauges consisted of a polyimide and metal constantan film, with temperature self-compensation for steel, and the strain gauge grid had an area of 1 mm^2 and electrical resistance of 120 Ω. Strain gauges used for this study had a gauge factor of 2.13. The gauge factor is a proportional constant for the electrical resistance variation and strain. For strain gauge attachment, enamel was etched with 35% phosphoric acid (Scotch Bond Etchant, 3M ESPE) for 30 seconds, rinsed with water, and air dried. Then, the strain gauge was bonded to the tooth structure using cyanoacrylate-based adhesive (Super Bonder, Loctite, São Paulo, SP, Brazil) and connected to a data acquisition system (ADS0500IP, Lynx, São Paulo, SP, Brazil). In addition, a control specimen with one strain gauge attached but not subjected to load application was mounted adjacent to the test tooth to compensate for dimensional alterations due to temperature fluctuations from the gauge electrical resistance or local environment.[42]

Table 2: *Number of Elements and Nodes for Each Model*

Model	Nodes	Elements
Sound tooth (SO)	2,473,693	1,709,931
Unrestored NCCL (UN)	2,444,468	1,688,610
Restorative technique with unique material (GI, FR, CR, and LD)	2,475,971	1,710,237
Restorative technique with two materials (CL)	2,479,488	1,711,624

Abbreviations: SO, sound tooth; UN, unrestored noncarious cervical lesions; GI, glass ionomer; FR, flowable composite resin; CR, nanofilled composite resin; LD, lithium disilicate ceramic; NCCL, noncarious cervical lesions; CL, nanofilled composite resin core associated with a lithium disilicate laminate.

The 25 sound teeth were subjected to a nondestructive axial (Al) and oblique (Ol) 0- to 150-N ramp-load at 0.5 mm/min, applied using a 4.0-mm diameter sphere and knife-shaped tip, respectively, in a mechanical testing machine (DL 2000, EMIC, São José dos Pinhais, PR, Brazil). All sound specimens were then submitted to 200,000 cycles (2

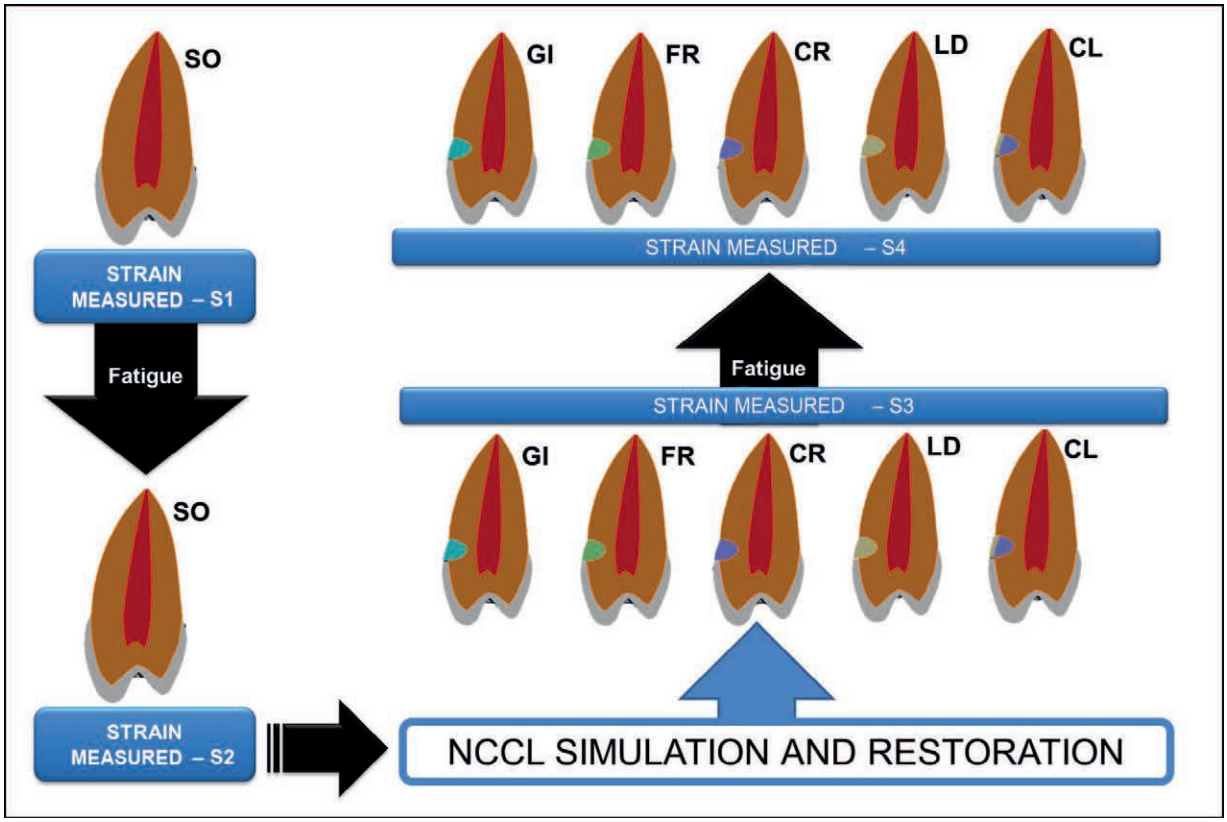

Figure 3. *Strain gauge test and mechanical fatigue scheme.*

Hz) of Ol on the palatine cusps (50 N), simulating approximately 10 months of clinical service.[43] Following mechanical aging, the specimens were resubmitted to Al and Ol up to 150 N, as described before, and the strains were measured (Figure 3).

Then, the strain gauges were removed and saucer-shaped NCCLs were simulated in the buccal wall of all specimens using diamond burs (No. 3118, KG Sorensen, São Paulo, SP, Brazil), creating 2.5-mm deep and 2.5-mm wide cavities. Afterward, the specimens were divided into five groups according to the materials used to restore the NCCLs (n=5): GI, FR, CR, LD, and CL. Selective etching of enamel was performed using 35% phosphoric acid for 15 seconds (Scotch Bond Etchant, 3M ESPE), and a self-etching adhesive system (Scotch Bond Universal, 3M ESPE) was used for hybridization, except with the GI group. The GI (Riva Light Cure, SDI, Victoria, Australia), FR (Filtek Z350 XT Flow, A3 Shade, 3M ESPE), and CR (Filtek Z350 XT, A3 Shade, 3M ESPE) were inserted in 2.0-mm increments, photoactivated for 20 seconds using an LED curing unit (Radii-Call, SDI) with a 1200 mW/cm^2 output, until complete filling of the cavity. For the indirect restorations, impressions of the NCCLs were made using vinyl-polysiloxane (Express, 3M ESPE), and stone casts were poured in type IV dental stone (Durone IV, Dentsply, Petrópolis, RJ, Brazil). For the CL specimens, a composite resin core was built, and after taking impressions and pouring stone casts as described previously, 0.5-mm ceramic laminates were obtained. All LD and CL specimens were restored using lithium disilicate glass-ceramic veneers or laminates, respectively (IPS e.Max Press, MO 1 shade, Ivoclar Vivadent, Schaan, Liechtenstein). The internal surfaces of the ceramic restorations were etched with 9.5% hydrofluoric acid for 20 seconds (Condicionador de Porcelanas, Dentsply). Then, the surfaces were rinsed with water for 30 seconds, and 35% phosphoric acid was applied for 60 seconds (Scotch Bond Etchant, 3M ESPE), followed by water rinsing and air drying. A silane coupling agent (Monobond Plus, Ivoclar Vivadent) was actively applied to the restoration and left to react for 1 minute, followed by luting with a light-cure resin cement (Variolink Veneer, Ivoclar Vivadent) that was light activated for 60 seconds.

New strain gauges (PA-06-038AB-120LEN, Excel Sensors) were then attached to the restorations, as described previously. All restored specimens were again submitted to Al and Ol up to 150 N for strain measurements. Sequentially, specimens were resubmitted to 200,000 cycles (2 Hz) of Ol on the palatine cusp (50 N). Finally, the strain of the specimens was measured for both compressive loading types (Al and Ol) up to 150 N after the second mechanical aging. The strain values were recorded at 4 Hz during the compressive loading, and the data were obtained from strain gauges through data analysis software (AqDados 7.02 and AqAnalisys). The strains were analyzed using a three-way analysis of variance (ANOVA) and a Tukey test for comparisons among study factors and their interactions (Occlusal Loading × Restorative Technique × Mechanical Fatigue). One-way ANOVA and paired *t*-test were used for comparisons between groups before and after restorative procedures (Sound Tooth × NCCL + Restoration). All tests were performed at a 95% confidence level.

RESULTS

The stress distribution for all models under the different restorative conditions and loading directions is presented in Figures 4 through 6. The variation in occlusal loading induced pronounced differences in the stress distribution, regardless of the presence of an NCCL or the restorative material type. For the SO model, Al resulted in homogeneous stress distribution, with no stress peaks located in the tooth structure (Figure 4: SO/Al). Ol resulted in high stress concentrations at the cervical area from the buccal and palatal regions for the SO model, mainly in the dentin and enamel at the CEJ (Figure 4: SO/Ol). The presence of an NCCL changed the stress pattern of the UN model when compared with SO, mainly for the dentin at the bottom of the lesion and in the occlusal wall of enamel (Figure 4: UN/Al). Higher stress concentration was observed when the loss of structure (UN) was combined with Ol (Figure 4: UN/Ol).

Irrespective of the restorative technique and occlusal loading, the replacement of lost tooth tissue with adhesive restorations recovered biomechanical behavior closer to the SO model. For the Al, similar behaviors were verified for all restorative materials (Figure 5). However, when restored models were obliquely loaded on the palatal cusp (Ol), some differences were observed among the restorative materials. The GI model showed lower stress concentration in the restorative material, similar to FR. The LD model presented higher stress concentrations in the restorative material. CR and CL showed similar stress distributions, with biomechanical behavior comparable with the SO model (Figure 6).

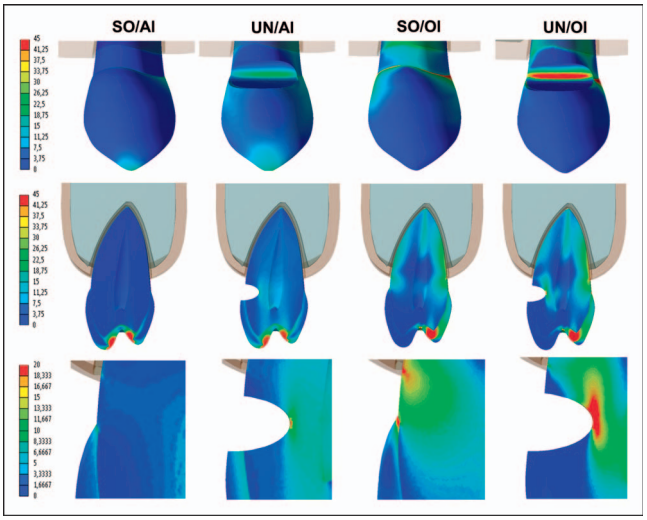

Figure 4. *Stress distribution by von Mises stress for sound tooth (SO) and unrestored NCCL (UN) according to the loading condition: Al, axial loading; Ol, oblique loading.*

The mean strain values for all groups under the different loading conditions are shown in Tables 3 and 4. The deflection of the restorative materials verified by the strain gauge test was significant only for the GI and LD groups, depending on the occlusal loading. Regardless of the restorative material and occlusal loading, strain increased with mechanical fatigue. When comparing the effect of different restorative materials, groups involving restorative techniques with ceramics (LD and CL) showed lower strain values, irrespective of occlusal loading type or fatigue. CR presented intermediate strain values, similar to FR for both Al and Ol. GI showed the highest strain values for Al and Ol, whereas there was no statistically significant difference for the FR group when evaluating Al (Table 3).

When analyzing, by a paired statistics method, the strain of sound tooth samples compared with their respective restored technique group, GI and CR showed higher strain for both occlusal loadings, irrespective of mechanical fatigue. The specimens restored with FR presented similar strain to SO/Ol before fatigue. After cyclic loading, the same specimens showed higher strains. LD presented similar strain to SO, regardless of loading type and fatigue. CL showed lower strains when compared to SO prior to fatigue for both loading types. However, after fatigue, CL showed no differences to the SO group

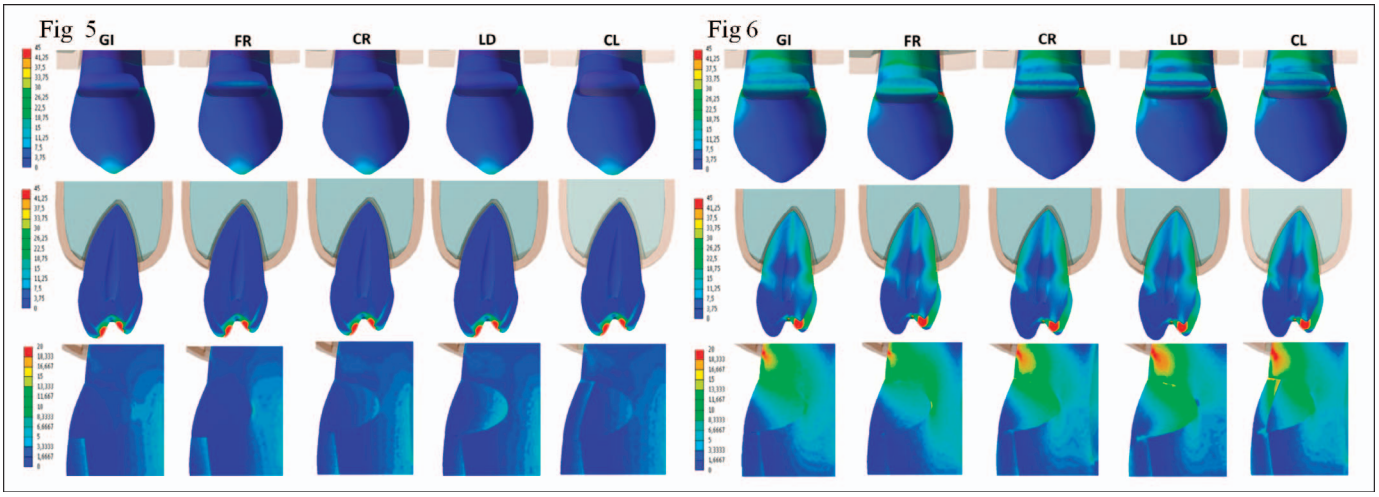

Figure 5. *Stress distribution by von Mises stress with axial loading according to the restorative materials.*
Figure 6. *Stress distribution by von Mises stress with oblique loading according to the restorative materials.*

(Table 4). The FEA strain measurements presented similar patterns when comparing the values obtained experimentally using the strain gauge test before fatigue, validating both methodologies (Figure 7).

DISCUSSION

According to the results, the hypothesis was rejected because the different restorative materials with distinct elastic moduli, different occlusal loading types, and the presence of cyclic loading changed the biomechanical behavior of maxillary premolars affected by NCCLs.

Despite the ease of using direct techniques for restoring NCCLs,[14] etiological factors such as acid degradation and attrition, among others, can also affect direct restorative materials.[15,16] When associated with stress accumulation during function,[17,18] these factors may result in low survival rates for direct restorative procedures in NCCLs.[17,18] Mechanical fatigue increased the strains on the restoration for all materials and loading conditions in the current study. However, when comparing the teeth before (sound) and after restoration following the fatigue process, higher strains were detected for the direct restorative materials, irrespective of loading type. Alternatively, groups restored with indirect materials (LD and CL) presented similar strains when compared with SO after mechanical fatigue.

Another point of concern is related to the surface roughness of direct restorations. The presence of NCCLs may occur concomitantly with root exposure,[44] leading to a strong relationship between gingival tissues and cervical restorations. Thus, restorative techniques using materials with smoother and better surface polishing would favor subsequent periodontal treatment.[32] Indirect ceramic restorations have excellent surface polishing because they are submitted to several polishing steps, in addition to the glazing process, which ensures

Table 3: *Mean Strain Values (μS) and Standard Deviation (SD) Comparing Restorative Technique × Occlusal Loading × Mechanical Fatigue*[a]

Restorative Technique	Oblique Loading		Axial Loading	
	Immediately	Mechanical Aging	Immediately	Mechanical Aging
GI	604.5 (66.5) A,a*	825.3 (172.7) A,b*	363.3 (163.7) A,a	442.4 (171.3) A,b
FR	290.1 (74.5) B,a	410.4 (121.2) B,b	265.2 (83.5) AB,a	324.9 (16.2) AB,b
CR	233.2 (57.3) B,a	315.5 (92.0) B,b	188.0 (45.0) B,a	272.8 (92.0) B,b
LD	156.4 (61.5) C,a*	180.4 (47.9) C,b*	63.7 (15.1) C,a	81.6 (21.6) C,b
CL	85.6 (22.2) C,a	109.1 (31.0) C,b	50.8 (13.8) C,a	87.6 (29.0) C,b

Abbreviations: SO, sound tooth; UN, unrestored noncarious cervical lesions;; GI, glass ionomer; FR, flowable composite resin; CR, nanofilled composite resin; ; LD, lithium disilicate ceramic; NCCL, noncarious cervical lesions;CL, nanofilled composite resin core associated with a lithium disilicate laminate.
[a] *Uppercase letters for vertical comparisons (restorative techniques). Lowercase letters for horizontal comparisons (mechanical aging).*
* *Significant influence of the occlusal loading for horizontal comparisons. Three-way analysis of variance and Tukey test;* $p < 0.05$.

Table 4: *Mean Strain Values (μS) and Standard Deviation (SD) Comparing Sound Tooth × NCCL + Restoration*

Restorative Technique	Occlusal Loading	Mechanical Aging	SO Strain	Material Strain	*p* Value
GI	Axial load	Immediately	85.7 (9.6)	363.3 (163.7)	.013*
		Fatigue	150.0 (34.8)	442.4 (171.3)	.016*
	Oblique load	Immediately	254.3 (36.5)	604.5 (66.5)	<.001*
		Fatigue	285.2 (43.9)	825.3 (172.7)	.001*
FR	Axial load	Immediately	75.2 (24.1)	265.2 (83.5)	.010*
		Fatigue	135.0 (38.2)	324.9 (16.2)	<.001*
	Oblique load	Immediately	200.0 (80.5)	290.1 (74.5)	.128
		Fatigue	174.8 (49.3)	410.4 (121.2)	.010*
CR	Axial load	Immediately	93.5 (37.2)	188.0 (45.0)	.003*
		Fatigue	118.1 (76.7)	272.8 (92.0)	.019*
	Oblique load	Immediately	149.2 (36.7)	233.2 (57.3)	.049*
		Fatigue	163.0 (81.0)	315.5 (92.0)	.014*
LD	Axial load	Immediately	90.1 (30.7)	63.7 (15.1)	.07
		Fatigue	103.4 (33.9)	81.6 (21.6)	.143
	Oblique load	Immediately	163.1 (53.4)	156.4 (61.5)	.453
		Fatigue	164.2 (56.9)	180.4 (47.9)	.374
CL	Axial load	Immediately	81.4 (15.5)	50.8 (13.8)	.014*
		Fatigue	138.4 (46.2)	87.6 (29.0)	.103
	Oblique load	Immediately	155.1 (43.1)	85.6 (22.2)	.012*
		Fatigue	244.7 (109.1)	109.1 (31.0)	.058

Abbreviations: SO, sound tooth; UN, unrestored noncarious cervical lesions;; GI, glass ionomer; FR, flowable composite resin; CR, nanofilled composite resin; ; LD, lithium disilicate ceramic; NCCL, noncarious cervical lesions;CL, nanofilled composite resin core associated with a lithium disilicate laminate .
** Significant difference between sound tooth (before) and NCCL + restoration (after). One-way analysis of variance and paired t-test; p < 0.05.*

notably smoother surfaces.[45] Improvements in the surface smoothness of direct restorative materials is a constant research subject for glass ionomers and composite resins.[16,21,46] However, direct restorations, especially ionomer-based materials, still present poor surface roughness.[21] On the other hand, direct materials do not damage periodontal tissues if restoration finishing and polishing is performed properly, allowing for a satisfactory restoration and surface roughness.[32]

Occlusal loading influenced the stress distribution and strain patterns. Loading applied on the palatine cusp was the main factor responsible for concentrating stresses in the tooth, especially at the cervical region. Ol also resulted in higher stress concentration on the material when compared with Al. The first condition can clearly represent what happens in clinical situations of teeth with premature contacts, which is relevant for the formation and progression of NCCLs.[8,47] This finding was also verified using the strain gauge test, when specimens were submitted to cyclic loading simulating a nonphysiologic occlusal contact, resulting in higher deformation. The present results support the importance of performing occlusal adjustments prior to restoring NCCLs. Even when NCCLs are already restored, fine adjustments of occlusal contacts should be properly performed to reduce stress concentration in dental structures. Lower stress accumulation at the cervical region and restoring the tooth may benefit the longevity of the selected restorative protocol.[48]

The loss of tooth structure at the cervical area is considered a relevant factor in the modification of the biomechanical behavior of teeth. It is important to observe the mechanical properties of dental tissues and restorative materials when restoring the loss of tooth structure. The ideal material should recover the biomechanical behavior of the lost structure, resulting in a similar mechanical pattern to sound tooth structures.[13,24] Flowable composite resins have been indicated for restoring NCCLs due to their low viscosity, which is commonly obtained by reducing the filler content and/or modifying agents, which facilitates material insertion and adaptation to the cavity walls.[3] However, this material has a low elastic modulus and an increased organic matrix, which promotes lower stress concentration; this tends to increase polymerization shrinkage and result in higher residual shrinkage stress on dental tissues.[49,50]

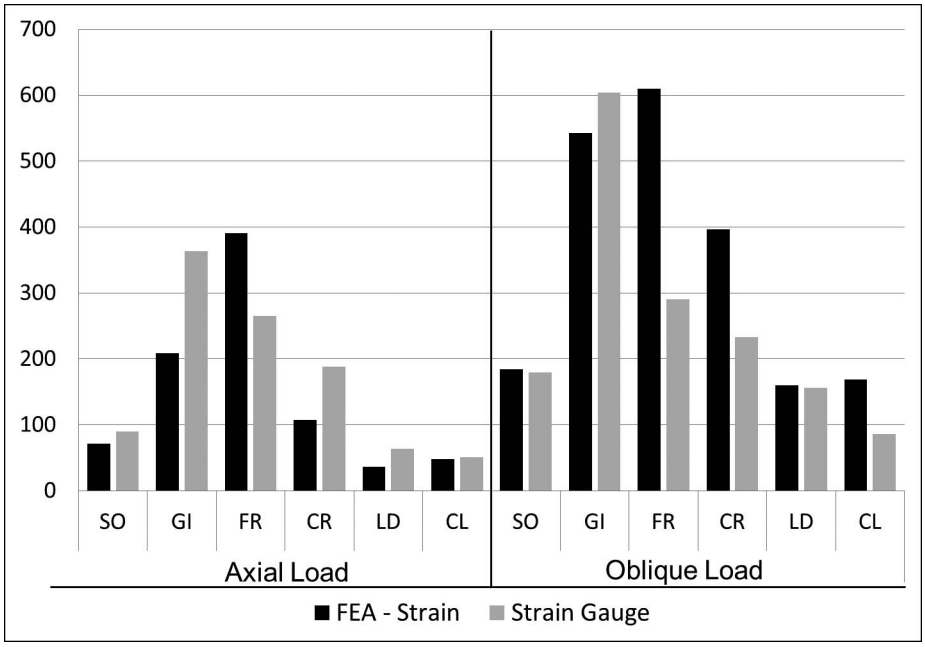

Figure 7. *Strains measured by FEA and strain gauge method on the same region of teeth.*

Glass ionomers and resin-modified ionomers also appear as options for restoring NCCLs because they present adhesion to tooth structures, acceptable biocompatibility and esthetics, and provide a good relationship with dentin hypersensitivity.[48] Glass ionomers have advantages, such as low viscosity, fluoride release, and an easy insertion technique.[14] However, poor strength and hardness are the main disadvantages in using GIs when restoring NCCLs.[51] In addition, FRs and GIs do not present similar mechanical properties when compared with dentin or enamel. These materials are more prone to deformation due to their lower elastic moduli, accumulating lower stress. For this reason, tooth structures can experience higher stress and strain concentration when restored with these materials.

Most conventional composite resins present similar elastic moduli to dentin; adhesive restorations can help to compensate for this stress generated by dental tissue loss.[52] Although widely used, composite restorations present some limitations, such as polymerization shrinkage, marginal discoloration, microleakage, and postoperative sensitivity.[14,19,20] The strain presented by the nanofill composite used to restore NCCLs in this current study was similar to that observed for the flowable composite restorations. The lithium disilicate glass ceramic, unlike composite resins, presented an elastic modulus that was closer to enamel. Because this material is more rigid, the restorations accumulated more stress and did not present high strains. Thus, when NCCLs are restored using only LD, higher stress concentration may occur on the gingival wall of the lesion.

The restorative technique combining composite resin and ceramic for restoring NCCLs is based on the principle of recovering the biomechanical behavior of sound tooth using materials with similar properties to the lost tissues. Therefore, a composite resin core was used, simulating lost dentin, and a thin ceramic laminate was used to simulate enamel in the cervical region. The results for the CL group confirmed this principle, presenting a stress and strain pattern similar to a sound tooth. Despite the excellent results obtained with the association of a composite resin core and ceramic laminate, the use of nanofilled composite resin alone also presented as a good alternative when restoring NCCLs. Moreover, high success rates were observed for NCCLs restored with direct composite restorations,[53] and restorative techniques using ceramics are usually more expensive.

Nondestructive methods are useful for analyzing the biomechanical behavior of teeth associated with dental tissue loss, distinct occlusal conditions, and different restorative materials.[13,54] FEA and the strain gauge test allow for the evaluation of the effects caused by the different treatments during physiological and nonphysiological occlusal situations, analyzing the relation between stress and strains. Thus, different factors can be evaluated in the same specimen, preventing destructive damage.[13,55,56] However, these methodologies present some limitations. For FEA, the use of homogeneous

and static models does not consider the consequences of polymerization shrinkage; and the strain gauge test also presents limitations, given that it only performs measurements of deformations on the external surface, without reflecting internal structure deformations. High standard deviations were verified with this technique, due to specimen variability. Because of these limitations, correlation of the methodologies is important for validating the results. Further clinical studies that take into consideration the restorative aspects evaluated in this investigation would be of benefit.

CONCLUSION

Within the limitations of this *in vitro* study, it was concluded that the presence of NCCLs associated with Ol resulted in high stress concentration in the cervical region. Ol was the main factor affecting the biomechanical behavior of teeth with NCCLs. Restorative techniques using materials with similar mechanical proprieties for the replacement of the lost tooth structures showed stress-strain patterns that were similar to sound teeth when subjected to an axial loading.

Acknowledgements

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Regulatory 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 was #539.002.

Conflict of Interest

The authors declare no conflict of interest.

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Proximal Contact Repair of Complex Amalgam Restorations

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Clinical Relevance

The repair of an open proximal contact in an otherwise functional, intact existing complex amalgam restoration may be an acceptable alternative to replacement.

SUMMARY

The carving of a complex amalgam restoration may occasionally result in light proximal contact with the adjacent tooth. The purpose of this study was to investigate the strength of complex amalgam restorations repaired with a proximal slot amalgam preparation. Extracted human third molars of similar coronal size were sectioned 1 mm apical to the height of the contour using a saw and were randomly distributed into 9 groups of 10 teeth each. One pin was placed at each line angle of the flattened dentinal tooth surface. A metal matrix band was placed and an admixed alloy was condensed and carved to create a full crown contour but with a flat occlusal surface. A proximal slot was prepared with or without a retention groove and repaired using a single-composition spherical amalgam 15 minutes, 24 hours, one week, or six months after the initial crown condensation. The specimens were stored for 24 hours in 37°C water before fracture at the marginal ridge using a round-ended blade in a universal testing machine. The control group was not repaired. The mean maximum force in newtons and standard deviation were determined per group. Data were analyzed with a 2-way analysis of variance as well as Tukey and Dunnett tests (α=0.05). Significant differences were found between groups based on type of slot preparation (p=0.017) but not on time (p=0.327), with no significant interaction (p=0.152). No significant difference in the strength of the marginal ridge was found between any repair group and the unrepaired control group (p>0.076). The proximal repair strength of a complex amalgam restoration was not significantly different from an unrepaired amalgam crown. Placing a retention groove in the proximal slot preparation resulted in significantly greater fracture strength than a slot with no retention grooves. Time of repair had no significant effect on the strength of the repair.

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INTRODUCTION

The repair rather than replacement of defective restorations is practiced more frequently today in dental practices. Complete replacement of restorations has the disadvantages of being time-consuming

with unnecessary removal of healthy tooth structure, enlargement of the preparation, the risk of converting the restoration to an indirect restoration, the possibility of major injuries to the pulp tissues, and more expense for the patient. Even though the repair of a functional, intact section of an existing amalgam restoration has been accepted as a practical alternative, it requires sound judgment.

Multiple clinical studies have demonstrated that repairing an existing amalgam restoration is a viable alternative to replacement. Martin and others[1] supported through their five-year clinical study the concept that repair is as effective as total replacement of restorations with localized defects and reduces biological costs to the patient. Minimally invasive treatments of defective amalgam restorations presented similar results to the restorations that were replaced.[1] A clinical study by Smales and others[2] also indicated that repair of local defects in amalgam restorations is an effective alternative to total replacement, at least over a five-year period. A recent prospective, blind, randomized clinical study[3] showed similar results between repaired and replaced Class I and II amalgam restorations after 10 years.

Roggenkamp and others[4] suggested that freshly mixed amalgam added to existing amalgam restorations as a means of repair may be expected to join at nearly the original strength with sufficient condensation time (one and a half seconds) and heavy pressure (four pounds). Under the condensation pressure used in their study, the addition of new amalgam to smooth previously set amalgam surfaces (up to seven years) resulted in shear-bond forces not statistically different from the intact control.[4] Virtually all (94%) of the bonds tested resulted in cohesive rather than adhesive failures. Condensation was done with a consistent force of 22.5 MPa, which is approximately 4 times the force a dental practitioner would apply clinically when condensing an amalgam restoration when deliberately trying to use extra force.[4]

The proper technique of condensing amalgam to the surface of an old amalgam is critical in establishing a bond between a new amalgam and old amalgam restoration. Shen and others[5] showed condensation pressure should be applied vertically to the repair surface whenever possible, or the size of the condenser should be smaller than the repair site in order to exert maximum pressure on the repair surface. Their study[5] also concluded that when repair of an amalgam restoration is carried out, an amalgam material of different composition should be used to achieve greater repair strength. Reducing the dimension of the amalgam repair site has also been shown to improve repair strength.[6] As the diameter of the condenser decreases along the width of the repair site, the axial condensation pressure exerted on the freshly triturated repair amalgam increases. Ogura and others[7] recommend that a spherical alloy should be selected as the repair material due to its high plasticity, which could facilitate the adaptation between new and old amalgam. The modes of measurement of the interfacial bond between new and existing amalgam include tensile,[8] shear,[9-12] and flexural strength values.[13,14] All of these studies, however, have reported that the bond strength of the repaired amalgam was significantly less than that of the intact specimen (less than 50%). If high condensation forces had been used, the strength of the repaired amalgam may not have been significantly less than that of an intact specimen. In addition, the use of heavy pressure upon condensation may infuse the available mercury in the fresh amalgam into the substrate amalgam, potentially precluding the use of spherical alloy.[4]

Fruits and others[15] also concluded the strength of the repaired amalgam was about 40% of that of unrepaired amalgam. Leelawat and others[16] revealed similar results when testing the shear and flexural strengths of the repaired amalgam. There were higher shear bond and flexural strength values when Amalgambond (Parkell, Edgewood, NY, USA) was applied to the prepared surface of the existing amalgam. Even with this treatment, however, the strength values were less than 50% of those of the amalgam control group.[16]

Hadavi and others[8] concluded that fractures in the repaired amalgam always occurred at the junction between old and new amalgam. The repaired amalgam exhibited a reduced tensile strength when compared with intact restorations. The authors[8] also suggested that when an amalgam repair is anticipated, precise mechanical retention must be prepared in the tooth and in the remaining amalgam restoration to complement the union between old and new amalgam alloys. Where the amalgam repair is in functional occlusion, the additional retention is critical to the longevity of the restoration.[8]

All of these studies have compared old amalgam repaired with new amalgam. However, very little investigation has been done to compare the repair of the newly condensed amalgam with newly triturated amalgam. The repair of new amalgam with new amalgam may become necessary when the clinician

discovers a defect after the removal of the matrix band or an open contact is discovered after condensing a full cuspal-coverage amalgam. Bagheri and others[17] revealed when new amalgam was repaired, the samples that were repaired five minutes after the initial condensation had 84% of the tensile strength of the controls. The 15-minute samples had 59% of the tensile strength of the controls, whereas the 30-minute, 60-minute, and 24-hour samples had tensile strength that was less than 50% of the controls. The study concluded that in the repair of the newly condensed (beyond 15 minutes) amalgam restorations, precise mechanical retention must be added.[17] However, in a study by Roggenkamp and others,[4] the *in vitro* shear bond strength at 15 minutes was not statistically different from the unrepaired control using Valiant PhD amalgam under heavy pressure and a one-and-a-half–second condensation time. Fifteen minutes is in the range of immediate chairside repairing of an amalgam accomplished at the same appointment.

No research has evaluated the strength of a complex amalgam after repair of a proximal contact using a slot preparation in human teeth. The purpose of this study was to investigate the strength of complex amalgam restorations repaired with newly triturated amalgam and added mechanical retention. The site of repair was the proximal contact area of a full cuspal-coverage amalgam restoration, and the mechanical retention was achieved through a slot preparation with or without retention grooves. The first null hypothesis to be tested was that there would be no difference in marginal ridge strength between the repaired marginal ridge and the unrepaired control group. The second and third null hypotheses were that there would be no difference in marginal ridge strength based on time of repair or type of slot preparation, respectively.

METHODS AND MATERIALS

Human third molar teeth were collected, stored in 0.5% chloramine-T, and used within six months of extraction. A total of 90 caries-free maxillary and mandibular third molars of similar coronal size were collected. The groups that were tested are shown in Table 1.

Table 1: *Groups Based on Time of Repair and Type of Preparation*

Group	Repair Time	Preparation Type
1	No repair (control)	None
2	15 min	Slot
3	15 min	Grooved slot
4	24 h	Slot
5	24 h	Grooved slot
6	1 wk	Slot
7	1 wk	Grooved slot
8	6 mo	Slot
9	6 mo	Grooved slot

A diamond saw (Isomet, Buhler, Lake Bluff, IL, USA) was used to section the crowns of the teeth to a level of 1 mm below the height of contour. A uniform smear layer was created on the flat dentin surfaces using 10 passes on 600-grit carbide paper. One retentive regular TMS pin (Coltene, Cuyahoga Falls, OH, USA) was placed at each line angle of the flat, sectioned dentinal tooth surface about 1 mm from the dentinoenamel junction. The pinhole was prepared using a slow-speed handpiece (Midwest, Shorty, Dentsply, Milford, DE, USA) by aligning the drill parallel to the external surface of the tooth. The teeth were mounted in the posterior sextant of a ModuPRO Endo (Acadental Inc, Mission, KS, USA) typodont next to a dentoform tooth that was part of the typodont using a heavy vinyl polysiloxane material (Regisil PB, Dentsply). The ModuPRO Endo is a typodont system with a socket area that allows the placement of an extracted tooth. The dentoform tooth served to provide a proximal contour during the condensation of amalgam as well as a point of reference for the height of the complex amalgam restoration. A No. 1 adult matrix band (Henry Schein, Melville, NY, USA) was placed and secured with a Tofflemire matrix retainer (Henry Schein). A wedge was used to separate the teeth (see Figure 1).

An admixed amalgam (Dispersalloy, Dentsply) was triturated per the manufacturer's instructions and incrementally placed and condensed with a Densco Condensaire (WaterPik Inc, Ft Collins, CO, USA). The matrix was removed and the amalgam was carved to contour with a flat occlusal surface to a height of 5 mm. The tooth specimen was removed from the ModuPRO (Acadental Inc). Each group consisted of 10 specimens. All specimens were stored in a lab incubator in 100% humidity at 37°C for 15 minutes, 24 hours, one week, or six months after condensation and carved before the proximal surface was prepared with a box and repaired with new amalgam.

The slot preparation was created at the proximal contact either with or without retention grooves. The slot was prepared using a No. 330 bur in a high-speed handpiece (Starbright, Star Dental, Lancaster, PA, USA). The occluso-gingival height of the proximal slot was 4 mm. The proximal preparation

Figure 1. *A flattened tooth with 4 pins was mounted into a typodont. A matrix band and wedge was placed and an admixed alloy was condensed and carved to create a full crown but with a flat occlusal surface.*

had a mesiodistal dimension of 2 mm and a buccolingual dimension of 3 mm at the occlusal surface and 4 mm at the gingival floor. The gingival floor was flat and perpendicular to the long axis of the tooth. The lingual and facial walls converged to the occlusal. The slot preparations were measured with a digital micrometer. For the grooved-slot preparations, the retention grooves were 0.5 mm in diameter and opposed each other to form a dovetail effect. The grooves were placed at the axio-pulpal line angles with a No. 169L bur using the high-speed handpiece. The grooves extended from the gingival floor to the occlusal surface and were prepared to be parallel buccolingually to each other (see Figure 2).

After the slot preparations were created, the tooth specimens were placed back into ModuPRO (Acadental Inc). A No. 1 adult matrix band was placed and secured with a Tofflemire matrix retainer (Henry Schein) and a wedge. A single-composition spherical amalgam (Tytin, Sybron Kerr, Orange, CA, USA) was triturated per the manufacturer's instructions, incrementally placed and condensed with hand condensation, and carved to contour in the slot preparation. The specimen was removed from the ModuPRO (Acadental Inc) and mounted in polyvinyl chloride pipe with dental stone and bis-acryl resin (Integrity, Dentsply). The tooth specimens were stored in a lab incubator in 100% humidity at 37°C. Twenty-four hours after repair, each specimen was removed from storage. A No. 6 round bur in a high-speed handpiece was used to produce a small flat area of consistent size and depth in the middle of the marginal ridge. The flat area was created to allow the stabilization of a smooth, round-ended blade. The blade was attached to the upper member of a universal testing machine (Instron 5943, Instron, Norwood, MA, USA), and the tip was lowered onto the flattened area of the marginal ridge. A compressive force was applied parallel to the specimens' long axis using a crosshead speed of 5 mm/min (see Figure 3).

Failure strengths of the restorations were recorded in newtons. A mean and standard deviation was determined per group. Data were analyzed with a 2-way analysis of variance (ANOVA) with Tukey *post hoc* tests to evaluate the marginal ridge strength of

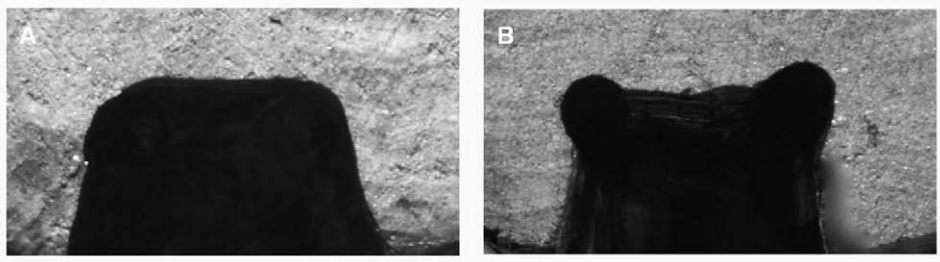

Figure 2. *After 15 minutes, 24 hours, one week, or six months after the initial crown condensation, slot preparations were made in the amalgam crowns with (A) or without (B) retention grooves. The specimens were placed back into the typodont, a matrix band and wedge were placed, and the slot preparations were restored with spherical amalgam.*

Figure 3. *The specimens were removed from the typodont and mounted in polyvinyl chloride pipe. After 24 hours of storage, the specimens were mounted in a universal testing machine. A round-ended blade was lowered onto the marginal ridge. The mean maximum force in newtons and standard deviation were determined per group (n=10).*

repaired complex amalgam restorations based on time of repair (4 levels) or type of preparation (2 levels) (α=0.05). Data ware also analyzed with the Dunnett test to compare differences between the control and the other eight groups (α=0.05). Fracture patterns were observed under 10× magnification using a stereomicroscope (SMB-1B, Nikon, Melville, NY, USA) . Four fracture patterns of the amalgam restoration were recorded among the groups: internal fracture; partial amalgam fracture of marginal ridge; complete amalgam fracture of marginal ridge; and bulk amalgam crown fracture.

RESULTS

The 2-way ANOVA found significant differences between the groups based on type of slot preparation (p=0.017) but not on time (p=0.327), with no significant interaction (p=0.152). Placing a retention groove in the proximal slot preparation resulted in significantly greater fracture strength than a slot with no retention grooves. The Dunnett test found no significant difference between any repair group and the unrepaired control (p>0.076; see Table 2). Four fracture patterns were observed among groups (see Figure 4). The control group was associated with more bulk amalgam crown fractures, and the prepared groups were associated with a mix of internal, partial, and complete amalgam fractures of the marginal ridge (see Figure 5).

Table 2: *Mean Fracture Strength in Newtons (n=10)*

Time	Mean Fracture Strength, N (Standard Deviation)	
	Slot	Grooved Slot
15 min	1323.8 (292.8)	1805.4 (481.2)
24 h	1278.3 (463.2)	1697.6 (273.8)
1 wk	1486.1 (572.5)	1433.1 (405.0)
6 mo	1646.2 (303.7)	1735.4 (520.5)
Control (unrepaired)	1324.6 (389.4)	

DISCUSSION

Minimal-intervention dentistry, such as repair of localized defects of restorations, could increase the longevity of amalgam restorations and reduce patient stress regarding treatment time and cost.[1-3] Most previous laboratory studies observed the repair of old amalgam with newly triturated amalgam. In some clinical situations, the repair of the newly condensed amalgam restoration may become necessary. However, a study by Bagheri and others[17] determined that when newly condensed amalgam was repaired after 15 minutes or later, precise mechanical retention must be prepared in the newly condensed amalgam restorations to compensate for diminished tensile strength of the repaired amalgam. If an open contact is discovered in a full cuspal-coverage amalgam restoration, should the clinician replace the restoration or should a slot-type box be prepared and repaired with newly triturated amalgam? This study demonstrated that the marginal ridge strength of a repaired proximal contact is not significantly different from the strength of an intact complex amalgam crown. Also, time of repair did not have a significant effect on marginal ridge strength. Therefore, the first and second null hypotheses were not rejected. However, the repaired amalgam with a grooved-slot preparation had significantly higher fracture strength than the repaired amalgam with only a slot preparation. Therefore, the third null hypothesis was rejected. This study supports the concept that when an amalgam repair is planned,

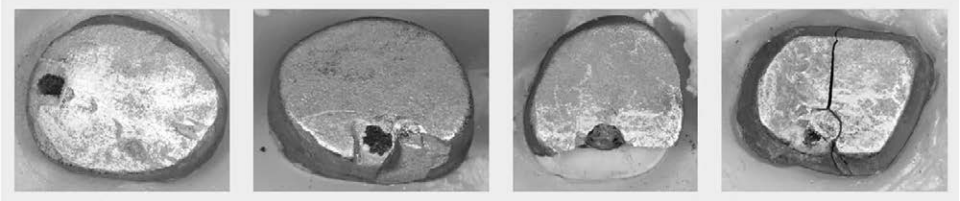

Figure 4. *Fracture patterns were recorded (left to right): internal amalgam fracture; partial amalgam fracture of marginal ridge; complete amalgam fracture of marginal ridge; amalgam crown fracture.*

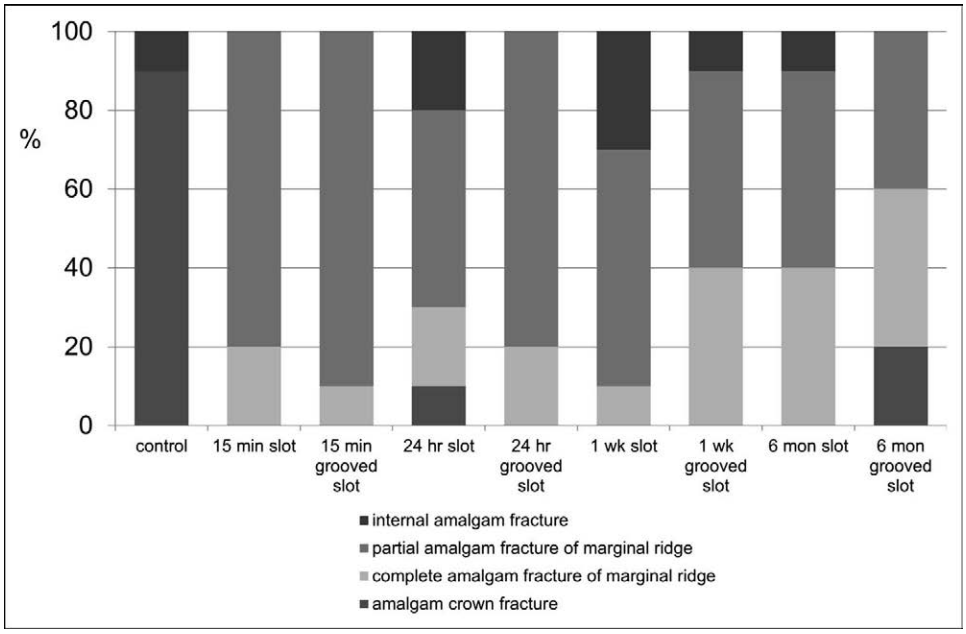

Figure 5. *Fracture pattern percentage per group.*

greater retentive features should be accomplished in the preparation to complement the union of the new and old amalgam.[8] However, heavy forces of condensation during the repair procedure may reduce the need for retentive features.[4]

The present study did not show a biological risk for the teeth: There were no catastrophic tooth fractures in any of the groups. The prepared groups were associated with a mix of internal, partial, and complete amalgam fractures of the repaired marginal ridge. However, the control group was associated with more bulk amalgam crown fractures and no fractures of the marginal ridge. Hadavi and others[8] also observed that fractures in the repaired amalgam always occurred at the junction between old and new amalgam. This finding could be related to the fact that the interface between the newer and older amalgam at the marginal ridge is the weakest point of the amalgam crown. However, the newer-to-older amalgam interface may not have been the weakest point of the amalgam crown if heavy forces were used during condensation, as found in the study by Roggenkamp and others.[4]

Although not statistically different, it was observed that the repairs completed with Tytin amalgam (spherical alloy) in the grooved-slot preparation displayed higher fracture strength than the amalgam crown control group, which was completed using Dispersalloy (Dentsply) amalgam (admixed alloy). The trend toward greater fracture strength could have been due to the higher mechanical properties of the spherical alloy compared with the admixed alloy and increased surface area with the retention grooves[18]; although, Shen and others[6] attributed the higher repair strength of a spherical alloy to the fact that the spherical amalgam is more plastic than admixed amalgam immediately after trituration. The extra plasticity may result in better wetting of the repair surface and higher repair-strength values.[6]

The carving of a complex amalgam restoration may occasionally result in light proximal contact with the adjacent tooth. Complete replacement of restorations has the disadvantages of being time-consuming with unnecessary removal of healthy tooth structure, enlargement of the preparation, the risk of converting the restoration to an indirect restoration, the possibility of major injuries to the pulp tissues, and more expense for the patient. This is the first study to evaluate the fracture strength of the marginal ridge of a repaired complex amalgam restoration. This investigation found no significant difference in fracture strength between repaired and unrepaired amalgam and suggests that the repair of a light proximal contact in an otherwise functional, intact, existing complex amalgam restoration may be an acceptable alternative to replacement.

CONCLUSIONS

The proximal repair strength of a complex amalgam restoration was not significantly different from that of an unrepaired amalgam crown. Placing a retention groove in the proximal slot preparation resulted in significantly greater fracture strength than a slot with no retention grooves. Time of repair had no significant effect on the strength of the repair.

Acknowledgements

The authors would like to acknowledge Mr Dan Sellers for his assistance with the universal testing machine. Funding for the study was provided by the 59th Clinical Research Training Division, Joint Base San Antonio-Lackland, TX.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Wilford Hall Ambulatory Surgical Center Institutional Review Board. The approval code for this study is FWH20140034N.

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. The authors do not have any financial interest in the companies whose materials are discussed in this article.

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In Vitro Evaluation of Marginal Adaptation of Direct Class II Composite Restorations Made of Different "Low-Shrinkage" Systems

C Shahidi • I Krejci • D Dietschi

Clinical Relevance

The use of bulk-filling restorative materials should lead to a restoration marginal quality comparable to conventional and so-called low-shrinkage restorative materials in enamel and acceptable marginal quality in dentin over a short to medium period of clinical use.

SUMMARY

The present study evaluated the influence of various low-shrinkage restorative systems in class II direct composite restorations following simulated occlusal loading. Forty MOD class II cavities were prepared on freshly extracted human lower third molars with proximal margins located mesially 1.0 mm coronal to and distally 1 mm apical to the cementoenamel junction. The samples were randomly distributed into five experimental groups corresponding to the following restorative systems: a conventional resin composite (Tetric) as active control group, a low-shrinkage composite (Extra Low Shrinkage [ELS]) alone or combined with its corresponding flowable version (ELSflow) used as a 1- to 1.5-mm liner, a bulk-filling flowable composite (Surefil SDR) covered by a 1-mm layer of restorative composite (Ceram-X), and a restorative bulk-filling composite (SonicFill). All specimens were submitted to 1,000,000 cycles with a 100N eccentric load into saline. Tooth restoration margins were analyzed semiquantitatively by scanning electron microscopy before and after loading. The percentage of perfect adaptation to enamel varied from 94.15% (SonicFill) to 100% (ELS) before loading and from 69.22% (SonicFill) to 93.61% (ELS and ELSflow) after loading. Continuous adaptation to cervical dentin varied from 22.9% (Tetric) to 79.48% (SDR/Ceram-X) before loading and from 18.66% (Tetric) to 56.84% (SDR/Ceram-X) after loading. SDR/CeramX and SonicFill showed the best cervical dentin adaptation.

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INTRODUCTION

The detrimental impact of resin composite polymerization shrinkage on restoration interface quality and stability has been recognized since the early use of this material and has led to the recommendation of various compensating procedures such as incremental methods,[1-5] the use of ceramic inserts,[6] the application of base/liners,[2,3,7-9] or indirect techniques.[10] The aforementioned, traditional restorative solutions shared the objective of limiting the development of marginal or internal defects, which can affect restoration behavior and longevity.

The use of incremental techniques was validated by several long-term clinical reports, demonstrating also the satisfactory clinical behavior of hybrid and microhybrid composite technology[11-13]; however, these methods, which are considered the "standard of care" for managing polymerization stresses in medium to large class II cavities, all have been at times considered cumbersome and stimulated the dental industry and clinicians to look for alternative and simplified operative protocols. Various technological solutions were then investigated, including improved filler technology (blend of fillers and/or increase of filler load); improved, novel matrix structure with reduced shrinkage (ie, silorane)[14,15]; use of stress-decreasing compounds within the resin matrix[16,17]; changes in light-initiation technology to increase curing depth; and use of sonic vibrations and energy to favor flow and adaptation of highly filled resin composite (SonicFill, patent US7014462 B1). Today, however, little information exists about the real clinical benefit of these new technologies used alone as a simplified filling method or in combination with some of the aforesaid stress control concepts.

The true challenge for simplified restorative systems based on a bulk-filling approach or the application of a thick flowable composite base underneath a single layer of restorative material is to demonstrate both satisfactory initial quality and medium- to long-term behavior (marginal, surface, and restoration bulk integrity) when compared with conventional composite technology and clinical protocols. So far, the available short-term clinical trials do not demonstrate fully convincing performance of either silorane technology or bulk-fill techniques on permanent teeth.[18-20] The short- and medium-term performance of bulk-filling technique proved satisfactory in only two studies dealing with the treatment of primary teeth.[21,22] The quantity and consistency of clinical evidence are clearly insufficient to unconditionally recommend these new systems.

To achieve optimal long-term performance, the requirements will be first to manage polymerization stress buildup following restoration placement and then to demonstrate suitable behavior of restored teeth to repeated oral strains though appropriate mechanical properties. Actually, the reaction to functional loading and hydrolytic degradation will define the material resistance to fatigue and subsequent interface and restoration breakdown.[23] The fatigue behavior of restorations and the occurrence of adhesive or cohesive failures depend on some important material properties such as flexural strength, fracture toughness (K_{Ic}), and elasticity modulus (E).[24-26] It was shown, for instance, that the latest property has a crucial impact on stress development within the tooth restoration system.[2,3,7,27,28] The use of an elastic base or liner with low E modulus, acting as a stress-breaker element within the restoration, has been extensively evaluated since the first works by Davidson and coworkers[27-29] and largely validated *in vitro*.[30,31] When using new simplified restorative systems featuring distinctive physicochemical characteristics, the potential impact of the above mentioned parameters on restoration quality and behavior is unknown and justifies additional investigations. In consideration of a rather well-established consensus suggesting medium- to long-term observation periods (about three to five years) to discriminately appraise the clinical performance of various operative protocols and material choices,[32-35] the use of *in vivo*, preclinical trials such as fatigue testing[36-39] appears particularly suitable today for evaluating new, simplified restorative protocols.

The aim of this *in vitro* study was to test the hypothesis that low-shrinkage restorative systems have the potential to improve restoration adaptation after simulated *in vitro* occlusal stressing, as compared with a traditional microhybrid restorative composite applied with a classical, well-documented incremental technique. The quality of the different interfaces was also evaluated to identify the restoration's most vulnerable areas.

METHODS AND MATERIALS

Specimen Preparation

Forty freshly extracted human third molars were used for this study. Samples were collected anonymously, and their use complied with all local human subject oversight according to the Swiss Human Research Act, under article 2.

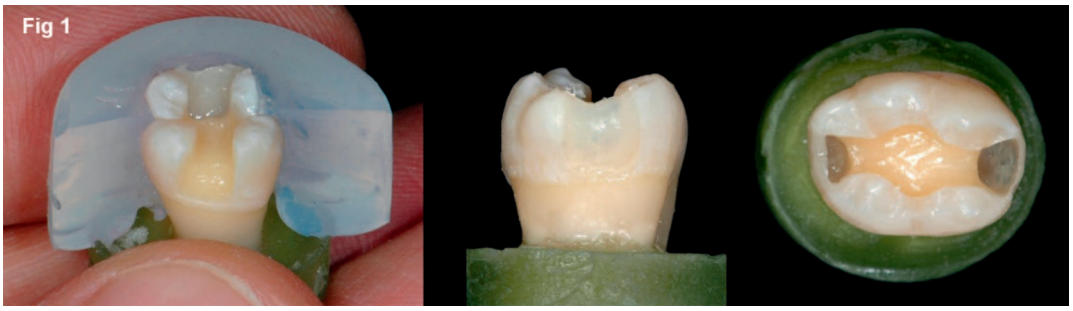

Figure 1. *Transparent silicone matrix used to improve restoration adaption, shorten finishing steps, and avoid margin overhangs and tedious, damaging finishing.*

The inclusion criteria were an absence of carious lesions, a complete root formation, and no visible tooth defect resulting from the extraction. The teeth were stored in a sodium azide solution (0.2%) at 4°C until the experiment onset. For each specimen, the root length was adjusted to fit in the test chamber of the mechanical loading device (Department of Cariology, Endodontics & Pedodontics, Laboratory of Electronics of the Medicine Faculty, University of Geneva). After the specimen was properly positioned, it was fixed with light-curing composite on a metallic holder (Baltec, Balzer, Liechtenstein); then, the root base was embedded with self-curing acrylic resin to complete the tooth stabilization, leaving the last 3 mm toward the cementoenamel junction free to provide proper access to restorative procedures.

A new, original technique was used to box proximal cavities, with the aim to facilitate material placement, improve restoration adaptation, and shorten finishing steps. Then, an impression of the tooth was made prior to cavity preparation with a putty silicone (Memosil 2, Heraeus Kulzer, Hanau, Germany) placed in a 2 cm diameter cylindrical plastic tube used as tray. After setting, the tube was removed and all the silicone material overlaying the tooth occlusal surface trimmed off before slicing this impression in two parts buccolingually (Figure 1). To simulate the use of a metallic matrix such as commonly employed, aluminum foil was adapted with a burnisher to each proximal surface of the silicone impression just before tooth preparation and application of the filling material, as further described.

Class II cavities (MOD) were then prepared, with the proximal margins located mesially 1.0 mm coronal to and distally 1 mm apical to the cementoenamel junction (Figure 2). The dimensions of the slightly divergent preparations were 4.0 mm in width and 2.0 mm in depth at the bottom of the proximal box and 3.0 mm in width and depth for the occlusal isthmus, all walls being only slightly divergent. The cavities were prepared using preparation diamond conical burs with round tips under profuse water spray (80 μm grain size, ISO 856/018, Strauss & CO, Ra'Anana, Israel). All enamel margins were beveled with a fine diamond flame bur (40 μm grain size, ISO 862/010, Strauss & CO) under air spray. The 40 prepared teeth were randomly assigned to one of the five study groups (one active control group and four experimental groups), corresponding to the combination of restorative materials and procedures listed in Table 1 and 2.

Restorative Procedures

After completion of the preparation, a 30 second selective enamel etching with 35% H_3PO_4 gel was performed (Ultraetch, Ultradent, South Jordan, UT, USA) prior to the treatment of all cavity surfaces with a two-step self-etch system (SEBond, Kuraray, Tokyo, Japan). Four restorative composites claiming to exhibit low shrinkage or to be used for a simplified filling approach (SureFil SDR flow and CeramX Mono+, DeTrey-Dentpsly, Constance, Germany; ELSflow and ELS, Saremco, St-Gall, Switzerland; and SonicFill, Kerr, Orange, CA, USA) were tested and compared with a well-established hybrid composite brand (Tetric, Vivadent, Liechtenstein), used here as the active control group (Table 1). The following restorative protocols were applied.

SDR Group—The cavities were filled with SureFil SDR flow until 1 to 1.5 mm (measured with a periodontal probe) below the occlusal surface, creating a base of about 3 to 4 mm thickness proximally. The remaining volume/surface was filled with the restorative material CeramX Mono+ in one increment.

ELS1 Group—A 1 to 1.5 mm thick lining of ELSflow (as measured with a periodontal probe) was applied over the entire preparation, including

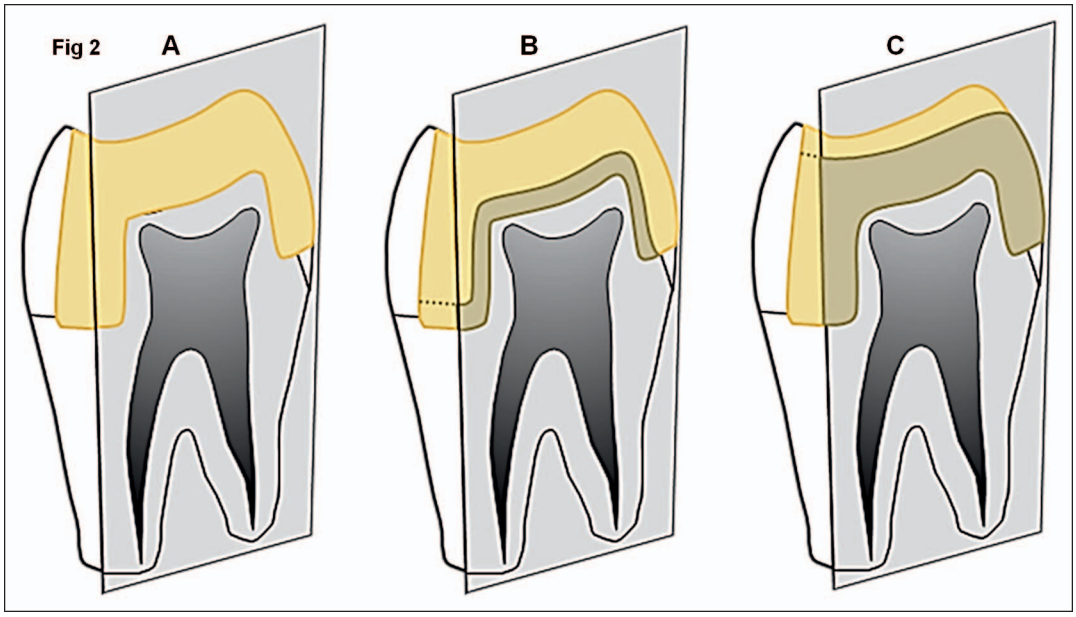

Figure 2. *Experimental restorative configurations: (A): Composite restorations made of a single material (layered or bulk; groups TET, ELS2, and SOF). (B): One-millimeter flowable composite lining underneath a restorative composite (ELS 1). (C): A ≥4-mm lining flowable material underneath a thinner layer of restorative material (SDR).*

both enamel and dentin cervical margins; the remaining cavity volume was filled with the ELS restorative composite using in proximal areas a technique similar to the three-sited light-curing technique (one layer cervically, followed by two more vertical layers)[2,3] and horizontal layers of 1.5 mm in the occlusal area.

Sonicfill Group—The entire cavities were filled in one step using the SonicFill handpiece (Kavo, Biberach, Germany) to facilitate flow of the material into the cavity.

ELS2 and Tetric—The three-sited curing technique was applied in the proximal preparations while the remaining occlusal volume was also filled in three steps, including two first oblique increments, followed by one last horizontal increment.

Both linings (Surefil SDR flow and ELSflow) were light cured for 20 seconds with continuous irradiation mode (Bluephase, Vivadent, in HIP mode: power output=1200 mW/cm^2). For all other protocols, each increment or composite bulk was light cured for 20 seconds as well, using the aforementioned irradiation conditions. All proximal cavities were restored using the silicone mold, as previously described (Figure 1). In all groups and for all aforementioned techniques, the final increment was sculpted with a hand instrument (DD1/DD2, Composculp Set, Hu-Friedy, Chicago, IL, USA) prior to final light curing, in order to ease finishing and also reduce mechanical stress on the margins.

The finishing and polishing followed immediately the restorative procedures and were performed under 10× magnification (Leica MZ6 microscope, Nidau, Switzerland). The finishing/polishing of occlusal surfaces was performed with 40 μm flame and pear-shape diamonds (ISO 368/013; Strauss & CO) followed by silicone points used under abundant water spray (Brownie; Shofu, Kyoto, Japan); for the proximal surfaces, discs of decreasing roughness

Table 1: *Combination of Products (Adhesive, Liners, and Restorative Materials) Among the Control and Four Experimental Groups*

Groups	Adhesive	Lining	Restorative
TET (ctr)	SEBond (Kuraray)	No	Tetric (Vivadent)
SDR	SEBond (Kuraray)	SDR flow (Dentsply)	CeramX mono+ (Detrey-Dentsply)
ELS1	SEBond (Kuraray)	ELSflow (Saremco)	ELS (Saremco)
ELS2	SEBond (Kuraray)	No	ELS (Saremco)
SOF	SEBond (Kuraray)	No	SonicFill (Kerr)

Table 2: *Composition and Relevant Physical Characteristics of Composites Under Evaluation (Manufacturer's Data)*

Product	Filler Content, %W	Matrix Composition	E Modulus, GPa	Shrinkage, %V	Batch No.
Tetric (Ivoclar)	81	BisGMA, TEGMA, UDMA	11.5	2.5 (24 h)	M31685
SDR (Dentsply)	68	Mod UDMA, TEGMA, EBPADMA	5.5	3.5 (24 h)	1005004013
Ceram-X mono (Dentsply)	76	Methacrylate modified polysiloxane DMA	8.5	2.3 (30 min)	1005004013
ELSflow (Saremco)	NA	BisGMA, EBPADMA	8.0	3.2 (30 min)	09.2013-74
ELS (Saremco)	75	BisGMA, BisEMA	9.0	1.3 (1 min)/2.4 (4 h)	10.2013-14
SonicFill (kerr)	83.5	BisGMA, UDMA	11.5	1.7 (12 h)	3691651

Abbreviations: %V, percentage of volumetric shrinkage; %W, weight percentage.

(rough, medium, fine, and extra fine; Sof-Lex Pop On XT, 3M, St Paul, MN, USA) were used at low speed ($\leq$1500 rpm) and with reduced pressure to limit frictional stress on the restoration margins.

Mechanical Loading

The stress test was carried out 24 hours after restoration placement and finishing, the restored teeth being kept in saline, at room temperature, during this interval as well as during the test phase inside the fatigue device chambers (University of Geneva). The teeth's pulpal cavity was penetrated buccally or palatally with a tube (sealed with Dentin Bonding Agent), which was connected to a simulated pulpal circulation of saline under a pressure of 14 cm H_2O[40]; the simulated pulpal pressure was then applied only after the restorative procedures. All specimens were submitted to 1,000,000 cycles with a 100 N eccentric occlusal load. The axial force was exerted at a 1.5 Hz frequency following a one-half sine wave curve. These conditions are taken to simulate about 4Y of clinical service,[37,38] Restored teeth were contacted by antagonist artificial cusps, made of stainless steel with a hardness similar to natural enamel (Vickers hardness: enamel=320-325; selected steel=315); the diameter of the cusps was 4 mm and contacted the restoration's occlusal surface on the restoration's proximal fossa. By having the specimen holder mounted on a hard rubber disc, a sliding movement of the tooth was allowed between the first contact on an inclined plane and the central fossa. The function of this device was similar to that of the machine developed by Krejci and coworkers.[37,38,]

Specimen Evaluation

Before the fatigue test, as well as after completion of the loading phase, the restoration's margins were cleaned with fine pumice before etching using diluted H_3PO_4 gel to remove finishing/polishing smear and improve the readability of the replicas under scanning electron microscopy (SEM; the solution was prepared with 35% H_3PO_4 gel mixed with three times its volume of distilled water). The restoration surfaces and margins were then treated by gentle brushing of this solution for four seconds, followed by thorough water rinsing. Impression of the samples was made with polyvinylsiloxane (President Light Body, Coltene/Whaledent AG, Alstätten, Switzerland), which served for the fabrication of gold-sputtered epoxy resin replicas (Epofix, Struers, Rødrove, Denmark).

The proximal mesial and distal aspects of the restorations were examined while the occlusal adaptation was not taken into consideration. The approximal mixed adaptation (Appr. M) refers to the entire mesial restoration adaptation (including both enamel and dentin margins). The cervical dentin adaptation (Cerv. D) refers to the specific cervical dentin adaptation on the mesial preparation side. The approximal enamel adaptation (Appr. E) refers to the distal, full enamel restoration margin adaptation. Each tooth restoration interface was analyzed quantitatively by SEM (Digital SEM XL20, Philips, Eindhoven, the Netherlands) using a standardized evaluation method.[41,42] The restoration margins were observed at a standard 200× magnification, and each margin segment, or subsegment, was delimitated by a digital marker and attributed a quality criteria, as described further (Figure 3). A dedicated software then computed the respective percentages of continuous or defective margin for the three different areas under evaluation applying those quality criteria: continuity or overfilling, underfilling, marginal opening, marginal restoration, and tooth fracture (Figure 1). When necessary for the assessment accuracy, higher magnifications were used (up to 1000×). The results for the restoration marginal adaptation before and following the loading test were expressed as percentages of continuity; the other parameters that describe

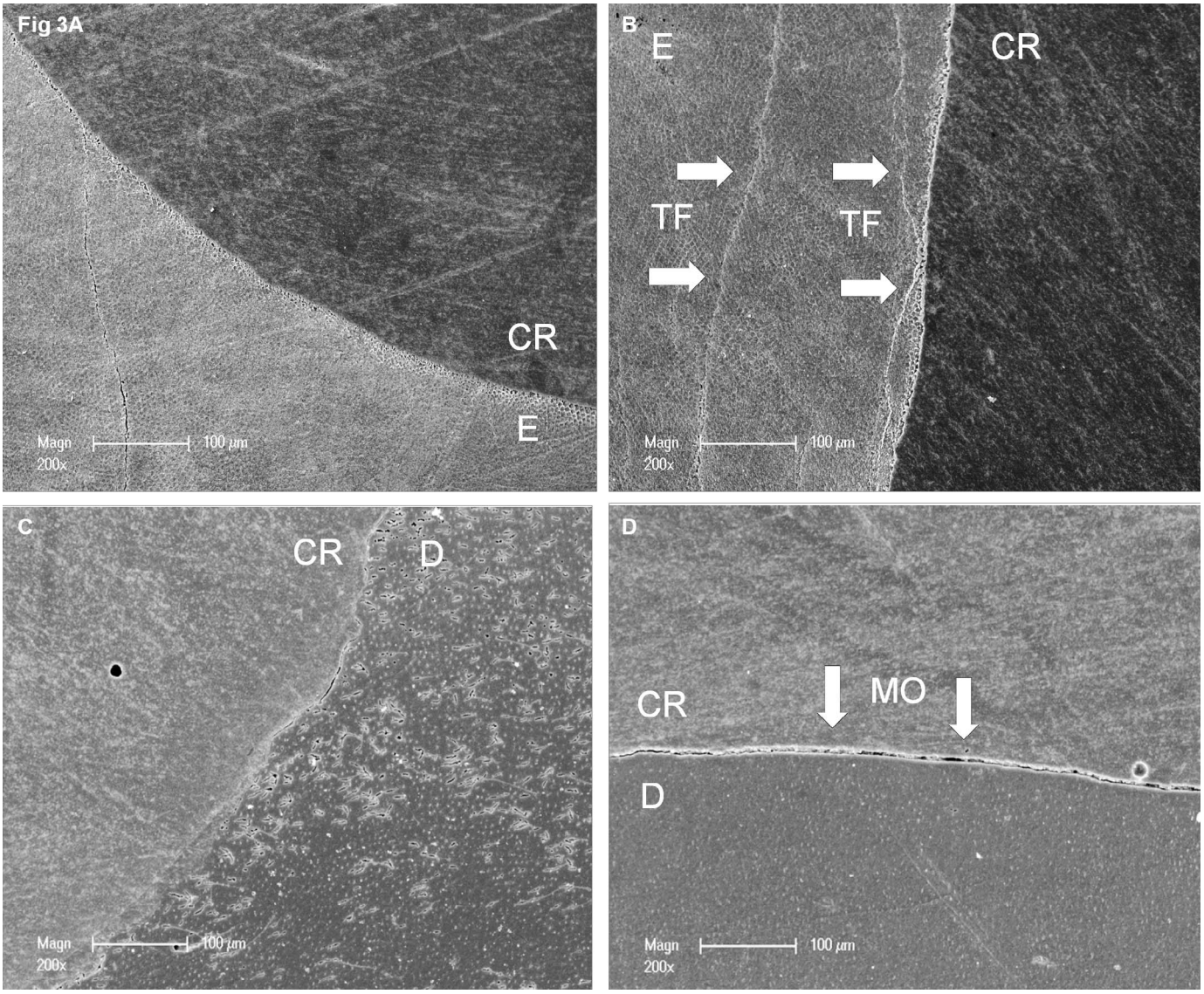

Figure 3. *Typical marginal appearance and quality under SEM, following cycling loading (one million cycles at 100N and 1.5-Hz loading frequency). NB, restoration margins were cleaned with a short etching with diluted phosphoric acid to improve replica quality and readability; CR, composite restoration; E, enamel; D, dentin. (A): Enamel margin in continuity. (B): Enamel marginal with tooth fracture. (C): Dentin margin in continuity. (D): Dentin margin with marginal opening.*

defective margin sections were pooled together and accounted for noncontinuous adaptation and were not analyzed individually. Percentages were calculated as the ratio between the cumulated distance of all segments with a continuous margin and the whole interface length. The restoration occlusal adaptation was not assessed.

All results of the SEM analysis were submitted to a parametric statistical analysis. An analysis of variance and Bonferroni post hoc test served for comparing the intergroup marginal continuity percentages for the different restoration areas, before and after the loading test (Instat, GraphPad Software, La Jolla, CA, USA). A paired *t* test served to compare the intragroup marginal adaptation percentages between pre- and postloading conditions (Instat, GraphPad Software). All tests were carried out at a 5% level of significance.

RESULTS

The study results, expressed as percentages of continuous marginal adaptation before and after loading, are presented in Table 3 together with their statistical analysis.

Table 3: *Percentages of Continuous Marginal Adaptation (±SD) for the Four Segments Under Evaluation, Pre- and Postcycling Loading*[a]

Groups	TET	ELS1	ELS2	SDR	SOF	ANOVA
Approximal enamel preloading	96.9% *(3.6)*	98.1% *(2.2)*	100% (0.00)	97.5% *(5.0)*	94.1 *(8.2)*	*f*=1.642
						p=0.1856 NS
Approximal enamel postloading	92.8%a *(8.1)*	91.2%a *(4.9)*	93.6%a *(8.0)*	92.7%a *(13.1)*	69.2b *(27.1)*	*f*=4.154
						p=0.0074 **S****
t-test	*t*=2.064 NS	*t*=3.634 **S****	*t*=2.261 NS	*t*=1.384 NS	*t*=2.792 **S***	
Approximal mixed preloading	82.7%a *(9.7)*	97.2%b *(1.4)*	97.0%b *(3.1)*	92.4%a,b *(9.5)*	90.4%a,b *(8.2)*	*f*=5.347
						p=0.0018 **S****
Approximal mixed postloading	77.0% *(9.6)*	89.2% *(7.5)*	83.0% *(12.0)*	87.0% *(11.6)*	79.0% *(10.2)*	*f*=1.996
						p=0.1166 NS
t-test	*t*=2.013 NS	*t*=3.400 **S***	*t*=3.229 **S***	*t*=3.185 **S***	*t*=2.757 **S***	
Cervical dentin preloading	22.1% *(12.0)*	60.4%a *(20.5)*	64.2%a *(23.0)*	79.5%a *(17.7)*	78.3%a *(15.0)*	*f*=13.255
						p<0.0001 **S****
Cervical dentin postloading	18.7%a *(11.4)*	29.6%a,b *(18.2)*	29.7%a,b *(23.6)*	56.8%b *(24.0)*	51.1%b *(15.3)*	*f*=5.682
						p=0.0012 **S****
t Test	*t*=2.335 NS	*t*=5.132 **S****	*t*=5.844 **S****	*t*=4.190 **S****	*t*=3.441 **S***	

Abbreviation: ANOVA, analysis of variance.
[a] *Groups with same letter are not statistically different. *p<0.01; **p<0.001.*

Enamel Adaptation (Distal Side)

The preloading proximal enamel adaptation presented proportions of continuity varying from 94.15% (SonicFill) to 100% (ELS), with no significant difference. After loading, those proportions decreased to values varying from 69.22 (SonicFill) to 93.61 (ELS + ELSflow). The change was significant for SonicFill and ELS only while the postloading distal adaptation of SoniFill was significantly lower than the other four groups.

Mixed Margin Adaptation (Mesial Side)

The preloading mixed proximal adaptation presented proportions of continuity varying from 82.72 (Tetric) to 97.24 (ELS + ELSflow), with Tetric adaptation being significantly inferior to ELS/ELSflow or ELS groups. After loading, those proportions decreased to values varying from 77.0% (Tetric) to 89.19 (ELS + ELSflow); the reduction in percentages of continuity was significant in all groups except for Tetric. The postloading adaptation values in mixed proximal margins did not show any significant difference among groups.

Dentin Margin Adaptation (Mesial Side)

The preloading cervical dentin adaptation presented proportions of continuity varying from 22.09% (Tetric) to 79.48% (SDRflow + CeramX), with the Tetric value being significantly inferior to the other groups. After loading, the continuity values dropped to percentages varying from 18.66% (Tetric) to 56.84% (SDRflow + CeramX); the reduction in percentages of continuity was significant for all products except for Tetric. The postloading adaptation of Tetric in cervical dentin was significantly inferior to SDRflow/ceramX and SonicFill.

DISCUSSION

Phenomena such as nanoleakage, leakage, pulpal complications, and secondary caries, which are induced by interface breakdown, account for a significant part of clinical failures observed in all types of direct posterior restorations.[11-13] Then, evaluating the behavior of adhesive restorations with natural tissues under simulated function, pulpal pressure, and moist environment helps in approaching the reaction of a restoration to the most important oral cavity strains and to monitor the tooth-composite interface stability and degradation as well.[36-39] This *in vitro*, preclinical research approach is well established and has been used previously in numerous studies evaluating the *in vitro* quality of class II restorations.[30,31,39]

Overall, the materials and restorative techniques under evaluation presented satisfactory adaptation to approximal enamel (distal) and to mixed enamel-dentin margins (mesial) before and after loading (>90% continuous margins), with the exception of SonicFill and Tetric. Actually, the restorations made with SonicFill showed more postloading marginal microfractures in plain enamel margins whereas the

restorations made with Tetric showed a high proportion of dentin marginal gaps, pre- and postloading. The higher occurrence of marginal enamel microfractures reported for SonicFill might be related to the higher stiffness of this product (highest E modulus among tested products; see Table 1), which likely increases stress transmission to the restoration margins, especially when used with a bulk-filling approach. The possible clinical impact of such adverse finding, commonly observed in previous *in vitro* fatigue studies, has not been clarified.[9,43]

The most critical margin area was as usual the cervical dentin, where the multilayered restorations made of a traditional microhybrid (Tetric) and a so-called "low-shrinkage" microhybrid (ELS) used alone or in combination with a flowable resin composite liner (ELS + ELSflow) did not perform satisfactorily, as shown by significantly lower continuity proportions in pre- (Tetric only) and postloading conditions. In studies evaluating marginal adaptation in similar *in vitro* conditions, Tetric[44] and Tetric Ceram,[45] which are both microhybrids, and Tetric EvoCeram (nanohybrid)[44] placed with other adhesives presented percentages of postloading continuity varying from 83.3% to 94.6% in enamel and from 56.2% to 74.6% in dentin, which is markedly higher than in the present study for dentin adaptation (18.66%). Kwon and others[46] using the same adhesive with Tetric Ceram obtained 78.7% of continuity in mixed dentin and enamel margins, which is nearly identical to the result of the present study, with 77.0% continuous margins for the multilayered Tetric class II restorations. When considering the adaptation to dentin, the performance of Tetric then fell below the range of published data for multilayered direct composite restorations tested in similar laboratory environments. As the present study protocol involved a higher cycling load (100N instead of 49-50N or varying forces from 50 to 100N), or a higher number of cycles (one million instead of 100,000 to 600,000), it could have had a more damaging and discriminative effect on dentin adaptation, although some unidentified confounding factor could also account for such surprisingly low performance.

In two recent studies, SDR flow combined with Ceram-X in class II restorations submitted to a similar fatigue test (but with a reduced number of cycles) presented postloading percentages of continuity in dentin of 50.3% and 64.9%, depending on the adhesive tested,[43] and 42.9%,[47] with those values being rather close to those reported in the present study. SonicFill presented percentages of continuity in dentin, postloading, of 51.1% in the present study and 61.7% in the other study.[47] While these percentages can be considered apparently satisfactory, such a proportion of continuous margin remains largely inferior to the best combination of adhesive, flowable resin liner, and restorative composite, peaking at 90.8% in a similar test environment for indirect composite restorations[9] or to classical multilayered full-composite restorations with continuous adaptation in dentin reaching 74.6% to 78%.[48]

The E modulus and volumetric shrinkage are of particular interest[49,50] as both properties are known to significantly affect polymerization stresses. Actually, for a given volumetric shrinkage, a lower elasticity modulus will help to reduce stresses. On the other hand, the material's stiffness (as defined by its E modulus) also influences restoration adaptation following loading because of distinct deformation and stress absorption capacity.[9,44,48] In this study, the SDR group (SDR flow E modulus=5.5 GPa) presented a better dentin and overall adaptation than the group with the ELSflow liner (E modulus=8 Gpa); considering a volumetric shrinkage within the same range, the material's stiffness is potentially the influential property. Considering again the interaction between shrinkage stress and E modulus, the similar results obtained with the ELS restorative material alone or combined with the ELSflow become more logical and suggest that using a lining with an almost similar E modulus (ELSflow and ELS E modulus are, respectively, 8 and 9 GPa) is unlikely to give any biomechanical advantage to the restoration's adaptation, although it facilitates and quickens clinical procedures.

Another influential parameter is the respective cavity and increment/layer C-factor (configuration factor),[36] which justified many evidenced-based restorative protocols.[30,31] The rationale for placing a flowable liner underneath direct class II composite restorations is then based on a low C-factor of this first layer, promoting reduced stress buildup at the cavity-restoration interface, combined also with the superior stress absorption capability of a flowable resin composite.[46,51-54] In consideration of the C-factor, simplified layering techniques (bulk-filling or extended flow base) do, however, not show any particular advantage.

In an attempt to summarize the main parameters that explain the present study's findings, the better performance of the SonicFill and SDR/Ceram-X groups in cervical dentin is potentially linked to, respectively, the low volumetric shrinkage of SonicFill and the low E

modulus of SDR flow base/Ceram-X systems. As well, the rather unsatisfactory results of the TET and ELS and combined ELS/ELSflow groups are to be attributed to the higher volumetric shrinkage and E modulus of Tetric and the intermediate E modulus and medium/high volumetric shrinkage of ELS/ELSflow materials, which overcame the theoretical advantage of using a multilayering approach.

The present study tested materials and filling methods in medium-size cavities, which could have been more favorable to a low E modulus material (ie, SDR flow) because of the less critical reinforcement role played by the restoration. In larger cavities or in biomechanically compromised teeth, such nonvital teeth, long-term functional loading, and higher occlusal forces (ie, bruxism) could have a more detrimental effect on restoration adaptation because of increased tooth structure deformation. More research is then needed to confirm the relationship between cavity size, material stiffness, and long-term restoration biomechanical behavior.

CONCLUSIONS

An *in vitro* evaluation of marginal adaptation following medium-term functional loading simulation (one million cycles) was used for evaluating new filling methods and materials proposed for class II restorations.

In this preclinical test, medium-size class II restorations made with a traditional layering approach and flowable composite resin liner or simplified filling methods presented satisfactory adaptation to proximal enamel, whereas in cervical dentin, the bulk-filling technique (SonicFill) and extended flow base (SDR flow + Ceram-X) showed the best adaptation. Reported values of continuity were, however, inferior to many direct or indirect composite restorations using traditional microhybrid composites, as reported in previous, similar *in vitro* trials. As some of the new, simplified restorative systems exhibit singular composition and physical characteristics, extended fatigue tests and clinical trials will be needed to assess their performance, in particular in larger cavities and with higher functional stresses. The use of new materials for bulk filling or a simplified restorative protocol cannot yet be unconditionally recommended, based on their *in vitro* performance.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Swiss Human Research Act, under Article 2.

Conflict of Interest

The authors declare that they have no financial, professional, or other personal interests that could influence the results or position presented in this article.

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Effect of Different Protocols in Preconditioning With EDTA in Sclerotic Dentin and Enamel Before Universal Adhesives Applied in Self-etch Mode

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Clinical Relevance

A shorter ethylene diamine tetra-acetic acid (EDTA) application time of 30 seconds with a sonic device produced a higher bond strength when compared with the conventional two-minute EDTA application in sclerotic dentin.

SUMMARY

Objectives: The aim of this study was to investigate the effect of different protocols of 17% ethylene diamine tetra-acetic acid (EDTA) conditioning on the etching pattern and immediate bond strength of universal adhesives to enamel and sclerotic dentin.

Methods and Materials: Forty bovine teeth with sclerotic dentin and 20 human third molars were randomly divided into eight groups resulting from the combination of the main factors surface treatment (none, two-minute EDTA conditioning manual application, 30-second EDTA manual application, 30-second EDTA sonic application) and adhesives systems (Scotchbond Universal Adhesive [SBU] and Prime & Bond Elect [PBE]). Resin-dentin and enamel-dentin bond specimens were prepared and tested under the micro-

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tensile bond strength (μTBS) and microshear bond strength (μSBS) tests, respectively. The etching pattern produced on the unground enamel and the sclerotic dentin surfaces under the different protocols and adhesive systems was evaluated under scanning electron microscopy.

Results: For enamel, only the main factor adhesive was significant ($p<0.0001$), with SBU showing the highest μSBS. In sclerotic dentin, the lowest mean was observed for the group without EDTA application and the highest mean in the group with EDTA application with the sonic device for 30 seconds. Regardless of the EDTA protocol, the highest means of μTBS were observed for SBU ($p<0.05$).

Conclusions: EDTA conditioning improves the bonding performance of universal adhesives in the self-etch mode on sclerotic dentin, mainly when applied for 30 seconds with the aid of a sonic device. EDTA pretreatment also improves the retentive etching pattern of enamel, but it does not result in higher enamel bond strength.

INTRODUCTION

The primary aim of dentin bonding systems is to provide retention of restorative materials to the dental structure as well as to seal the dentin substrate. Fortunately, the immediate bonding effectiveness of most current adhesive systems is quite favorable,[1] but their effectiveness is often evaluated based on their ability to bond to sound dentin.

While sound dentin is frequently encountered in dental practice, a variety of other pathologically altered dentin substrates to include caries-affected and sclerotic dentin are also encountered.[2-5] Irrespective of the bonding strategy used, bonding to sclerotic dentin presents a challenge, which often results in diminished bond strengths.[2-6] This is due to partial or total obliteration of dentinal tubules with mineral crystals and to the presence of an acid-resistant hypermineralized layer.[5,7]

In fact, previous studies suggested that bonding to human sclerotic dentin could be improved by changing the adhesive protocol that is normally employed for sound dentin. Several methods have been suggested to include phosphoric acid pretreatment when using self-etch systems,[8,9] roughening of the sclerotic dentin surface with diamond burs,[6,8,10,11] increased application times of adhesive systems,[9] and preconditioning dentin with weak acids such as ethylene diamine tetra-acetic acid (EDTA).[11-13]

Among these strategies, preconditioning with EDTA seems a very promising approach. EDTA acts as a chelator and produces a shallow demineralization of the dentin,[14,15] which is likely responsible for the high immediate bond strength of self-etch adhesives to sound and sclerotic dentin.[8,14] In addition, EDTA conditioning also has an inhibitory effect on the matrix-bound endogenous metalloproteinases (MMPs) of demineralized dentin,[16,17] producing more stable adhesive interfaces in sound[14] and sclerotic[13] dentin. Furthermore, previous EDTA application can increase the bond strength of self-etch adhesives to enamel,[18,19] which solves one of the main drawbacks of self-etch adhesives.[20,21]

While the use of EDTA pretreatment has shown promise, it does involve an extra step and up to two minutes extra procedural time; one of the touted benefits of the current simplified adhesive systems is time saving.[13,16,22] Shorter EDTA application times and/or "active" EDTA application may allow the benefits of EDTA use with reduced time requirements. Previous studies have reported that agitation of EDTA in root canal systems provides more effective removal of smear layer and debris.[23-25]

Sonic and ultrasonic application of adhesives may improve the longevity of bonds to tooth structure without greatly increasing time requirements for bonding. This technology has existed since 1950 and is used in periodontics and endodontics.[26] The technique can be used in the application of dental adhesive systems with potential benefit of improved bond strengths.[27] The vibration at high speed favors the fluid dynamics in the substrate as well as eliminates bubbles and solvent evaporation.[28]

To bring the benefits of prior application of EDTA to the most difficult-to-treat substrates—such as sclerotic dentin and enamel—the use of a prototype sonic device (SMART, FGM Dental Products, Joinville, SC, Brazil) may be beneficial in the application of EDTA to sclerotic dentin and enamel prior to the use of self-etch adhesives (compared with manual application).

The aim of this study was to investigate the effect of different preconditioning protocols with EDTA on the bond strength and etching pattern prior to application of universal adhesive systems to enamel and sclerotic dentin. This study tested two null hypotheses: 1) different preconditioning protocols with EDTA do not affect the bonding effectiveness of adhesives to sclerotic dentin and b) different precon-

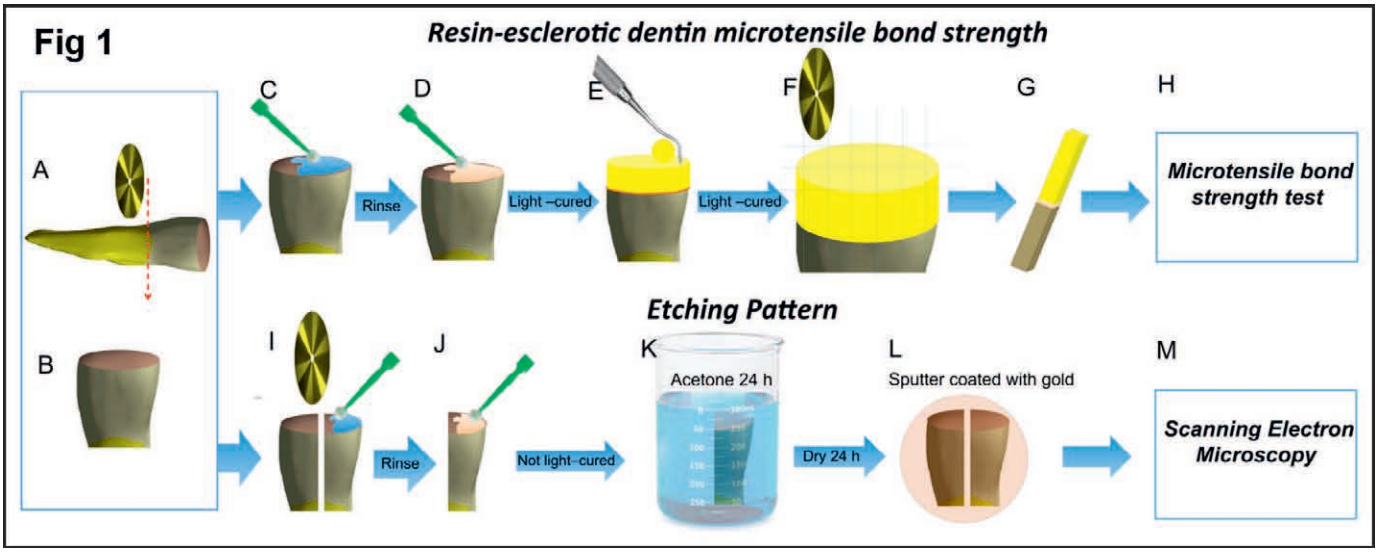

Figure 1. *Schematic drawing showing specimen preparation and microtensile bond strength testing. (A): The roots of all bovine teeth were removed by sectioning at the cementum-enamel junction. (B): Sclerotic tooth surface ready to receive the adhesive protocols. (C): Ethylene diamine tetra-acetic acid (EDTA) application according to each group. (D): Application of the universal adhesive systems (Scotchbond Universal Adhesive or Prime & Bond Elect) according to guidelines of the manufacturers. (E): Composite resin crowns were constructed. (F): Specimens were cut perpendicularly with a low-speed diamond saw to obtain resin-dentin specimens (G) for microtensile testing (H). (I): For the etching pattern, the teeth were cut in half; in one half, the adhesive was applied, and in the other half, the sclerotic dentin surface was left intact. (J): In the halves in which the adhesive protocol was performed, the groups received the application of EDTA; the adhesive application was per the manufacturer's instructions, except that the adhesive was not light cured. (K): Specimens were stored in an acetone bath for 24 hours (K), to remove all the resin monomers from the surface. (L): Samples were dehydrated for 24 hours, sputter coated with gold, and examined by scanning electron microscopy (M).*

ditioning protocols with EDTA do not affect the bonding effectiveness of adhesives to enamel.

METHODS AND MATERIALS

Resin-Dentin and Resin-Enamel Bond Strength

Sclerotic Dentin—Forty extracted bovine incisors with exposed sclerotic dentin in the incisal area due to abrasion/erosion and with an extremely vitreous appearance[12] were used in this part of the study. Bovine substrate was used instead of human teeth, as it is easy to obtain and has been considered a reliable substitute for human teeth.[29]

The teeth were extracted from the mandibles of three-year-old animals that had been slaughtered on a commercial scale for meat consumption. After harvesting, they were stored in distilled water at 4°C for no longer than one week before being used in this experiment. The roots of all teeth were sectioned, and the coronal pulp was removed (Figure 1).

The teeth were then embedded in chemically activated resin, with the exposed dentin surfaces parallel to the horizontal plane (Figure 1). The teeth were randomly assigned into eight experimental groups (n=5) according to the combination of the main factors "adhesive system" (two levels) and "surface treatment" (four levels), using a table of random numbers. A person not involved in the research protocol performed the randomization procedure using computer-generated tables.

In the control group, adhesives were applied as per the manufacturers' instructions (Table 1). In the group EDTA 30 seconds, a solution of 17% EDTA (Biodinâmica, Ibiporã, PR, Brazil) was applied manually with a microbrush actively for 30 seconds. In the group EDTA two minutes, the same solution was applied manually on the sclerotic dentin surfaces with a microbrush actively for two minutes. In the group EDTA 30 seconds + sonic, the 17% EDTA solution was applied as in the earlier group, but the solution was agitated with a sonic device. For this purpose, the microbrush was first attached to the prototype of the Smart sonic device (FGM Dental Products, Joinville, SC, Brazil).

The prototype produced an oscillating vibration of 10,200 rpm or 170 Hz as measured by the Blackman-Harris sound method.[27] The Smart device (FGM) has five different oscillating frequencies (144.5, 150, 170, 223.5, and 267.5 Hz). This study employed the middle frequency of the device. After application of the EDTA in the experimental groups, the sclerotic dentin surfaces were copiously rinsed with water for

Table 1: *Adhesive Systems, Batch Number, Composition, and Mode of Application*

Adhesive (Batch Number)	Composition	Self-etch Strategy
Scotchbond Universal Adhesive (SBU) (130811)	1.Adhesive: MDP phosphate monomer, dimetacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	1. Apply the adhesive to the entire preparation with a microbrush and rub it actively for 20 s
		2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent is evaporated completely[a]
		3. Light cure for 10 s at 1200 mW/cm^{2}[b]
Prime & Bond Elect (PBE) (523652)	1. Adhesive: Mono-, di-, and trimetacrylate resins; PENTA diketone; organic phosphine oxide; stabilizers; cetylaminehydrofluoride; acetone; water	1. Apply generous amount of adhesive to thoroughly wet all tooth surfaces
		2. Agitate for 20 s
		3. Gently dry with clean air for at least 5 s; surfaces should have a uniform, glossy appearance[a]
		4. Light cure for 10 s at 1200 mW/cm^{2}[b]

Abbreviations: MDP, methacryloyloxydecyldihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; PENTA, dipentaerythritolpenta acrylate monophosphate.
[a] *As per the manufacturers' instructions; as reported by manufacturers' material safety data issue or technical profile.*
[b] *The intensity of light curing was standardized for all materials.*

two minutes, and the surfaces were only slightly dried with an air stream without dehydration.

After that, the adhesive systems Scotchbond Universal Adhesive ([SBU] 3M ESPE, St Paul, MN, USA) and Prime & Bond Elect ([PBE] Dentsply, Milford, DE, USA) were applied by a single and calibrated operator according to the manufacturers' instructions (Table 1). After the bonding procedure, all teeth received a microhybrid composite restoration (Opallis, FGM Dental Products, Joinville, SC, Brazil) in three increments of 1 mm. Each increment was light cured for 40 seconds using a light-curing unit set at 1200 mW/cm^{2} (Radii Cal, SDI Limited, Bayswater, Victoria, Australia). The specimens were stored in water at 37°C for 24 hours.

To perform the microtensile bond strength test (μTBS), the specimens were cut perpendicularly with a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) to obtain resin-dentin specimens (0.8 mm^{2} cross-sectional dimensions on average) from each tooth for microtensile testing (Figure 1). The bonded area was measured to the nearest 0.01 mm with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan). If premature debonding occurred during sectioning, the number of specimens was recorded. Specimens were attached to a Geraldelli jig[30] with cyanoacrylate adhesive and stressed under tension (Kratos Dinamometros, Cotia, SP, Brazil) at 0.5 mm/min until failure. Bond strengths were calculated by dividing the load at failure by the cross-sectional bonding area.

The failure mode of the specimens was classified as adhesive/mixed if failure occurred at the resin-dentin bond interface with or without cohesive failure of the neighboring substrates and as cohesive if the failure occurred at the substrate (resin or dentin). The classification was done under a stereomicroscope at 40× magnification (Olympus SZ40, Tokyo, Japan).

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

The roots of all teeth were removed by sectioning at the cementum-enamel junction. The dental crowns were then sectioned in the diagonals across the long axis of teeth to produce four enamel specimens (buccal, lingual, and proximal; Figure 2).[31] Eighty enamel specimens, originating from 20 teeth, were ground wet with No. 180 and 600-grit SiC paper for 60 seconds, and each surface was mounted in a polyvinyl chloride ring filled with acrylic resin (AutoClear, Dent Bras, Pirassununga, SP, Brazil), showing the buccal, lingual, and proximal enamel surface on the top of the cylinder. Enamel surfaces were ground flat for development of uniform tensile forces during microshear loading.

For the microshear bond strength (μSBS), the delimitation of the bonding area was performed according to Shimaoka.[32] Four to six perforations with an internal diameter of 0.8 mm were made in an acid-resistant double-faced adhesive tape (Adel-

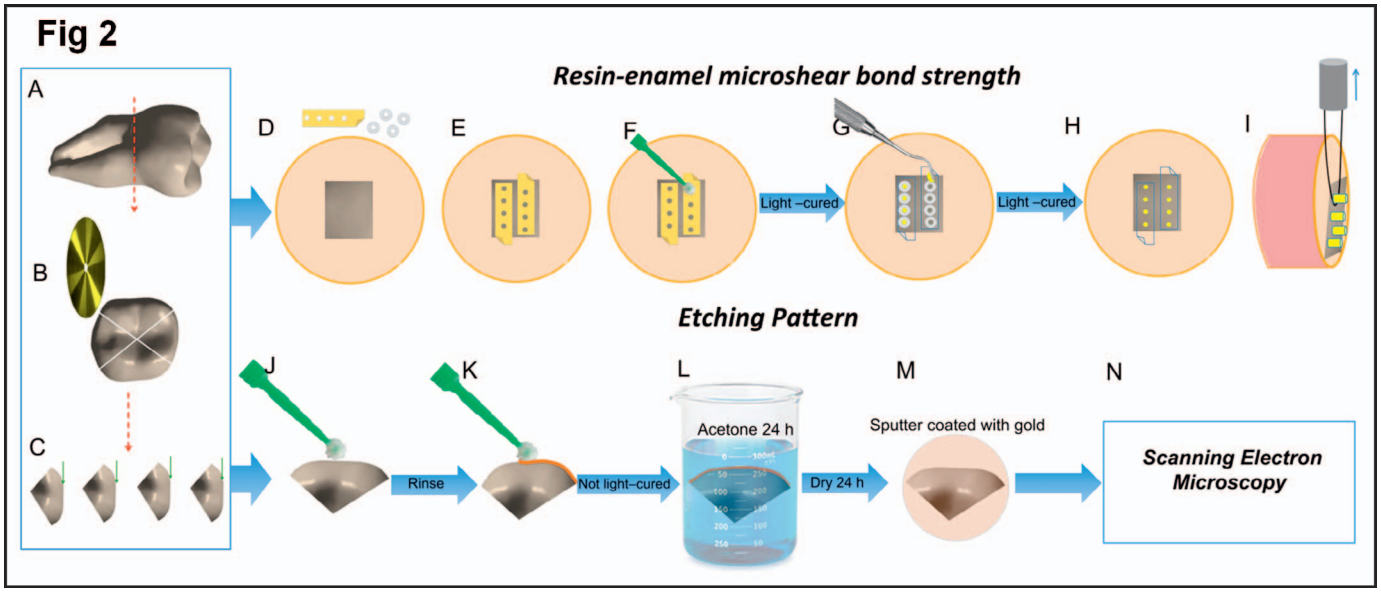

Figure 2. *Schematic drawing showing specimen preparation and microtensile bond strength testing. (A): The roots of all human teeth were removed by sectioning at the cementum-enamel junction. (B): Dental crowns were then sectioned in diagonals across the long axis of teeth. (C): Four enamel specimens (buccal, lingual, and proximals) were produced. (D, E): For resin-enamel microshear bond strength, each enamel specimen was mounted on a PVC ring filled with acrylic resin (displaying the enamel surface on the top of the cylinder); a double-faced adhesive tape was then attached to the enamel specimens to delimit the bonding area. (F): Adhesive application and light curing. (G): The Tygon tubes were placed on the enamel surface, and the lumen was filled with composite resin and light cured. (H): After storage, Tygon tubes and adhesive tapes were removed, leaving bonded resin composite cylinders only on the enamel surface. (I): Each tooth was placed in a jig and assembled in a universal testing machine for microshear bond strength testing with an orthodontic loop around the composite resin specimens. (J): For the etching pattern, the application of the EDTA protocols was done as performed in each group. (K): Adhesives systems (Scotchbond Universal Adhesive or Prime & Bond Elect) were applied, except that the adhesive layer was not light cured after application. (L): Specimens were stored in an acetone bath for 24 hours, to remove all the resin monomers from the surface. (M, N): Then, the samples were dehydrated for 24 hours, sputter coated with gold, and examined by scanning electron microscopy.*

bras Ind. e Com. Adesivos Ltda, SP, Brazil) with a hygienic Ainsworth-style rubber dam punch (Coltene, Alstätten, Switzerland). This adhesive tape was then attached to the enamel specimens (Figure 2).

The variation in the number of perforations was due to the different dimensions of the ground enamel specimens. Before adhesive application, all specimens were randomized into different groups as described for the sclerotic dentin (Figure 2). A single and calibrated operator applied the universal adhesive systems on enamel and sclerotic dentin.

After the application of the adhesive system, four to six polyethylene transparent Tygon tubes (Tygon Medical Tubing Formulations 54-HL, Saint Gobain Performance Plastics, Akron, OH, USA), with the same internal diameter of the perforations and a height of 0.5 mm, were positioned to face over the double-faced tape, ensuring that their lumen coincided with the circular areas exposed by the perforations (Figure 2).

Resin composite (Opallis, FGM Dental Products, Joinville, SC, Brazil) was carefully packed into each tube, and a clear Mylar matrix strip was placed over the filled Tygon tube and pressed gently into place. The composite was light cured for 40 seconds using a light-emitting diode light-curing unit set at 1200 mW/cm^2 (Radiical, SDI Limited, Bayswater, Victoria, Australia). These procedures were carried out under magnifying loupes.

After storage of the specimens in distilled water for 24 hours at 37°C, the Tygon tubes were carefully removed with a blade, exposing the composite cylinders (Figure 2). Each specimen was examined under a stereomicroscope at 10× magnification. The bonded cylinder was discarded if there was evidence of porosities or gaps at the interface.

The specimens were attached to a shear-testing fixture (Odeme Biotechnology, Joaçaba, SC, Brazil) and tested in a universal testing machine (Kratos IKCL 3-USB, Kratos Equipamentos Industriais Ltda, Cotia, SP, Brazil). Each specimen was positioned into the universal testing machine, and a thin orthodontic wire (0.2-mm diameter) was looped around the base of each composite cylinder. The orthodontic wire contacted the composite resin cylinder at half of its circumference (Figure 2).

The setup was kept aligned (resin-enamel interface, the wire loop, and the center of the load cell) to ensure the correct orientation of the shear forces.[27] The cross-head speed was set at 1 mm/min until failure. The μSBS values (MPa) were calculated by dividing the load at failure by the surface area (mm^2). The failure mode analysis was performed under a stereomicroscope at 100× magnification (Olympus SZ40) and classified as cohesive in enamel or resin composite, adhesive or mixed, which included adhesive and cohesive failure of the neighboring substrates.

Etching Pattern Examined by Scanning Electron Microscopy (SEM)

Sclerotic Dentin—Twenty-four additional bovine teeth were used for this part of the experiment. Teeth were randomly distributed using a table of random numbers into the same eight groups tested in the microtensile protocol. The teeth were cut perpendicular to their long axes using a slow-speed diamond saw (Isomet) to obtain two dentin halves. One half was used to evaluate the degree of dentin obliteration (control group), while the other half was treated according to one of the eight groups (n=3) described earlier in the μTBS test (Figure 1).

Initially, the specimens were then immersed in distilled water and left in an ultrasonic bath for five minutes to remove the debris from the surface.[33] Immediately after, the application was performed with EDTA for each experimental group of adhesives as described in Table 1, except that the adhesive layer was not light cured after application.

Each surface was rinsed with an acetone bath for 24 hours, to remove all resin monomers from the surface before being dehydrated for 24 hours in a desiccator containing colloidal silica. Finally, the samples were sputter coated with gold (Sputtering SCD050, BalTec, Balzers, Liechtenstein) and examined by SEM (SSX-550, Shimadzu, Tokyo, Japan) at 12 kV operated in secondary electron mode.

Enamel Substrate—Eight additional human teeth were used for this part of the experiment. The roots of all teeth were removed by sectioning at the enamel-cementum junction. The dental crowns were then sectioned in the diagonals across the long axis of teeth to produce four enamel slices (buccal, lingual, and proximal),[31] totaling 32 enamel specimens. In this part of the study, the enamel specimens were not ground as previously described and because we aim to evaluate the etching pattern in the most challenging condition (Figure 2).

First, the specimens were immersed in distilled water and left in an ultrasonic bath for five minutes to remove the debris from the surface.[33] Immediately after, the application was performed with EDTA for each experimental group of adhesives as described in Table 1, except that the adhesive layer was not light cured after application.

After that, the surfaces were immediately stirred in acetone for 24 hours to dissolve the monomers from the enamel surface.[34] All specimens were then allowed to dry for 24 hours in a desiccator, mounted on Al stubs, sputter coated with gold (Sputtering SCD050, Bal-Tec), and examined under the SEM (SSX-550, Shimadzu) at 12 kV operated in secondary electrons mode.

Statistical Analysis

All resin-dentin μTBS and resin-enamel μSBS values obtained from the same dentin and enamel surface, respectively, were averaged for statistical purposes.[35,36] The data from the μTBS and μSBS were then submitted to two-way analysis of variance (adhesive systems vs. surface treatment) and post hoc Tukey's test at a level of significance of 5%. The etching pattern produced on enamel and dentin substrates was evaluated only qualitatively.

RESULTS

Resin-Dentin Bond Strength

Approximately 12 to 20 resin-dentin bonded specimens were obtained from each tooth. The failure modes of all experimental groups are shown in Table 2. Most of the specimens (87%) presented adhesive/mixed failures. Dentin and resin cohesive failures were rarely observed. A small number of premature failures (1.6%) were observed. No significant difference was observed among groups (data not shown, chi-square test, $p>0.05$).

Only the main factors adhesive system ($p=0.01$) and surface treatment ($p<0.001$) were statistically significant. For both adhesives, the lowest mean μTBS values were observed when the adhesives were applied according to the manufacturer's directions (Table 3). Compared with the control group (without EDTA application), manual EDTA application either for 30 seconds or two minutes resulted in higher resin-dentin bond strength (Table 3; $p<0.05$). However, for both adhesives, the highest μTBS values were observed when the EDTA was applied for 30 seconds with the sonic device (Table 3).

Table 2: *Number of Specimens and Percentage (%) of Failure Types for All Experimental Groups in Sclerotic Dentin*

Adhesive System	Failure Type	Surface Treatment			
		Control	EDTA 30 s	EDTA 2 min	EDTA 30 s + Sonic Device
SBU	A	58 (93.6)	41 (82.3)	50 (80.4)	42 (76.3)
	D	0 (0)	3 (5.9)	2 (3.2)	2 (3.6)
	R	2 (3.2)	3 (5.9)	6 (9.5)	8 (14.5)
	M	2 (3.2)	3 (5.9)	5 (6.9)	2 (3.6)
	PF	0 (0)	0 (0)	0 (0)	1 (2)
PBE	A	42 (85.7)	43 (76.9)	42 (77.9)	49 (80.3)
	D	3 (6.1)	2 (3.5)	6 (11.1)	2 (3.3)
	R	0 (0)	4 (7.1)	5 (9.3)	4 (6.6)
	M	4 (8.2)	4 (7.1)	0 (0)	5 (8.2)
	PF	0 (0)	3 (5.4)	2 (1.7)	1 (1.6)

Abbreviations: A, adhesive failure; D, dentin cohesive failure; EDTA, ethylene diamine tetra-acetic acid; M, mixed failure; PBE, Prime & Bond Elect; PF, premature failure; R, resin cohesive failure; SBU, Scotchbond Universal Adhesive.

Resin-Enamel Bond Strength

Approximately four to six resin-enamel bonded specimens were obtained from each tooth. Table 4 shows the percentage of fracture patterns found in each group. Most of the specimens (92%) presented adhesive/mixed failures. No cohesive failure in enamel or cohesive failure in resin was observed for either adhesive. A small number of premature failures (8.3%) were observed. No significant difference was observed among groups (data not shown, chi-square test, $p>0.05$). Table 5 shows the mean values and standard deviations of the μSBS according to the experimental groups to be addressed. The cross-product interaction adhesive system versus surface treatment ($p=0.754$) as well as the surface treatment ($p=0.11$) were not statistically significant. Only the main factor adhesive was significant ($p<0.0001$), with SBU showing the highest μSBS when compared with PBE.

Etching Pattern on Sclerotic Dentin

The etching pattern produced by the different protocols on sclerotic dentin can be seen in Figures 3 and 4. The presence of a hypermineralized dentin substrate, with obliterated dentin tubules in the sclerotic dentin, can be confirmed by Figures 3A and 4A.

Without any additional treatment, the application of the universal adhesives in the self-etch mode produced dentin substrates with dentin tubules obliterated by mineralized deposits (Figures 3B and 4B). This is more evident for the PBE adhesive (Figure 4B).

The use of EDTA for 30 seconds under manual application (Figures 3C and 4C) improved the etching pattern, but mineralized deposits can still be seen as in the control groups. On the other hand, EDTA pretreatment for two minutes, followed by adhesive application, produced a dentin substrate with more open dentin tubules, with only some of them obliterated by mineralized deposits (Figures 3D and 4D). The application of EDTA for 30 seconds with the sonic device (Figures 3E and 4E) produced a dentin substrate that resembles that of the EDTA two minutes. Although mineralized deposits can still be seen in the EDTA 30 seconds + sonic group, they are located deeper in the dentin tubules.

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

Adhesive System	Surface Treatment				Main Factor Adhesive System[a]
	Control	EDTA 30 s	EDTA 2 min	EDTA 30 s + Sonic Device	
SBU	32.8 ± 2.8	40.3 ± 4.2	49.3 ± 4.7	54.4 ± 1.8	44.2 ± 9.0 A
PBE	28.4 ± 0.9	40.6 ± 1.7	40.9 ± 9.4	49.9 ± 3.7	40.0 ± 9.2 B
Main factor surface treatment*	30.6 ± 3.0 c	40.5 ± 3.2 b	45.1 ± 8.6 b	52.2 ± 3.7 a	—

Abbreviations: EDTA, ethylene diamine tetra-acetic acid; PBE, Prime & Bond Elect; SBU, Scotchbond Universal Adhesive.
[a] *Uppercase letters indicate comparison of the main factor adhesive, and lowercase letters indicate comparison between the main factor surface treatments. Different uppercase or lowercase letters indicate groups statistically different (analysis of variance, Tukey's test, $p<0.05$).*

Table 4: *Number of Specimens and Percentage (%) of Failure Types for All Experimental Groups in Enamel*

Adhesive System	Failure Type	Surface Treatment			
		Control	EDTA 30 s	EDTA 2 min	EDTA 30 s + Sonic
SBU	A	60 (78.5)	59 (94.2)	67 (87.6)	72 (98.3)
	D	0 (0)	0 (0)	0 (0)	0 (0)
	R	0 (0)	0 (0)	0 (0)	0 (0)
	M	16 (19.0)	7 (3.0)	9 (12.4)	0 (0)
	PF	4 (2.5)	5 (2.8)	0 (0)	3 (1.7)
PBE	A	49 (65)	62 (90.1)	45 (75.0)	58 (71.5)
	D	0 (0)	0 (0)	0 (0)	0 (0)
	R	0 (0)	0 (0)	0 (0)	0 (0)
	M	4 (6.5)	3 (1.9)	3 (5.0)	16 (25.5)
	PF	26 (28.5)	9 (8.0)	12 (20.0)	5 (3.0)

Abbreviations: A, adhesive failure; D, dentin cohesive failure; EDTA, ethylenediaminetetraacetic acid; M, mixed failure; PBE, Prime & Bond Elect; PF, premature failure; R, resin cohesive failure; SBU, Scotchbond Universal Adhesive.

Etching Pattern on Enamel

The etching pattern produced by the different protocols on enamel can be seen in Figures 5 and 6. In enamel, the application of SBU produced a shallow selective enamel demineralization (type I pattern) with some areas of unetched enamel (Figure 5A). However, this was not observed in the PBE group (Figure 6A). The use of EDTA in all groups produced a deeper demineralization of the enamel prism cores (Figures 5B,D and 6B,D), even for the PBE group (Figure 6), with fewer areas of no selective enamel etching. Differences between the different EDTA protocols are not very evident for both adhesives. The etching pattern produced by the SBU was visually more retentive than that of the PBE for both enamel and dentin (Figures 3 through 6).

DISCUSSION

Sclerotic dentin is a challenging substrate for dentin bonding. It is much more resistant to dissolution by acidic monomers because of the presence of a hypermineralized surface layer and partial/total obliteration of the dentinal tubules.[5] The presence of mineralized deposits inside the dentinal tubules hinders the formation of resin tags and reduces the thickness of the hybrid layer in the intertubular dentin.[8,37,38] These are possible reasons why lower bond strength values are observed in this substrate as compared with sound dentin to self-etch adhesives.[39,40]

Our study showed that pretreatment of sclerotic dentin with EDTA produced a significant increase in the immediate μTBS values for both universal adhesives, which led us to reject the first null hypothesis. This is in agreement with other published studies that reported higher μTBS values for self-etch adhesives when applied in EDTA-treated dentin substrates.[6,14,15]

Unlike previous studies, our study evaluated several different EDTA application protocols, to include a novel sonic application. We observed that the two-minute application time, as already tested in the literature,[13,16] produced μTBS values similar to a shorter manual application time of 30 seconds, which requires reduced clinical time. This means that clinicians can shorten the application time of the

Table 5: *Microshear Bond Strength (μSBS in MPa) Values (Means ± Standard Deviations) for the Different Experimental Groups*[a]

Adhesive System	Surface Treatment				Main Factor Adhesive System
	Control	EDTA 30 s	EDTA 2 min	EDTA 30 s + Sonic Device	
SBU	15.4 ± 1.2	15.2 ± 1.5	16.3 ± 1.6	14.4 ± 1.3	15.3 ± 1.4 A
PBE	9.3 ± 1.3	9.4 ± 0.8	10.2 ± 0.9	9.5 ± 0.8	9.6 ± 1.0 B
Main factor surface treatment	12.4 ± 1.2 a	12.3 ± 1.3 a	13.3 ± 1.3 a	12.0 ± 1.1 a	—

Abbreviations: EDTA, ethylene diamine tetra-acetic acid; PBE, Prime & Bond Elect; SBU, Scotchbond Universal Adhesive.
[a] *Uppercase letters indicate comparison of the main factor adhesive, and lowercase letters indicate comparison between the main factor surface treatments. Different uppercase letters indicate groups statistically different (analysis of variance, Tukey's test, $p<0.05$). Similar lowercase letters indicate groups statistically similar (analysis of variance, Tukey's test, $p>0.05$).*

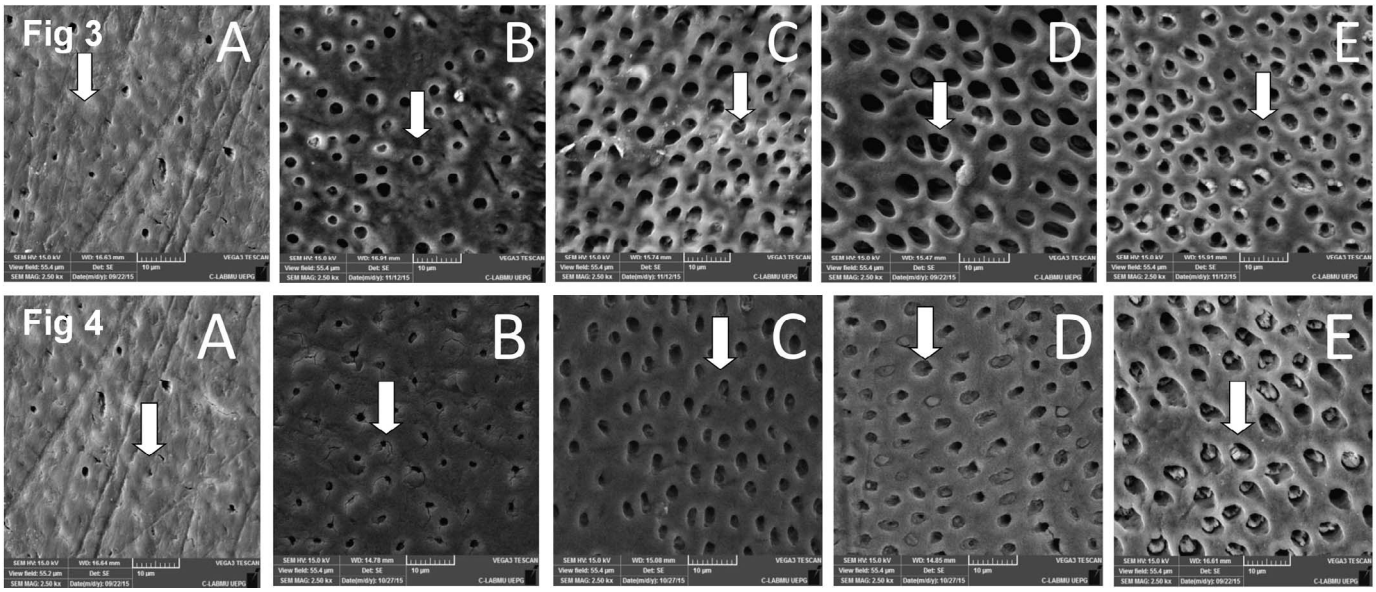

Figure 3. *Etching pattern of sclerotic dentin after the different protocols of bonding with the Scotchbond Universal Adhesive (SBU). In (A), we can observe the sclerotic dentin surface without any type of dentin treatment. There are a high number of obliterated dentin tubules (white arrow) in the dentin substrate. In (B), the sclerotic dentin surface was treated only with the universal adhesive in the self-etch mode. Although the dentin tubules are more evident, they are still obliterated by mineralized deposits (white arrow). In (C), the sclerotic dentin was treated with ethylene diamine tetra-acetic acid (EDTA) for 30 seconds with manual application before adhesive application. Dentin tubules are opened, but there are mineralized sclerotic deposits (white arrow) inside all of the dentin tubules. In (D), the sclerotic dentin was treated with EDTA for two minutes before adhesive application. The dentin tubules are much more opened, and only a few of them are obliterated by mineralized deposits (white arrow). In (E), dentin was treated with EDTA for 30 seconds with sonic application. This surface looks like the EDTA two-minute group with dentin tubules opened and less mineralization inside the dentin tubules (white arrow).*

Figure 4. *Etching pattern of sclerotic dentin after the different protocols of bonding with Prime & Bond Elect (PBE). In (A), we can observe the sclerotic dentin surface without any type of dentin treatment. There is a high number of obliterated dentin tubules (white arrow) in the dentin substrate. In (B), the sclerotic dentin surface was treated only with the universal adhesive in the self-etch mode. Although the dentin tubules are more evident, they are still obliterated by mineralized deposits (white arrow). In (C), the sclerotic dentin was treated with ethylene diamine tetra-acetic acid (EDTA) for 30 seconds with manual application before the adhesive protocol. Dentin tubules are opened, and sclerotic mineralized deposits can be seen practically in all dentin tubules (white arrow). In (D), the sclerotic dentin was treated with EDTA for two minutes before adhesive application. The dentin tubules are much more opened (white arrow). In (E), dentin was treated with EDTA for 30 seconds with sonic application. Dentin tubules are evident and opened, although some mineralized deposits can still be seen, but they are located deeper in the dentin tubules (white arrow).*

EDTA without compromising the bond strength values to this substrate.

The higher μTBS values of the EDTA-treated group can be attributed to the structure of the chelating ability of the EDTA molecule. The presence of four carboxylic acid groups produces the sequestration of metal ions of dental substrates and causes the selective dissolution of hydroxyapatite.[14] In dentin, EDTA removes the surface smear layer, which is a natural barrier to the penetration of acidic primers,[19,41] and creates a cleaner substrate, with a more retentive etching pattern (Figures 3 and 4) than that produced by the self-etch without previous EDTA application. This allows for better interaction of the self-etch adhesive with the sclerotic dentin substrate.

Unlike phosphoric acid etching (which also removes the smear layer but leaves collagen fibers exposed and perhaps prone to degradation), use of EDTA produces only a partial dissolution of hydroxyapatite. EDTA leaves residual apatite crystals in the collagen matrix and may make collagen more resistant to denaturation,[14,42] producing dentin-bonded interfaces that are less prone to degradation over time.[14,15]

Interestingly, the present study demonstrated that the application of EDTA for only 30 seconds with the aid of a sonic device produced a higher μTBS than the other EDTA protocols. Sonic vibration propagates pressure waves because of the stimulation of the EDTA molecules. The agitated molecules are able to reach areas beyond those where the bristles of the microbrush can touch. The high-speed vibration of the microbrush creates pressure waves and shear forces in the adhesive.[27] It also generates microscopic bubbles that are forcefully propelled against surfaces to which the adhesive solution is applied, increasing the dissolution of the smear layer, due to the fluid dynamics of

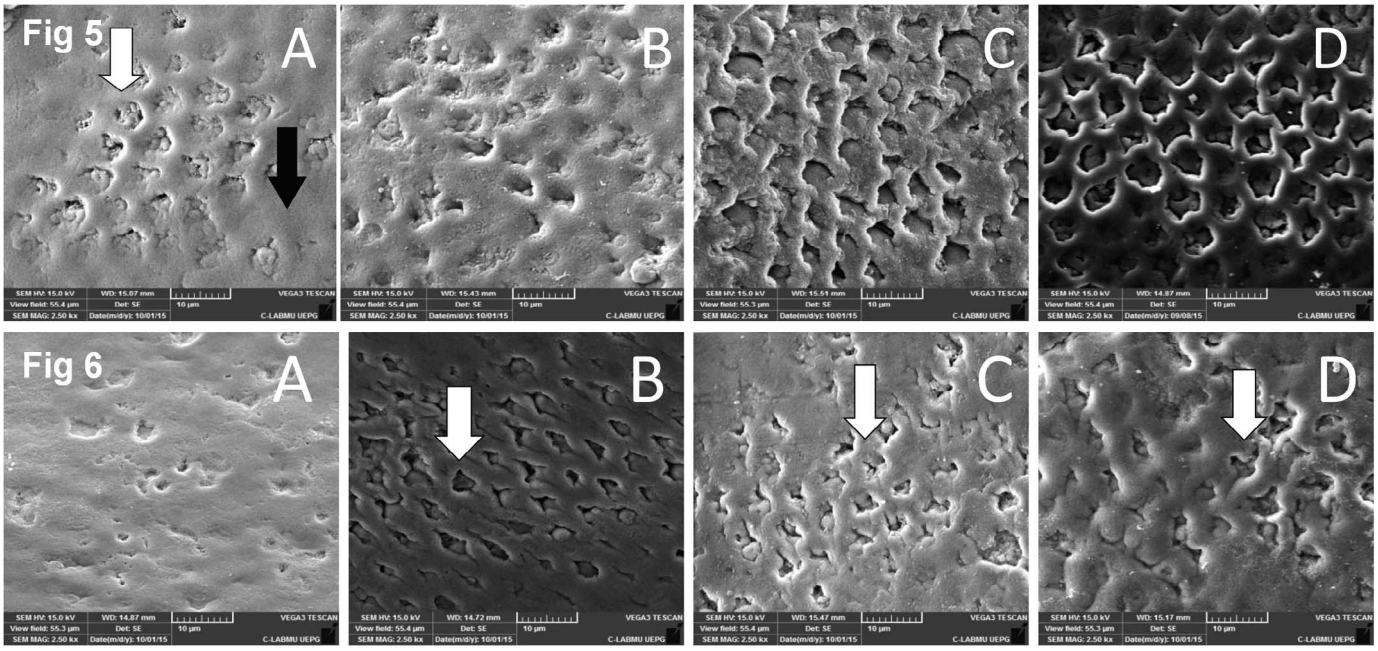

Figure 5. *Etching pattern of enamel after the different protocols of bonding with the Scotchbond Universal Adhesive (SBU). In (A), the adhesive was applied without any pretreatment. One can observe a very smooth selective etching of prism cores (white arrow, type 1 pattern), with some areas without selective demineralization (black arrow). In (B), ethylene diamine tetra-acetic acid (EDTA) was manually applied for 30 seconds. The etching pattern is somewhat better than that of image A, with a shallow demineralization of the prism cores and just some islands without selective enamel etching. In (C), EDTA was applied for two minutes before adhesive application. The same type of etching pattern was observed, but the demineralization of the prism cores is deeper, and there is no area of unetched enamel. In (D), EDTA was applied for 30 seconds with a sonic device. Some prism cores were demineralized deeper, similar to the EDTA two-minute group.*

Figure 6. *Etching pattern of enamel surface after the different protocols of bonding with the Prime & Bond Elect (PBE). In (A), the adhesive was applied without any pretreatment. One can observe a very smooth surface without any selective pattern of etching. In (B), ethylene diamine tetra-acetic (EDTA) was manually applied for 30 seconds. In (C), EDTA was applied for two minutes before adhesive application. In (D), EDTA was applied for 30 seconds with a sonic device. One can observe in all groups where EDTA was applied a very shallow demineralization of the prism cores (white arrow) and some islands of unetched enamel.*

acid on the sclerotic surface.[27] This allows better removal of the mineralized deposits inside the dentin tubules than the application of EDTA for 30 seconds with manual application. Use of the sonic application device generated the highest μTBS values.

Although there is no study that evaluated the EDTA application on sclerotic dentin with this sonic device, which prevents us from further comparison with the literature, an earlier study reported that the application of EDTA as an endodontic irrigant, with the sonic device, was more efficient at removing more of the smear layer inside the root canal than the conventional application.[43] In coronal dentin, there are reports that the use of this sonic device increases the resin-dentin μTBS of adhesive systems[27,44] and also reduces the permeability of the adhesive layer.[27]

Apart from the increases in the resin-dentin μTBS in the present and earlier studies by EDTA etching,[14,15,41] previous authors demonstrated that 17% EDTA applied for two minutes can reduce the activity of MMPs of human dentin.[16] The chelating activity of EDTA promotes the sequestration of zinc and calcium ions that act as potential activators of MMPs,[45,46] thereby minimizing its action on the hybrid layers.

The benefits of EDTA pretreatment in dentin were also confirmed in a recent randomized clinical trial. A higher retention rate of composite resins was observed in noncarious cervical lesions bonded with a self-etch adhesive when the dentin was pretreated with EDTA 17% for two minutes.[13]

In the current study, EDTA pretreatment of enamel did not show any benefits. None of the different types of surface treatment affected the μSBS values of either adhesive, leading us to accept the second null hypothesis. This finding is in agreement with previous studies[18,19] that observed that EDTA pretreatment in enamel was not effective at improving the bond strength of self-etch adhesives.

Although a more retentive etching pattern was observed in enamel after EDTA pretreatment, in

none of the conditions was this pattern similar to that produced by phosphoric acid etching,[47,48] which is considered the gold standard etchant for enamel. The EDTA pretreatment and self-etch application allowed for some selective demineralization of the enamel, with preferential dissolution of the enamel prism cores. However, this procedure did not produce microporosities within the prism cores and peripheries, as seen with phosphoric acid etching.

For both enamel and dentin, clear differences among the adhesives were observed. Most currently available universal adhesives contain at least one functional acidic monomer, which has chemical bonding potential. According to the manufacturer's information, SBU contains two components with this potential: methacryloxydecyl phosphate (MDP)[49] and methacrylate-modified polyalkenoic acid copolymer,[31] while PBE contains only dipentaerythritol-penta acrylate monophosphate as the functional monomer. This twofold mechanism of demineralization of the substrates may be responsible for the better etching potential of SBU than PBE in enamel and dentin in the present study as well as in previous studies.[31,50]

Although monomers with potential bonding to calcium are also presented in the composition of PBE,[51] chemical bonding alone is not enough to provide a strong bonded interface; the calcium salt produced by this chemical interaction should also be stable in an aqueous environment, such as that produced by MDP.[49,52] Furthermore, the interaction of MDP with hydroxyapatite is significantly stronger and within a clinically reasonable application time. This is reflected in the higher microtensile strength to dentin and enhanced sealing potential for the prevention of nanoleakage and thus extend bonding longevity.[53] Hydroxyapatite, however, needs to remain available at the partially demineralized dentin surface; this may also explain the greater difficulty with the effectiveness of self-etching adhesive in sclerotic dentin.

One should not deny that EDTA preconditioning includes an extra clinical step to the already complicated bonding protocol, but this study showed that with the aid of a sonic application, the extra time for EDTA preconditioning may be reduced even further than the application times recommended so far while keeping the benefits of this protocol. However, when available for purchase, the sonic device will add some additional cost to clinicians, which may be seen as a clinical limitation to the implementation of its use.

Further clinical trials should be performed to validate the results of the current *in vitro* study.

CONCLUSIONS

In sclerotic dentin, a shorter EDTA application time of 30 seconds can yield similar results to those produced by the conventional two-minute EDTA application in dentin. However, a 30-second application of EDTA in combination with a sonic device produced the highest resin-bond strengths to sclerotic dentin. The more visibly retentive etching pattern produced in enamel after EDTA pretreatment did not result in improved bond strengths to enamel.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local oversight committee guidelines and policies of UEPG. The approval code for this study is 110.234.

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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Novel Microscalpels for Removing Proximal Composite Resin Overhangs on Class II Restorations

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Clinical Relevance

Microscalpels and scalpel numbers 12 and 15 can be efficiently used to remove excess composite material in accessible interproximal areas. Scalpels seem to be able to cut composite resin in a material-specific manner without damaging tooth surfaces or resin material surfaces.

SUMMARY

Introduction: Limited access to interdental spaces complicates removal of excess material when placing class II composite resin restorations. Evidence-based recommendations on interproximal finishing are rare. We present novel microscalpels for this indication. The aim of the study was to test their fracture strength and cutting ability and to compare microscalpels with the use of a scaler, oscillating devices (G5-ProShape, G5-Proxocare), finishing strips and scalpels of sizes 12, 15, and 21 in a standardized *in vitro* model.

Methods and Materials: Fracture strength (LOAD) and cutting forces (CUT) of microscalpels were evaluated at different angles (15, 30, 60, and 75 degrees; n=30 each) in a universal testing machine. Devices were compared *in vitro* using standardized composite overhangs. Marginal quality (QUAL; n=30) and quantity of excess/deficit (QUAN; n=30) were evaluated using scanning electron microscopy (SEM) for each device (explorative data analysis, Student *t*-test or analysis of variance; *post hoc* Scheffé).

Results: Microscalpels showed the highest LOAD (95.8 [5.0] N) (mean [standard deviation]) and easiest cutting (CUT) (7.6 [1.5]) at 15 degrees. At all angles, LOAD was significantly higher than CUT ($p<0.001$). Perfect margins were seen most often with scalpel size 12 (QUAL: 37% relative frequency), while most excess (73.4%) was observed with finishing strips. QUAN was lowest with microscalpels (19.3 [4.4] μm) and highest with finishing strips (116.0 [18.8]). Use of scalers led to fractures and crack formation.

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Conclusion: Microscalpels are able to cut composite at a lower force than necessary to fracture the blades at all angles. Small and/or curved scalpels yield the best-quality margins.

INTRODUCTION

Currently, posterior composite resin restorations are widespread treatment options for all cavity sizes. They show good survival when patient, operator, and material parameters are considered adequately.[1-5] Previous limitations in bonding techniques have long been overcome, and even proximal contacts can be adequately reconstructed with novel types of sectional matrices applied with separation rings.[6-8] Concerning layering techniques, elaborate concepts have been presented,[9] while with regard to finishing and polishing, experimental and case reports and descriptions of technical procedures are available.[10-16]

However, the description of finishing procedures focuses mainly on easily accessible surfaces[10] and easily accessible cavity classes (class V[17]), whereas the finishing of proximal surfaces is not specified at all. In general, instruments recommended for marginal finishing are, for example, fine or ultrafine finishing diamonds, which seem to be superior to carbide finishing burs.[15] Even the polishing direction of instruments has been tested and is reported to be superior when moved from the restoration to the tooth surface.[18] Yet those techniques and instruments are not suitable for the interproximal area or are applicable only under extremely limited circumstances.

The lack of interproximal polishing approaches is becoming a more profound problem when looking at the fact that class II composite resin restorations carry a distinct risk of having proximal overhangs.[19] Such proximal overhangs can irritate the periodontium and jeopardize periodontal health.[20-28] Opdam and others[29] reported that up to 43% of margins were overfilled in class II composite resin restorations. Further, overfilled or overhanging margins can also be seen with various other insertion techniques and matrix systems.[29-33] Very early reports on proximal finishing recommended the application of burs, stones, and flexible finishing strips and discs,[16] oscillating devices (eg, Roto-Pro and EVA), and sonic and ultrasonic devices (eg, Cavitron and Sonic Scaler).[34,35] Since those early publications, no novel or innovative instruments for interproximal excess removal have been developed or tested. To address this shortcoming, it is believed that there is great potential in dental scalpels, which

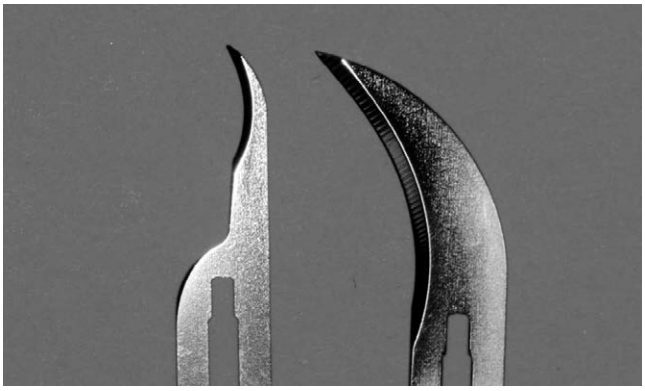

Figure 1. *Microscalpel (left) and scalpel number 12 (right).*

are available in various shapes and sizes. At present, their main field of use is in surgery, and they remain of minor importance in restorative dentistry. The use of a scalpel with blade number 12 has already been reported to be helpful for the removal of composite resin excess after adhesive cementation of ceramic blocks.[36] In addition, Morgan and others[37] reported the use of blade number 12 for removing excess composite from the proximal area. Very recently, Kup and others[38] described the use of scalpel number 15 in shaping composite resin as a "material-selective" and "tooth-friendly" way to finish dental composites in anterior teeth. A critical review from our group described the application of scalpel number 12 in the course of a novel two-step application technique for subgingival composite resin restorations.[39] There are increasing data suggesting that blades numbers 12 and 15 are especially suitable for material-selective shaping and finishing of composite resin restorations. Having gained clinical experience with scalpels over many years, our research team had the idea of further reducing the size of the scalpel blades used. Inspiration came from microscalpels, which are used in ophthalmological microsurgery. In collaboration with a company (Trinon Titanium GmbH, Karlsruhe, Germany), a prototype microscalpel (Figure 1) was developed in our department. The delicate blade appeared promising with regard to interproximal accessibility and excess removal efficiency. However, it remained to be ascertained whether the small blade could withstand the high working forces necessary to cut composite resin material without fracturing. This research question was the first aim of our study.

The second aim was to compare traditional proximal finishing devices competitively with the novel device to see if it provides any superior benefit. After screening the literature, it was decided to

include plastic flexible finishing strips, oscillating polishing devices, standard scalpels (numbers 12, 15, and 21), and a scaler.

A brief rationale for this choice is presented next. Abrasive finishing strips are recommended in state-of-the-art textbooks and reviews.[40-42] However, these are not capable of reaching concavities of the proximal tooth surfaces due to a "bridging effect." In spite of this, it was included since they are easy to use and widespread. The oscillating polishing device G5-ProxoCare (SDC Switzerland SA, Grancia, Switzerland) was also included in the study. This is similar to the EVA system (KaVo Dental GmbH, Biberach an der Riss, Germany),[34,35] with both the G5-ProxoCare and the EVA system being powered by air pressure, which induces oscillation of a polishing file at the instrument's head.[43] The polishing files are delicate and abrasive on only one side, allowing for comparatively good access to the interdental space without damaging the neighboring tooth surface. The second oscillating device G5-ProShape (SDC Switzerland) was originally introduced as an instrument for orthodontic enamel slicing. It is composed of a metal finishing strip fixed to a plastic frame holder. Its efficient enamel removal prompted the manufacturer to extend the indication to interproximal removal of excess restorative material, and consequently it was included in the present investigation. Scalers are designed for the removal of calculus and plaque from tooth surfaces. Being a hand instrument used in prophylaxis and periodontal treatment, they provide ergonomic handling and good accessibility into interdental spaces even in the farthest posterior locations. Therefore, they have proven convenient to use for the removal of excess bonding material in restorative dentistry.[44-46] The choice of scalpel numbers 12 and 15 is based on literature reports[36-38] and personal clinical experience.[39] Scalpel number 21 is a comparatively large blade not primarily suitable for interdental working and acted as a kind of negative control in our setting.

The following research questions were raised and addressed by this experimental study:

1. Are novel microscalpels safe to use without the risk of fracture?
2. Is there any superior instrument for removing composite resin excess in the interproximal area?

METHODS AND MATERIALS

To answer the first research question, the maximum load strength (LOAD) and cutting ability (CUT) of microscalpels used at different working angles were investigated.

Table 1: *Instruments Tested in the Study*

Instrument	Manufacturer
Scaler S204S7	Hu-friedy Mfg Co, LLC, Tuttlingen, Germany
G5-ProShape grey (60 μm)[a]	SDC Switzerland SA, Bioggio, Switzerland
G5-Proxocare 1760 (60 μm)	SDC Switzerland
Scalpel blade number 12	Feather, Osaka, Japan
Scalpel blade number 15	Feather
Scalpel blade number 21	Feather
SofLex Finishing Strips (Coarse[b])	3M ESPE, Seefeld, Germany
Microscalpel[a]	Trinon Titanium GmbH, Karlsruhe, Germany

[a] *Prototype.*
[b] *No further specification of grit by manufacturer.*

To address the second research question, traditional instruments for proximal excess removal (Table 1) were compared with microscalpels in an *in vitro* model on standardized composite overhangs. Marginal quality (QUAL) and quantity of excess or deficit (QUAN) were evaluated.

LOAD Testing

In clinical use, instruments can be used at different angles on tooth surfaces, requiring the application of gradual forces. To simulate a clinical procedure, microscalpels were tested at four different angles (15, 30, 60, and 75 degrees). The test specimens were made from Eppendorf tubes with aluminum rods polymerized with composite resin perpendicular to the tube axis (Figure 2). Scalpel holders were fixed to a universal testing machine (Zwicki1120, Zwick GmbH & Co, Ulm, Germany) at each of the four angles tested. To test the maximum load, the scalpel was moved up steadily (10 mm/min) parallel to the tube axis toward the aluminum rod until fracture. The maximum load at fracture was displayed in a load/time graph over 90 seconds, and the maximum force was determined as LOAD (N). At each angle, the measurements were repeated 30 times.

CUT Testing

The angle influences the cutting ability of the blade. To test these forces, the same experimental setup was used. Here, specimens were composed of Eppendorf tubes with standardized composite resin overhangs of 150 μm (TetricEvoCeram, Ivoclar Vivadent, Schaan, Liechtenstein), which were fabri-

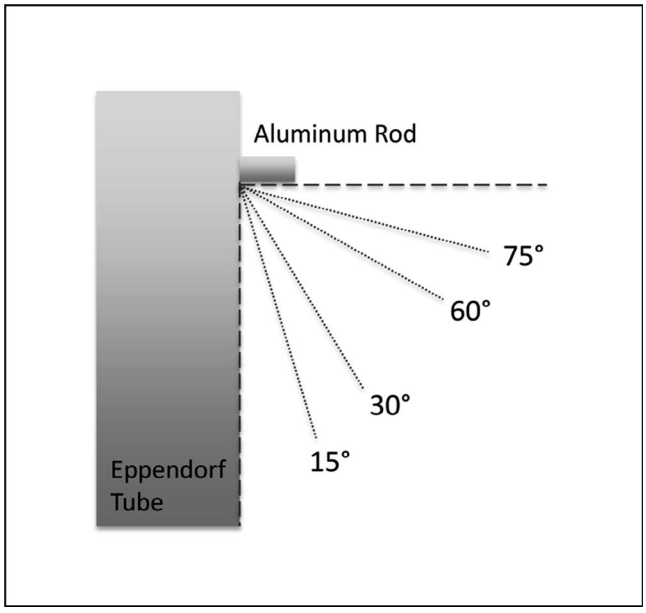

Figure 2. *Schematic diagram of the experimental setup for LOAD testing. The scalpel blade was moved steadily up to the aluminum rod with a velocity of 10 mm/min until fracture.*

cated in a standardized manner using a molding frame. The test procedure was run similarly as described above at all four angles (n=30 measurements per angle). The maximum force necessary to cut the composite resin overhang was displayed in a load/time graph over 90 seconds and determined to be CUT (N).

QUAL/QUAN Experimental Setup

Eight devices for interdental excess removal were tested (Table 1). One hundred and twenty extracted caries- and restoration-free human molars were used to fabricate *ex vivo* models. Teeth were cleaned and optically screened (magnification 2.3×, dental magnification loupes) for cracks, fissures, or flaws in the proximal surfaces. Teeth were stored in 50% ethanol solution until use. They were then randomly distributed into eight groups (n=15 per group) providing a total of up to 30 restorations (mesial and distal sides of the tooth) per group. Human and artificial teeth (Frasaco GmbH, Tettnang, Germany) were set in 120 models simulating rows of teeth. The models allowed for fixation in a phantom head of a dental simulation unit, thus making the application of the test instruments clinically more realistic. For the standardized cavity preparation, we used sonic preparation devices (Sonic Sys 3, SONICflex prepgold, KaVo Dental). The proximal preparation margins were located within the enamel. Proximal composite resin overhangs of 150 μm (mesio-distal thickness) were

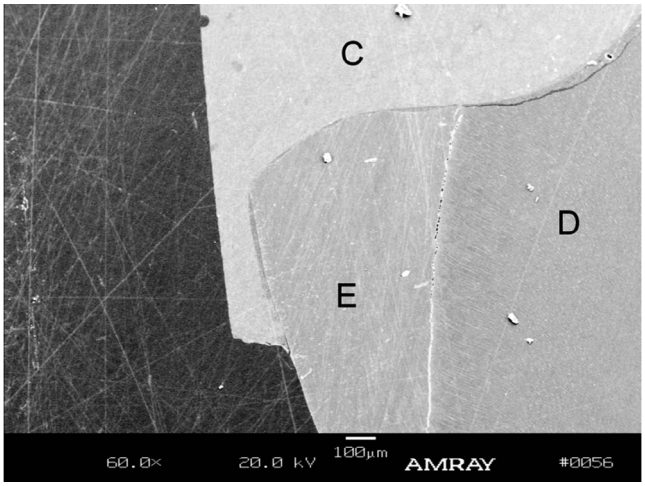

Figure 3. *SEM image of the standardized composite resin overhang of 150 μm (E, enamel; D, dentin; C, composite resin restoration).*

created by applying three layers of metal matrix band (Tofflemire, Kerr, Raststatt, Germany, thickness 50 μm) to maintain the distance between the circumferential matrix band and the proximal tooth surface cervical to the proximal cavity margin. With this technique, we were able to produce standardized overhangs similar to those resulting from insufficiently wedged matrices (Figure 3). Cavities were etched (Email Preparator, Ivoclar Vivadent, 30 seconds enamel, 15 seconds dentin) and treated with the adhesive system according to the manufacturer's instructions (OptiBond FL, Kerr Hawe, Bioggio, Switzerland). Composite resin was applied in layers of a maximum of 2-mm thickness (Tetric Evo Ceram, Ivoclar Vivadent), and each layer was polymerized (Bluephase, Ivoclar Vivadent, 1200 mW/cm^2, 40 seconds). One experienced dentist carried out interproximal excess removal for a limited time period of two minutes before teeth were removed from the model and further analyzed.

QUAL Analysis

Teeth were cut through the center in the buccolingual direction, resulting in two halves containing one restoration each. Specimens were dehydrated through ascending grades of ethanol, fixed to an SEM holder, air-dried in a desiccator at approximately 20 hPa, and sputter coated with a 30-nm layer of gold (S150, Edwards, Marburg, Germany). For evaluation of the proximal restoration margins, specimens were magnified (30×) in an SEM (1810D, Amray, Bedford, MA, USA). One blinded examiner visually evaluated the marginal qualities by assigning one grade to each specimen. Grading of qualities was 1) perfect: continuous margin between

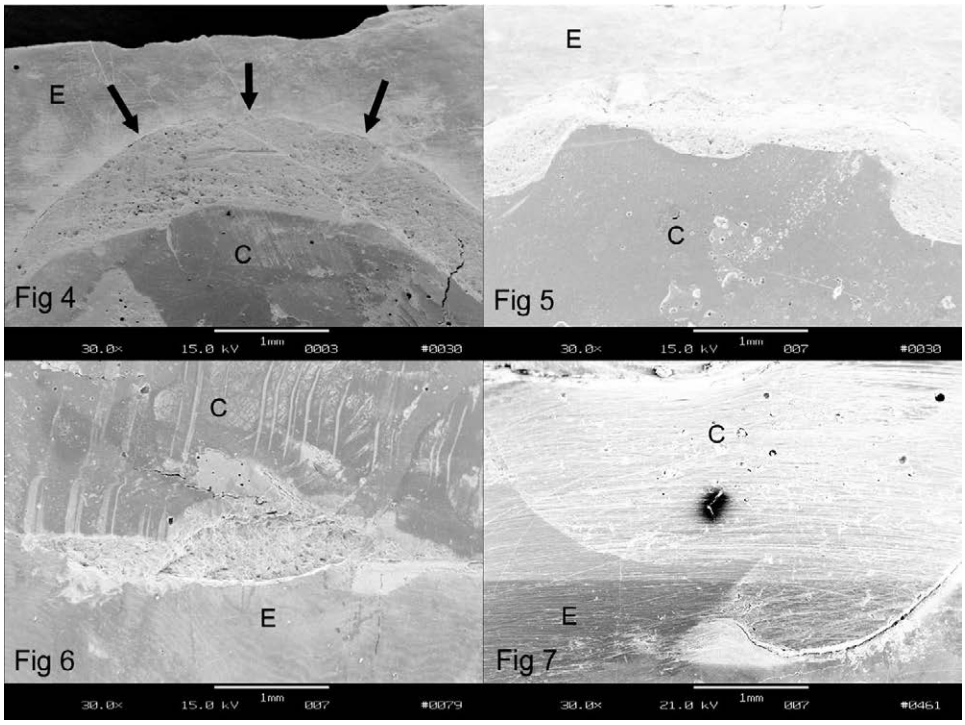

Figure 4. *SEM picture of margin quality (QUAL) graded as "perfect" after finishing with a microscalpel (E, enamel; C, composite resin; arrows, restoration margin).*
Figure 5. *SEM picture of margin quality (QUAN) graded as "excess" after finishing with scalpel number 21 (E, enamel; C, composite resin).*
Figure 6. *SEM picture of margin quality (QUAN) graded as "deficit" after finishing with a scaler (E, enamel; C, composite resin).*
Figure 7. *SEM picture of margin quality (QUAN) graded as "combination" after finishing with a finishing strip (E, enamel; C, composite).*

restoration and enamel surface without deficit or excess (Figure 4), 2) excess: excess composite resin material in relation to enamel margin visible (Figure 5), 3) deficit: deficit in composite resin material in relation to enamel margin visible (Figure 6), and 4) combination: deficit and excess of composite resin material at different sites of the visible margin (Figure 7).

QUAN Analysis

The same specimens were then removed from the SEM holder and embedded in methyl methacrylate (Paladur, HeraeusKulzer, Hanau, Germany). Three mesio-distal tooth slices parallel to the tooth axis and perpendicular to the proximal restoration margin (thickness 1.0 mm) were obtained with a water-cooled microtome saw (1600, Leitz, Bensheim, Germany). Surfaces were ground with wet silicon-carbide abrasive paper of descending grit (to 4000 grit), fixed to the specimen holder, and sputter coated again with a 30-nm layer of gold (S150, Edwards). SEM analysis (1810D, Amray) was performed at 60× magnification. The distance between tangents to restoration and enamel surface was measured (Figure 8) (analySIS Program, Soft imaging System, Emsis, Münster, Germany). Three measurements on each of the 30 specimens were averaged, resulting in a total of 30 values per group.

Statistical Analysis

Data were documented in Excel (Microsoft Excel 2010) and analyzed using SPSS (IBM SPSS Statistics for Windows version 20). Metrical data were normally distributed. Explorative data analysis was performed by calculating means and standard deviations for each group. Groups were compared using Student *t*-test and analysis of variance together with *post hoc* tests (Scheffé, level of significance $p>0.05$). Nominal data (QUAL) are presented as relative frequencies per group. Quantitative marginal analysis (QUAN) yielded positive measured values ("excess") as well as negative measured values ("deficit"). Perfect marginal quality (Figure 9) would thus be represented by a measured value of zero. Any excess or deficit was rated inferior; therefore, positive and negative values were processed as absolute values in the sense of a functional amount.

RESULTS

LOAD and CUT Testing

LOAD varied between 22.22 (1.96) N (mean [standard deviation]) and 95.83 (4.96). CUT values were between 7.61 (1.49) and 36.18 (9.61). Comparison of LOAD and CUT revealed that LOAD was significantly higher than CUT (Table 2) at all angles. The difference between LOAD and CUT was highest at an angle of 15 degrees. LOAD steadily decreased from 15

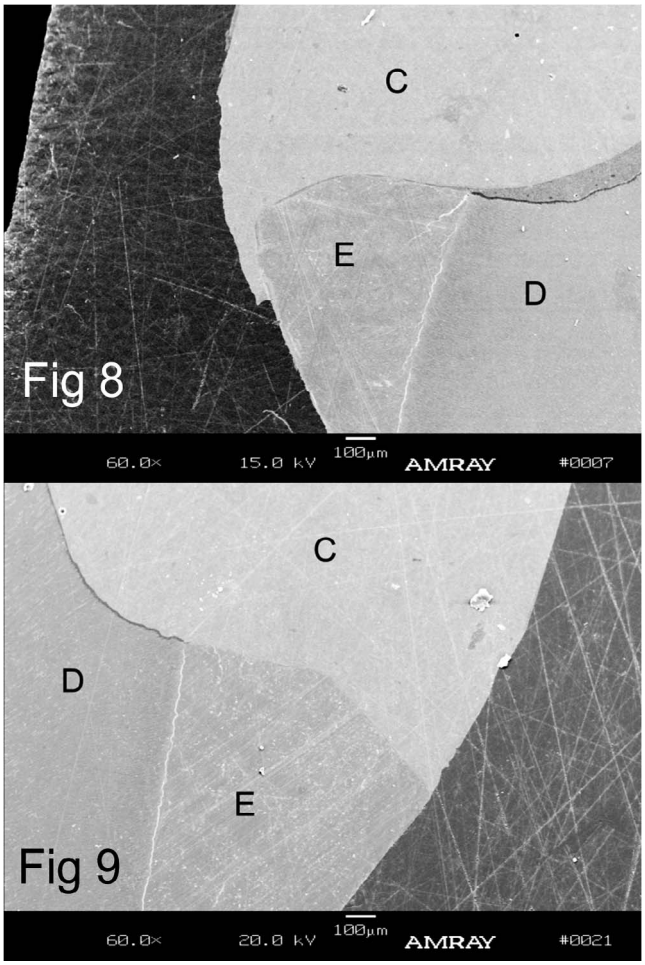

Figure 8. *SEM picture of residual overhang (QUAN) finished with scalpel number 21 (E, enamel; D, dentin; C, composite resin restoration).*
Figure 9. *SEM picture of perfect excess removal (QUAN) obtained with a microscalpel (E, enamel; D, dentin; C, composite resin restoration).*

degrees to more than 30 degrees and from 60 degrees to 75 degrees. CUT displayed a distinct outlying value at 60 degrees, where the forces for cutting were comparatively high. We assumed that this effect was seen due to the blade getting stuck in the resin and therefore not being able to cut freely any more.

QUAL Analysis

The greatest number of perfect margins was found with scalpel number 12 (relative frequency 36.7%), followed by the microscalpel and G5-ProShape (31.0% for both). Use of a scaler produced the lowest proportion of perfect margins (6.6%), and no perfect margins were seen with scalpel number 21 (0%). An excess of composite resin was most frequently seen with finishing strips (73.4%), whereas a scaler created the most deficits on margins (26.7%) (Figure 10).

QUAN Analysis

Absolute values for excess and deficit varied from 19.3 (4.4) µm (mean [standard deviation]) (microscalpel) to 116.0 (18.8) (finishing strips) (Table 3). Analysis of variance yielded significant differences between groups (*post hoc* Scheffé, $p=0.05$), showing that microscalpels, scalpel numbers 12 and 15, G5-ProShape, and G5-ProxoCare performed significantly better than finishing strips.

DISCUSSION

To our knowledge, this is the first study that presents data on a novel interproximal finishing device and that systematically compares eight different instruments in an experimental setup.

We evaluated the load strength of novel microscalpels in comparison to their cutting ability on standardized composite resin overhangs at different angles. The blades could be loaded to a significantly higher extent than that of the forces necessary for blade fracture. Our results revealed that the optimal working angle was 15 degrees.

To obtain access into the interproximal area, the idea was to scale down the size of scalpel blades,

Table 2. *Microscalpel Data for LOAD and CUT Testing at Different Angles, Mean (Standard Deviations in Parentheses), Minimum and Maximum Values, and p-Values (Two-Tailed Significance) from t-Tests (Paired)*

Angle (n=30, each)	LOAD (N)		CUT (N)		*p*-value
	Mean (SD)	Min/Max	Mean (SD)	Min/Max	*t*-Test, Two-Tailed Significance
15 degrees	95.83 (4.96)	87.00/104.00	7.61 (1.49)	5.66/12.60	<0.001
30 degrees	76.97 (9.59)	50.90/94.30	8.92 (2.82)	4.40/15.90	<0.001
60 degrees	57.86 (7.30)	34.60/69.80	36.18 (9.61)	18.50/48.90	<0.001
75 degrees	22.22 (1.96)	18.70/28.00	11.17 (4.01)	5.40/18.90	<0.001

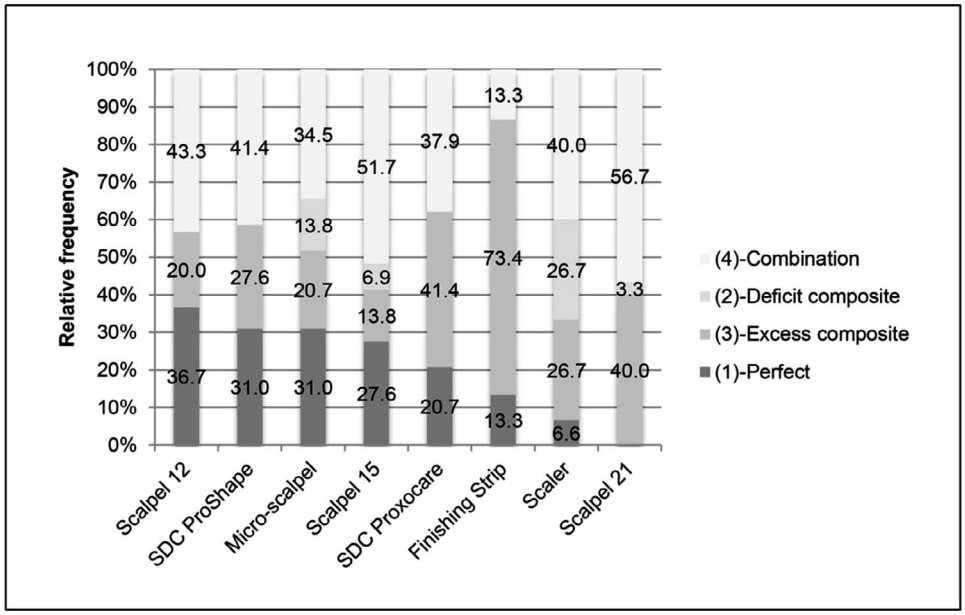

Figure 10. *Relative frequencies of margin qualities (QUAL) evaluated by SEM (n=30 per group).*

allowing for more maneuverability within the limited space. A more delicate blade, however, is prone to fracture more easily, especially when high forces are applied at unfavorable angles. However, in our tests the microscalpel proved to be robust enough to remove excess resin. It showed load-bearing values significantly higher than the forces necessary for fracture at all angles. Common dental scalpels are extremely robust and display high load strength. Pilot tests with scalpel blade numbers 12, 15, and 21 showed that their LOAD was beyond the testing range of our experimental setup, leading not to fractures of the blades but rather to destruction of the setup or to distortion of the holder (internal data). Therefore, a comparison of microscalpels with standard scalpels in terms of LOAD was not possible in our setup. Nevertheless, the data allow us to conclude that microscalpels are able to cut composite overhangs without the risk of overloading and/or fracture of the blades.

Table 3. *Absolute Values for Excess/Deficit (Mean and Standard Deviation [SD] in μm) Measured by SEM on Specimen Slices (n=30 Values per Group), Group Comparison With Analysis of Variance and* Post Hoc *Testing (Scheffé Procedure,* p=*0.05)*

Groups (n=30)	Mean (SD) (μm)	Min/Max (μm)
Microscalpel	19.3 (4.4)$_{A}$[a]	0.0/84.3
Scalpel 12	32.1 (7.6)$_{A}$	0.0/151.8
Scalpel 15	36.9 (11.6)$_{A}$	0.0/299.4
G5-ProShape	37.5 (10.2)$_{A}$	0.0/261.0
G5-Proxocare	53.6 (12.7)$_{A}$	0.0/307.0
Scalpel 21	60.3 (9.8)$_{AB}$	0.0/191.0
Scaler	60.5 (9.3)$_{AB}$	0.0/201.7
Finishing strip	116.0 (18.8)$_{B}$	0.0/412.3

[a] *Superscript letters designate subgroups with statistically significant differences.*

The rationale for the development of this microscalpel was the need for an optimized interproximal working device. Its shape should be such that both concave and convex surface anatomy can be worked on. Its size should be adequate to maneuver and work properly within the interdental space. In addition, a suitable instrument should work efficiently within a clinically acceptable amount of time. Concerning the time necessary for removal of excess material, Spinks and others[46] identified the fastest method as a motor-driven diamond tip. Three minutes were sufficient for complete excess removal. In comparison, in the same test, a sonic scaler needed seven minutes and a curette 15 minutes for the procedure. For this step of the restorative procedure, time periods longer than two to three minutes are clinically unsatisfactory since they entail undesirable delay for the treatment process. In this study, we chose two minutes as the time period to be investigated and tested different devices to assess how they would perform in removing excess within that time. Our results demonstrate that none of the tested instruments were able to create perfect margins in all specimens within the two-minute time period. The greatest proportion of perfect margins was produced by scalpel number 12 with a relative frequency of 36.7 percent. Scalpel number 21 was obviously too large to be able to reach into the

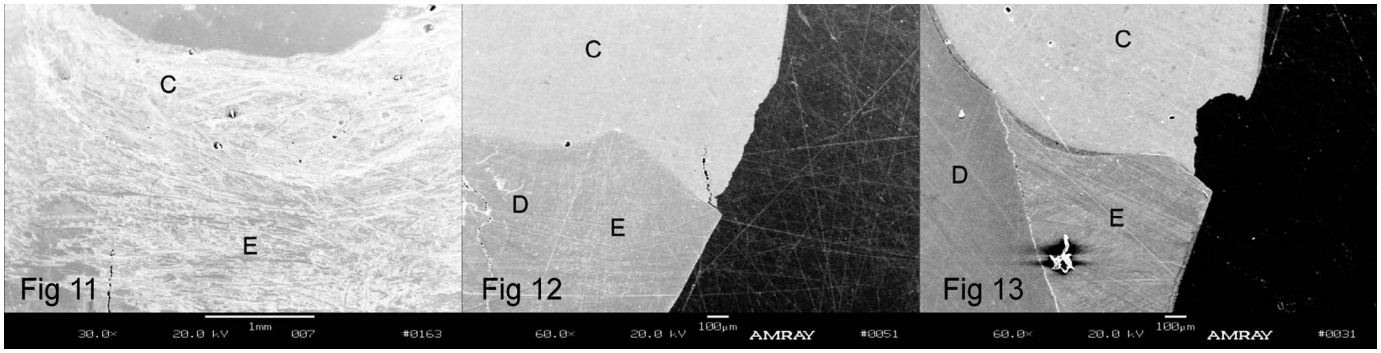

Figure 11. *SEM picture of distinct scratches and horizontal lines after use of G5-Proxocare (E: enamel; C, composite resin).*
Figure 12. *SEM picture of margin deficit and crack formation after scaler application (E, enamel; D, dentin; C, composite resin restoration).*
Figure 13. *SEM picture of large-scale marginal fracture after scaler application (E, enamel; D, dentin; C, composite resin restoration).*

interdental space and consequently failed to do the job. A strikingly high percentage of margins with excess were seen with finishing strips (73.4%, relative frequency). Even when coarse grit was used, they seem to be inadequate for the removal of distinct composite resin overhangs in the given time.

Since the goal of polishing and finishing should be to reconstruct the anatomically correct tooth shape, we graded both excess and deficit as "unfavorable" and transformed the negative and positive values into absolute values adding up to the final values of QUAN excess/deficit. The outcomes closest to the anatomically correct tooth shape, resulting in the lowest QUAN values, were displayed by microscalpels and scalpel numbers 12 and 15. The oscillating devices produced QUAN values in the midrange. Our study used them with coarse grit (60 μm), making efficient excess removal possible. Examination of the SEM pictures, however, shows distinct scratches and horizontal lines after their use (Figure 11). Similarly, Whitehead and others[47] saw enamel surface destruction after finishing with diamond finishing strips. They concluded that such roughness would be difficult to polish and promote the development of calculus, staining, and periodontal or cariological problems. Using scalpels is similar or more efficient, yet the blades seem to work in a much more material-selective manner than the oscillating devices, which strip enamel surfaces just as efficiently as restorative surfaces.

The efficient and material-selective cutting of composite resin makes scalpels preferable for excess removal in interdental spaces. However, another crucial aspect needs to be taken into consideration. Common scalpel holders are not angled like ergonomic scalers or curettes. This makes their use in the farthest posterior interdental spaces impossible. Depending on the mouth opening of the patient and the anatomical circumstances of teeth and tooth positions, the mesial aspect of the first molar is the farthest surface that can be reached. For interdental spaces posterior to those, the development of angled scalpel holders is necessary. For the moment, it seems likely that an ergonomically superior scaler would be used for this task. We saw, however, that their use resulted in high levels of composite resin deficits. Visual inspection of SEM images showed that frequent crack formation (Figure 12) and rough disruptions (Figure 13) in the marginal area were seen only in this group. Sharp scalers seem not to cut composite resin but rather to tear out pieces of composite, leaving rather disrupted surfaces. Clinical experience supports this idea. The scaler hooks onto the excess material, and considerable force is necessary to remove it. Large fragments are torn from the restoration, leaving rough breakout areas, and even further polishing might not be capable of smoothing these defects. Marginal breakdown, leakage, or secondary caries are possible long-term risks as a result. In this article, for the first time, the effects of the use of scalers to remove composite resin overhangs is demonstrated with examples of marginal breakouts and crack formation revealed by SEM.

As already mentioned in the introduction, research on this topic is rare, and comparable studies are not available. There are only a few publications that hint at the superiority of scalpel application in restorative dentistry. First, Pratten and others[48] analyzed surface qualities in anterior and posterior composite resin restorations and recommended the use of scalpel number 15 for proximal finishing since it yielded surface characteristics similar to those of carbide burs. They described that the scalpel blade can cleave the resin in a manner

similar to the cutting of a rotating carbide bur. Second, Anami and others[36] reported that after adhesive cementation of ceramic blocks, the surface roughness and biofilm adhesion were least with the brush technique and subsequent polishing and after scalpel number 12 application.

In our study, the adequate sample size and the reproducible setup allowed reliable data collection. In the clinical setting, tooth anatomy, limited interdental accessibility, and the possibility of adjacent structures blocking instrument access might complicate the procedure, resulting in possible variations in values. Yet within the limitations of the study, we can conclude the following:

1) Novel microscalpels can be used for cutting composite resin without the risk of blade fracture.
2) Small and/or curved scalpels were superior to the other devices tested with regard to removing excess composite material in the presented setup.

CONCLUSION

There is a clinical need for the development and testing of interproximal finishing instruments. Microscalpels and/or standard curved scalpels provide good accessibility into the interproximal area and allow for material-selective removal of excess material. Oscillating finishing devices are universally and efficiently applicable, yet coarse-grit instruments carry the risk of enamel removal, creating a rough surface texture. A scaler can cause distinct large-scale fractures or crack formation in the marginal area, while plastic flexible finishing strips are not sufficient as exclusive finishing instruments.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Ethikkommission der Medizinischen Fakultät Heidelberg. The approval code for this study is S-034/2010.

Conflict of Interest and Sources of Funding Statement

The authors declare that they have no financial or other relationships that might lead to actual or potential conflict of interest. This study was self-funded by the authors and their institutions in its major parts. SDC Switzerland SA (Bioggio, Switzerland) provided G5-ProShape and G5-Proxocare, and Trinon Titanium GmbH (Karlsruhe, Germany) provided microscalpel blades for this investigation.

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Enamel Mineral Content Changes After Bleaching With High and Low Hydrogen Peroxide Concentrations: Colorimetric Spectrophotometry and Total Reflection X-ray Fluorescence Analyses

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Clinical Relevance

Hydrogen peroxide concentrations and the composition and application protocol of each bleaching agent can influence calcium and phosphorous content in enamel, with the results varying according to the methodology used.

SUMMARY

The purpose of this study was to evaluate the calcium (Ca) and phosphorous (P) content in enamel bleached with high and low concentrations of hydrogen peroxide (HP) using Total Reflection X-Ray Fluorescence (TXRF) and colorimetric spectrophotometry (SPEC). Forty-eight sound human third molars were used.

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Their roots were embedded in polystyrene resin and immersed for seven days in an artificial saliva solution. Then they were distributed into six groups to receive the bleaching treatments. The agents of high HP concentration (for in-office use) evaluated were Whiteness HP Maxx/FGM (35% HP), Whiteness HP Blue/FGM (35% HP, 2% calcium gluconate), Pola Office+/SDI (37.5% HP, 5% potassium nitrate), and Opalescence Boost/Ultradent (38% HP, 1.1% ion fluoride, 3% potassium nitrate); these agents were applied to enamel in three sessions. The agents of low HP concentration (for home use) evaluated were Pola Day/SDI (9.5% HP) and White Class 10%/FGM (10% HP, potassium nitrate, calcium, fluoride), and these agents were applied for 14 days. Enamel microbiopsies were evaluated by TXRF and SPEC analysis before the bleaching treatment (baseline), during the treatment, and 14 days after the end of the treatment. For TXRF, the Kruskal-Wallis test showed that Ca and P were not influenced by agent ($p>0.05$). For SPEC, Pola Office+, Opalescence Boost, Pola Day, and White Class 10% caused a decrease of Ca over time; there was a significant decrease of P over time to Pola Office+ and White Class 10%. The Spearman test showed no correlation between the Ca ($p=0.987$; $r^2=-0.020$) and P ($p=0.728$, $r^2=0.038$) obtained by SPEC and TXRF. For TXRF and SPEC, changes in Ca and P during bleaching occurred independently of the HP concentration used.

INTRODUCTION

Hydrogen peroxide (HP) is the most widely used agent for tooth bleaching, and it can be used either at high concentrations (35% to 40%) using in-office techniques or at low concentrations (5% to 10%) for at-home bleaching.[1,2] Despite the application of faster bleaching protocols, which reduce the contact time between bleaching agent and tooth structure, changes in enamel and dentin micromorphology may occur, such as the presence of erosions and/or porosity[3,4] and changes in mineral content[5-8] as well as in surface microhardness,[3,6,9-12] which may be related to the composition of bleaching agents, concentration, pH values, and different application techniques.

Considering the increased risk of enamel demineralization or loss of mineral content due to teeth bleaching, the addition of calcium (Ca) and/or fluoride to the composition of bleaching gels could be beneficial.[4,9,11,13-19] Borges and others[17] observed that adding 2% calcium gluconate to a hydrogen peroxide gel resulted in reduced enamel erosion, with a significant increase in enamel microhardness after bleaching with hydrogen peroxide at 35% enriched with calcium and fluoride.[18]

However, Oliveira and others[20] found no significant increase in enamel microhardness when using a bleaching agent at low concentration (10% carbamide peroxide) enriched with calcium fluoride for home use. Soares da Costa and others[16] showed that high-concentration chairside bleaching treatments with or without calcium decreased enamel microhardness. Basting and others[7] measured calcium and phosphate content via enamel microbiopsy and spectrophotometry,[5,21] verifying that the mineral content within the human dental enamel structure following high-concentration bleaching was generally similar whether or not calcium was featured in its composition. Consequently, questions remain regarding the advantages of incorporating calcium and fluoride in the composition of high- or low-concentration bleaching agents as a result of the contradictory results observed in different studies.

Various methods of analysis can be applied to evaluate changes in dental substrate in terms of mineral content, such as Fourier Transform infrared spectroscopy,[8] energy-dispersive X-ray spectroscopy,[6,11] Fourier Transform Raman spectroscopy,[22-24] atomic absorption spectroscopy,[13,25] surface microhardness,[9,12,22,26,27] induced plasma mass spectrometry,[14] total reflection X-Ray fluorescence,[25] and spectrophotometric or colorimetric analysis.[5,7] Total reflection X-ray fluorescence analysis (TXRF), which causes exposure of ions through the phenomenon of fluorescence,[25] allows the study of the atomic and molecular structure of matter, identifying and quantifying chemical elements, especially in small volumes.[28] This could be advantageous when using the enamel microbiopsy technique,[5,7] especially in clinical studies that aim to preserve the dental structure when evaluating changes in mineral content.[5] By using the technique of enamel microbiopsy, the method of colorimetric spectrophotometry (SPEC) may also be useful to quantify the minerals extracted from the dental substrate;[5,7] however, there are no studies comparing these methods.

Therefore, the aim of this study was to evaluate the Ca and phosphate (P) contents in human enamel before, during, and after treatment with bleaching

agents containing either high- or low-concentration hydrogen peroxide with or without calcium and/or fluoride. Mineral content was evaluated by both SPEC and TXRF, and the two methods were also verified for correlation.

METHODS AND MATERIALS

Experimental Design

The samples were comprised of aqueous solutions of mineral obtained from the enamel surface via microbiopsy from 48 human third molars distributed into the following study factors (n=8):

- Bleaching agents containing hydrogen peroxide with or without calcium and/or fluoride on six levels. Four levels of high-concentration bleaching agents: Whiteness HP Maxx/FGM (35% hydrogen peroxide); Whiteness Blue HP/FGM (35% hydrogen peroxide + 2% calcium gluconate); Pola Office+/SDI (37.5% hydrogen peroxide + 5% potassium nitrate); Opalescence Boost/Ultradent (38% hydrogen peroxide + 1.1% fluoride and 3% potassium nitrate)—and two levels of low-concentration bleaching agents: Pola Day/SDI (9.5% hydrogen peroxide, + 5% fluoride); White Class 10%/FGM (10% hydrogen peroxide + potassium nitrate + calcium + fluoride);
- Treatment times on six levels: T0 = control: before applying the bleaching agent (baseline); T1 = immediately after the first application of the bleaching agent; T2 = seven days after starting treatment; T3 = after the second application of high-concentration hydrogen peroxide or after the eighth application of low-concentration hydrogen peroxide; T4 = after 14 days of starting treatment; T5 = after the third application of high-concentration hydrogen peroxide or after the 15th application of low-concentration hydrogen; and T6 = 15 days after the completion of treatment.

Ca and P values were obtained from the enamel microbiopsy samples in micrograms per milliliter (μg/mL) using SPEC and TXRF. For TXRF, only T0, T1, T3, T5, and T6 were evaluated, with n = 3.

Tooth Preparation and Dental Bleaching

Forty-eight unerupted freshly extracted human third molars were cleaned using a rotary brush, pumice, periodontal curettes, and scalpel blades and were stored in 0.1% thymol solution for up to six months. The teeth showed no evidence of cracks, wear, or fractures, and the coronal enamel was free from spots.

Polyvinyl chloride rings measuring 20 mm in diameter and 20 mm in height were used to embed the teeth in acrylic resin (Maxi Rubber, Diadema, SP, Brazil). The roots of the teeth were immersed into the resin 2 mm apically to the cementoenamel junction, maintaining the long axis of the tooth perpendicular to the horizontal plane.

The teeth were randomly divided into six groups (n=8) according to high or low hydrogen peroxide concentration (Table 1). Prior to bleaching, the teeth were stored at 37°C ± 1°C in a bacteriological incubator (Odontobrás Ind E Com Equip Med Odont Ltda, Ribeirão Preto, SP, Brazil) immersed in 20 mL of artificial saliva solution for seven days before the application of bleaching agents. The artificial saliva solution used was the same reported in previous studies.[7,26,29]

The teeth were removed from the artificial saliva and dried with jets of air for five seconds to perform the initial microbiopsy T0 (baseline). The bleaching agents were then applied following the instructions and application times recommended by the manufacturers (Table 1). The pH value of the bleaching agents was measured using a benchtop pH meter (MS Tecnopon Special Equipment Ltd, Piracicaba, SP, Brazil) at different times (baseline and after seven, 15, 30, and 40 minutes), coinciding with half of the time and total application time for each bleaching agent. The bleaching agent was dispensed directly from the syringe or after mixing the freshly prepared gel.

Immediately upon completion of the first bleaching application an enamel microbiopsy was taken (T1). Teeth receiving the high-concentration hydrogen peroxide treatment were stored separately in bottles containing 20 mL of artificial saliva for seven days, changing the solution every two days. The bleaching treatment was repeated twice for a total of 15 days (three clinical sessions, each session intercalated by a seven-day immersion in artificial saliva). Enamel microbiopsies from these groups were taken at the times established in the experimental design. The teeth receiving low-concentration hydrogen peroxide were also stored separately in bottles containing 20 mL of artificial saliva, changing the solution every two days. The bleaching agents were applied daily for a total of 15 days. The teeth were removed from the storage solution and dried prior to daily application of the bleaching treatment and enamel biopsies at certain times, as established in the experimental design.

Table 1: *Treatment Agents, Composition, Protocol of Use, and pH Values*

Hydrogen Peroxide	Commercial Brand/ Manufacturer (City, State, Country) Lot No.	Composition (According to the Manufacturer)	Time Application in Each Session/ Total No. of Sessions or Total Days of Treatment	pH Values				
				Baseline	7 min	15 min	30 min	40 min
High concentrations	Whiteness HP Maxx/FGM (Blumenau, PR, Brazil) P72199	35% Hydrogen peroxide, thickener, dyes, glycol, inorganic filler, deionized water	15 min × 3 = 45 min/3 sessions	5.70	5.54	5.40	—	—
	Whiteness HP Blue Calcium/FGM (Blumenau, PR, Brazil) 150514	35% Hydrogen peroxide, thickener, violet dye, neutralizing agents, 2% calcium gluconate, glycol, deionized water	40 min/3 sessions	8.0	—	8.09	—	7.89
	Pola Office+/SDI (Melbourne, Victoria, Australia) 132020	37.5% Hydrogen peroxide, 5% potassium nitrate	8 min × 4 = 32 min/3 sessions	7.41	8.21	7.27	—	—
	Opalescence Boost/ Ultradent Products (South Jordan, Utah, USA) DOO6A	38% Hydrogen peroxide, 1.1% fluoride, 3% potassium nitrate	15 min × 3 = 45 min/3 sessions	6.98	7.01	6.74	—	—
Low concentrations	Pola Day 9.5%/SDI (Melbourne, Victoria, Australia) P130308Z	9.5% Hydrogen peroxide, <47% additives, 30% glycerol, 20% water, 0.1% flavoring, potassium nitrate	30 min/15 d	6.07	—	6.07	6.13	—
	White Class 10%/ FGM (Blumenau, PR, Brazil) 140114	10% Hydrogen peroxide, neutralized carbopol, 5% potassium nitrate, sodium fluoride, aloe vera, calcium gluconate (concentration not available), stabilizing, moisturer, deionized water	30 min/15 d	5.49	—	5.47	6.00	—

Enamel Microbiopsy Technique

Enamel microbiopsy was performed according to the method described by Amaral and others[5] and Basting and others.[7] The tooth surfaces used for biopsy were buccal, lingual, and proximal for each tooth, which were rinsed with water for five seconds and air-dried for five seconds. Each biopsy was taken from a different surface every time. The solutions containing the microbiopsies were frozen at −18°C (CRM 32ABANA, Consul Biplex Frost Free 320, Multibrás SA Appliances, Joinville, SC, Brazil). When carrying out chemical analyses, the samples were thawed at room temperature.

Chemical Analyses Via Spectrophotometry

The concentrations of Ca and P were established following the method previously published by Amaral and others[5] and Basting and others.[7] Ca concentration was determined via an endpoint colorimetric Arsenazo III method, in which calcium reacts with Arsenazo III in acidic medium to form a blue color complex, in which intensity is proportional to calcium concentration in the sample, using the calcium-Arsenazo III kit (K051, Bioclin, Belo Horizonte, MG, Brazil). P concentrations were determined by the principle that phosphorus from mineral phosphates is transformed into phosphomolybdate, which is then

reduced by alpha-amino-naphthol-sulfonic acid to a blue reaction product, the color intensity of which is proportional to the inorganic phosphorus content in the sample. The quantification of Ca and P concentrations was performed in duplicate. Wavelength absorbance was evaluated in a Universal Microplate Reader (ELX800UV, Bio-Tek Instruments Inc, Winooski, Vt, USA) at 630 nm and concentrations were obtained in μg/mL.

Chemical Analyses Via TXRF

The mineral solutions obtained from the microbiopsy were taken to the Brazilian Synchrotron Light Laboratory for analysis. As a result of the large number of samples obtained as well as the time needed for each analysis, a decision was made to include n = 3 for T0, T1, T3, T5, and T6 in the benefit of optimization and rational use of the equipment. The three samples from each group were randomly selected for evaluation.

A standard Scandium solution (20 ppm) was added to each sample to serve as a known reference for comparison for each sample. Five microliters of each solution was applied to the acrylic reflector of the equipment in triplicate. The reflector was then repositioned into its specific compartment in the Total Reflection X-Ray Fluorescence equipment so that the analysis using Synchrotron Radiation could be performed. The concentrations of Ca and P were quantified in μg/mL.

Statistical Analysis

The concentrations of Ca and P obtained by SPEC were analyzed using the mixed model method (PROC MIXED) via repeated-measures analysis of variance (ANOVA) and Tukey-Kramer test (α=0.05) (SAS Institute Inc, Cary, NC, USA; Release 9.3, 2011). For Ca and P concentrations obtained by TXRF, neither data homoscedasticity nor normality were obtained, even after data transformation. Nonparametric methods were therefore used: Kruskal-Wallis, Friedman, and Wilcoxon tests with Bonferroni correction. The Spearman correlation test was used to verify the correlation between the concentrations of Ca and P obtained via the SPEC and TXRF methods (α=0.05) at the matched evaluation times (T0, T1, T3, T5, and T6) (SPSS 20, SPSS Inc, Chicago, Ill, USA).

RESULTS

The data obtained by SPEC and analyzed by two-way ANOVA applied to Ca concentrations showed an interaction between the factors 'hydrogen peroxide with or without calcium and/or fluoride' and 'treatment times' (p=0.0001). Ca concentrations were not significantly influenced by the type of bleaching agent used (p=0.2822) but were influenced by time (p<0.001). The Tukey-Kramer test showed that at any time point Ca concentrations between the different bleaching agents did not differ (Table 2). For the bleaching agents Pola Office+ Opalescence Boost, Pola Day, and White Class, a significant reduction in calcium concentration was observed over time, with values at T6 significantly lower than those at T0. For Whiteness HP Maxx and Whiteness HP Blue and Calcium no significant difference in calcium concentration was observed at different time points. Regarding the concentration of P, two-way ANOVA showed no interaction between the factors 'hydrogen peroxide with or without calcium and/or fluoride' and 'treatment times' (p=0.0691). P concentrations were not significantly influenced by the type of bleaching agent used (p=0.0833), but they were affected by time (p<0.0001). The Tukey-Kramer test showed that at any time point, the concentration of P between different bleaching agents did not differ (Table 2). For the Pola Office+ agent, a significant reduction in the concentration of P was observed over time, with values at T2, T3, T5, and T6 being lower than values at T0. Regarding the White Class agent, a significant reduction in the concentration of P was observed at T4 when compared to T0. As for the remaining bleaching agents, no significant difference was observed over time.

Regarding the results obtained by TXRF, the Kruskal-Wallis test showed that at each time point Ca concentration was not significantly affected by the bleaching agent used (p>0.05) (Table 3). Only for the bleaching agent Pola Office+ did the Friedman test show that time significantly influenced the concentration of Ca (p=0.027), with no difference between T5 and T6, whereas these concentrations were lower than the concentrations at T1 and T3. At T0, the concentration of Ca was intermediate and did not differ over time (Wilcoxon multiple comparisons test with Bonferroni correction). For the remaining bleaching agents, time did not significantly influence the concentration of P (Friedman test, p>0.05). The Kruskal-Wallis test showed that P concentration was not significantly affected by the bleaching agent used at any time point, (p>0.05). Only for the bleaching agent Pola Day did the Friedman test show that time significantly influenced the concentration of P (p=0.043), with greater concentrations

Table 2: *Calcium and Phosphorous Concentration Means (±Standard Deviation Values) by Colorimetric Spectrophotometry (SPEC) According to Groups and Time (μg/mL)*[a]

Time	High Hydrogen Peroxide Concentration				Low Hydrogen Peroxide Concentration	
	Whiteness HP Maxx	Whiteness HP Blue Calcium	Pola Office+	Opalescence Boost	Pola Day	White Class
Calcium						
T0	10.34 ± 0.86 Aa	10.67 ± 0.89 Aa	11.10 ± 0.80 Aa	10.85 ± 1.03 Aa	10.91 ± 0.99 Aa	11.33 ± 0.99 Aa
T1	8.60 ± 0.64 Aa	8.98 ± 1.46 Aa	9.08 ± 0.80 Aab	8.46 ± 0.51 Ab	9.34 ± 0.69 Aabc	10.29 ± 1.92 Aab
T2	9.84 ± 0.73 Aa	9.81 ± 0.78 Aa	9.32 ± 0.87 Aab	9.15 ± 0.80 Aab	9.33 ± 1.06 Aabc	8.45 ± 0.98 Abcd
T3	9.76 ± 1.58 Aa	9.70 ± 1.45 Aa	9.51 ± 1.56 Aab	9.26 ± 1.66 Aab	7.83 ± 1.58 Ac	7.58 ± 0.60 Acd
T4	9.23 ± 1.41 Aa	10.16 ± 1.52 Aa	9.08 ± 1.06 Aab	10.12 ± 0.99 Aab	8.53 ± 0.73 Abc	8.61 ± 1.12 Abcd
T5	9.67 ± 0.48 Aa	9.04 ± 0.71 Aa	8.82 ± 0.48 Ab	9.29 ± 0.64 Aab	10.04 ± 1.43 Aab	9.36 ± 1.04 Abc
T6	9.37 ± 1.36 Aa	9.60 ± 1.16 Aa	8.99 ± 1.24 Ab	8.89 ± 1.45 Ab	8.00 ± 1.91 Ac	7.38 ± 0.40 Ad
Phosphorous						
T0	3.90 ± 0.94 Aa	3.55 ± 1.21 Aa	5.50 ± 1.98 Aa	3.52 ± 1.33 Aa	4.12 ± 1.30 Aa	4.52 ± 1.67 Aa
T1	3.13 ± 0.78 Aa	4.18 ± 0.91 Aa	4.68 ± 1.91 Aab	3.91 ± 0.85 Aa	4.32 ± 1.92 Aa	4.27 ± 1.75 Aab
T2	4.05 ± 1.54 Aa	2.86 ± 0.67 Aa	3.14 ± 0.93 Ab	2.71 ± 0.71 Aa	2.42 ± 0.71 Aa	2.94 ± 0.70 Aab
T3	2.66 ± 0.42 Aa	2.90 ± 0.66 Aa	2.85 ± 0.77 Ab	2.14 ± 0.41 Aa	2.80 ± 1.00 Aa	2.68 ± 0.99 Aab
T4	2.47 ± 0.76 Aa	2.84 ± 0.85 Aa	3.26 ± 0.57 Aab	2.42 ± 0.76 Aa	3.09 ± 1.42 Aa	2.38 ± 0.53 Ab
T5	3.15 ± 0.70 Aa	3.26 ± 0.70 Aa	2.82 ± 0.52 Ab	3.49 ± 1.11 Aa	2.68 ± 0.71 Aa	3.19 ± 1.20 Aab
T6	2.61 ± 0.87 Aa	2.59 ± 0.45 Aa	2.92 ± 0.74 Ab	3.10 ± 0.76 Aa	2.92 ± 0.99 Aa	2.66 ± 0.50 Aab

Abbreviations: T0, control: before applying the bleaching agent (baseline); T1, immediately after the first application of the bleaching agent; T2, seven days after starting treatment; T3, after the second application of high-concentration hydrogen peroxide or after the eighth application of low-concentration hydrogen peroxide; T4, after 14 days of starting treatment; T5, after the third application of high-concentration hydrogen peroxide or after the 15th application of low-concentration hydrogen; T6, 15 days after the completion of treatment.

[a] *Means followed by the same letter (capital letters in rows and lowercase letters in column) do not differ from each other ($p \leq 0.05$) for calcium (Ca) and phosphate (P) independently.*

at T1 when compared to other times, during which no significant differences in concentrations were observed (Wilcoxon multiple comparisons test with Bonferroni correction). For the remaining bleaching agents, time did not significantly influence the concentration of P (Friedman test, $p>0.05$) (Table 3). Figure 1 illustrates the comparisons of the mean Ca and P concentrations (μg/mL) obtained by SPEC and TXRF over time and study group.

The Spearman test revealed no correlation between the concentrations of Ca ($p=0.987$; $r^2=-0.020$) and P ($p=0728$; $r^2=0.038$) obtained by the SPEC and TXRF methods (Figure 2).

DISCUSSION

The results for the concentration of Ca and P by TRFX showed no differences among bleaching agents at any time point. Although no other studies have quantitated the mineral content of the enamel throughout the bleaching procedure using the same agents as those in the present study, thus preventing some comparisons, it is important to note that the results obtained using SPEC did not correlate with those obtained from TXRF via the Spearman test.

The TXRF method was used by Wang and others[25] when analyzing the amount of calcium leached from the enamel after treatment with a sodium chloride–based bleaching agent (Rapid White/Rapid White Products) and a hydrogen peroxide–based high-concentration agent at 38% (Opalescence Xtra Boost/Ultradent Products Inc). These authors reported that for the hydrogen peroxide–based agent, the presence of potassium in the solutions collected (by applying distilled water) decreased the detection limit of the method, in which the presence of calcium could only be evaluated via atomic absorption spectroscopy. Consequently, TXRF, though accurate, could yield results biased by contaminants in the solutions,[25] since there is a risk of bleaching gel residue on the enamel surface, which may have affected the detection of other elements. Despite the fact that calcium belongs to the group of alkaline earth metals in the periodic table, it can react with other elements when in contact with water, forming hydroxides,[30] thereby affecting the detection accuracy of this chemical element in the solution used. On the other hand, phosphorus is not a metal and

Table 3: *Calcium and Phosphorous Concentration Means (±Standard Deviation Values) by X-ray Fluorescence by Total Reflection (TRXF) According to Groups and Time (μg/mL)[a] (Ext.)*

Time	High Hydrogen Peroxide Concentration			
	Whiteness HP Maxx	Whiteness HP Blue Calcium	Pola Office+	Opalescence Boost
Calcium				
T0	2.26E-04 ± 2.87E-04 Aa	6.79E-05 ± 2.68E-05 Aa	1.90E-04 ± 2.21E-04 Aab	5.57E-05 ± 4.33E-05 Aa
T1	5.48E-05 ± 2.22E-05 Aa	1.30E-04 ± 1.47E-04 Aa	1.68E-03 ± 1.87E-03 Aa	7.02E-05 ± 5.90E-05 Aa
T3	5.47E-05 ± 1.16E-05 Aa	3.16E-03 ± 3.28E-03 Aa	5.46E-03 ± 9.28E-03 Aa	6.86E-05 ± 3.40E-05 Aa
T5	2.08E-04 ± 2.50E-04 Aa	5.57E-05 ± 5.34E-05 Aa	5.14E-05 ± 7.76E-06 Ab	6.23E-05 ± 6.34E-06 Aa
T6	7.87E-05 ± 4.84E-05 Aa	4.84E-05 ± 1.06E-05 Aa	4.44E-05 ± 2.15E-05 Ab	3.22E-03 ± 5.41E-03 Aa
Phosphorous				
T0	8.03E-05 ± 1.24E-04 Aa	4.47E-05 ± 5.92E-05 Aa	1.16E-04 ± 1.80E-04 Aa	1.70E-05 ± 1.45E-05 Aa
T1	1.48E-05 ± 8.66E-06 Aa	8.02E-04 ± 1.36E-03 Aa	1.24E-03 ± 1.37E-03 Aa	3.33E-04 ± 5.61E-04 Aa
T3	2.11E-05 ± 9.20E-06 Aa	6.54E-04 ± 4.07E-04 Aa	1.50E-03 ± 2.57E-03 Aa	1.08E-05 ± 5.52E-06 Aa
T5	8.92E-05 ± 1.34E-04 Aa	5.43E-05 ± 6.08E-05 Aa	8.20E-05 ± 1.23E-04 Aa	1.03E-05 ± 2.61E-06 Aa
T6	1.31E-05 ± 3.78E-06 Aa	1.29E-05 ± 6.38E-06 Aa	3.95E-05 ± 4.64E-05 Aa	1.11E-03 ± 1.88E-03 Aa

Abbreviations: T0, control: before applying the bleaching agent (baseline); T1, immediately after the first application of the bleaching agent; T2, seven days after starting treatment; T3, after the second application of high-concentration hydrogen peroxide or after the eighth application of low-concentration hydrogen peroxide; T4, after 14 days of starting treatment; T5, after the third application of high-concentration hydrogen peroxide or after the 15th application of low-concentration hydrogen; T6, 15 days after the completion of treatment.

[a] Means followed by the same letter (capital letters in rows and lowercase letters in column) do not differ from each other ($p \leq 0.05$) for calcium (Ca) and phosphorous (P) independently. Scientific notation: 1.0E-03" = 1.0×10^{-3}.

is part of the nitrogen group, which is highly reactive and highly oxidative when in contact with oxygen from the air. Additionally, P is an element with low electrical conductivity,[31] and these characteristics may have also influenced the results. In this respect, the standard deviations were high for all groups, illustrating that the coefficient of variation reflected high data dispersion and heterogeneity. Although the high variation of TXRF results would justify the unnecessary performance of the correlation analysis, the lack of a correlation emphasizes that one of the methods is preferable to the other.

When the SPEC method was used, the negative control group was established as the baseline data for each group, when no bleaching agent was applied. An interesting and important result can be observed for the Ca and P baseline values that show no differences among the groups (represented by the rows of Table 2). This result (no differences at the baseline time point) allowed comparisons among groups (represented by the columns of the Table 2) according to each treatment time. No difference was found in Ca and P concentrations at any time point for the different bleaching agents. Basting and others[7] assessed the Whiteness HP Maxx and the Whiteness HP Blue agents and also found similar results for the times evaluated in the present study using the same methodology.

From the high-concentration hydrogen peroxide–based bleaching agents for use in office, the Pola Office+ was shown to reduce the concentration of Ca and P over time. The Pola Office+ agent has slightly alkaline pH values, which would not be responsible for the change in mineral content. For this reason, one would expect the Whiteness HP Maxx agent, due to its acidic pH values (5.4 after 15 minutes of mixing, which is a value close to the critical pH for enamel demineralization), to have caused greater changes in mineral content,[9] which was not the case.

Hydrogen peroxide–based bleaching agents feature an acidic pH and are therefore recognizably superior in terms of product stability, which is important in terms of the degradation reactions of the bleaching agent. However, despite the risk of enamel erosion due to the low pH of the bleaching agents, Xu and others[24] and Sa and others[27] reported that such a characteristic has not been implicated in significant changes in enamel composition. Araujo and others[32] also observed that the acidic pH of hydrogen peroxide bleaching agents was not the cause of changes to enamel microhardness. This finding could be attributed to the composition of bleaching agents, which includes no calcium and/or fluoride, unlike other bleaching products. According to the manufacturer, the Pola Office+ agent only has potassium nitrate added to its composition, which is used to reduce the dentinal sensitivity caused by tooth bleaching.[33] Potassium nitrate would not,

Table 3: Extended.

Time	Low Hydrogen Peroxide Concentration	
	Pola Day	White Class
Calcium		
T0	4.79E-05 ± 8.58E-06 Aa	6.67E-05 ± 4.81E-05 Aa
T1	5.25E-05 ± 4.16E-05 Aa	6.27E-05 ± 1.45E-05 Aa
T3	5.21E-05 ± 3.44E-05 Aa	7.00E-05 ± 5.21E-05 Aa
T5	4.29E-05 ± 2.35E-05 Aa	8.27E-05 ± 6.27E-05 Aa
T6	1.06E-04 ± 1.25E-04 Aa	1.30E-03 ± 6.27E-05 Aa
Phosphorous		
T0	1.55E-05 ± 4.17E-06 Aa	9.64E-06 ± 6.44E-06 Aa
T1	7.36E-06 ± 7.96E-06 Ab	2.11E-04 ± 2.65E-04 Aa
T3	9.64E-05 ± 1.32E-04 Aa	5.80E-05 ± 4.46E-05 Aa
T5	1.60E-05 ± 3.96E-06 Aa	3.43E-05 ± 5.17E-06 Aa
T6	7.21E-05 ± 5.34E-05 Aa	3.71E-03 ± 2.11E-04 Aa

however, be expected to affect the mineral content of the enamel, unlike sodium fluoride, which is found in Opalescence Boost.

As a result of their mechanism of deposition onto the dental substrate,[33] sodium fluoride present in high-concentration hydrogen peroxide bleaching agents could contribute to the maintenance of the mineral content or even enamel remineralization.[9,18] Such an effect was not, however, observed for Opalescence Boost, which revealed a decrease in the concentration of Ca over the treatment period, even at pH values that were close to neutral. Phosphate concentrations were, however, similar for Opalescence Boost over time. A reduction in the concentration of Ca may be related to the mineral content of the bleaching agent, which is subsaturated in relation to the dental enamel, even when fluoride is added to its composition.

Only Whiteness HP Blue Calcium (containing calcium gluconate) and HP Maxx Whiteness (which displays neither calcium nor fluoride in its formulation) were among the high-concentration hydrogen peroxide agents that were able to maintain the concentrations of Ca and P in the enamel over time. In addition to containing neutralizing agents that could favor neutral pH values, Whiteness HP Blue Calcium features calcium in its composition in a saturated form, which would promote mineral deposition onto the enamel surface, or Ca incorporation into hydroxyapatite, preventing demineralization of the substrate, as shown by Borges and others[18] and Cavalli and others,[15] who evaluated agents containing calcium chloride, and Borges and others,[19] who

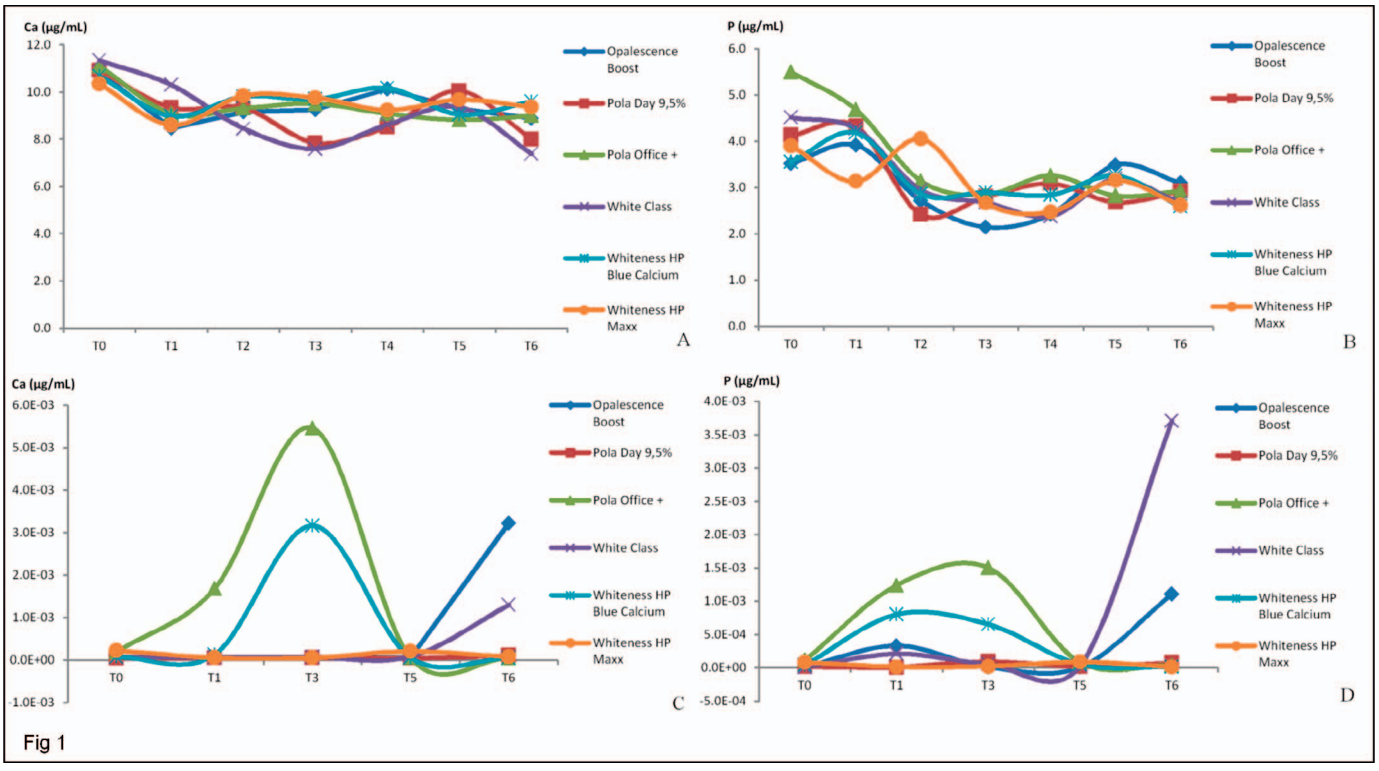

Figure 1. *Ca and P concentration means (in µg/mL) by colorimetric spectrophotometry (A and B) and total reflection X-ray fluorescence analyses (C and D) according to bleaching agents over time.*

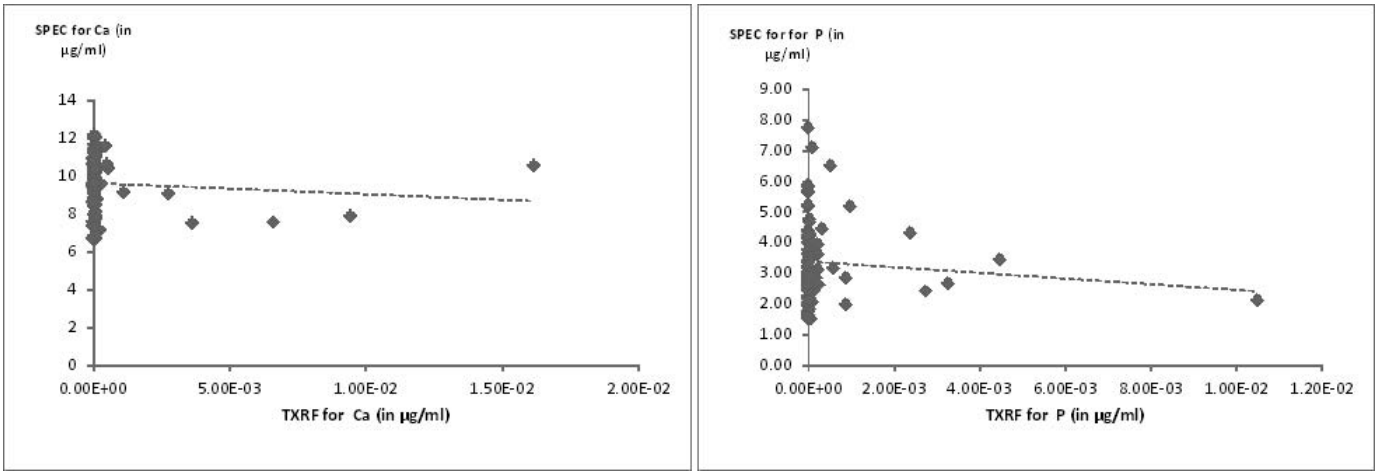

Figure 2. *Spearman correlation of Ca (A) and P (B) concentrations by colorimetric spectrophotometry and total reflection X-ray fluorescence analyses.*

used 2% calcium gluconate in 35% hydrogen peroxide. Calcium deposits on the enamel surface were observed via scanning electron microscopy when using Whiteness HP Blue Calcium,[16] which may reduce enamel susceptibility to erosion.[17] The beneficial effects of using bleaching agents containing calcium are still controversial, considering that no differences were observed regarding the enamel concentrations of Ca and P when using the Whiteness HP Maxx agent containing no calcium gluconate in its formulation. Since calcium gluconate is incompatible with strong oxidizing agents[34]—such as the free oxygen and perhydroxyl present in the byproducts released during the bleaching treatment[35]—the addition of this component to bleaching agents appears questionable, as observed by Basting and others.[7]

Regarding low-concentration hydrogen peroxide bleaching agents, higher concentrations of Ca[11,13,14] and P[11,14] have been observed in the enamel when compared to the high-concentration agents. The present study has shown, however, that both bleaching agents with low hydrogen peroxide concentration have caused a decrease in the concentrations of Ca. In terms of P, White Class has also caused a decrease in its concentration over time. Although both agents present acidic pH values, they feature remineralizing agents in their composition, which could avoid enamel demineralization. Regarding bleaching agents for home use, it is suggested[4,15] that the addition of calcium fluoride can minimize changes to the mineral content of the enamel. Fluoride is present in the composition of Pola Day, and calcium gluconate is part of the composition of White Class. One would therefore expect similar results with White Class to those observed with the high-concentration agent, which also contained calcium (Calcium Whiteness Blue HP) and did not cause changes in the concentrations of the minerals studied. Nonetheless, similar results on Ca and P concentrations for both agents containing calcium in the formulation were not observed.

Immersion in artificial saliva for 14 days after bleaching was not able to recover the initial concentration values of Ca and P in the enamel when using bleaching agents known to lead to changes in mineral content over time (Pola Office+, Opalescence Boost, Pola Day, and White Class). For home-use low–hydrogen peroxide agents, in particular, a significant reduction in Ca concentrations was observed. Other studies[6,11,12,16,26] using artificial saliva in the postbleaching period observed a recovery of the initial microhardness values due to the supersaturated minerals in the saliva, leading to enamel remineralization. Changes in mineral content may have been accompanied by erosive surface lesions, which may have prevented remineralization, or the post–treatment time was not sufficient to remineralize the tooth substrate.[16]

Considering the results of this study, one may suggest that changes in enamel calcium and phosphate contents during and after bleaching may occur regardless of the presence of fluoride or calcium in the composition of certain brands of bleaching agents, concentration of hydrogen peroxide and bleaching technique (office or home), or time of application of the bleaching agent and its pH, corroborating the study by De Abreu and others.[26] Changes in enamel mineral content may be related to the compositional characteristics of each bleaching gel brand, since not all manufacturers mention

all components and their percentages in the formulation. This study suggests that when choosing a bleaching approach, either in office with a high-concentration hydrogen peroxide agent or a home-bleaching method with a low-concentration hydrogen peroxide product, the agents should be selected according to their effects on the tooth structure, in which case Whiteness HP Maxx, Whiteness Calcium Blue HP, Opalescence Boost, and Pola Day were those that caused minimal or no change to the enamel concentrations of Ca and P.

Since no correlation was observed between the methods used to quantify the concentration of calcium and enamel phosphate, and in view of the limitations related to the accuracy of chemical element analysis by the TXRF method when using the enamel biopsy solution, as well as the laborious and restrictive evaluation protocols relating to the use of Synchrotron radiation beam, it can be suggested that the analysis by colorimetric spectrophotometry may yield more homogeneous results, with lower coefficients of variation as well as the possibility of using more repetitions per group.

CONCLUSIONS

Changes in enamel calcium and phosphate contents during and after bleaching may occur regardless of the presence of fluoride or calcium in the composition of certain brands of bleaching agents, concentration of hydrogen peroxide, and bleaching techniques used. Whiteness HP Maxx, Whiteness Calcium Blue HP, Opalescence Boost, and Pola Day caused minimal or no change to the enamel concentrations of calcium and phosphate. No correlation was observed between the methods (TXRF or colorimetric spectrophotometry) used to quantify the concentration of calcium and enamel phosphate.

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Regulatory Statement

This study was conducted in accordance with all of the provisions of the local human subjects' oversight committee guidelines and policies of São Leopoldo Mandic Research Ethics Committee. The approval code for this study is 32495614.0.0000.5374.

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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Whitening Efficacy of Whitening Mouth Rinses Used Alone or in Conjunction With Carbamide Peroxide Home Whitening

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Clinical Relevance

Several over-the-counter whitening products, such as whitening mouth rinses, are available to consumers. The bleaching efficacy of some mouth rinses may increase the longevity of carbamide peroxide home whitening outcomes over time.

SUMMARY

Objectives: The aim of this study was to compare the effectiveness of whitening mouth rinses on teeth previously whitened or not, exposed to food dyes.

Methods and Materials: One hundred twenty enamel-dentin specimens, 3 mm in diameter, were obtained from bovine incisors. The specimens were stained for 14 days in staining broth. After staining, the initial color reading was performed via a spectrophotometer CM-2600d (Konica Minolta). Half of specimens were submitted to whitening (10% carbamide peroxide [CP]) for 14 days. They were then divided into three groups and were submitted to cycles of staining (five minutes) and mouth rinses (two minutes) for 12 weeks, with the following: CP-LI, Listerine Whitening; CP-PL, Plax Whitening; CP-BP, bromelain + papain; CP-DW, deionized water. LI, PL, BP, and DW groups were submitted to the same cited cycles but with no prior bleaching. The color measurements were performed after four, eight, and 12 weeks of treatment with mouth rinses. Data were submitted to repeated measures analysis of variance (ANOVA) and Tukey's test for multiple comparisons, with significance level at 5%.

Results: The results showed that the CP-LI, CP-PL, LI, and PL groups had greater color change than did the others. The CP-BP and BP groups were similar to CP-DW and DW.

Conclusions: We therefore conclude that Listerine Whitening mouth rinse presented the

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highest bleaching effect, followed by Plax Whitening mouth rinse. Both maintained CP bleaching effect after 12 weeks of dye-rinse cycles. However, none of these rinses were able to produce whitening similar to CP. Bromelain- and papain-containing mouth rinses did not show bleaching effect, being similar to the control groups.

INTRODUCTION

Staining of extrinsic origin is an important factor among many others affecting the esthetics of teeth.[1] Staining is the result of extrinsic agents, and it affects the outer surface of teeth or it could be incorporated in the internal structure of teeth. Food dyes, beverages, tobacco, some mouthwashes, and drugs are etiologic agents responsible for chromogenic changes of teeth.[2] Dental prophylaxis eliminates the adsorbed stains on dental surfaces. If the pigments are incorporated in tooth structure, dental whitening techniques might be used.[3,4]

Besides the most common professional techniques for dental whitening (in-office and at-home supervised modalities), some whitening mouth rinses and tooth pastes are also available to consumers as over-the-counter (OTC) products.[5,6]

Mouth rinses were developed to aid controlling dental biofilm, which is mainly responsible for infections and the prevalence of dental caries and periodontal disease.[7] However, to satisfy the requirements of the current consumer society, whitening mouth rinses are freely sold, and advertisements convey the idea of achieving white teeth in a short period of time. They have become a very popular OTC whitening product due to their low cost, easy application, and wide availability in supermarkets and drugstores.[6] In their composition, there may be low concentrations of hydrogen peroxide, usually 1% to 2%[5] and according to Naik and others,[8] such concentrations usually do not cause gingival irritation.

Some studies have evaluated the effectiveness of whitening mouthwashes with hydrogen peroxide.[5,9-11] A study evaluated the efficacy and safety of a whitening mouth rinse (2% hydrogen peroxide) and whitening strips (10% hydrogen peroxide) that were used twice daily for one week. The results showed that, although both products had been well tolerated, the group treated with whitening strips experienced a significantly greater tooth color improvement than the whitening mouth rinse.[11] Torres and others[9] found the use of a 2% hydrogen peroxide mouth rinse resulted in whitening efficacy similar to that obtained with 10% carbamide peroxide (CP) at-home whitening. Jaime and others[10] and Karadas and others[12] reported that mouth rinses containing hydrogen peroxide were able to lighten the darkened human enamel, but to a lesser degree than the whitening produced by 10% CP. Different methodologies, and protocols of use, might have led to different results in those studies.

Due to the small number of studies evaluating the effectiveness of whitening mouthwashes and their association with home whitening, more studies on that issue are needed. Understanding the whitening mouth rinses would help clinicians know their effects and to clarify to patients whether they are needed or not.

Thus, this study aimed to compare the efficacy of whitening mouth rinses on teeth previously whitened or not, and exposed to food dyes. The null hypotheses tested were as follows: 1) the mouth rinses are not able to maintain the outcomes obtained with 10% CP whitening over the studied time period; 2) there is no difference in the whitening efficacy among the tested products; and 3) the mouth rinses are not able to promote a whitening effect similar to that of supervised home whitening.

METHODS AND MATERIALS

Freshly extracted and intact bovine incisors were stored until required in a 0.1% thymol solution and refrigerated at 4°C. Cylindrical enamel samples (3 mm diameter and 2 mm height: 1 mm of enamel and 1 mm of dentin) were prepared from the labial surface of the tooth using a trephine mill.

The specimens were stained in a staining broth (27 g of finely ground instant coffee; 27 g of finely ground instant tea; and 20 g of finely ground gastric mucin dissolved in 8 L of deionized water, 6 mL FD&C [Food, Drug, and Cosmetic] Red 40, 6 mL FD&C Yellow 5, and 750 mL red wine; adapted from Wozniak and others[13]). The specimens were stained for 14 days, under constant agitation.

The specimens were then positioned in a silicone mold with a cavity 6 mm in diameter and 3.1 mm in depth. On the bottom of the mold, there was a second-level cavity (3 mm in diameter and 0.1 mm in depth). The specimens were placed inside the internal cavity with the enamel surface directed toward the bottom of the mold, which was then filled with low-viscosity composite resin (Opallis Flow, FGM, Joinville, SC, Brazil). The specimens were attached to a metal holder, and the enamel surface

Table 1: *Products, Manufacturers, and Their Components*

Product	Manufacturer	Components
Whiteness Perfect 10%	FGM, Joinville, SC, Brazil	Carbamide peroxide, neutralized carbopol, potassium nitrate, sodium fluoride, humectant (glycol), deionized water
Listerine Whitening	KIK Custom Products, Etocicoke, Canada	Water, 8% alcohol, hydrogen peroxide 2%, sodium phosphate, poloxamer 407, sodium lauryl sulfate, sodium citrate, mint aroma, menthol, eucalyptol, sodium saccharin, sucralose
Colgate Plax Whitening	Colgate-Palmolive, São Bernardo do Campo, SP, Brazil	Water, sorbitol, ethanol, hydrogen peroxide 1.5%, poloxamer 338, polysorbate 20, methyl salicylate, menthol, sodium saccharin, CI 42090
Bromelain + papain	Experimental	Bromelain 0.5%, papain 0.25%, methylparaben, Propylparaben

was polished using sequential aluminum oxide abrasive papers (1200, 2400, and 4000 grit x96 FEPA-P, Struers, Ballerup, Denmark) in a polishing device (DP-10, Panambra, Sao Paulo, SP, Brazil). The specimens were immersed in an ultrasonic bath with deionized water for five minutes (Ultrasonic Cleaner, Odontobras, Ribeirão Preto, Brazil) for the removal of all waste. The prepared specimens were examined under a stereomicroscope (Stemi 2000-20×, Carl Zeiss, Tokyo, Japan) to certify the absence of cracks or other surface defects and then stored in deionized water to avoid dehydration.

Prior to treatment, the baseline L* values of each specimen were assessed under standardized ambient conditions, according to the Commission Internationale de l'Eclariage (CIE) L*a*b* system, using a spectrophotometer (CM2600d, Konica Minolta, Osaka, Japan) and an integrating sphere. The device was adjusted to use the D65 standard light source with 100% ultraviolet (UV) light and specular component included (SCI). The observer angle was set at 2°, and the device was adjusted to a small reading area (SAV). The color of each sample was measured three times and averaged. The results of the color measurements were quantified in terms of three coordinate values (L*, a*, b*), as established by the CIE, which locates the color of an object in a three-dimensional (3D) color space. The L* axis represents the degree of lightness within a sample and ranges from 0 (black) to 100 (white). Axis a* represents the degree of green/red color, whereas b* axis represents the degree of blue/yellow color within the sample. L* value of each specimen was used for stratified allocation among eight groups (n=15). The color was measured over white (L*: 84.95; a*: −0.38; b*: 2.93) standard background. Values alterations in L* (ΔL*), a* (Δa*), and b* (Δb*) were calculated from color measurements at baseline and after the whitening procedures. Total color change or variation in color perception of each specimen was calculated and designated by the abbreviation ΔE*. This parameter was calculated according to the following formula: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$

Table 1 shows all products used in this study, including manufacturers and their components. Half of the specimens were submitted to a whitening treatment with CP.

A 10% CP gel was used (Whiteness Perfect 10%, FGM, Joinville, SC, Brazil). The bleaching gel was applied over the surface of specimens in a 2-mm-thick layer (approximately 0.1 g) for eight hours. The gel was removed using a vacuum aspirator, and the specimens were stored in artificial saliva for 16 hours. The whitening procedure was repeated for 14 days. Artificial saliva was prepared according to Gohring and others[14]: 22.1 mmol/L $NaHCO_3$; 16.1 mmol/L KCl; 14.5 mmol/L NaCl; 2.6 mmol/L KH_2PO_4; 0.8 mmol/L H_3BO_3; 0.7 mmol/L $CaCl_2 \cdot 2H_2O$; 0.4 mmol/L KSCN; 0.2 mmol/L $MgCl_2 \cdot 6H_2O$; and mucin and adjusted to a pH of 7.0 with an HCl solution.

Twenty-four hours after the last application of bleaching gel, the color reading was performed. Then, they were submitted to dye-rinse cycles, according to the following groups (n=15): CP-LI, Listerine Whitening mouth rinse, 2% hydrogen peroxide; CP-PL, Colgate Plax Whitening mouth rinse, 1.5% hydrogen peroxide; CP-BP, experimental mouth rinse prepared using 40 g bromelain (Sigma-Aldrich, St. Louis, MO, USA), 20 g of papain (Vetec, Duque de Caxias, RJ, Brazil), 2 g of methylparaben, and 1.2 g of propylparaben, diluted in 8 L deionized water (0.5% bromelin and 0.25% papain); CP-DW, deionized water.

For the dye-rinse cycles, the specimens were immersed for five minutes in the staining broth;

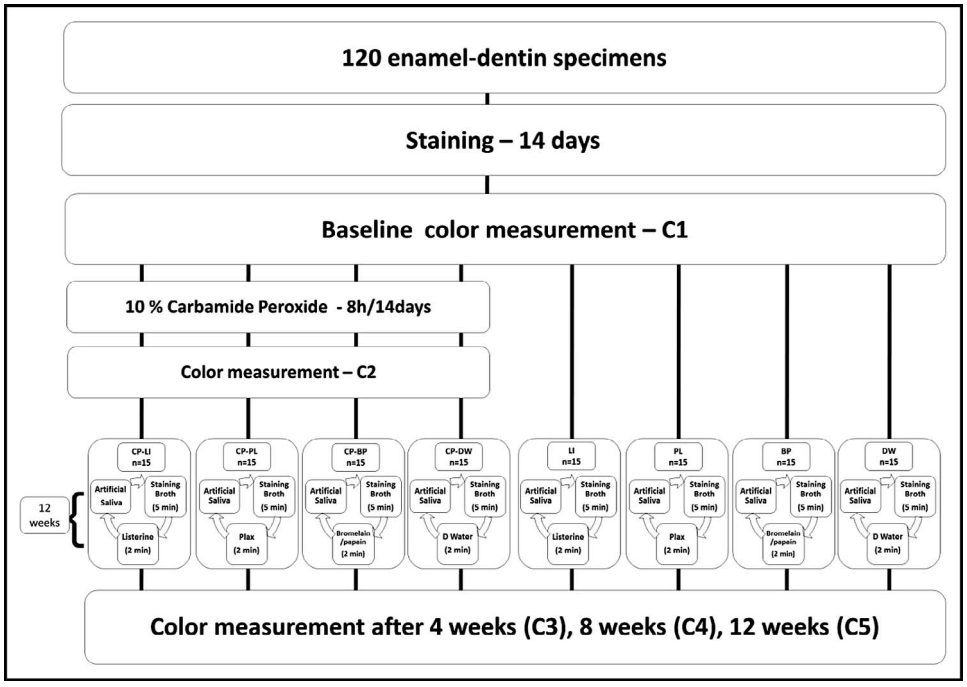

Figure 1. *Experimental design.*

subsequently, they were immersed for two minutes in the mouth rinses and then remained in artificial saliva. This cycle was repeated for 12 weeks. While immersed in the broth and mouthwash, the specimens were kept in constant agitation. Artificial saliva was replaced daily. Color reading was performed after four, eight, and 12 weeks.

The five-minute period in staining broth was recommended to simulate an extreme situation of daily consumption of food dyes, whereas two minutes in the mouth rinses represented the time recommended by the manufacturers.

Groups LI, PL, BP, and DW were submitted to the same cycles as groups CP-LI, CP-PL, CP-BP, and CP-DW were submitted, except for the submission to previous CP whitening. Figure 1 shows the study design.

During the whitening procedure and storage time in artificial saliva, all specimens were kept in a bacteriologic oven (ECB 11 Digital, Odontobrás, Ribeirão Preto, SP, Brazil) at 37°C. The staining and mouth rinse cycles were performed at room temperature (25°C).

With L*a*b* values after the teeth staining (C1, baseline values) and values obtained after different periods of tested treatments, it was possible to calculate ΔE* and then determine the efficacy after simulated home bleaching (ΔE^*_1), as well as treatments at four (ΔE^*_2), eight, (ΔE^*_3), and 12 weeks (ΔE^*_4).

The data obtained were statistically analyzed using Statistica for Windows (Statsoft, Tulsa, USA). Repeated measures analysis of variance (ANOVA) and the Tukey's test were applied at a significance level of 5%.

RESULTS

A descriptive table with ΔL*, Δb*, Δa*, and ΔE* values of all groups is presented as Table 2. At baseline, all color coordinate (L*, a*, and b*) means for each group were not statistically different.

The overall color change of specimens after whitening with 10% CP and after four, eight, and 12 weeks of dye-rinse cycles is shown in Table 3. The cross-product treatment versus time and the factors were statistically significant (p=0.001).

Concerning b* values, negative changes in Δb* values were observed, except for the CP-BP group at 12 weeks and BP and DW at all assessed periods. b* values decreased during the course of the experiment, reflecting a reduced yellowness in specimens.

Figure 2 shows reflectance curves of the mean values of the CP groups at baseline, after whitening, and after 12 weeks of dye-rinse cycles. The major reflectance was observed after bleaching; however, after 12 weeks, CP-LI still presented a level of reflectance similar to that after whitening. CP-BP showed decreased reflectance after 12 weeks of dye-rinse cycles. The major changes could be observed

Table 2: *Mean and Standard Deviation (SD) of ΔL*, Δa*, Δb*, and ΔE* for All Groups at All Measurement Times*

Group	Time	ΔL*		Δa*		Δb*		ΔE*	
		Mean	SD	Mean	SD	Mean	SD	Mean	SD
PC-LI	Bleaching	3.09	2.42	0.15	0.58	−5.05	1.45	6.41	1.34
	Four weeks	2.04	3.38	−0.19	0.52	−5.59	1.11	6.55	2.23
	Eight weeks	0.62	4.60	−0.08	0.60	−5.78	1.85	6.88	3.22
	12 weeks	0.27	3.98	0.01	0.58	−5.31	1.92	6.53	2.11
PC-PL	Bleaching	2.04	3.86	0.45	0.66	−5.30	1.49	6.84	1.52
	Four weeks	2.28	2.60	−0.12	0.56	−5.23	1.40	6.20	1.67
	Eight weeks	1.03	4.63	0.02	0.63	−4.71	1.62	6.24	2.76
	12 weeks	0.75	4.55	0.03	0.62	−3.32	1.62	5.09	2.89
PC-BP	Bleaching	4.70	1.33	0.44	0.43	−4.17	1.32	6.51	0.92
	Four weeks	2.96	1.27	−0.13	0.37	−2.24	1.65	4.03	1.35
	Eight weeks	0.22	1.96	0.00	0.46	−2.33	1.29	3.08	1.18
	12 weeks	2.95	2.20	0.52	0.51	1.52	1.80	3.90	2.02
PC-DW	Bleaching	3.63	2.28	0.23	0.46	−4.78	1.61	6.41	1.64
	Four weeks	3.11	1.50	0.04	0.54	−3.55	1.52	4.99	1.44
	Eight weeks	0.54	1.77	−0.12	0.55	−3.63	2.06	4.23	1.72
	12 weeks	2.31	2.73	0.26	0.59	−1.11	1.68	3.73	1.69
LI	Four weeks	0.42	1.02	−0.14	0.25	−1.67	1.37	2.33	0.62
	Eight weeks	2.70	1.60	−0.13	0.35	−1.20	1.97	3.63	1.37
	12 weeks	3.78	2.03	0.22	0.42	−0.96	2.15	4.46	1.99
PL	Four weeks	−1.51	2.05	0.56	0.56	−0.55	1.37	2.74	1.20
	Eight weeks	0.26	1.92	0.24	0.35	−0.27	0.87	1.89	0.99
	12 weeks	0.75	1.54	0.34	0.42	−0.64	0.95	1.89	0.90
BP	Four weeks	−2.70	1.93	0.28	0.38	3.53	1.91	4.91	1.73
	Eight weeks	−2.78	2.17	−0.31	0.46	1.82	1.40	4.02	1.21
	12 weeks	−1.35	1.86	0.66	0.60	3.92	2.05	4.75	1.63
DW	Four weeks	−1.64	1.24	0.42	0.37	0.99	0.80	2.32	0.83
	Eight weeks	−0.70	2.35	0.22	0.31	1.77	1.56	3.25	0.85
	12 weeks	−1.81	2.12	0.40	0.48	2.90	1.11	3.99	1.27

near 400-450 nm, which is the spectrum region corresponding to blue reflection.

Figure 3 presents ΔL* and ΔE* results of all groups submitted to dye-rinse cycles with no previous 10% CP whitening treatment. For ΔE*, the cross-product treatment versus time and the factors were statistically significant ($p=0.001$).

LI presented a positive ΔL* for the entire time period, showing color change direction toward the white region of L* axis and therefore whitening of the specimens. For PL, from eight weeks of rinse treatment, a whitening effect could be noted (positive ΔL*). However, for BP and DW, a darkening of the specimens could be observed (negative ΔL*).

One-way ANOVA and Dunnet's test were performed to compare the whitening effect of rinse treatment after 12 weeks with 10% CP. No rinse was able to produce the same whitening effect as that of 10% CP.

Table 3: *Mean and Standard Deviation of Color Changes After Bleaching and Four, Eight, and 12 Weeks of Dye-rinse Cycles*[a]

Group	Bleaching (ΔE^*_1)	4 weeks (ΔE^*_2)	8 weeks (ΔE^*_3)	12 weeks (ΔE^*_4)
CP-LI	6.41 (1.34) Aa	6.55 (2.23) Aa	6.88 (3.22) Aa	6.53 (2.11) Aa
CP-PL	6.84 (1.52) Aa	6.20 (1.67) ABab	6.24 (2.76) ABab	5.09 (2.89) Ab
CP-BP	6.51 (0.92) Aa	4.03 (1.35) Bb	3.08 (1.18) BCb	3.90 (2.02) Bb
CP-DW	6.41 (1.64) Aa	4.99 (1.44) ABab	4.23 (1.72) Cb	3.73 (1.69) Bb

[a] *Different capital letters mean significant differences among rows ($p<0.05$). Different lowercase letters mean significant differences among lines ($p<0.05$).*

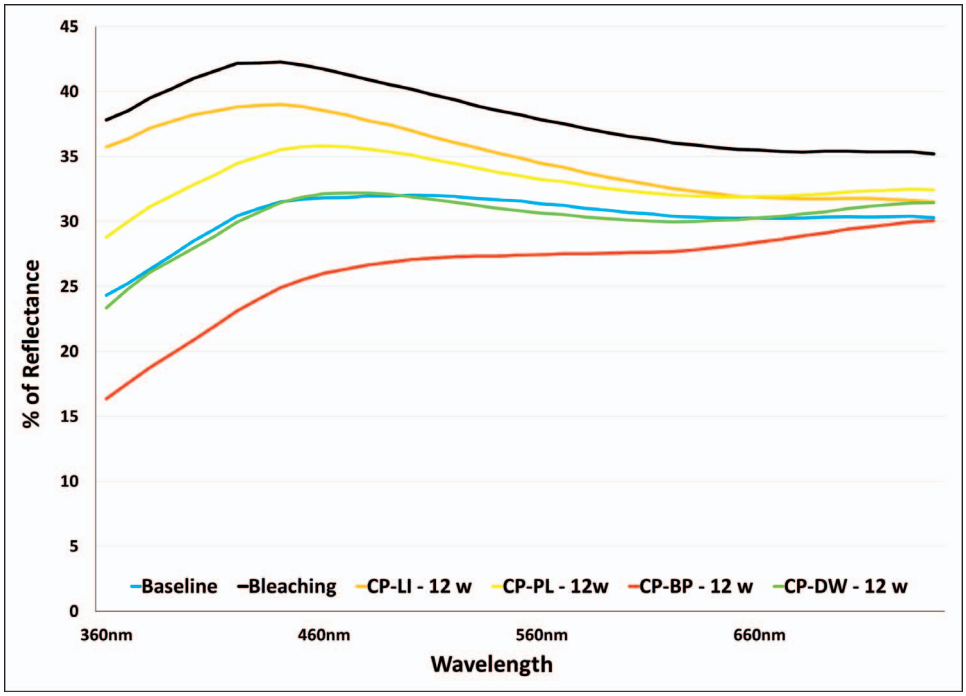

Figure 2. *Reflectance curves of CP subgroups.*

DISCUSSION

The null hypotheses tested in this study were that evaluated mouth rinses are not able to maintain the whitening outcome after whitening with 10% CP; there is no difference in whitening efficacy among the tested products; and they are not able to promote a whitening effect similar to that of supervised home whitening. According to the results, the first hypothesis was partially accepted, the second one was rejected, and the third hypothesis was accepted.

This study was performed according to the ADA Recommendations for Laboratory Testing Methods of Whitening agents.[15] The intention of staining was to standardize tooth discoloration to compare different bleaching treatments.

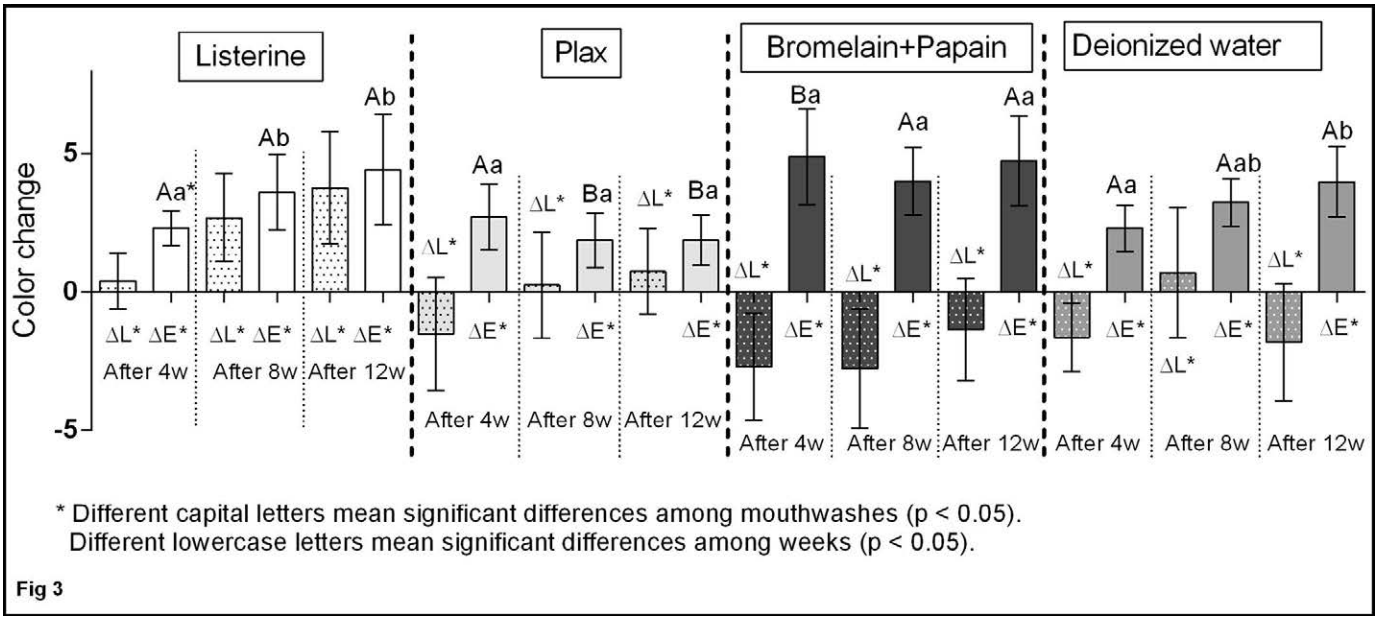

Figure 3. *Results of ΔL and ΔE for subgroups LI, PL, BP, and DW.*

After 14 days of 10% CP bleaching, the results showed that ΔL*, Δa*, and Δb* values confirmed the whitening effect for all groups (Table 2). The effectiveness of this whitening protocol is very well established in the literature; and the protocol has been successfully used since its introduction by Haywood and Heymann.[16] Some studies have noted the longevity of whitening obtained with 10% CP.[17-19] However, after one to two years of clinical follow-up, it was observed that the color of the tooth did not return to the baseline; a mild to moderate relapse might be observed. To provide the maintenance of the result obtained by conventional whitening over time, we tested the use of whitening mouth rinses after 10% CP treatment, associated to *in vitro* simulation of daily exposure to food dyes.

The whitening mouth rinses used in this study include hydrogen peroxide at different concentrations (Listerine Whitening, 2%; Plax Whitening, 1.5%) or bromelain and papain. Hydrogen peroxide is an effective oxidizer, with a known whitening effect at high concentration.[20,21] Bromelain and papain are proteolytic enzymes derived from pineapple (*Ananas comosus*) and papaya (*Carica papaya*), respectively.[22] Currently, toothpastes and mouth rinses that contain bromelain and papain in their composition are available on the market, but research about these products is still scarce.[22-24]

From the results, we found that Listerine Whitening and Plax Whitening were able to maintain the result obtained initially with 10% CP, for a simulated three-month period of dye-rinse challenge. The dye cycling represents a very intense challenge, and control group specimens, who were challenged by the dye but not exposed to mouth rinses had a significant relapse of color.

Specimens submitted solely to whitening mouth rinse treatments (LI, PL, BP, and DW groups) revealed a whitening effect for groups LI and PL after 12 weeks of treatment. The fact that Listerine Whitening and Plax Whitening mouth rinses have shown the best results, respectively, is probably due to the fact they contain hydrogen peroxide in their composition. Lima and others[5] and Torres and others[9] also performed studies with those rinses and obtained positive results. Joiner[25] stated that the effectiveness of a whitening technique depends on the concentration and exposure time to hydrogen peroxide.

CP-DW and DW control groups were expected not to show a whitening effect and in fact they did not. On the other hand, CP-BP and BP were expected to demonstrate such an effect, because previous studies with toothpastes containing the proteolytic enzymes bromelain and papain in their composition emphasized their whitening effectiveness.[22-24] Such studies, however, did not reveal the chemical composition, including the percentage of enzymes of the tested toothpastes. This may raise the hypothesis that there is another bleaching agent in these toothpastes or that bromelain and papain require a specific base for stability. We prepared an experimental 0.5% bromelain and 0.25% papain containing mouth rinse. That particular concentration was determined in a pilot study, with a lower concentration (0.1% for both substances) showing no whitening effect. Furthermore, when we searched for commercial products with bromelain and papain in their composition, the percentage of 0.5% bromelain and 0.125% papain was found for the toothpaste Janina Liquid Toothpaste Spray (Janina Ultra-White, London, England). The methylparaben and propylparaben were used as antimicrobial preservative agents. The whitening result obtained with LI and PL was lower than the result obtained by 10% CP. There is no consensus in the literature on this outcome.[9,10] Perhaps the methodology used in those studies and the dyes used could promote different results.

The use of whitening mouth rinses is recent, and few studies on this topic are available. Most of the studies, similar to the present one, are *in vitro* studies and aim to simulate a clinical situation. However, more clinical trials are needed so that we would ensure the effectiveness of these products and the best protocol to use them.

CONCLUSIONS

Within the limitations of this study, it is concluded that 1) the mouth rinses LI and PL were able to maintain the result obtained with 10% CP after 12 weeks of a dye-rinse challenge; 2) the mouth rinse Listerine Whitening presented the highest bleaching effect, followed by the mouth rinse Plax Whitening; 3) the bromelain and papain containing mouth rinse did not show a whitening effect, thus resembling the control group; and 4) the mouth rinses were unable to produce the same bleaching effect as 10% CP.

Acknowledgement

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of UNESP - São Paulo State University, São José dos Campos, Brazil.

Conflict of Interest

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

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Effect of Endocrown Pulp Chamber Extension Depth on Molar Fracture Resistance

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Clinical Relevance

Endocrown pulp chamber extension depth should not be greater than 2 mm.

SUMMARY

Purpose: The purpose of this study was to evaluate the effect of endocrown pulp chamber extension on mandibular molar fracture resistance.

Methods and Materials: A total of 36 recently extracted mandibular third molars of approximate equal size were sectioned at the facial lingual height of contour followed by endodontic access into the pulp chamber. The specimens were then randomly divided into three groups (n=12) and pulpal and root canal contents removed. Pulp chamber floors were established at 2, 3, and 4 mm from the occlusal table using a three-step etch-and-rinse adhesive and a flowable resin composite. The prepared specimens were then embedded in autopolymerizing denture base resin with surface area available for adhesive bonding determined using a digital recording microscope. Specimens were restored using a standardized template with a chairside computer-aided design/computer-aided manufacturing unit with the endocrown milled from a lithium disilicate glass–ceramic material. Restoration parameters of occlusal table anatomy and thickness were standardized with the only parameter difference being the pulp chamber extension depth. The endocrown restorations were luted with a self-adhesive resin luting agent and tested to failure after 24 hours on a universal testing machine, with force applied to the facial cusps at a 45° angle to the long axis of the tooth. The failure load was converted into stress for each specimen using the available surface area for bonding. Mean failure load and stress among the three groups was first subjected to the Shapiro-Wilk and Bartlett tests and then analyzed with an analysis of variance with the Tukey post hoc test at a 95% confidence level (p=0.05).

Results: The 2- and 4-mm chamber extension groups demonstrated the highest fracture resistance stress, with the 3-mm group similar to the 2-mm group. The 3- and 4-mm chamber

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extension group specimens demonstrated nearly universal catastrophic tooth fracture, whereas half the 2-mm chamber extension group displayed nonrestorable root fractures.

Conclusions: Under the conditions of this study, mandibular molars restored with the endocrown technique with 2- and 4-mm pulp chamber extensions displayed greater tooth fracture resistance force as well as stress. All groups demonstrated a high number of catastrophic fractures, but these results may not be clinically significant because the fracture force results are higher than normal reported values of masticatory function.

INTRODUCTION

Restoration of endodontically treated teeth remains a challenge because they represent a stark biomechanical difference compared with their vital counterparts, representing a multifactorial dissimilarity that includes changes in tissue composition and dentin microstructure and macrostructure as well as the evident loss of tooth structure.[1] Many different compensatory treatment strategies have been proposed, including intracoronal post systems, directly placed complex restorations, and adhesive considerations.[1] Full-coverage indirect crown restorations are usually the preferred method of most practitioners and have been reported to display an increased survival rate compared with directly placed restorations.[2,3] Amalgam and resin composite cores have traditionally served as crown foundation materials, with recent reports that resin cores present either equivocal[4] or increased longevity compared with cast metal cores.[5] When adequate sound-dentin-supported enamel is present, intracoronal bonded resin composite restorations have been suggested to demonstrate fewer fractures within endodontically treated teeth, whereas intracoronal posts have the potential to lessen fracture resistance due to root dentin removal.[2]

Dental computer-aided design/computer-aided manufacturing (CAD/CAM) is increasingly used for full-coverage ceramic restorations due to reported esthetics, marginal accuracy, and expedient restoration production, with the restoration relying more on adhesive technology for retention.[6-15] In restoring endodontically treated teeth some CAD/CAM proponents emphasize the endocrown method.[6] The endocrown prosthesis consists of a merged crown-core unit that is adhesively bonded to the remaining tooth structure and is purported to provide a more conservative option to traditional post-and-core restorative strategies as well as providing equitable results.[6-15] Furthermore, some CAD/CAM proponents anecdotally maintain that the endocrown bond to dentin is superior to the bond of a ceramic crown to either amalgam or composite core materials. Moreover, adhesive technology is purported to be able to compensate for traditional macroretentive preparation features that are required for aqueous-based luting agents.[9]

General guidelines for endocrown preparations include 2- to 3-mm cuspal reduction, 90° butt margins, smooth internal transitions, a 6° pulp chamber taper, a relatively flat pulp chamber floor with sealed radicular spaces, and supragingival margins when possible.[8-10,15] Although a flat pulp chamber floor may not be an absolute requirement, the authors feel that it could be more difficult to achieve a symmetrical pulp chamber taper without a stable internal reference. There are no definitive guidelines concerning the pulp chamber extension depth required for adequate retention and resistance form. However, one report suggests that a 2-mm extension into the pulp chamber is sufficient.[14] The purpose of this study was to evaluate the effect of CAD/CAM endocrown restorations with pulp chamber extension depths of 2, 3, and 4 mm on molar fracture resistance. The null hypothesis was that there would be no difference in fracture resistance among the three groups.

METHODS AND MATERIALS

The human mandibular third molars used in this study were collected from local oral and maxillofacial surgery clinics and had been removed as per routine clinical indications under local institutional review board–protocol approval.

A total of 36 recently extracted mandibular third molars of approximately equal size were sectioned with a slow-speed diamond saw (Buehler, Lake Forest, IL, USA) at the facial-lingual height of contour perpendicular to the tooth long axis. Access to the pulp chamber was accomplished using a high-speed handpiece (EA-51LT, Adec, Newburg, OR, USA) and a diamond bur (6847.33.016, Brassler USA, Savannah, GA, USA) with copious water spray. Pulpal remnants were removed with barbed broaches and gross instrumentation with hand files (Miltex, York, PA, USA). The prepared teeth were randomly subdivided into three groups (n=12) and received pulp chamber restorations to achieve symmetrical pulp chamber floors approximately parallel to and at depths of 2, 3, and 4 mm from the sectioned molar occlusal table using a three-step

etch-and-rinse adhesive (Optibond FL, Kerr Corporation, Orange, CA, USA) and a flowable resin composite (Tetric Evo Flow, Ivoclar Vivadent, Amherst, NY, USA). All materials were placed following manufacturer recommendations with light polymerization accomplished using a polywave LED-based visible light curing (VLC) unit (Bluephase G2, Ivoclar Vivadent) whose irradiance was verified (1000 mW/cm2) using a laboratory-grade laser power meter (10A-V1, Ophir-Spiricon, North Logan, UT, USA). The specimens were then prepared to final form with the same handpiece and diamond bur to the desired configurations, with the preparations modified as needed from measurements from the digital two-dimensional microscope (Hirox 7700, Hirox USA, Hackensack, NJ, USA). Preparation standardization was assisted with a locally established covariance upper limit of 25%.

Specimens were then embedded in auto-polymerizing denture base resin (Impak Self Cure, CMP Industries, Albany, NY, USA) and surface area available for adhesive bonding determined using a digital recording microscope (KH-7700, Hirox USA) (Figure 1). The chamber surface area was determined by measuring a polyvinylsiloxane-impression replica of the prepared chamber. Mean chamber depth was determined by the mean of six measurements obtained at the mesial, distal, and middle chamber extensions. The restored resin chamber floor was also included in the surface area determination. Specimens were scanned using a standardized template simulating clinical conditions (Figure 2a,b) using a chairside CAD/CAM unit (Cerec AC/Cerec MC XL, Sirona Dental Systems, Charlotte, NC, USA; version 4.2.4.72301), with the crown milled from a lithium disilicate glass–ceramic material (IPS e.Max CAD HT A2, Ivoclar Vivadent). The occlusal table was established approximately 4 mm in height for all specimens. This provided consistent coronal thickness and form for all restorations, with the only parameter difference being the pulp chamber extension depth. Crystallization firing was accomplished following manufacturer protocol in a dental laboratory ceramic furnace (Programat P700, Ivoclar Vivadent). The restoration's intaglio surface was then steam cleaned and dried, followed by a 5% hydrofluoric acid etch (IPS Ceramic Etching Gel, Ivoclar Vivadent) for 20 seconds. Etched surfaces were thoroughly rinsed with water for 15 seconds and dried with oil-free compressed air, after which two thin coats of a silane agent (Monobond Plus, Ivoclar

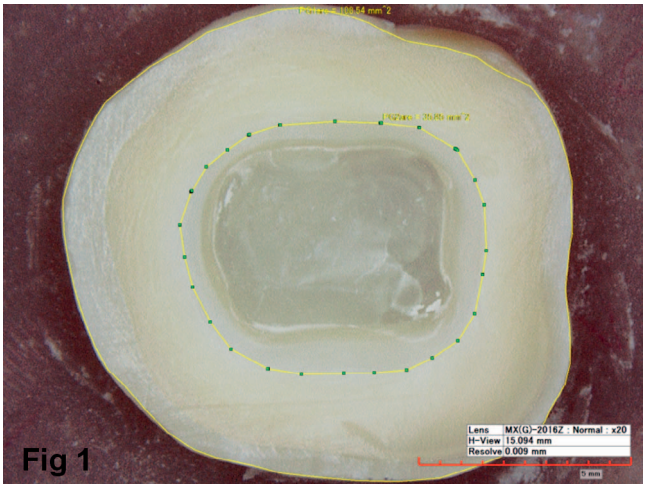
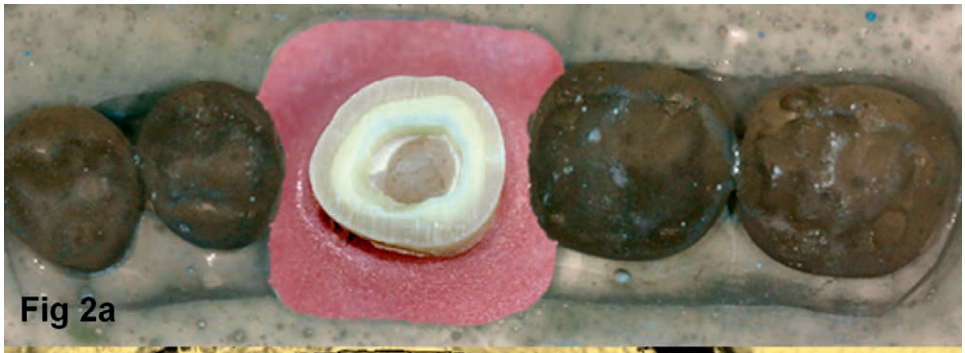


Figure 1. *Surface area assessment measurements.*

Vivadent) was applied with a microbrush to the treated surface for two 60-second intervals with the excess dispersed with a strong stream of air. The recipient tooth was prepared for cementation using pumice slurry in a prophylaxis cup (Extended Straight Attachment DPA, Preventech, Indian Trail, NC, USA) using a slow-speed handpiece (Midwest Shorty, Dentsply, York, PA, USA), rinsed thoroughly with water for 5 seconds, and then air dried. Restorations were cemented with a self-adhesive resin luting agent (Rely-X Unicem, 3M ESPE, St Paul, MN, USA) with firm digitally applied pressure and stabilization to allow the restoration to fully seat. A 2-second VLC tack cure was accomplished (Bluephase G2, Ivoclar Vivadent) with the excess cement removed, followed by each surface receiving additional light curing for 20 seconds. Restorations were then stored in dark conditions at 37°C ± 1°C and 98% ± 1% humidity.

Twenty-four hours after cementation, specimens were placed into a vise fixture mounted on a universal testing machine (RT-5, MTS Corporation, Eden Prairie, MN, USA) with the long axis of the tooth oriented at a 45° angle to the testing device. The facial functional cusps were loaded with a 3-mm diameter, hardened, stainless steel piston with a 0.5-m radius of curvature as described by Kelly and others[16] and were loaded at a rate of 0.5 mm per minute until fracture, with the failure load recorded in newtons. Failure load was converted into stress for each specimen using the available surface area for bonding. Each fractured specimen was also examined visually at 20× magnification (Hirox KH-7700, Hirox USA) to determine whether the failure was cohesive for the ceramic material, adhesive between the ceramic and the tooth structure, or



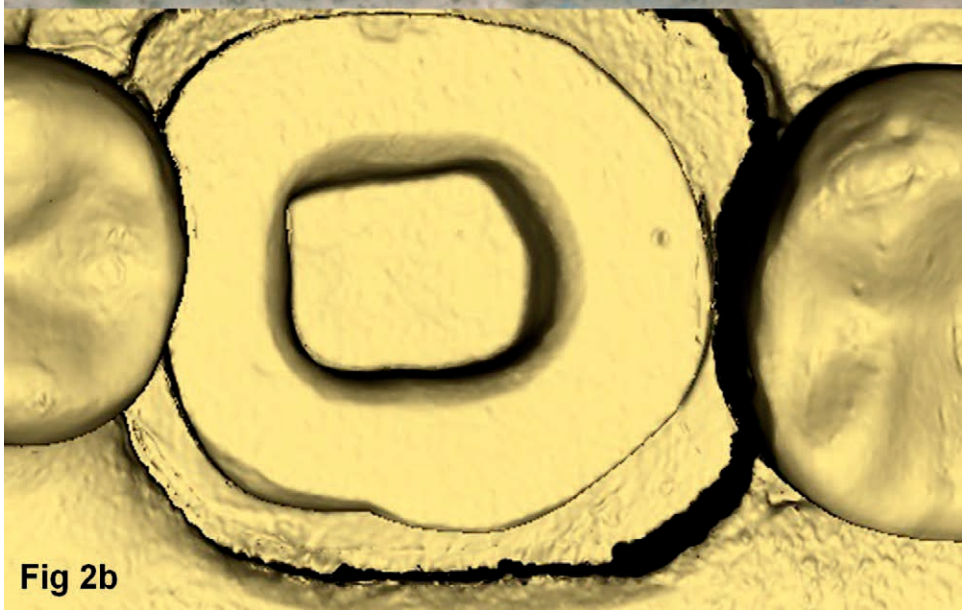


Figure 2a. *Prepared specimen in template for scanning.*
Figure 2b. *Resultant scanned image.*

fracture of the tooth material. Further failure analysis was accomplished with microradiographic tomography (microCT) (Skyscan 1172, Bruker microCT/Micro Photonics, Allentown, PA, USA), with samples scanned over a 180° radius with 13.6-μ resolution, 100-kV power, 100-mA resolution, aluminum filtration, and a 0.4° step size. Resultant individual images were recombined with software (nRecon, Bruker microCT), with resultant recombined images visualized using CTan and CTVox software (Bruker microCT). The mean failure force and stress values were evaluated by the Shapiro-Wilk and Bartlett tests, which verified both the normal distribution and variance equality. The mean data were then analyzed with an analysis of variance (ANOVA) with a Tukey post hoc test at a 95% confidence level (α=0.05).

RESULTS

The mean preparation features of each group are presented in Table 1. Consistency within the sample groups was achieved with relative success, indicated by the low covariance noted with the mean chamber extension depth and surface area covariance being less than 18%.

Resultant mean failure forces and stress are listed in Table 2. The ANOVA identified differences between the failure force groups (p=0.02) and the failure stress groups (p=0.008). The results of the *post hoc* testing are shown in Table 3. In regard to failure force, the 3-mm chamber extension groups demonstrated the lowest fracture resistance trend. It is interesting that the 4-mm chamber extension group demonstrated the highest fracture force resistance, which was contrasted by the 2-mm chamber extension group having the highest stress fracture resistance.

Failure mode results are identified in Table 4. The 3- and 4-mm chamber extension depth group specimens demonstrated nearly universal catastrophic tooth fracture, whereas eight of the twelve 2-mm chamber depth extension group demonstrated nonrestorable fractures.

Table 1: *Mean (Standard Deviation) of Tooth Preparation Parameters (n=12)*

Group (Chamber Extension Depth)	Mean Chamber Extension Depth (mm)	%COV	Surface area (mm^2)	% COV
2 mm	2.2 (0.05)	2.0	118.5 (16.6)	14.1
3 mm	3.1 (0.06)	1.9	139.2 (19.5)	14.3
4 mm	4.1 (0.09)	2.2	162.1 (28.1)	17.4

Abbreviation: COV, covariance.

DISCUSSION

The need for proper restoration of endodontically treated teeth is well known. Tang and others[2] reported that failure to provide permanent restorations after endodontic treatment resulted in greater than 65% tooth loss during a mean follow-up time of three years. They emphasized the need for expedient sealing of all endodontic access cavities followed by cuspal coverage restorations[2] to alleviate bacterial recontamination of obturated root canals, which has been reported to occur as quickly as 25 to 30 days after re-exposure.[17,18] The endocrown method offers an expedient clinical option in regard to the sealing and restoration of the endodontically treated tooth as well providing clinical alternatives in the situations of teeth with calcified, short, or dilacerated root canals.[7]

The endocrown method was first described by Pissis,[10] followed by Bindl and Mormann[6] who reported clinical results demonstrating a 95% endocrown survival rate over a mean recall rate of 26.6 months.[6] Lander and Dietschi[11] described that endocrowns could provide suitable restorations in situations with minimal compromised vertical height and ferrule. In an *in vitro* finite element analysis model, Dejak and Młotkowski[13] reported molars restored with endocrowns transferred less functional stress to dentin compared with molars restored with posts and cores.

In this study, we investigated the effect of endocrown chamber depth extension on mandibular molar fracture resistance. Specimens were prepared as uniformly as possible by one researcher with the mean specimen parameters previously listed in Table 1. Surface area available for adhesion was determined by the area measurement function of the digital measuring microscope (Figure 2). The authors feel that preparation standardization within each group was reasonably achieved, given that the chamber depth measurement covariance was approximately 2%, whereas surface area measurements covariance ranged from 14% to 17%. Furthermore, the increase in surface area associated with each chamber depth level was demonstrated to be a linear function (r^2=0.99), as shown in Figure 3.

Under the conditions of this study, the null hypothesis was rejected in that the 2- and 4-mm groups demonstrated higher resistance to failure as compared with the 3-mm chamber extension group. However, due to the similarity of the data ranges, the results may not be clinically significant. Perhaps the most clinically relevant findings of this study is revealed by the failure analysis. Ninety-two percent of the 3-mm chamber extension group and 83% of the 4-mm chamber extension group demonstrated catastrophic fracture, whereas 66% nonrestorable failures were observed with the 2-mm chamber extension group. Upon initial visual analysis, the 2-mm pulp chamber extension group was thought to comprise mostly restoration debonding accompanied with repairable tooth fracture (Figure 4). However, microCT imaging proved to be a valuable tool in failure mode assessment. Some specimens that were initially thought repairable on the basis of visual examination alone were found after microCT assessment to also contain irreparable root fractures, which, depending on location, may or may not be visible on a standard periapical film (Figure 5). Accordingly, after microCT evaluation a total of six

Table 2: *Mean (Standard Deviation) of Failure Force and Stress Results (n=12)*

Endocrown Chamber Extension Depth (mm)	Failure Force (N)	Failure Stress (MPa)
2	843.4 (106) AB[a]	7.29 (1.6) A
3	762.8 (240) A	5.33 (1.2) B
4	943.5 (110) B	6.04 (1.6) AB

[a] *Groups identified with same capital letter within each column are similar (Tukey,* p=*0.05).*

Table 3: *Tukey Multiple Comparison Test Results (n=12)*

Comparison	Failure Force	Failure Stress
2 vs 3 mm	$p = 0.377$*	$p = 0.007$
2 vs 4 mm	$p = 0.262$	$p = 0.109$
3 vs 4 mm	$p = 0.015$	$p = 0.471$

* p = *0.05.*

Table 4: *Failure Mode Analysis Results (n=12)*

Endocrown Chamber Extension Depth (mm)	Failure Mode		
	Adhesive	Restorable Fracture	Catastrophic Fracture
2	3	1	8
3	1	0	11
4	1	1	10

of the specimens with a 2-mm chamber depth were found to have catastrophic tooth fracture.

The results of the current study are comparable to that of Biacchi and Basting[8] who reported a mean endocrown failure load of approximately 675 N in preparations with pulp chamber extensions ranging from 2.0 to 2.7 mm, which also demonstrated tooth fracture in 90% of the specimens. However, results of the present study are much lower than those of Magne and others,[19] who reported a mean failure load of 2606 N for endocrowns with 1.5-mm chamber depth extensions, as well as of those of El-Damanhoury and others,[14] who found that endocrowns with 2-mm chamber depth extensions demonstrated a failure resistance of 1368 N. However, these studies' methodologies differed from that of the present study in that the loads to failure were applied at different vectors, axially and at 35°, respectively. However, failure mode results of this study compares favorably with that of El-Damanhoury and others,[14] who found that approximately 70% of the endocrowns demonstrated catastrophic failure. Also, fracture resistance reported by Carvalho and others[20] as well as Gresnigt and others[21] was higher than that found in the present study, but those previous studies involved axial loading after fatigue stressing the specimens. However, all of these findings should be considered with the fact that failure loads of this study as well as other studies are above the reported human functional load. Accordingly, molar region occlusal load has been reported to be near the range of 100 to 200 N and has been estimated as high as 965 N in situations of accidental occlusal contact, parafunction, and/or trauma.[22-29]

This is the first endocrown *in vitro* study that produced failure stress data that was based on tooth surface available for bonding. Failure stress calculation was undertaken to evaluate whether stress results, on the basis of surface area available for bonding, might serve to normalize failure force results that may be affected by different tooth specimen size. However, under the conditions of this study no clear normalization effect was noted. This aspect will continue to be evaluated with future studies, and more comparative studies will be required before definitive recommendations may be proffered. In addition, microCT analysis was found to be a valuable failure analysis tool, given that this modality allowed nondestructive failure analysis permitting fracture detection that was not usually not discernable visually and might be beyond

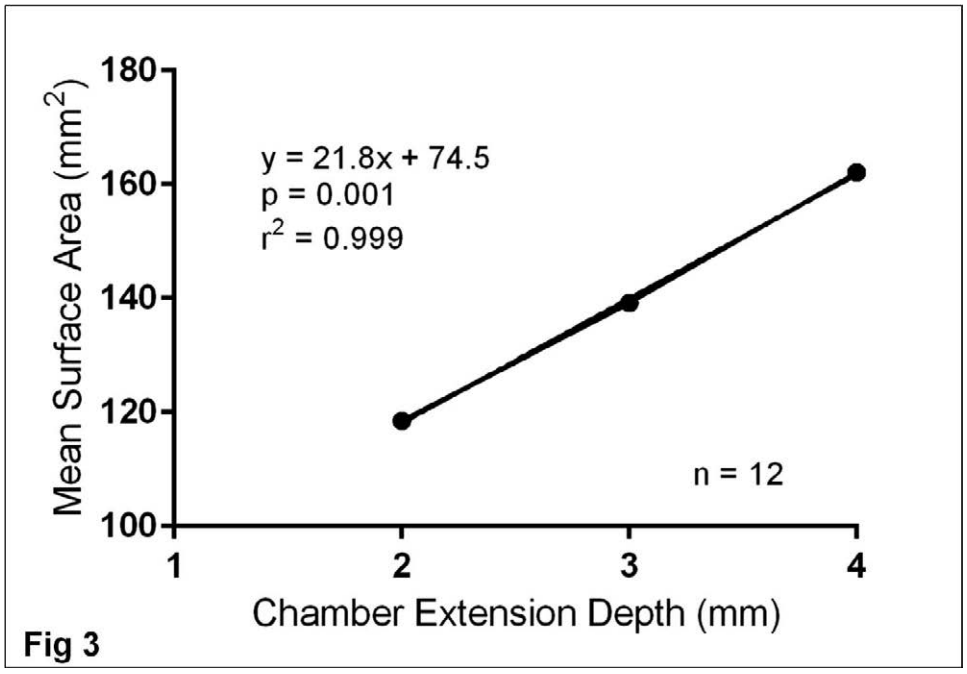
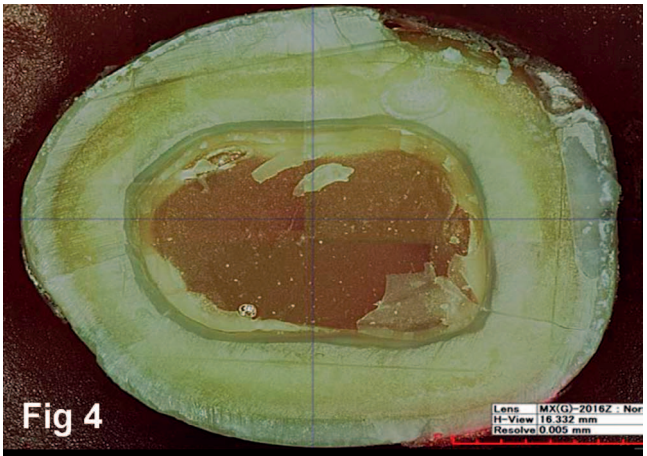


Figure 3. *Surface Area Increase Per Chamber Depth*



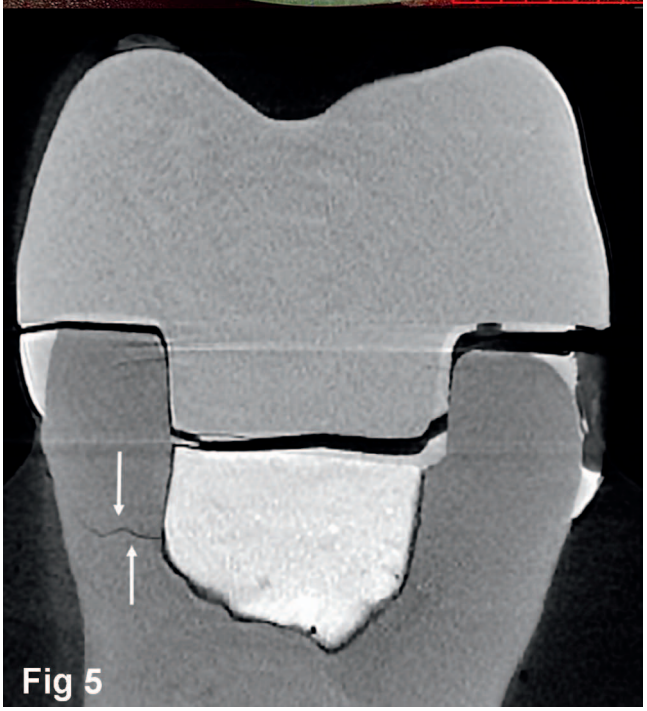


Figure 4. *Failure analysis visual assessment revealing restorable tooth damage.*
Figure 5. *MicroCT assessment of Figure 4 specimen. (Arrows denote fracture)*

detection with the usual clinical radiographic techniques.

One limitation of this study was the choice of a fixed crown height. Reports suggest that internal stresses to both the endocrown and supporting tooth structure increase as the force application location increases vertically from the endocrown buccal margin.[21,30] Accordingly, different force application heights would involve different lever-arm tipping forces as well as force vectors between the supporting tooth and restoration—all of which may change fracture load results. The authors chose to limit the force application level to evaluate solely the effect of different pulp chamber extension depths, but this load was applied at the same height as the marginal ridge to, one hopes, represent a worst-case scenario.

The findings of this laboratory study reinforces CAD/CAM proponents' recommendations of a 2-mm endocrown pulpal extension. However, this study identified a remarkable number of irreparable fractures, and this recommendation requires further investigation. Because the identified failure forces were above the published range reported with normal human function as well as parafunction, fatigue analysis is planned as well as research evaluating endocrown preparations with ferrule preparation features.

CONCLUSIONS

Under the conditions of this study, mandibular molars restored with the endocrown technique with 2- and 4-mm pulp chamber extensions displayed greater tooth fracture resistance. All groups demonstrated a high number of catastrophic fractures, but these results may not be clinically significant because the fracture force results are higher than normal reported values of masticatory function.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of 81 Medical Group. The approval code for this study is FKE20140012N.

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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Bonding Polycrystalline Zirconia With 10-MDP–containing Adhesives

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Clinical Relevance

10-MDP–containing adhesives are important for bonding resin cements to zirconia. However, various concentrations of this component need further investigation to determine the most optimal amount responsible for improving the bonding to Y-TZP.

SUMMARY

Objective: The objective of this study was to evaluate the influence of adhesives with different 10-MDP concentrations on the shear bond strength of a resin cement to zirconia.

Methods and Materials: Six experimental adhesives were prepared with the following composition: camphorquinone, 1,2-diaminobenzene, butylhydroxytoluene, diphenyliodonium hexafluorophosphate, 2-hydroxyethyl methacrylate triethylene glycol dimethacrylate, ethoxylated bisphenol A glycol dimethacrylate, urethane dimethacrylate, bisphenol A diglycidyl methacrylate, and ethanol. The 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer was added at 0wt%, 3wt%, 6wt%, 9wt%, 12wt%, or 15wt%. Three commercially available adhesives were evaluated: Single Bond Universal, Single Bond 2, and Signum Zirconia Bond. Resin cement cylinders made with RelyX Ultimate were bonded to yttria-stabilized tetragonal zirconia polycrystal with one of the evaluated adhesives and were subjected to the shear bond strength evaluation. Failure modes were analyzed with a stereoscopic loupe. Statistical analyses were performed with one-way analysis of variance and the Tukey's Honestly Significant Difference test (α=0.05). Pearson's was used to correlate the percentage of 10-MDP in the experimental adhesives and shear bond strength.

Results: There were significant differences between adhesives ($p<0.00001$). The highest shear bond strength values were obtained with the Signum Zirconia Bond and Single Bond Universal. Single Bond 2 showed the lowest values. There were no differences between experimental adhesives. All groups showed

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adhesives failures. A nonlinear correlation was found between bond strength and percentage of 10-MDP in experimental adhesives (r=0.872).

Conclusions: The commercially available adhesives indicated for bonding to zirconia showed the highest bonding values.

INTRODUCTION

The clinical success of all-ceramic yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) indirect restorations not only depends on the correct knowledge and handling of the material itself but also the use of an adhesive system associated with resin-based cement to provide satisfactory bonding of the prosthetic work to the dental structures.[1] Y-TZP is widely recognized for its excellent mechanical, physical, and thermal properties; biocompatibility; high fracture toughness; hardness; and wear resistance.[2] Although Y-TZP ceramics present all these excellent properties, the effectiveness of adhesive cementation procedures is still a problem, because Y-TZP ceramics cannot be conditioned by the application of hydrofluoric acid and conventional silane coupling agents due to the absence of silica and a glass phase.

In search of solutions, different procedures to improve the bond of the resin cement to the inner surface of zirconia have been tested, such as surface preparation with erbium-doped and yttrium-aluminum-garnet laser (Er: YAG), grinding with diamond rotary instruments, selective infiltration etching, surface roughening by aluminum oxide blasting of different particle sizes before or after sintering, surface roughening by alumina-silica particles before silanization, and application of a liner.[2-10] All these methods seek to improve the mechanical and micromechanical interlocking through the increase in the roughness of the surface. However, some of these treatments have proved to be ineffective, and, in several cases, they may cause surface damage.[11]

A different approach to improve the bond strength to zirconia is to develop a chemical interaction between the surface and the applied resin cement.[12] For this task, research has focused on the use of primers that contain phosphate monomers that have an affinity for metal oxides with 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) being one of the most used monomers.[13,14] Although important for bonding to both dental substrates and zirconia, the maximum concentration of 10-MDP monomers that should be added to dental adhesives still needs to be evaluated, as it has been suggested that the polymerization of camphorquinone (CQ)/amine-based adhesives may be negatively affected by the interaction between functional monomers such as 10-MDP and tertiary amines.[15] Therefore, it is important to evaluate how the incorporation of different concentrations of 10-MDP may improve or jeopardize the bonding of adhesive systems to zirconia.

It is well known that light-cured resin-based materials such as adhesives commonly contain CQ and tertiary amines that allow free-radical polymerization triggered by the irradiation with visible light. On the other hand, it has been suggested that the inclusion of a third component to the CQ/amine photoinitiating system such as iodonium salts could improve the photo-activation induced by visible light sources.[16]

10-MDP monomers are required to obtain bonding with acid resistant polycrystalline ceramic materials such as zirconia. It has been suggested that these monomers can interact with metal oxides, enabling chemical bonding of ceramic oxides with or without an additional coupling agent.[17] Thus, the purpose of the present study was to evaluate the influence of different 10-MDP concentrations on the shear bond strength of a resin cement to zirconia. For this purpose, 10-MDP was added to six experimental adhesives and compared with three commercially available materials. The null hypotheses evaluated were (1) the concentration of 10-MDP monomers in the experimental adhesives would not influence the shear bond strength; and (2) there would be no difference between the experimental and commercially available adhesives.

METHODS AND MATERIALS

Experimental adhesives contained of a mixture of 2-hydroxyethyl methacrylate (HEMA), triethylene glycol dimethacrylate (TEGDMA), ethoxylated bisphenol A glycol dimethacrylate (Bis-EMA), urethane dimethacrylate (UDMA), and bisphenol A diglycidyl methacrylate (Bis-GMA). Ethanol (10wt%), CQ (0.50wt%), 1,2-diaminobenzene (DABE) (1.00wt%), iodonium salt diphenyliodonium hexafluorophosphate (DPIHP) (0.45wt%), and butylhydroxytoluene (BHT) (0.20wt%) were added. Five concentrations of 10-MDP were added to this basic adhesive: 3wt%, 6wt%, 9wt%, 12wt%, or 15wt%. Materials were used without further purification. As controls, three commercially available adhesive systems were evaluated: Single Bond 2 (not indicated for bonding to zirconia), Single Bond Universal

Table 1: *Materials and Composition*

Adhesive (manufacturer)	Composition	Instructions for use
Adper Single Bond 2 (3M ESPE, Sumaré, SP, Brazil)	BisGMA, HEMA, dimethacrylates, silica nanofiller, ethanol, water, photoinitiator system and methacrylate functional copolymer of polyacrylic and polyitaconic acids	(a) Apply two to three consecutive coats of adhesive to the ceramic surface for 15 seconds with gentle agitation using a fully saturated applicator; (b) gently air thin for five seconds to evaporate solvents; (c) light cure for 10 seconds
Single Bond Universal (3M ESPE, Seefeld, Germany)	MDP phospate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane	(a) Clean the surface with alcohol and dry it with compressed air; (b) apply with a microbrush to the surface for 20 seconds; (c) apply compressed free oil air for five seconds; (d) light cure for 10 seconds
Signum Zirconia Bond (Heraeus Kulzer, Hanau, Germany)	Signum zirconia bond I[a]: Acetone, 10-MDP, acetic acid. Signum zirconia bond II[a]: methyl methacrylate, diphenyl(2,4,6- trimethy lbenzoyl)phosphine oxide. MMA, initiators	(a) Clean the surface with alcohol and dry it with compressed air; (b) Signum Zirconia bond I is dispensed and applied with a suitable brush to the entire surface and air dried for five seconds; (c) Signum Zirconia bond II is applied and light cured for 40 seconds

[a] *Obtained from the manufacturer's safety data sheet.*

(indicated for bonding to zirconia), and Signum Zirconia Bond (indicated for bonding to zirconia). Table 1 describes the composition, manufacturers, and instructions for the use of these adhesives.

Polycrystalline ceramic blocks (IPS e.max ZirCAD, Ivoclar Vivadent, Schaan, Liechtentein) were cut into 2-mm-thick slices with a cutting machine (Isomet 1000 Low Speed, Buehler, Lake Bluff, IL, USA) using a diamond disc (15LC diamond no. 11-4254, Buehler) at a speed of 275 rpm under constant water irrigation. Four ceramic slices were used in each group. The slices were polished with sequential sandpaper discs (grit sizes, #800, #1000, and # 1200; K2000 Polishing Paper, Exact, Nordestedt, Schleswing-Holstein, Germany) using a metallographic polishing machine (Exact, Nordestedt) to standardize the ceramic surfaces. Forty slices were prepared.

After surface standardization, the ceramic slices were sintered (INFIRE Oven HTC Speed, Sirona Dental Systems, Long Island City, NY, USA) according to the recommendations of the ceramic manufacturer. The slices were then randomly divided into nine groups and embedded in acrylic resin. The surfaces were polished with sequential sandpaper discs (grit sizes #800 to #1200; K2000 Polishing Paper, Exact) to remove any acrylic resin that might have covered the samples. Samples were washed under copious deionized water cooling and dried with oil-free air with a pressure of 40 psi, at a standardized distance of 15 cm and an angle of 45°. Experimental adhesives were applied as follows: all the ceramic surfaces were cleaned and dried; adhesives were applied with a microbrush and remained untouched for 20 seconds; oil-free air with pressure of 40 psi was applied at 90° from a distance of 15 cm for five seconds to evaporate solvents; and adhesives were light cured for 10 seconds with an LED device (VALO Cordless, Ultradent Products, South Jordan, UT, USA) operating at an irradiance of 1100 mW/cm^2. Commercially available adhesives were applied as per manufacturers' instructions and were light cured with the same device.

Surgical catheters with an internal diameter of 1.40 mm and a height of 1 mm were used for the resin cement cylinders (RelyX Ultimate, 3M ESPE, St. Paul, MN, USA). Four cylinders per ceramic surface were prepared, resulting in 16 for each group. The cement was mixed according to the manufacturer's recommendations, inserted into the catheters, and light cured for 20 seconds with the VALO curing device with irradiance of 1100 mW/cm^2. After light curing, the surgical catheters were kept untouched for 10 minutes before being removed with #11 scalpel blades (Embramed, Jurubatuba, SP, Brazil) to expose the resin cement cylinders. Samples lost during removal of the catheters were counted and considered as 0 MPa in the statistical analysis.

The samples were stored in deionized water for 24 hours at 37°C. After storage, the samples were subjected to shear bond strength evaluation using a 0.2-mm wire-loop positioned as close as possible of the adhesive interface (Figure 1) and a universal testing machine (Instron 3342, Illinois Tool Works, Norwood, MA, USA) with a 500-N load cell operated at a crosshead speed of 0.5 mm/min.

Data were analyzed with one-way analysis of variance and the Tukey Honestly Significant Differ-

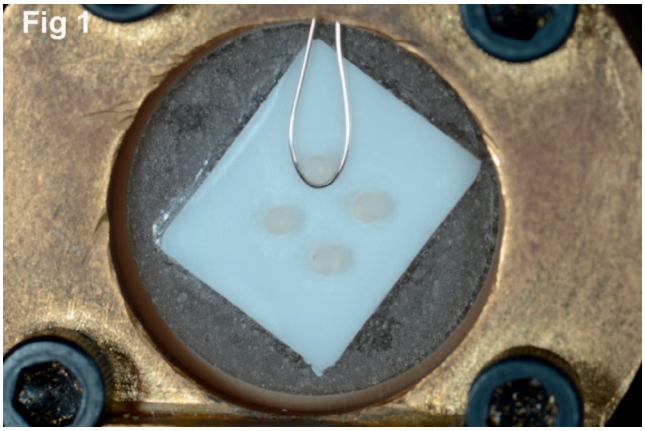

Figure 1. *Example of samples adapted to the universal testing machine before the shear bond strength evaluation.*

ence test. A global significance level of 5% was adopted. Pearson correlation analysis was used to determine whether there was a correlation between the percentages of 10-MDP in the experimental adhesives and shear bond strength. The failure modes were evaluated with a stereoscopic loupe.

RESULTS

Mean values, number of lost samples for each group, and SDs for the shear bond strength are shown in Table 2. There were significant differences between groups (F=6.5741; p<0.00001). The highest shear bond strength values were obtained with the Signum Zirconia Bond and Single Bond Universal. There were no differences between the experimental adhesives. Single Bond 2 showed the lowest values. The failure mode was adhesive for all specimens. A nonlinear correlation was found between bond strength and percentage of 10-MDP in the experimental adhesives (r=0.872; Figure 2).

Table 2: *Mean Values and SDs for Shear Bond Strength*

Adhesive	Shear bond strength, MPa	Sample losses (% of 16 samples)
Single Bond 2	6.06 ± 5.78 A	4 (25%)
Single Bond Universal	14.02 ± 6.5 BC	2 (12.5%)
Signum Zirconia Bond	20.86 ± 6.11 C	0 (0%)
0% 10-MDP	7.89 ± 8.38 AB	6 (37.5%)
3% 10-MDP	9.34 ± 6.75 AB	2 (12.5%)
6% 10-MDP	12.56 ± 7 AB	1 (6.25%)
9% 10-MDP	13.43 ± 6.62 AB	3 (18.75%)
12% 10-MDP	10.7 ± 6.08 AB	2 (12.5%)
15% 10-MDP	11.81 ± 4.2 AB	0 (0%)

Different letters represent statistically significant differences (p<0.05).

DISCUSSION

The present study evaluated the shear bond strength of a resin cement to zirconia after the application of 10-MDP–containing experimental and commercially available adhesives. Although a nonlinear correla-

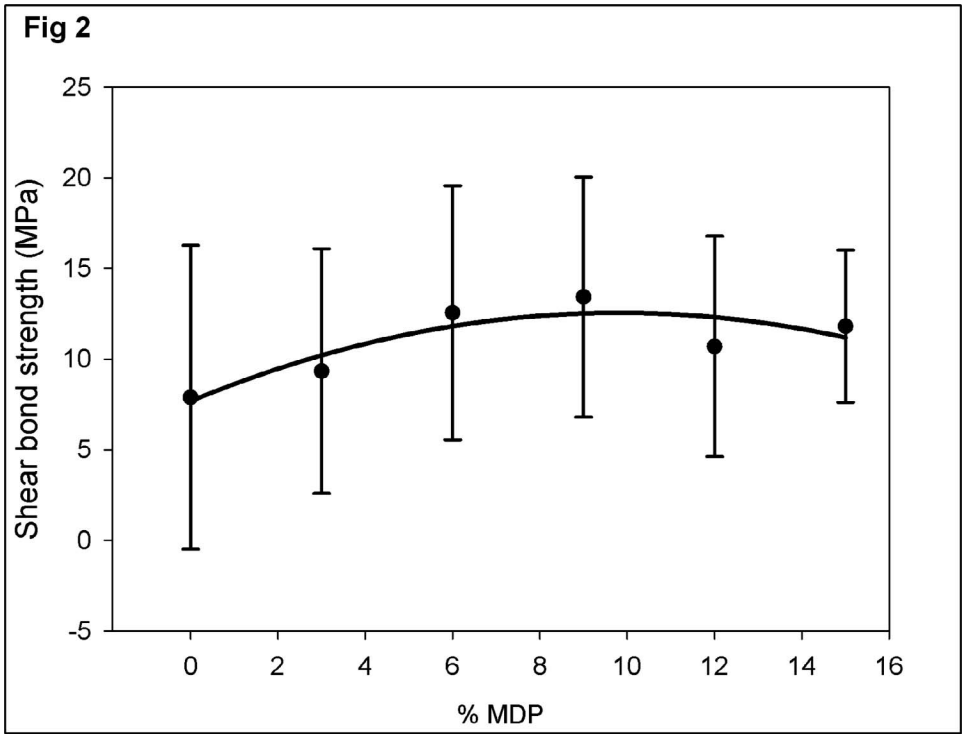

Figure 2. *Pearson correlation analysis. A nonlinear correlation was found between bond strength and percentage of 10-MDP in the experimental adhesives* ($y = -0.0506x^2 + 0.9927x + 7.6836$; r=0.872).

tion was found between bond strength and percentage of 10-MDP added to the experimental adhesives (Figure 2), in this study, the first null hypothesis was accepted because no statistically significant difference was found between experimental adhesives. The second null hypothesis was rejected, because there were differences between experimental and commercially available adhesives.

In the present study, no ceramic pretreatment was conducted. Several pretreatments have been suggested and studied in the literature to improve the adhesion between Y-TZP and resin cements, As a standard method, the aluminum oxide sandblasting technique has been described.[10,18] Different advantages of this technique include increasing surface roughness, wettability, and surface energy of the ceramics, which improve the micromechanical retention.[18] On the other hand, Y-TZP ceramics may suffer a phase transformation from tetragonal to monoclinic, which may be detrimental for the durability and mechanical properties of this ceramic.[10] As the present study was designed to address the bond strength of different adhesive systems without the interference of surface-related characteristics such as roughness, no sandblasting or any other mechanical surface treatment was conducted. Additionally, to better understand the influence of the different adhesives evaluated, after they were embedded in acrylic resin, Y-TZP discs were polished with #1200 sandpaper. However, because Y-TZP ceramic restorations are obtained by milling technologies, higher bond strength values would be expected due to the increased roughness.

Another point to be addressed is the fact that in the present study the light activation of the resin cement was conducted directly, without the influence of a ceramic material between the light source and the resin cement. Although brands of Y-TPZ materials may present different degrees of translucency, the light attenuation caused by the ceramics may play a role on the properties of the resin cement.[19-21] Although the interposition of ceramic materials between the light source and the resin cement has the effect of decreasing the degree of conversion and the hardness of the resin cement,[19] it has been suggested that the thickness of the interposing ceramic material influences the light transmittance, and as ceramic thickness increases, longer irradiation periods are required.[21] Besides the composition of the adhesive and the microstructure and thickness of the interposing ceramic material, it is known that the bonding between the ceramics and the dental substrate also depends on the proper curing of the resin cement, and factors such as type of resin cement, characteristics of the light curing device, and light activation protocol could play a major role. For this reason, the results of the present study could be different not only if the ceramics were interposed but also if other resin cements and curing protocols were evaluated.

Regarding the shear bond strength method, some points should also be discussed. As described in the literature, two experimental designs, shear and tensile tests both at the micro- or macro-scale, are commonly used in this type of study.[22] In general, the shear bond strength test is used because it is simpler than tensile tests. On the other hand, shear tests have a nonhomogeneous stress distribution at the adhesive interface.[23] For this reason, absolute numerical results obtained from one study cannot be directly compared to another. It should be noted that, despite the described limitations regarding the bond strength evaluations, different materials are similarly ranked, as correlations between different methods have been found.[24,25]

In the present study, the shear bond strength evaluation was conducted on 24-hour water-stored samples. Although a nonlinear correlation was found between bond strength and percentage of 10-MDP in the experimental adhesives (Figure 1), it was shown that the concentration of 10-MDP monomers on the experimental adhesives did not influence the bond strength of the evaluated resin cement, whereas the two commercially available adhesives indicated for bonding to zirconia that also contain 10-MDP showed a better performance. These results are in agreement with others studies.[17,26,27] It is, however, important to note that, although no long-term storage or aging was conducted in the present study, the differences between materials containing different 10-MDP concentrations could be demonstrated if a long-term evaluation of the bond strength was conducted. Other studies confirm that active parts of 10-MDP react with the zirconia surface, but this is vulnerable to instability after aging.[1,28]

For a better interpretation of this study, it is important to understand how the 10-MDP monomer works. Chemically, this monomer bonds to oxide metals and tooth substrates. It has an amphiphilic structure with the vinyl group as the hydrophobic half and the phosphate group as the hydrophilic half.[29] The 10-MDP monomer is an effective bonding agent between the resin cement, zirconia, and other metal oxide materials.[30] Authors such as Yoshida and others[17] evaluated the interaction of hydroxyl groups of the phosphate half with the hydroxyl groups on the

zirconia surface through van der Waals forces or hydrogen bonds. Because of these studied properties, many commercial products such as adhesives, primers, and resin cements have incorporated 10-MDP in their composition, seeking to improve the bonding properties to Y-TZP ceramic materials.[31]

Signum Zirconia Bond is a 10-MDP–containing material especially designed for the purpose of bonding resin cements to Y-TZP ceramics. This bonding material is delivered by two different bottles. MDP monomers, acetic acid, and acetone are present in bottle 1, whereas bottle 2 contains diphenyl phosphine oxide and methyl methacrylate. In the present study, this material has shown increased shear bond strength. Other studies have also shown similar results.[18,32] According to Ural and others,[33] the key factor of this material could be the methyl methacrylate, which establishes primary bonds with the methacrylate present in the resin cement and improves the bond strength in that way. Thus, a further study with more focus on MDP and methyl methacrylate interaction is suggested. Although important for bonding resin cements to zirconia, the 10-MDP concentration alone is not the only factor to be considered.

CONCLUSIONS

According to the results of the present study, it may be concluded that the commercially available adhesives indicated for bonding to Y-TZP showed the highest bonding values, and the concentration of 10-MDP on experimental adhesives was not significant.

Acknowledgements

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Bauru School of Dentistry, University of São Paulo, Brazil.

Conflict of Interest

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

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Departments

Faculty Positions

The University of Kentucky College of Dentistry is seeking applications for a full-time tenure-track faculty position at the academic rank of assistant, associate, or full Professor in the Department of Oral Health Practice, to serve as Chief of the Division of Restorative Dentistry.

As our growth in the areas of education, service and clinical care has been accelerating during the last two years, we are turning now our attention into building up our faculty membership. Responsibilities will include oversight of the operative dental curriculum, mentoring division faculty, as well as some clinical and didactic teaching. The College is looking for an individual with strong clinical skills and research background to provide leadership for its restorative dentistry program. Private practice experience is also desirable. Applicants must have a DDS/DMD from a program accredited by the Commission on Dental Accreditation or equivalent, and preferably additional training in operative dentistry. The applicant should qualify for an unrestricted or faculty-limited Kentucky dental license. Teaching experience and board certification in operative dentistry are desirable.

Salary and rank will be commensurate with qualifications and experience. Review of applications will begin immediately and continue until the positions are filled.

To apply, please attach a letter of interest and curriculum vitae. Also provide the names and contact information for three references when prompted in the application. This information may be utilized to solicit recommendation letters from your references within the employment system.

Inquiries regarding the position may be submitted via email to Dr. Robert Kovarik, Senior Associate Dean at rekova2@uky.edu. Please note that, to be considered an applicant for this position, you must submit an application via UK's online employment system.

Errata:

Operative Dentistry apologizes for the following errors in the manuscript, "Biological Effects of Provisional Resin Materials on Human Dental Pulp Stem Cells" published as an online only article attached to volume 42 issue 2 pp. e81-e92.

There were multiple errors in the author contact list. A corrected version is presented here:

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The lot numbers in Table 1 were incorrect and are shown corrected in this table:

Table 1: *Provisional resin materials tested in this study.*

Product	Code	Manufacturer	Lot number	Composition
Snap	SN	Parkell Inc	59308	Poly ethyl methacrylate (PEMA)
Jet	JE	Lang Dental	28789	Poly methyl methacrylate (PMMA)
Luxatemp	LT	DMG	726443	Bis-acrylic composites
Revotec LC	RL	GC America Inc	1610151	Urethane Dimethacrylate (UDMA)
Vipi block	VB	Madespa	0000023806	Poly methyl methacrylate (PMMA)

Lastly, the following acknowledgement should have appeared with the article:

"This research was supported by Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Science, ICT & Future Planning (NRF-2015R1C1A1A01052127)."

Errata:

Operative Dentistry apologizes for the table 3 error in the manuscript, "Relined Fiberglass Post: Effect of Luting Length, Resin Cement, and Cyclic Loading on the Bond to Weakened Root Dentin" published as an online only article attached to volume 41 issue 6 p. e178. The correct table is shown here:

Table 3: *Pull-out Bond Strength Means (MPa) and Standard Deviations Between the Different Luting Lengths Without and With Cyclic Fatigue Loading*[a]

Luting Length, mm	Cyclic Fatigue Loading	n	Mean	Standard Deviation
5	Without	20	6.98 A	1.18
	With	20	4.78 B	1.32
10	Without	20	6.17 A	0.88
	With	20	6.03 A	0.99

[a] *Different letters indicate statistically different means according to Tukey test ($p < 0.05$).*

Online Only Articles

On occasion we receive manuscripts that we would like to publish, but do not have the page room to include in the print journal. For the full article, please go to www.jopdentonline.org or enter the provided address into your address bar.

The Survival of Class V Composite Restorations and Analysis of Marginal Discoloration

J-H Kim • J Cho • Y Lee • B-H Cho

Clinical Relevance: Type of resin composite, the presence of occlusal wear facets, and bleeding on probing were associated with the longevity of class V composite restorations. Surface refurbishment and the use of visual magnification are recommended when evaluating aged composite restorations.

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The Survival of Class V Composite Restorations and Analysis of Marginal Discoloration

J-H Kim • J Cho • Y Lee • B-H Cho

Clinical Relevance

Type of resin composite, the presence of occlusal wear facets, and bleeding on probing were associated with the longevity of class V composite restorations. Surface refurbishment and the use of visual magnification are recommended when evaluating aged composite restorations.

SUMMARY

The aims of this retrospective clinical study were to analyze the longevity of class V composite restorations and compare the results obtained from clinical and laboratory evaluation of marginal discoloration. A total of 186 restorations were evaluated with modified US Public Health Service criteria. Longevity and associated variables were analyzed with the Kaplan-Meier method and a Cox proportional hazard model. Restorations with marginal discoloration were additionally evaluated using digital photographs and epoxy resin replicas under a stereomicroscope. The mean survival time was 15.0 years, with five- and 10-year survival rates of 95.5% and 83.1%, respectively. Z250 had a higher risk of failure (hazard ratio=7.01, 95% confidence interval=2.07-23.72) than Z100. In addition, the presence of occlusal wear facets and bleeding on probing were associated with an increased risk of failure of the restorations. However, the use of an adhesive system (Scotchbond Multi-Purpose or Clearfil SE Bond) did not affect the longevity of the restorations. The results of laboratory evaluation were significantly different from clinical evaluation ($p<0.001$, McNemar test). Among 55 restorations rated as Bravo in the clinical evaluation, 24 restorations (43.6%) were determined to have penetrating discoloration on laboratory evaluation. When evaluating aged composite restorations, surface refurbishment and the use of a microscope are recommended, which will be helpful in determining the need for timely repair or replacement.

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INTRODUCTION

Resin composites are effective materials for restoring damaged or decayed teeth, with the advantages of good esthetics, high surface gloss, and clinically acceptable wear resistance.[1,2] Contemporary dental adhesives used with resin composites show favorable immediate results in terms of retention and sealing of the tooth-composite interface.[3-5] However, some clinical studies showed a significant reduction in dentin adhesion over time and reported relatively short lifetimes of failed composite restorations.[6,7]

The tooth-composite interface is the weakest point of composite restorations.[8] None of the current adhesive systems are capable of completely eliminating marginal leakage, at least in the long term.[1,6,8] Marginal deterioration and recurrent caries are the most common causes of failure of composite restorations.[9,10] Adhesion degradation over time commonly manifests as a marginal discoloration. Hayashi and others[11] reported that marginal deterioration and discoloration were important predictors of the failure of composite restorations. Accordingly, the margins should be carefully examined to evaluate the clinical status of the restorations and determine their prognosis.

Visual rating scales are preferred for the clinical evaluation of existing restorations. The US Public Health Service (USPHS) criteria has been used in most clinical studies with slight modifications.[7,12-14] The USPHS criteria include the assessment of marginal discoloration, based on the assumption that penetrating discoloration is associated with microleakage and the development of secondary caries. The assessment of margins is a difficult decision for dental practitioners, but is usually performed with the naked eye in practice. Furthermore, there is a lack of literature on the effectiveness and accuracy of clinical evaluation for marginal discoloration.

Resin composites have been widely used for restoring class V lesions; however, the restoration of class V lesions seems to be rather difficult because class V lesions often have a low retentive cavity shape and cervical margins lying on dentin or cementum that are unfavorable for resin bonding.[15,16] Some researchers have reported unsatisfactory clinical performance of resin composites in class V lesions.[6,7] Although clinical procedures for restoring class V lesions are relatively simple compared with those of class II and III restorations, it has been suggested that the clinical outcome of class V composite restorations is affected by various factors, such as type of resin composite, adhesive system, tooth type, and operator.[3,14]

In this retrospective clinical study, we assessed the survival of class V composite restorations and prognostic factors for their longevity. In addition to clinical evaluation of restorations using modified USPHS criteria, laboratory evaluation using epoxy resin replicas and a stereomicroscope was performed to determine the modes of marginal discoloration. It was hypothesized that there would be no differences between results obtained from the clinical and laboratory evaluations for marginal discoloration.

METHODS AND MATERIALS

Subjects and Clinical Evaluation Procedures

Forty-six patients who had received cervical restorations with resin composites more than 1 year ago at the Department of Conservative Dentistry of the Seoul National University Dental Hospital were recalled between April 1, 2014 and February 28, 2015. The study protocol was approved by Institutional Review Board of the Seoul National University Dental Hospital (IRB No., CRI14007). Written informed consent was obtained from every patient. Previous dental records were examined to collect information about the patients (sex, age, medical and dental history) and restorative materials (adhesive system and resin composite) by one investigator. Patients with severe systemic diseases or dental problems such as severe chronic periodontitis or bruxism were excluded. On the day of clinical examination, two other experienced and calibrated investigators evaluated class V composite restorations using the modified USPHS criteria, which included assessment of retention, marginal discoloration, marginal adaptation, anatomic form, and secondary caries (Table 1). Interexaminer agreement was assessed using Cohen's κ. Clinical evaluations were performed by the naked eye and a dental explorer. In case of disagreement between the investigators, consensus was reached by reexamination and discussion. Clinical photographs of the teeth were taken with a digital camera, and silicone rubber impressions were obtained to make epoxy resin replicas.

Longevity of the Restorations and Associated Factors

Survival rates of the restorations were analyzed using the Kaplan-Meier method. Restorations receiving a score of Charlie for any category of the

Table 1: *Modified USPHS criteria used to evaluate class V composite restorations*

Category	Rating	Criteria
Retention	Alpha	Restoration is present
	Charlie	Restoration is partially or totally lost
Marginal discoloration	Alpha	No discoloration
	Bravo	Superficial staining
	Charlie	Deep staining penetrating in a pulpal direction
Marginal integrity	Alpha	No detectable gap
	Bravo	Detectable gap with an explorer
	Charlie	Marginal crevice requiring replacement
Anatomic form	Alpha	Restoration continuous with existing anatomic form
	Bravo	Restoration discontinuous with existing anatomic form but clinically acceptable
	Charlie	Sufficient material is lost to exposed dentin
Secondary caries	Alpha	No caries present at the margin of the restoration
	Charlie	There is evidence of caries at the margin of the restoration

modified USPHS criteria were counted as a failure, and their lifespan was defined as the period from the date of the initial treatment to the date of examination. When a restoration was replaced or the tooth received further treatment, such as crown and extraction, the lifespan of the restoration was defined as the period from the date of the initial treatment to the date of retreatment. A Cox proportional hazard model was used to identify factors with possible influence on the longevity of the restorations. The following 12 variables were analyzed as possible predictors of the longevity of the restorations: patient sex and age, adhesive system (Scotchbond Multi-Purpose, 3M ESPE, St. Paul, MN, USA, and Clearfil SE Bond, Kuraray, Osaka, Japan), resin composite (Z100 and Z250, 3M ESPE), jaw site (upper or lower), tooth type (incisor, premolar, or molar), occlusal wear facet (present or absent), lateral occlusion scheme (canine-guided or group function), gingival and plaque index (the Silness-Löe index[17]), bleeding on probing (BOP; present or absent), and brushing stroke (horizontal or rolling). All variables were entered simultaneously into the model, and the Wald test was used to assess the significance of covariates. The relative hazard ratios (HRs) with respective 95% confidence intervals (CIs) were determined.

Laboratory Evaluation of Marginal Discoloration Modes

The restorations, which were rated Bravo or Charlie for marginal discoloration in the clinical evaluation, were additionally investigated using digital photographs and epoxy resin replicas under a stereomicroscope (SZ4045, Olympus Optical Co Ltd, Tokyo, Japan) at 40× magnification. Under the microscope, marginal defects were evaluated whether they were a mere chipping or were accompanied by a crevice between the composite and tooth. In addition, the marginal discoloration mode was reevaluated and classified as superficial or penetrating. When a discoloration was observed in association with a marginal crevice or a detached tooth-restoration interface, it was classified as a penetrating discoloration. Statistical comparison between the results of marginal discoloration obtained from the clinical evaluation at the chairside and the laboratory evaluation was performed using the McNemar test (α=0.05). Additionally, the results from the laboratory evaluation were analyzed using the χ^2 test to identify an association between the presence of marginal defect and marginal discoloration modes (α=0.05).

RESULTS

The Cohen's κ statistics showed excellent agreement between the two examiners (κ=0.95). Clinical data were collected from a total of 217 cervical restorations from 46 patients during the survey. One patient and 31 restorations were excluded from the analysis due to loss of restoration (n=24), re-restoration with a full coverage crown (n=3), or tooth loss (n=4) at an unknown date. Finally, 186 cervical restorations from 45 patients were analyzed for the purposes of this study. Twenty patients (44.4%) were male and 25 patients (55.6%) were female. Patient age at treatment ranged from 23 to 76 years, with a mean age of 61.4 ± 9.7 years. Fifty-five (29.6%) restorations were placed on anterior teeth, 100 (53.7%) restorations on premolars, and 29 (15.6%) restorations on molars. Ninety-nine (53.2%)

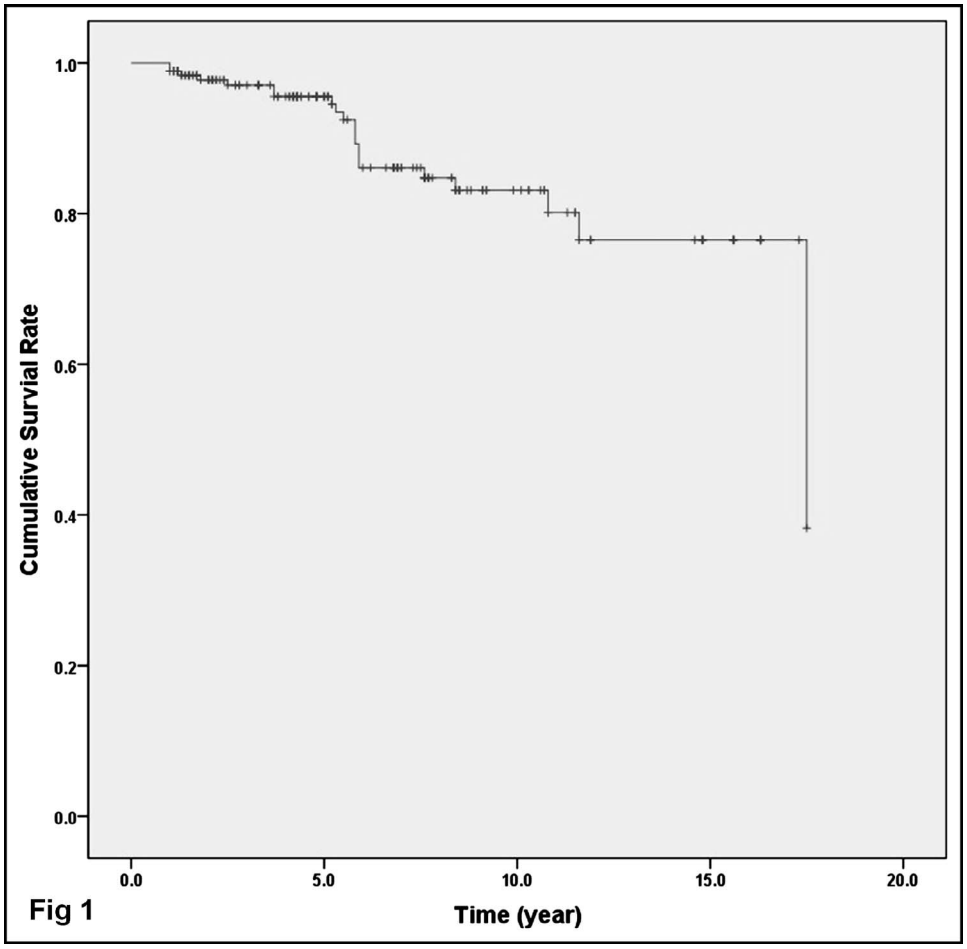

Figure 1. *Cumulative survival estimates of class V composite restorations using the Kaplan-Meier method.*

restorations were placed on upper teeth and 87 (46.8%) restorations on lower teeth.

Thirteen restorations (7.0%) failed and were replaced before the examination. Seven restorations were rated as Charlie for marginal discoloration, and one restoration was rated as Charlie for marginal adaptation. Consequently, 21 restorations (11.2%) were considered failed at the time of examination. Figure 1 shows the cumulative survival rates of the class V composite restorations. The estimated mean survival time of the restorations was 15.0 years (SE=0.53), with estimated five- and 10-year survival rates of 95.5% and 83.1%, respectively.

The Cox proportional hazard model revealed that type of resin composite, occlusal wear facet, and BOP significantly affected the longevity of class V composite restorations (Table 2). Z250 resin composites were more likely to fail than Z100 resin composite, corresponding to a HR for failure of 7.01 (95% CI=2.07-23.72, p=0.002). The restorations of teeth with occlusal wear facets and BOP had 6.65- and 4.58-fold increased risk of failure (95% CI=1.85-23.88, p=0.004 and 95% CI=1.46-14.33, p=0.009,

Table 2: *Prognostic variables affecting the longevity of class V composite restorations and their relative hazard ratio*

Variables	Relative HR	95% CI		Wald	*p*-value
		Lower	Upper		
Resin composite					
Z100	1.00				
Z250	7.01	2.07	23.72	9.791	0.002
Occlusal wear facet	6.65	1.85	23.88	8.425	0.004
Bleeding on probing (BOP)	4.58	1.46	14.33	6.819	0.009

Table 3: *Comparison of results obtained from clinical and laboratory evaluations for marginal discoloration*

Clinical evaluation	Laboratory evaluation		Total
	Superficial discoloration	Penetrating discoloration	
Modified USPHS rating			
Bravo	31	24	55
Charlie	1	7	8
Total	32	31	63

There was a significant difference between results obtained from the clinical and laboratory evaluation ($p<0.001$, McNemar test).

Table 4: *Association between the presence of marginal defects and marginal discoloration modes*

Presence of marginal defect under magnification	Marginal discoloration		Total
	Superficial discoloration	Penetrating discoloration	
No	16	18	34
Yes	16	13	29
Total	32	31	63

There was no association between the presence of marginal defects and marginal discoloration modes ($p>0.05$, χ^2 test).

respectively). However, patient sex and age, adhesive system, jaw site, tooth type, lateral occlusion scheme, gingival and plaque index, and brushing stroke did not affect the longevity of the restorations.

Sixty-three restorations, which were rated Bravo or Charlie for marginal discoloration on clinical evaluation, were evaluated in the laboratory for marginal discoloration mode using the digital photographs and epoxy resin replicas. The results of the laboratory evaluation were significantly different from those of the clinical evaluation ($p<0.001$, McNemar test; Table 3). Among 55 restorations rated as Bravo in the clinical evaluation, 24 restorations (43.6%) were determined to have penetrating discoloration by the laboratory evaluation. On the other hand, 87.5% of restorations rated as Charlie were determined to have penetrating discoloration by the laboratory evaluation. There was no association between the presence of marginal defects and marginal discoloration modes ($p>0.05$, χ^2 test; Table 4).

DISCUSSION

In this retrospective cross-sectional clinical study, we assessed the longevity and clinical status of 186 resin composite restorations. Cross-sectional retrospective studies are less satisfactory compared with controlled prospective trials in terms of methodologic criteria for validity and quality.[9] The major drawback of a cross-sectional design in this case is that various factors associated with restoration procedures such as cavity shape and preparation technique are not well controlled. However, cross-sectional designs are able to examine a large number of restorations in a relatively short survey period. Most prospective clinical studies have included a limited number of cases due to a high research cost and a considerable dropout rate.[18,19] Moreover, a few prospective clinical studies assessed composite restorations over a period of five years.[7,19-22] In the present study, the mean age of the assessed 173 restorations (excluding 13 restorations that failed before the examination) was 6.4 years, with a maximum of 17.5 years based on the date of examination. The medium- to long-term outcomes give more valuable insight into the clinical effectiveness of composite restorations than a short-term clinical trial. Such practice-based studies may reflect the real-life performance of restorative treatment more accurately than a well-controlled trial under ideal conditions.

Kaplan-Meier survival analysis indicated that the mean survival time of the restorations was 15.0 years (SE=0.53), and the estimated 10-year survival rate was 83.1%. Some short- to medium-term clinical trials have reported low retention rates of resin composite restorations over time.[6,7] However, recently, several long-term studies showed high retention rates and excellent clinical performance of class V composite restorations.[4,19,21,22] Wilder and others[19] reported a high retention rate of 89% of class V restorations with a three-step etch-and-rinse adhesive after 12 years. Similar high retention rates of 89.5% were reported for a two-step self-etch adhesive (Clearfil SE Bond) after 13 years by Peumans and others.[22] The excellent clinical performance of composite restorations can be largely attributed to improvement in the formulation of dentin adhesives, which accompanies proper dentin hybridization.

In the present study, we did not find any significant differences between the clinical performance of Scotchbond Multi-Purpose and Clearfil SE Bond. Scotchbond Multi-Purpose, a three-step etch-and-rinse adhesive, includes a water/ethanol-based primer and solvent-free bonding agent. The bonding effectiveness of Scotchbond Multi-Purpose to dentin has been shown to decrease with artificial aging in several *in vitro* studies.[23,24] However, the solvent-free bonding agent provides a stable and durable adhesion to acid-etched enamel and may overcome problems associated with hydrolytic degradation of the adhesive-dentin interface.[1,6] On the other hand,

Clearfil SE Bond is a two-step self-etch adhesive composed of a self-etch primer and a separate hydrophobic resin.[25,26] Clearfil SE bond is less sensitive to hydrolytic and enzymatic degradation of the hybrid layer compared with etch-and-rinse adhesive systems.[27] However, unsatisfactory bonding to enamel has been pointed out as a shortcoming of "mild" self-etch adhesives in several *in vitro* studies.[28,29] Well-controlled clinical studies, however, revealed a good clinical performance of Clearfil SE Bond even on unprepared enamel.[1,22] Considering that it is less technique sensitive and has a lower incidence of postoperative sensitivity compared with that associated with etch-and-rinse adhesives, Clearfil SE Bond should be the material of choice for restoring cervical lesions.

According to the Cox proportional hazard model, the Z250 resin composite had a higher risk of failure (HR=7.01, 95% CI=2.07-23.72) than Z100. Some researchers have claimed that a material with a lower elastic modulus is able to flex with the tooth and as a result shows a higher retention rate for class V restorations than a material with a higher elastic modulus.[30] However, recent clinical trials on class V restorations that compared performance of composites with different elastic modulus revealed no differences in retention rates between materials.[5,13,21] The elastic modulus of Z250 (6.9 GPa) was lower than that of Z100 (11.3 GPa).[31] Thus, the assumption that a material with a low elastic modulus would be favorable for cervical lesions was not supported by the results of the present study, as well as previous clinical studies. [5,13,21] The elastic modulus may be less significant for the retention of cervical restorations with current dentin adhesive systems. On the other hand, a material having mechanical properties similar to that of dentin may form a monolithic structure with the tooth and distribute stress evenly throughout the body of the restoration and the adhesive interface.[32,33] Z100 has an elastic modulus similar to that of dentin (mean, 13.2 GPa) compared with Z250.[34] This is likely to improve the clinical performance of Z100 for the cervical lesion. Another possible explanation for the difference between the clinical performance of Z100 and Z250 could be their chemical composition. Z100 contains a matrix resin consisting of bisphenol A glycol dimethacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA), whereas in Z250, the majority of Bis-GMA and TEGDMA is substituted by ethoxylated bisphenol A glycol dimethacrylate (Bis-EMA) and urethane dimethacrylate (UDMA). Several *in vitro* studies reported that UDMA-based composites more easily undergo softening in water or oral simulating fluids than do Bis-GMA–based composites.[35,36] Deterioration of mechanical properties with aging could result in the relatively high failure rate of Z250 restorations. However, well-designed *in vitro* and *in vivo* studies comparing the two composites are required to give an exact explanation of the difference in their clinical performances.

Class V restorations of teeth with occlusal wear facets had a 6.65-fold increased risk of failure than those of teeth without wear facets (95% CI=1.85-23.88). Occlusal wear facets are associated with heavy occlusal or parafunctional forces, which concentrate at the cervical region. Repetitive compressive/tensile stresses caused by tooth flexure promote marginal breakage and dislodgement of cervical restorations.[30,37] According to studies that reported associations between occlusal interferences and non-carious class V lesions, occlusal adjustment may reduce the failure of class V restorations.[37,38] However, the effectiveness of occlusal adjustments is not supported by substantial evidence. Occlusal adjustment should be considered in cases where interferences are clearly present.

Interestingly, teeth with BOP had an increased risk of failure for class V composite restorations (HR=4.58, 95% CI=1.46-14.33). Cervical restorations are contiguous with the marginal gingiva and thus would have an effect on periodontal health. Well-finished and contoured composite restorations are clinically biocompatible and do not adversely affect the marginal gingiva.[39] However, the deterioration of marginal adaptation and gap formation are associated with increased plaque accumulation and gingival inflammation.[40] The association between BOP and an increased failure of class V restorations can be explained by marginal deteriorations of the restorations, which result in bacterial accumulation and persistent gingival inflammation manifested by BOP. Regular checkups to correct marginal defects and smooth the surfaces of the composite restorations will improve the longevity of the restorations and overall periodontal health.

A considerable number of clinical trials have used the USPHS criteria with slight modifications and demonstrated their validity and usefulness.[1,7,13,14,19,38] However, there is a concern regarding examination rating accuracy, in particular for marginal discoloration. Marginal discoloration is a major reason for replacement of composite restorations; thus, an accurate evaluation of marginal discoloration is of the utmost importance.[9,11] Evaluations of existing

restorations have been performed chairside with the naked eye in most clinical studies. In the present study, we were able to determine modes of marginal discoloration by carefully inspecting the location of discoloration and the status of tooth-restoration interfaces in laboratory evaluation. Laboratory evaluation revealed that 43.6% of restorations rated Bravo for marginal discoloration had penetrating discoloration (Table 3). In contrast, with regard to restorations rated Charlie, the results of laboratory evaluation were consistent with those of clinical evaluation. No alterations such as polishing or refurbishing were done before the clinical evaluations. The underestimation of marginal discoloration status could be attributed to the fact that plaque and/or superficial staining interfere with detecting the presence of penetrating discoloration. When evaluating aged restorations, the use of magnifying equipment, such as eye loupes and operating microscopes, and removal of surface stains are recommended for improving the differential diagnosis between superficial staining and penetrating discoloration; the latter is a predictor for failure of restorations and requires intervention such as replacement.

Based on the results of the laboratory evaluation, there was no association between the presence of marginal defects and marginal discoloration modes (Table 4). Most marginal defects were a clinically acceptable chipping fracture at the thin margin of the composite rather than a crevice between the tooth and composite. This result implies that marginal discoloration is not necessarily associated with marginal microleakage, which is mainly characterized by the presence of penetrating discoloration. In the case of marginal chipping with no evidence of microleakage, repair or refurbishment instead of total replacement can be a conservative treatment option for a defective restoration. Total replacement readily leads to loss of a significant amount of tooth structure.[41] In addition, at the restorative stage of class V lesions, avoiding composite flash or overhang extending over the preparation margin may help to reduce chipping fractures at the margin.

CONCLUSIONS

Within the limitations of the present study, the Z250 resin composite had a sevenfold increased risk of failure compared with Z100 (95% CI=2.07-23.72), whereas the type of adhesive system (Scotchbond Multi-Purpose and Clearfil SE Bond) did not affect the longevity of the restorations. Occlusal wear facets and BOP were associated with an increased risk of failure of class V composite restorations. There was a significant difference between the results of the clinical and laboratory evaluations for marginal discoloration. Microscopic examination and refurbishment of aged restorations should be considered before determining their prognosis and treatment options for repair or replacement.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Seoul National University Dental Hospital. The approval code for this study is CRI14007.

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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